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UV, SEM, Powder XRD Studies of Acetoacetanilide (AAC) Crystal

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Abstract: Non linear optics is concerned with the proper conglometric symmetrical applications in different fields. Organic NLO's are predominant type of materials with proper SHG/THG. Here the present paper deals with the second order NLO material acetoacetanilide with UV-Vis Spectral study, powder XRD study and SEM study.
Key words: Acetoacetanilide, UV-Vis spectral study, SEM micrographs and XRD, NLO.

1. Introduction

Nonlinear optics is concerned with the interaction of electromagnetic fields with various media to produce new electromagnetic fields altered in phase, frequency or amplitude from the incident fields. Second harmonic generation (SHG) is an example of second-order nonlinear optical (NLO) process. (1-7). In the present study, bulk crystal larger in size than previously reported crystals of acetoacetanilide (AAC) has been grown and Powder x-ray diffraction study, scanning electron microscope (SEM), optical study were conducted and detailed report has been presented in this paper.

2. Experimental Procedure

2.1 Determination of solubility

The principal experimental factor that defines the growth rate of a crystal is the dependence of solubility of the substance on temperature. Solubility data of a material govern the amount of the material, which will be available for the growth and thereby defines the total size limit. Hence, the analysis of the solubility of a material in a particular solvent is an essential criterion in solution growth. Before proceeding with the growth of larger size crystals, the solubility of acetoacetanilide in acetone was determined at different temperatures. The commercially available materials are less pure (AR grade). In order to improve the purity of the raw material, repeated recrystallization processes were carried out. The solubility of AAC in acetone was measured at six different temperatures (30, 35, 40, 45, 50 and 55 ^{o}C). The solubility data was determined by dissolving the synthesized salt of AAC in 100 ml of double distilled water at a constant temperature with continuous stirring.

After attaining the saturation, the equilibrium concentration of the solute was analyzed gravimetrically. Fig.1 shows the solubility curve of AAC at different temperatures.

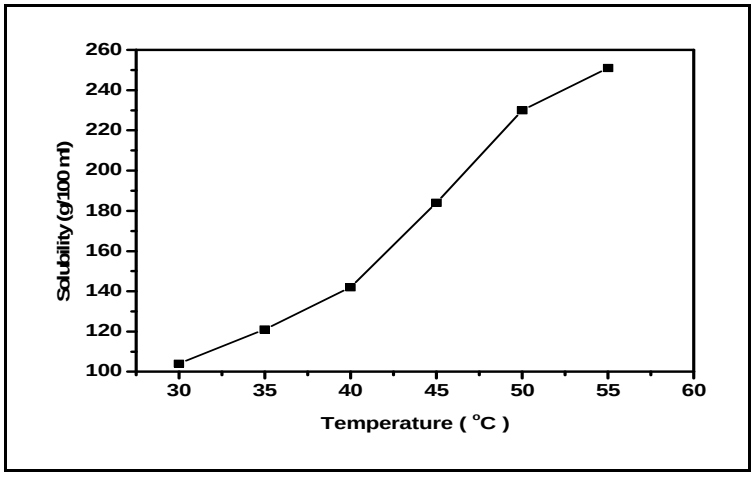

Fig.1 Solubility curve of AAC

2.2 Growth of AAC crystal

Single crystals of AAC were grown by slow solvent evaporation technique at room temperature. Saturated solution was prepared according to the solubility data (Fig.1), using the recrystallized salt of AAC. The solution was stirred for one day using magnetic stirrer and then filtered using filter paper. The solution was then kept at room temperature to evaporate the solvent. Seed crystals of AAC were formed due to spontaneous nucleation in a period of 7-10 days. Good transparent single crystals were harvested from the mother solution. The grown single crystals are shown in Fig.2.

