



ISSN NO. 2320-5407

Journal homepage: <http://www.journalijar.com>

INTERNATIONAL JOURNAL  
OF ADVANCED RESEARCH

## RESEARCH ARTICLE

## Growth And Characterization of Ginger Extract Doped KDP Crystal.

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### Manuscript Info

#### Manuscript History:

Received: 11 January 2015

Final Accepted: 25 February 2015

Published Online: March 2015

#### Key words:

UV-Vis spectroscopy, FTIR spectroscopy, powder X-ray diffraction studies, NLO measurements

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### Abstract

Pure and Ginger extract doped KDP crystals were grown from aqueous solution by natural evaporation method. The grown Ginger extract doped KDP crystals were characterized by UV-Vis spectroscopy, FTIR spectroscopy, Powder X-ray diffraction studies, Second harmonic generation efficiency measurements, Thermo gravimetric, differential thermal analysis (TA / DSC) etc. Ginger extract doped KDP crystals were found to be highly transparent and full faced. The experimental results evidence the suitability of the grown crystal for optoelectronic applications.

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## INTRODUCTION

The numerous applications of the nonlinear optical (NLO) crystals in the vast field of science and technology made the process of search of the new NLO crystals and improvements in the properties of the known crystals a never ending process. KDP crystal is a widely used and thoroughly studied NLO crystal [1–4]. Many researchers have tried to modify the properties and growth rate of the KDP crystal by either changing the growth conditions or by adding different impurities [5–7]. The NLO and other properties of the crystal have been improved by doping of organic impurities. Potassium dihydrogen phosphate (KDP) is one of the first and best-known electro-optic crystals. It is a member of a broad family of isomorphous compounds having a generic composition  $MY_2XO_4$ , where  $M = K$ ,  $Y = H$  and  $X = P$ . KDP crystal has considerable interest among several research workers because of its wide frequency conversion efficiency, good UV transmission and high damage threshold against high power laser.

Ginger is one of the world's top ten favored spices. Its molecular formula is  $C_{11}H_{14}O_3$  and IUPAC name is 4-(4-hydroxy-3 methoxy phenyl)-2-butanone. Ginger has been promoted for cancer treatment "to keep tumors from developing". It was found to be more effective than Placebo for treating nausea caused by sea sickness, morning sickness and chemotherapy.

In the present investigation the ginger extract was added to the KDP and it was observed that the SHG efficiency was enhanced by 1.1 times that of Pure KDP. In the present work pure and ginger extract doped KDP crystals were grown by slow evaporation technique and the grown crystals are subjected to X-ray diffraction, FTIR spectroscopy, UV-visible spectral analysis, second harmonic generation, Thermo gravimetric, differential thermal analysis (TA / DSC) etc and the corresponding results have been compared with pure KDP crystal from literature.

## II. Experimental Procedure and Characterization

Potassium Dihydrogen Phosphate (KDP) is a well-known inorganic salt, which has been purified by repeated recrystallization using the method of dissolving in distilled water. Then the solution of KDP salts have been prepared by stirring well for three hours constantly using magnetic stirrer, till the salts have been fully dissolved in water. The prepared solution has been transferred into two clean petri dishes and kept for crystallization at room

temperature in a quiet place. Within four days the nucleation took place and a seed crystal in petri dish has been obtained.

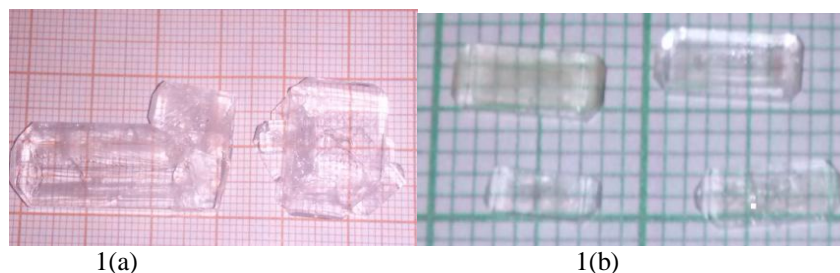


Figure 1. Photograph of the grown crystals of (a) Pure KDP and (b) Ginger extract doped KDP

To obtain ginger extract doped crystal, ginger extract was added to the saturated mother solution. Highly transparent and full faced crystals were obtained within three weeks. The pure KDP and Ginger doped KDP crystals grown are colorless and are shown in Figure 1 (a) and 1(b) respectively. A slow evaporation method has been employed to grow ginger extract doped KDP crystal [8].

