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Growth of non-linear optical γ-glycine single crystals and their characterization

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Abstract

The non-linear optical γ-glycine single crystals were grown by low temperature solution growth methods from aqueous solutions with incorporation of known amount of sodium chloride as additive. Crystals were grown from pure aqueous solutions of glycine with a range of sodium chloride concentration. The change in morphology of the α-form of glycine with sodium chloride concentration and the occurrence of γ form at a critical concentration of sodium chloride were studied. The form of crystallization was confirmed by X-ray power diffraction method and the optical transparency of the γ-glycine in the UV–Vis–near IR region was studied by recording the optical transmittance spectrum.
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1. Introduction

Crystal growth of organic non-linear optical materials has significantly attracted in recent years because of their high value of non-linear optical coefficients. Glycine, a well-known amino acid, crystallizes in three different polymorphs: α, β, and γ [1–3]. While α and β forms crystallize in centrosymmetric class, the γ-glycine crystallizes in non-centrosymmetric space group $P3_2$ making it a potential candidate for non-linear optical applications especially for effective optical second harmonic generation [3,4]. It is reported that its second harmonic energy conversion efficiency is about 1.5 times that of KDP [5]. α and β forms crystallize from pure aqueous solutions but γ form results from aqueous solutions with selected acetic or alkaline incorporations. β form is a most unstable. In the present work, single crystals of γ-glycine were grown from aqueous solutions with incorporation of a range of sodium chloride (NaCl) concentration by low temperature solution growth methods. The influence of NaCl addition on the form of crystallization and morphologies of the crystals were studied. X-ray powder diffraction was used to confirm the form of crystallization and the optical transparency was studied by UV–Vis–near IR spectroscopy.

2. Experimental

The commercially available analytical grade glycine (amino acetic acid: NH_2CH_2COOH), sodium chloride and double distilled (DD) water were used for the crystal growth experiments. NaCl salt of different weights in the range from 1 to 12 g in 100 ml of double distilled water were dissolved in separate but similar vessels and glycine salt of known weight were added in each of the vessels, stirred well for about 6 h and saturated solutions were prepared at room temperature. Also, a pure aqueous solution was prepared in a similar vessel for comparison. The variation of solubility of glycine with NaCl concentration

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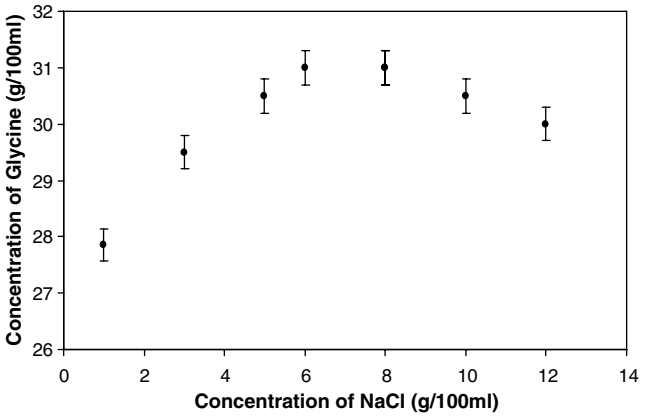

Fig. 1. Variation of glycine solubility in DD water with NaCl concentration.

was determined by gravimetric method and is indicated in Fig. 1.

The solutions were filtered with Borosil No. 1 filter sheets and kept in a dust free atmosphere for evaporation. Nucleation was observed in the interval of 2–10 days from solutions with different concentrations of NaCl; while higher mixing concentrations of NaCl yield quick nucleation and higher rate, smaller concentrations yield slower nucleation with lower rate. The nucleated solutions were allowed for further growth and then harvested. Morphology of the resulted crystals from NaCl incorporated solutions was compared with the one resulted from pure solution. X-ray powder diffraction (XRD) spectrum of the crystals were recorded on an automated analytical MPD model diffractometer using Cu Kα radiation of wavelength $\lambda = 1.5406$ Å by step scanning between 10° and 70° in 2θ. Optical transmittance spectrum was recorded for the grown crystals in the region 200–3000 nm by 500 Scan UV–Vis–NIR spectrometer.

