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Growth and characterization of Imidazolium adipate crystal

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Abstract

Imidazolium adipate (IMA) crystal was grown by slow evaporation solution growth technique by taking methanol as a solvent at room temperature. The grown crystals were analyzed for various characterizations like single crystal and powder X-Ray diffraction analysis to study the lattice parameters and crystalline nature of the IMA crystal. Fourier Transform Infra-Red (FTIR) spectrum was recorded to study the various modes of functional group present in the crystal. UV-vis-NIR spectrum was recorded to analyze the transmittance and band gap energy of the crystal. Vickers microhardness study was performed to study the mechanical strength of the crystal. Dielectric study was carried out to find the dielectric loss and dielectric constant of IMA.

Keywords: Imidazolium adipate, FTIR, optical properties and Vickers microhardness.

1 Introduction

Nonlinear optical (NLO) materials with weak nonlinear absorption but strong nonlinear refraction has attracted considerable attention because of their potential uses in the optical signal processing devices. The crystals which has low absorption, low reflectance and low refractive index in the transmission spectrum region which make the suitable material for antireflection coating in solar thermal devices. [1] Imidazole has a supreme importance because of its nitrogen heterocycle and have a significant property in the field of biology. Molecular structure imidazole transfers the proton in living systems to act as a weak acid and as a base [2] Due to the aromatic heterocyclic compound, Imidazole is a potential candidate for second-order NLO applications. Owing to the delocalization of π electron present in the molecule, hyper polarizability is enhanced which results in high optical non linearity so that the potential applicability of the material is more. [3]. Adipic acid or 1, 6-dioic acid is an odorless, white crystalline material belongs to dicarboxylic acid. It dissolves in organic solvents, and the main application is to produce nylon and polyurethane.[4] Few research articles have been reported Imidazole L tartaric acid [5], urea adipic acid [1] and electrical conductivity of imidazolium adipate[2] etc. In the present work we have grown an IMA crystal by slow evaporation solution growth technique. The structural, optical, mechanical and dielectric studies have been reported in this paper.

2 Experimental method

Imidazolium adipate (IMA) crystal was grown by taking Imidazole (Merk) and Adipic acid (Merck) in 1:1 ratio. The calculated amount of salt was dissolved in methanol and stirred for 2 hours to attain a homogenous solution then the solution was filtered, covered and placed in the shelve for few weeks. After 23 days IMA crystals were harvested, the grown IMA crystal size is 12 x 2 x 1 mm^3. Figure 1 shows the photograph of IMA crystal.

Fig 1. Photograph of IMA crystal

3 Results and Discussion

3.1 Powder X-ray diffraction analysis

The powder X-ray diffraction pattern was recorded for IMA crystal using a X-ray diffractometer with Cu Kα (λ= 1.540598 Å) radiation over the range 10-70° at a scan rate of 1°/min. Figure 2 shows the X-ray diffraction pattern of IMA.

3.2 Single crystal XRD

Single crystal XRD of IMA crystal was analyzed by an ENRAF NONIUS CAD4 instrument. The lattice parameters of IMA crystal were shown in the table 1. The grown

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IMA crystals belong to Triclinic system with P-1 space group.

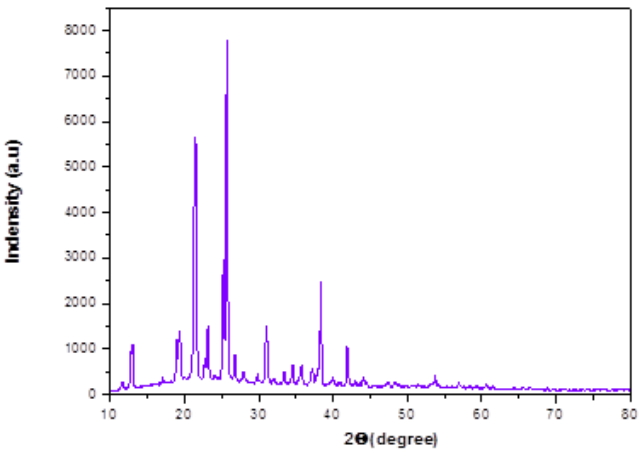

Fig 2. XRD pattern of IMA crystal

Table 1. shows the lattice parameters of IMA crystal.

Lattice parameters	Reported [2]	IMA crystal
a [Å]	7.484	7.50
b[Å]	8.934	8.94
c[Å]	10.313	10.33
V[Å^3]	576.38	580
α	104.92	104.88
β	109.01	109.12
ϒ	106.55	106.40

3.3 Fourier Transform infrared spectral analysis

The functional and vibrational spectrum of Imidazolium adipate (IMA) crystal was analyzed by using Perkin Elmer FT-IR spectrum 2 instrument in the wavenumber region of 400 to 4000 cm^{-1}. The assigned NH stretching vibration is arises at 3144 cm^{-1}.[6] The expected CH_3 asymmetric stretching is in the frequency of 2941 cm^{-1}.

