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Original research article

A comparative study on the effect of L-tartaric acid and sodium bicarbonate on the growth and characterization of KDP single crystals

S. Nirmala Sri Devi, E.K. Girija *

Department of Physics, Periyar University, Salem, Tamil Nadu, India

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ABSTRACT

Potassium dihydrogen phosphate (KDP) is one of the efficient inorganic single crystals employed in second harmonic generation (SHG) conversion applications. Present work describes the growth and characterization of optically transparent single crystals of KDP and KDP in presence of two different concentrations of L-tartaric acid and three different concentrations of sodium bicarbonate by slow evaporation solution growth technique at ambient temperature. FT-IR analysis did not reveal any main vibrations of LTA or $NaHCO_3$. The UV–Vis–NIR study suggest that the crystals are highly transparent in the region 373–1200 nm and percentage of transmittance slightly increased in case of KDP crystals grown in presence of 0.1 M% sodium bicarbonate. Improvement in SHG efficiency was observed in KDP grown in presence of 0.1 M% L-tartaric acid. Thermal stability of the KDP grown in presence of impurities showed marginal increase when compared to pristine KDP.

1. Introduction

Potassium dihydrogen phosphate (KDP) in the crystalline form is an optical material, widely used in frequency conversion because of its high conversion efficiency and high laser damage threshold. This crystal is of special interest due to its wide applications as ferroelectric, piezoelectric, pyroelectric as well as laser modulator material. Due to excellent piezoelectric and nonlinear optical properties, it is used as a standard material to characterize the noninear optical (NLO) response of other crystals. It is widely applied as laser radiation converter and Q-switch in laser fusion for its high electro optic and nonlinear coefficient, wide frequency conversion and high damage threshold against high power laser [1–3].

On account of various applications, growth of KDP crystals in presence of impurities as additives/dopants is increasingly interesting because of the fact that impurities have various effects both in habit modification and crystal properties [4–8]. Several reports are available in the literature on the effect of various organic, inorganic and semiorganic additives on KDP single crystals [9–13].

L-tartaric acid (LTA, $C_4H_6O_6$) is a NLO organic material and its second harmonic generation (SHG) efficiency is comparable to that of KDP. Also the laser damage threshold value of LTA has been found to be higher than KDP [14]. Metal ion dopants such as Ni^{2+}, Al^{3+} and Na^{+} in KDP was found to enhance the linear optical and NLO properties, thermal and mechanical stabilities when compared to pristine KDP [13,15]. Mn^{2+} ion in KDP was already reported to influence the optical properties

* Corresponding author.
E-mail address: girijaeaswaradas@gmail.com (G. E.K.).

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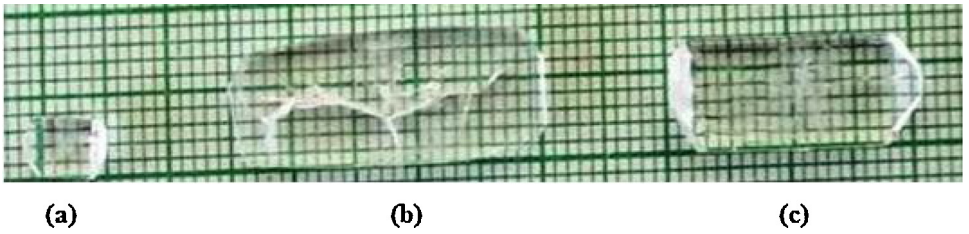

Fig. 1. Photograph of the grown crystals (a) pristine KDP, KDP grown in presence of (b) 0.1 M% and (c) 1 M% LTA.