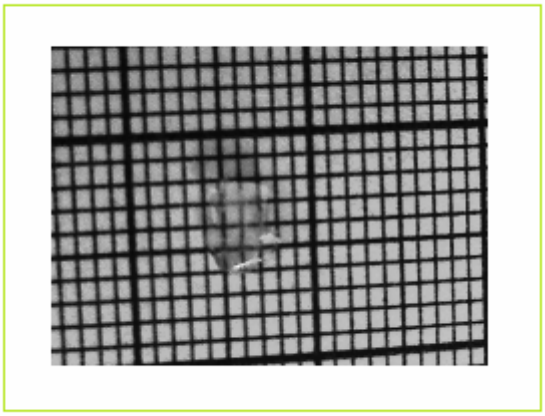

Fig.2 Photograph of as grown AAC crystal

3. Characterizations

The grown seed crystals were powdered and used to study the properties using various techniques. Powder X-ray diffraction analysis (PXRD) was carried out using an X-ray diffractometer, with monochromatic nickel filtered CuK_{α} (λ = 0.15406 nm) radiation. The optical absorption spectrum was recorded in the range of 200-800 nm using VARIAN CARY 5E UV-Vis-NIR SPECTROPHOTOMETER and SEM by microscope with high resolution.

3.1 Powder X-ray diffraction analysis

The grown crystals were made as fine powder and subjected to powder x-ray diffraction analyses. The sample was scanned over the range 10 - 80° at the rate of one degree/minute. The input voltage and current were 35 kV and 30 mA respectively, and the slit width was 0.1 mm. The recorded powder X-ray diffraction

pattern of AAC is shown in Fig.3. The differences in the peak amplitudes can be attributed to the different sizes and orientations of the powdered grains. The diffraction pattern contains various reflections corresponding to various crystallographic planes. All the observed reflections were indexed. The (*h k l*) planes satisfy the general reflection conditions of space group observed from single crystal XRD. The calculated lattice parameter values indicate that the AAC is orthorhombic crystal system and the space group is $P2_12_12_1$. The sharp peeks of the pattern have been observed due to the good quality of crystalline nature.

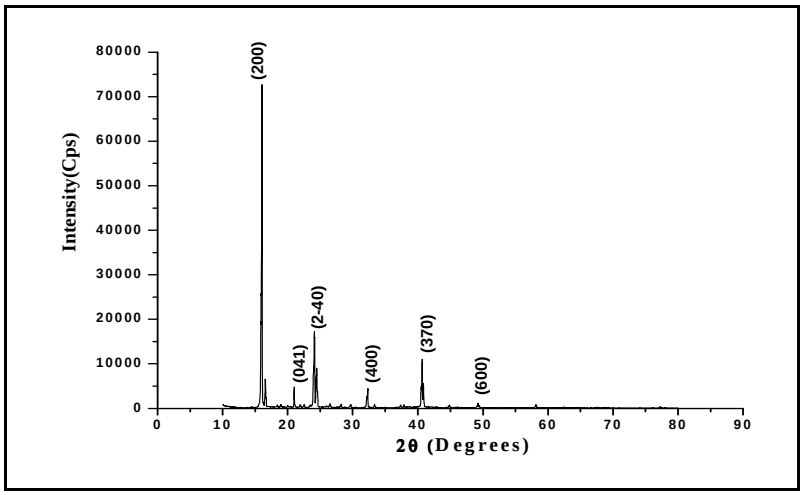

Fig.3 Powder X-ray diffraction pattern of AAC

3.2 Optical absorption spectrum

The optical absorption spectrum of AAC was recorded using a crystal of thickness 1 mm. It is evident from the spectrum (Fig.4) that AAC has its cut-off wavelength around 350 nm. Present work has low value of cut-off wavelength when compared with reported value of acetoacetanilide crystals (8). The absorbance is found to be nearly equal to zero in the entire visible region, which is a desirable property for the crystals used for NLO applications.

