



Synthesis and characterization of adipic acid based glycine crystals

¹R. Indhumathi, ²A.Sinthiya

¹Department of Physics, Thanthai Hans Roever College (Autonomous), Perambalur.

²PG and Research Department of Physics, Srimad Andavan Arts and Science College (Autonomous), Trichy.

* Corresponding Author - sinhi@andavancollege.ac.in

ABSTRACT

Single crystals of adipic acid based glycine were grown by slow evaporation method at room temperature. From Powder X-ray diffraction (XRD) pattern, the crystal particle size were calculated by using Scherrer Equation. The functional groups were identified by using FT-IR spectrum.

KEYWORDS: Glycine adipic acid, powder x-ray diffraction, FTIR spectrum

1. INTRODUCTION

Crystal growth is a very important of material sciences and engineering. In the field of molecular electronics the organic material are demanded because of their versatile functionalities. The intermolecular connections in organic materials affect novel functionalities such as conducting, dielectric and optical proper-ties. In organic materials charge transfer process i.e transfer of electron between donor and acceptor molecules play critical roles [1]. Glycine is the simplest amino acid and it forms several new compounds with other organic as well as inorganic materials. Recently, several complexes of glycine have been reported, viz., glycine sodium nitrate [2], di-glycine picrate [3], glycine lithium sulphate [4], bis(glycine) lithium nitrate [5], etc. Various methods including biosynthesis [6-8], supercritical method [9,10,11] have been elaborated for the clean synthesis of adipic acid. However, they were too expensive or too difficult to apply to manufacturing processes. Their catalytic property were studied in the presence of surfactants as phase-transfer agents [12] or synthesize surfactant-type polyoxo metalates [13,14]. Although surfactants can enlarge the yield of adipic acid, they are relatively expensive. Hence, in the present work, the synthesis, development, crystal structure and characterization of the organic material of adipic acid based glycine (GAA) is reported. It is interesting to grow GAA (Glycine adipic acid) crystals

on the basis of crystals grown by adding acetic acid with amino acids [15]. The amino acid glycine (NH₂CH₂COOH) is the only protein forming amino acid without centre of chirality [16]. Adipic acid is of great importance commercially in that it is used in the manufacture of nylon [17]. It is therefore, highly desirable to develop more environmentally friendly methods for the manufacture of GAA.

2. EXPERIMENTAL PROCEDURE

Commercially available glycine and adipic acid was taken in the stoichio-metric ratio 1:1 and were dissolved in double distilled water. The mixture of solutions was stirred well for 1 hr to get a homogeneous solution by using magnetic stirrer till a clear solution was obtained. The solution was filtered in a clean dry beaker using whatmann filter paper. After filtration, the beaker was covered by a perforated aluminium foil paper. The filtrated was then allowed to crystallize in dust free environment at ambient temperature. Crystallization took place about 48 days under the experimental conditions.

3 RESULT AND DISCUSSION

3.1 POWDER X-RAY DIFFRACTION ANALYSIS

Powder X-ray diffraction (XRD) analysis was carried out to confirm the crystalline nature of the sample and also to determine the purity of the GAA crystal. XRD patterns were recorded for the centrifuged and dried samples using x-ray diffractometer (RIGAKU ULTIMA III) using Cu K_α radiation ($\lambda = 1.5406 \text{ \AA}$). The recorded XRD pattern of GAA is shown in Figure 1. The appearance of sharp and strong peaks confirms good crystalline nature of the grown GAA crystal. The average crystallite size (L) of the synthesized GAA have been deduce as 63 nm. They have been obtained from the full width at half maximum (FWHM) of the most intense peak of the crystal by the Scherrer equation.

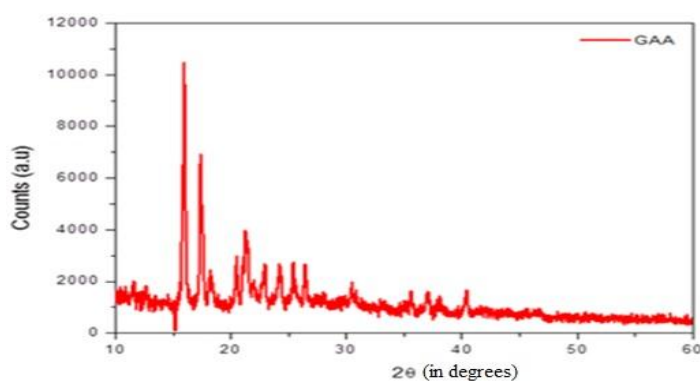


Figure 1 Powder x-ray diffraction pattern of GAA

3.2 FTIR SPECTRUM ANALYSIS

The FT-IR spectrum of the synthesized compounds was measured in the 4000 - 400 cm^{-1} region using on SPECTRUM RX I spectrophotometer (Perkin Elmer). The FT-IR spectrum is shown in Figure 2. The sharp peak at 3039 and 2958 cm^{-1} may be assigned to $-\text{NH}_2$ of glycine and $-\text{OH}$ stretching frequency of CO (OH). The peaks at 2753 to 2880 cm^{-1} are attributed to the CH stretching mode vibrations [17]. The most intense peak at 1696 cm^{-1} indicate the C=O [18].