### III. Results and Discussion

#### 1. Powder X-ray Diffraction (XRD) Analysis

X-ray diffraction technique is a powerful tool to analyze the crystalline nature of materials. Powder X-ray diffraction analysis was carried out by using PANalytical X-Ray diffractometer with CuK $\alpha$  radiation ( $\lambda=1.5406 \text{ \AA}$ ). The samples were scanned over the range  $10^\circ - 80^\circ$ . The XRD pattern of grown ginger extract doped KDP is shown in Figure 2.

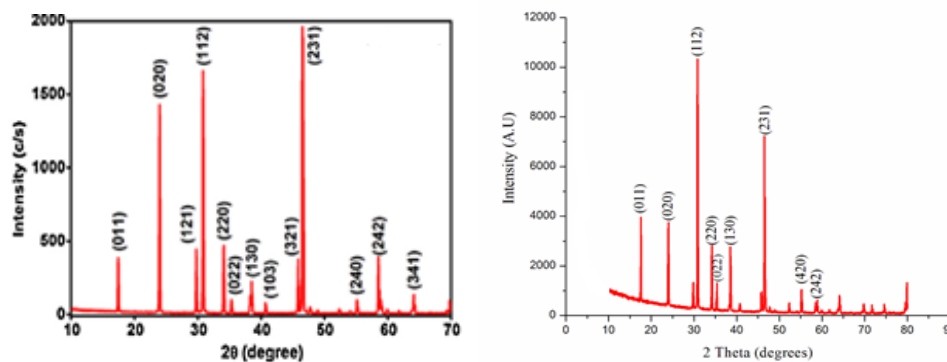


Figure 2. Powder XRD pattern of (a) pure KDP and (b) ginger extract doped KDP.

#### 2. Optical property study

The optical properties of a material are important, as they provide information on the electronic band structures, localized states and types of optical transitions. For optical applications, especially for second harmonic generation, the crystal should be highly transparent in a considerable range of wavelengths. Optical transmission spectra were recorded for the grown crystals using a SHIMADZU UV-Spectrometer 1601 in the range of 200 to 800 nm. The recorded absorbance spectra and reflectance spectra of ginger extract doped KDP crystals in the wavelength range 200–800 nm are shown in Figure 3(a) and Figure 3 (b) respectively. It can be seen that the crystal has sufficient transmission in the entire visible region. The cutoff wavelength was found to be 275 nm. Optically polished crystal plates of 1.5 mm thickness were used for the measurement.

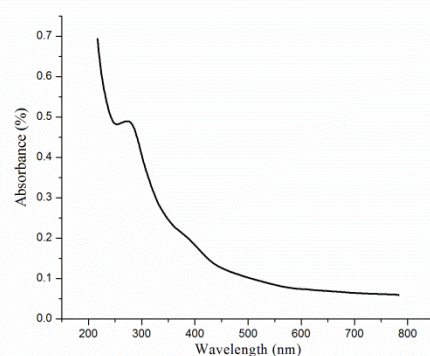


Figure 3 (a). Absorbance spectra of ginger extract doped KDP crystal.

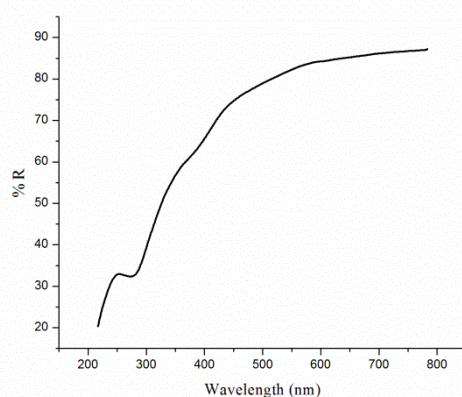


Figure 3 (b). Reflectance spectra of ginger extract doped KDP crystal.

### 3. FTIR spectral studies

The Infrared spectral analysis is effectively used to understand the chemical bonding and it provides information about the functional groups present in the synthesized compound. The FTIR spectra have been recorded in the range  $400$  to  $4000\text{ cm}^{-1}$ . The FTIR spectra of ginger extract doped KDP is shown in Figure 4. The observed frequencies and their assignments for pure and ginger extract doped KDP crystals are listed in Table 1.