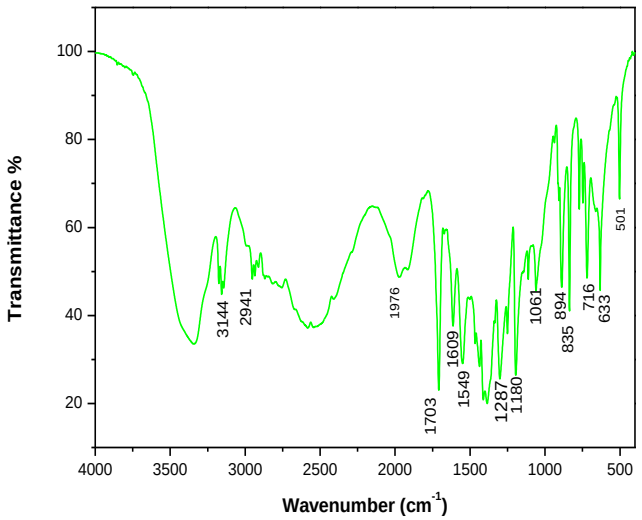

Fig 3. Shows FTIR spectrum of IMA crystal

In this spectrum 1703 cm^{-1} represents the C=O stretch and the 1609 cm^{-1} is the C=C stretching vibration respectively. The peak at 1549 and 1362 cm^{-1} are corresponds to NO_2 Asymmetric stretching and CH_2 bending vibration. The modes of O-H plane bending and NH rocking are containing the following wave numbers 1287 and 1180 cm^{-1}. The C-C stretching and CH bending vibration of sharp peaks are obtained in the frequencies 894 and 835 cm^{-1}. The frequency at 627 cm^{-1} is containing the C-N-O bending vibration. Figure 3. shows the FTIR Spectrum of IMA crystal.

3.4 UV –Visible spectral analysis

The optical property of the grown IMA crystal was analyzed by Varian Cary Bio 50 spectrometer in the wavelength ranges from 200- 800 nm. The lower cutoff wavelength is about 242 nm and the transmittance of IMA crystal is around 90%. The good transmittance of IMA crystal shows that it is a possible material for optoelectronic application.[7] Figure 4 shows the UV-vis-NIR spectrum of IMA crystal.

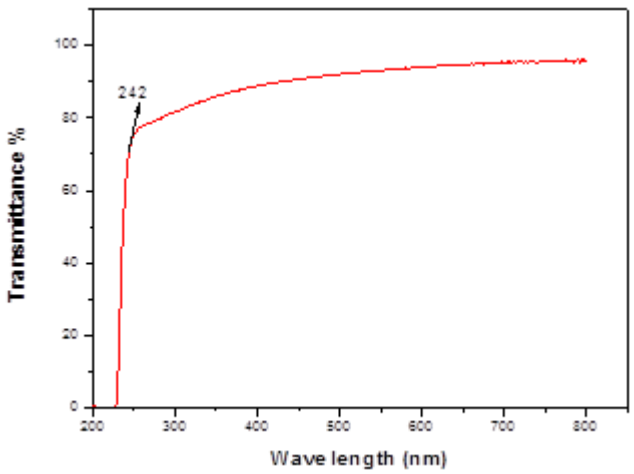

Fig 4. UV-Visible spectrum of IMA

3.5 Microhardness

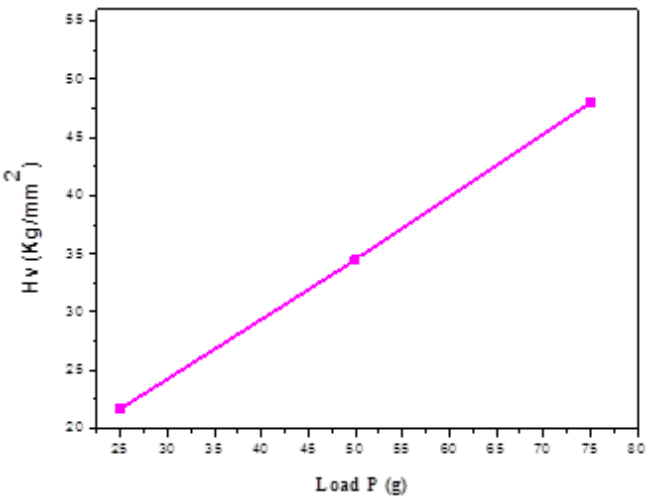

Fig 5. Plot of Vickers microhardness against load for IMA

The Vickers's Micro hardness of IMA crystal was calculated by the formula Hv=[1.8544/d²] kg/mm². Where microhardness number is denoted as Hv. P and d are the applied load and average diagonal length of the indentation. The time kept for indentation is 5s. the Vickers micro hardness of IMA at 25, 50 and 75g are 21.5, 34.5 and 47.2 kg/mm². Beyond 75g of a load a significant crack developed on the surface of the crystal. Therefore, it is suggested that IAA crystal can be potential candidate for the device below 75g of load. Figure 5 shows the plot of Vickers microhardness against load for IMA crystal.