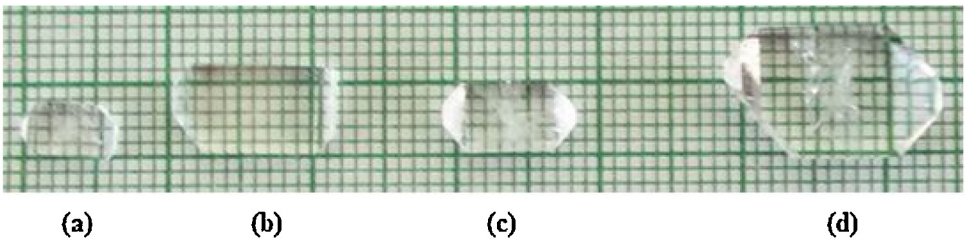

Fig. 2. Photograph of the grown crystals (a) pristine KDP, KDP grown in presence of (b) 0.1 M%, (c) 1 M% and (d) 10 M% of $NaHCO_3$.

[16]. In some cases the substitution of metal dopants into the lattice is also found to retard the growth process [17]. On the other hand, the substituted impurities may also lead to a reduction in the free energy, which results in an increase in crystal growth rate [18]. In the present work, the influence of two different concentrations of LTA and Na^+ ion (in three different concentrations) in KDP crystal and their properties were investigated.

2. Experimental details

KDP, L-tataric acid (LTA) and sodium bicarbonate ($NaHCO_3$) were procured from Merck (GR grade). Double distilled water was used as the solvent for the growth of the crystals by slow evaporation solution growth technique (SEST). A saturated aqueous solution of KDP was prepared and was stirred for 4 h using magnetic stirrer for homogenization. KDP grown in presence of impurities was prepared by dissolving 0.1 M% and 1 M% of LTA, 0.1 M%, 1 M% and 10 M% of $NaHCO_3$ in saturated solution of KDP and again stirred for 2 h. Pristine and impurity added KDP solutions were filtered and taken in clean beakers, covered with perforated polythene sheet and allowed for slow evaporation at ambient temperature.

3. Characterization

Powder X-ray diffraction (XRD) patterns of the grown crystals were recorded using Rigaku Mini Flex II desktop X-ray diffractometer with CuK_α radiation of wavelength 1.5406 A° in the range $20^\circ \leq 2\theta \leq 60^\circ$. FT-IR of solid phase samples were analyzed using Perkin-Elmer RX1 in the range 400 – 4000 cm^{-1} using KBr pellet technique at room temperature. UV spectrum was traced with Lambda 35 UV–Vis–NIR spectrometer in the wavelength range 190 – 1100 nm at room temperature. Differential thermal analysis and thermo gravimetric analysis curves were recorded for the grown crystals using a simultaneous thermal analyzer TGA7 (Perkin Elmer), Q500 Hi-Res V20 in the temperature range from room temperature (RT) to 913 °C at a heating rate of 10 °C/min. Composition of sodium ion in KDP single crystal was determined by inductively coupled plasma optical emission spectroscopy (ICP–OES) in Perkin Elmer Optima 5300 DV spectrometer. Kurtz SHG test was performed to find the NLO property using a Q-switched Nd:YAG laser (1064 nm, Quanta ray series with input energy: 2.9 mJ/pulse).

4. Results and discussion

When compared to pristine KDP, crystals grown in presence of impurities are found to be large in size. Photograph of the grown crystals are shown in Fig. 1. and Fig. 2. and their dimensions are found to be $7 \times 4 \times 3$ (pristine KDP), $26 \times 9 \times 4$ (0.1 M% LTA-KDP), $21 \times 7 \times 4$ (1 M% LTA-KDP), $12 \times 6 \times 3$ (0.1 M% $NaHCO_3$-KDP), $9 \times 5 \times 3$ (1 M% $NaHCO_3$-KDP) and $14 \times 9 \times 4$ (10 M% $NaHCO_3$-KDP). The grown crystals are found to be stable, colorless and transparent in the ambient temperature.

4.1. XRD

The XRD patterns of pristine KDP, KDP crystals grown in presence of LTA and $NaHCO_3$ are shown in Fig. 3. Comparison of XRD patterns with the JCPDS data for KDP [No. 84-1557] revealed the presence of almost all the peaks in the grown crystals without any additional peaks which indicates the phase purity of the samples. The sharp peaks of XRD patterns indicate the