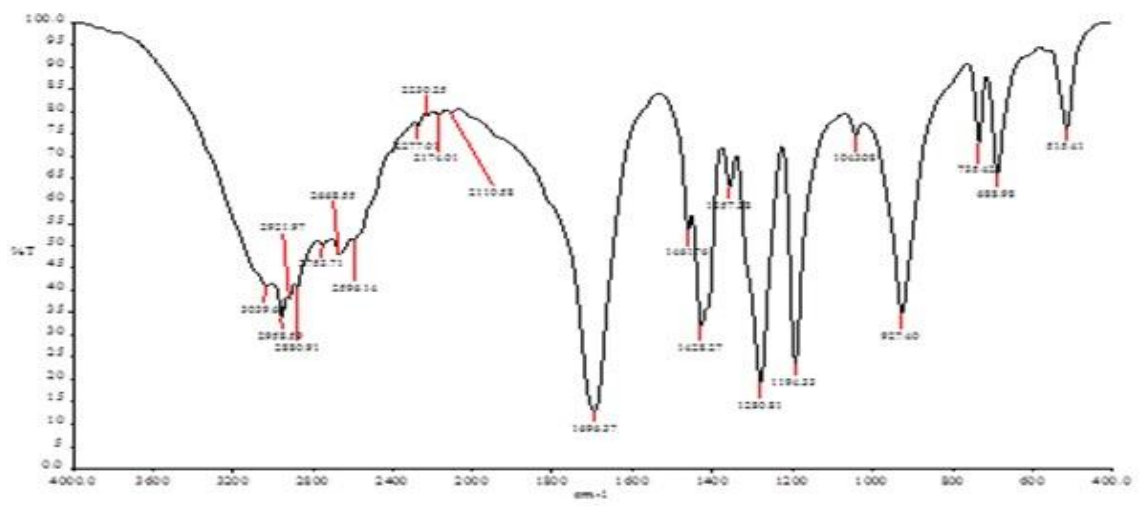


Figure 2: FTIR spectrum of GAA

The asymmetric and symmetric stretching modes of $-\text{COO}$ groups are confirmed by the peaks at 1461, 1428, 1357 and 1280 cm^{-1} [19]. FT-IR spectral analysis thus identifies the presence of functional group in the crystal material of GAA.

3.3 CONCLUSION

A single crystal of adipic acid based glycine (GAA) was grown by slow evaporation technique. The single crystal was characterized by powder X-ray diffraction and the functional groups were confirmed by FT-IR spectrum analysis.

ACKNOWLEDGEMENT

The authors thank the management of Srimad Andavan Arts and Science College (Autonomous) for the entire support given to complete this project.

REFERENCE

- [1.] Jyotsna R Pandey, Growth and optical properties of organic GOA crystals, American Journal of Engineering Research (AJER),3, 30-36 (2014),
- [2.] M. Narayan Bhat, S.M. Dharmaprakash, J. Cryst. Growth, 235, 511-516 ,(2002),
- [3.] M. Shakir, S.K. Kushwaha, K.K. Maurya, M. Arora, G. Bhagavannarayana, J. Cryst. Growth., 311, 3871-3875 (2009).

- [4.] M.R. Suresh Kumar, H.J. Ravindra, S.M. Dharmaprakash, J. Cryst.Growth., 306, 361-365(2007),
- [5.] J. Baran, M. Drozd, H. Ratajczak, A. Pietraszko, J. Mol. Struct., 927,43-53 (2009),.
- [6.] K.M. Draths, J.W. Frost, J. Am. Chem. Soc., 116, 399-400 (1994).
- [7.] W. Niu, K.M. Draths, J.W. Frost, Biotechnol. Progr., 18, 201-211 (2002).
- [8.] J.M. Thomas, R. Raja, J.B.F.G. Ohnson, T.J. O Connell, G. Sankar, T.Khimiya, Chem. Commun., 1126-1127 (2003).
- [9.] E.J. Beckman, Environ. Sci. Technol., 37, 5289-5296 (2003).
- [10.] Z. Hou, B. Han, L. Gao, Z. Liu, G. Yang, Green Chem., 4, 426-430 (2002).
- [11.] A.J. Bailey, W.P. Grieth, B.C. Parkin, J. Chem. Soc. Dalton Trans., 1833-1837 (1995).
- [12.] E. Antonelli, R. DAloisio, M. Gambaro, T. Fiorani, C. Venturello, J. Org.Chem. 63, 7190-9206 (1998).
- [13.] W. Zhu, H. Li, X. He, Q. Zhang, H. Shu, Y. Yan, Catal. Commun., 9,551-555 (2008).
- [14.] X. Shi, J. Wei, J. Mol, Catal. A: Chem., 229, 13-17 (2005).
- [15.] T. Pal and T. Kar, Optical, mechanical and thermal studies of nonlinear optical crystal l-arginine acetate, Materials Chemistry and Physics, 91,343-347 (2005).
- [16.] N. Vijayan, S. Rajasekaran,G. Bhagavannarayana et al., Growth and characterization of nonlinear optical amino acid single crystal: L-alanine, Crystal Growth & Design, vol. 6, no. 11, 2441-2445 (2006).
- [17.] R. M. Silverstein,G. C. Bassler, and T. C. Morrill, Spectrometric Identification of Organic Compounds, John Wiley& Sons, New York, NY, USA,(1981).
- [18.] R. M. Silverstein and F. X.Webster, Spectroscopic Identification of Organic Compounds, John Wiley& Sons, New York, NY, USA, (1998) [6th edition].
- [19.] G. Herzberg, Infrared and Raman Spectra of Polyatomic Molecules, VanNostran Reenhold, New York, NY, USA, (1945).