3.6 Dielectric studies

The IMA crystal was subjected to dielectric studies at the different temperature using a HIOKI 3532 LCR HITESTER in the frequency region from 50 Hz to 5 MHz. The dielectric constant (ε_r) and dielectric loss have been calculated [8] using equations (1) and (2)

$$\varepsilon = {Cd}/{\varepsilon_o A} \quad \text{----------(1)}$$

$$\varepsilon = \varepsilon \tan \delta \quad \text{----------(2)}$$

Where

d is the thickness of the sample
A is the area of the sample
C is the capacitance
ε_o is permittivity of free space (8.854 x 10^{-12}).

The dielectric constant of IMA crystal at lower frequency is found to be having high values, when we proceed further it decreases with increase in frequencies. The high dielectric constant at low frequencies are arising because of the presence of all types of polarization. If the frequency increases, dielectric loss was decreases. It can be considered as good chemical homogeneity of the crystal with less defects. So, we may use this material for device fabrication application in the field of optoelectronics. Figure 6 & 7 shows the dielectric loss and dielectric constant of IMA crystal.

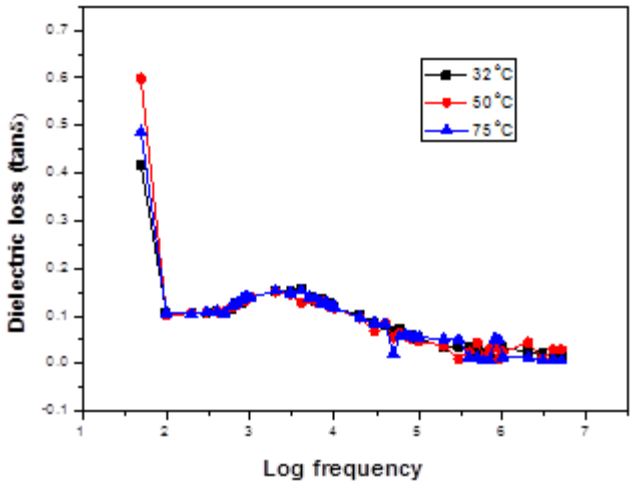

Fig 6. shows the Log frequency Vs Dielectric loss of IMA crystal

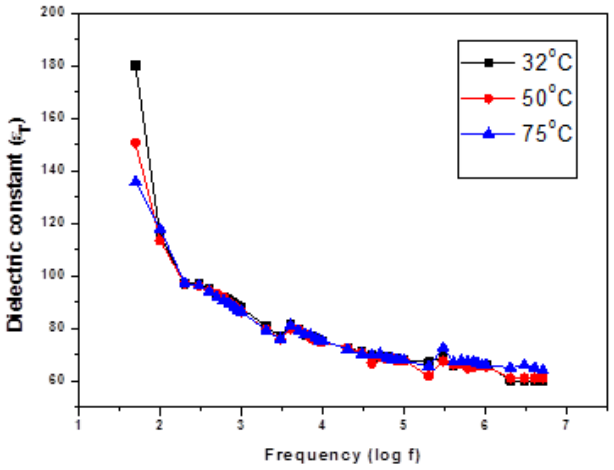

Fig 7. shows the Log frequency Vs Dielectric loss of IMA crystal

4 Conclusion

The Imidazolium adipate (IMA) crystal was grown by slow evaporation solution growth technique by taking methanol as a solvent at room temperature. The grown crystal was about 12 x 2 x 1 mm³. The single crystal XRD reveals very well matches with the reported paper. The Fourier Transform Infra-Red (FTIR) analysis shows various modes of functional group present in the crystal. UV-vis-NIR spectrum shows that the lower cutoff wavelength is about 242 nm and the transmittance of IMA crystal is around 90%. Hence, we may use this crystal for optoelectronic application. Vickers microhardness study reveals that the crystal can withstand the load of about 75g above which significant cracks appears in the crystals. From the Dielectric study it is found that when the frequency increase there is a decrease in the Dielectric loss which shows that the crystal has less defects. Hence this crystal is a potential candidate for opto electronic applications.

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