3. Results and discussion

3.1. Growth of α-form and the effect of NaCl concentration on morphology

Morphology of the grown crystals depends very much on the concentration of NaCl in the solution. Photograph of some of the crystals grown from pure aqueous solution is shown in Fig. 2. These have the usual morphology of α-glycine with comparatively long growth along the 'c' direction than 'a'. NaCl incorporation changes the actual morphology to the extent that it enhances the growth along the 'a' direction and reduces the growth along 'c'. Because of this, the end morphology of the crystals appears more elongated along 'a' direction than that of 'c'. This morphological change is more when the NaCl concentration increases. Photograph of some of the crystals grown from solution with 5 g NaCl concentration is shown in Fig. 3 for comparison.

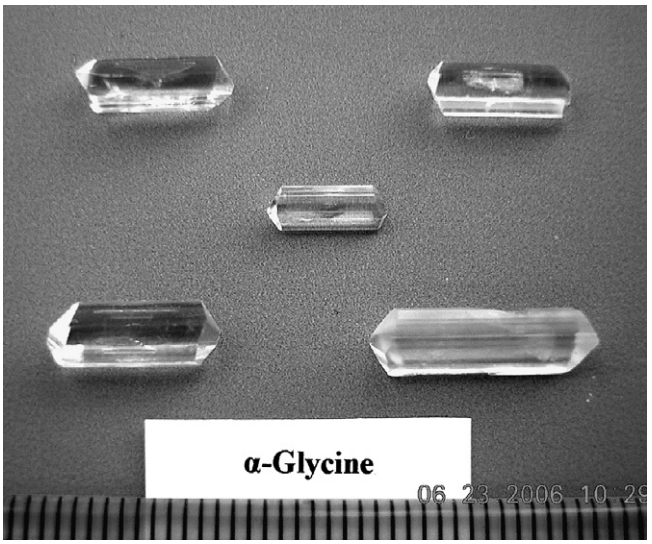

Fig. 2. α-Glycine single crystals grown from pure aqueous solution.

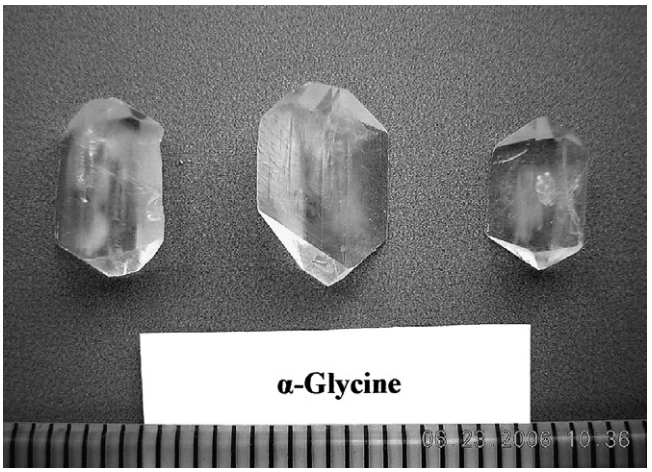

Fig. 3. α-Glycine single crystals resulted from solution with 5 g of NaCl concentration in 100 ml of water.

The variation of crystal length along 'a' and 'c' directions of the grown crystals with NaCl concentration is shown in Fig. 4. This situation continuous until the NaCl concentration has reached a critical level (about 8 g in 100 ml of DD water) in the solution and the crystals resulted out of these solutions are α-form. This is confirmed by XRD.

3.2. Growth of γ-form and optimum level of NaCl concentration

Above the critical concentration of NaCl (>8 g) the solution yield crystals with different morphology and also belong to γ-form. Photograph of some of the crystals resulted from aqueous solution with 8 g NaCl concentration is shown in Fig. 5. The resulted crystals have trigonal end with smooth facets at one end and truncated rough facets on the opposite end, which clearly indicates that the

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