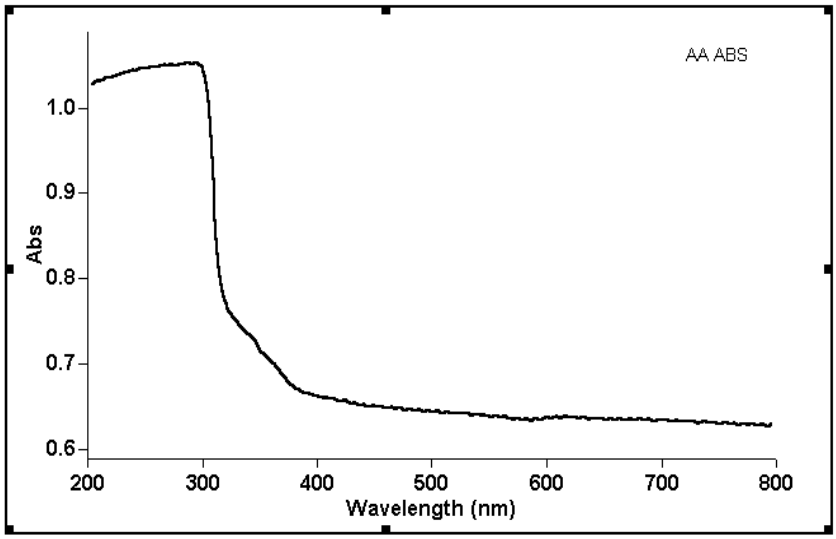

Fig.4 Optical absorption spectrum of AAC

3.3 SEM analysis

The surface morphology of the as grown sample of AAC was investigated using a scanning electron microscope (SEM). Basically the scanning electron microscope employs an electron beam of about 10^{-10} amp at about 20 keV. The as grown crystals with well defined planes and of smooth surface were selected for the SEM analysis and no polishing was done. The SEM micrographs of AAC taken at room temperature with different magnification are shown in Fig 5a-c; which suggests the existence of grain boundaries in the sample. It is clear

from the SEM micrographs of AAC (Fig.5a) that the crystal surface contains voids of irregular size. The presence of valley and cracks are predominantly seen on the surface of the crystal. In Fig. 5a, layer like growth pattern is seen on the crystal. Traces of micro nucleations are also observed. Fig. 5b shows many clusters of micro crystals on the surface of LADN crystal. Figs. 5a-c illustrate that the crystal surface on the whole appear smooth but there are also few isolated islands and as like a steps over the surface.

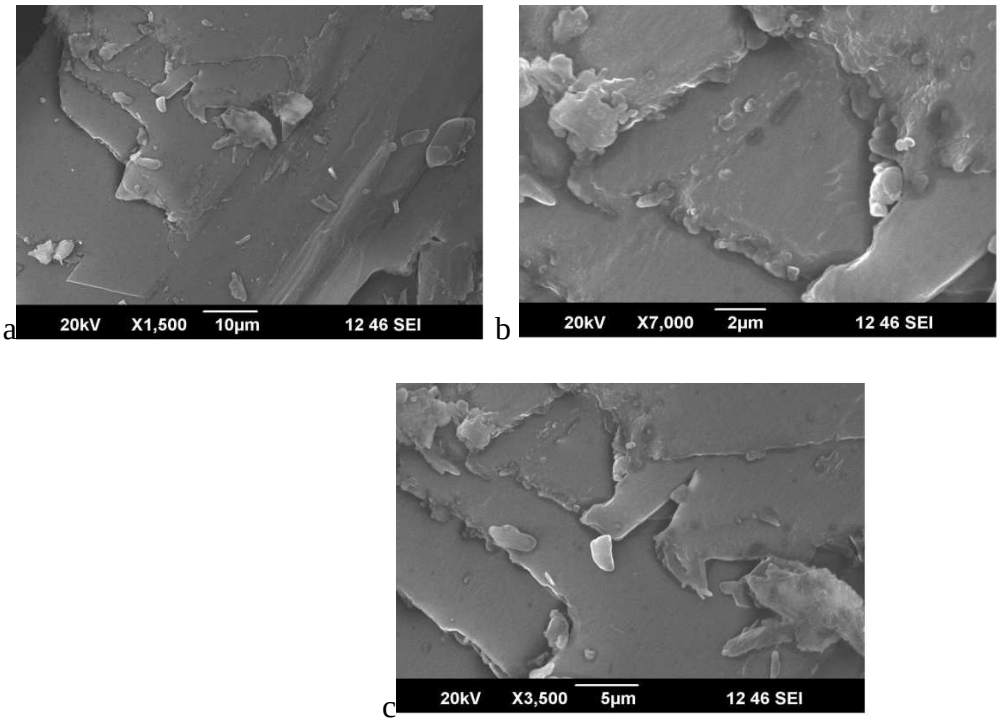

Fig. 5 SEM micrographs of AAC

4. Conclusion

Good quality AAC single crystals were grown by slow solvent evaporation technique at room temperature in a period of 7-10 days. A solvent of acetone is used for the growth process. The grown crystal was confirmed by powder XRD analysis. The powder XRD data proves that AAC crystal belongs to orthorhombic in structure with a non centro symmetric space group $P2_12_12_1$. Optical absorption studies confirm the UV cut-off wavelength of AAC at 300 nm. The SEM micrographs indicate the surface morphology of AAC crystal.

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