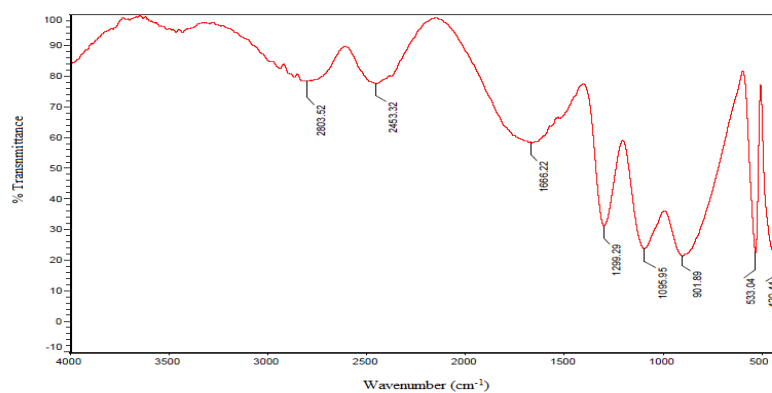


Figure 4. The FTIR spectra of ginger extract doped KDP

Table 1. The observed frequencies and their assignments for pure and ginger extract doped KDP crystals

Frequency ( $\text{cm}^{-1}$ )		Functional group assignments
KDP	Ginger extract doped KDP	
3615	-----	O-H stretching hydrogen bonded
3320	-----	O-H stretching
3155	-----	O-H stretching
2914	2803	P-O-H stretching
2329	2453	P-O-H bending
-----	1666	Carboxylic acid
1306	1299	P=O stretching
-----	1095	P-O stretching
-----	901	P-O-H stretching
531	533	HO-P-OH bending

#### 4. Second Harmonic Generation Efficiency Measurements

The first and the most widely used technique for confirming the SHG from prospective second order NLO materials is the Kurtz powder technique [9] to identify the materials with non-centrosymmetric crystal structures. The SHG conversion efficiency of ginger extract doped KDP crystal was studied using a 1064 nm Nd:YAG laser. The schematic of the experimental set-up is shown in Figure 5. The samples of ginger extract doped KDP were made in powder form. To make relevant comparisons with known SHG materials, KDP was also ground and sieved into the same particle size range. The powdered samples were filled air-tight in separate micro-capillary tubes of uniform bore of about 1.5mm diameter. A Q-switched, fundamental laser beam of 1064 nm wavelength from a Nd:YAG laser was made to fall normally on the sample cell. The power of the incident beam used was 4.4mJ/pulse. The input laser beam was allowed to pass through an IR reflector and then directed on the micro-crystalline powdered samples packed in a capillary tube. The photodiode detector and oscilloscope arrangements measured the light emitted transmitted by the sample. The SHG radiations of 532nm (green light) transmitted through the sample were collected by a photomultiplier tube (PMT-Hamamatsu-model R 2059). The optical signal incident on the PMT was converted into voltage output at the CRO (Tektronix-TDS 305213). Output intensity of SHG gives relative values of NLO efficiency of the material. It is found that the efficiency of SHG is 1.1 times of that of standard KDP.

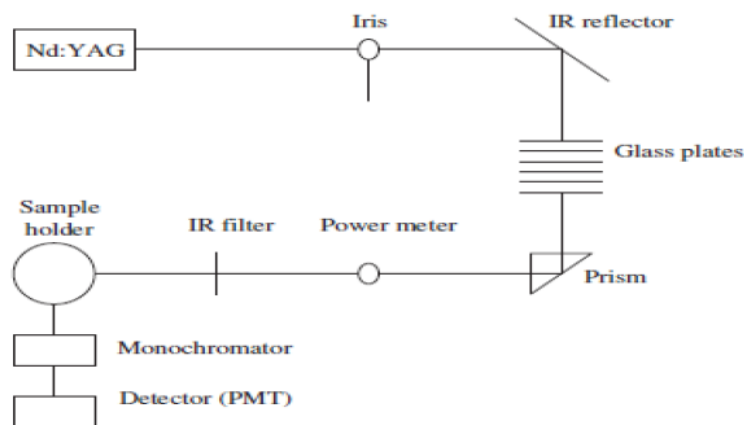


Figure 5. Schematic arrangement of the experimental setup used for measuring the SHG efficiency.

#### 5. TGA and DTA studies

To analyze the thermal stability [10-11] and to confirm the melting point of the material, the thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) were carried out using PL-STA 1500 thermal analyzer at a heating rate of  $20^\circ \text{C min}^{-1}$  in air. The TGA-DTA spectra of ginger extract doped KDP crystal is shown in Figure 6.

The recorded TGA curve of ginger extract doped KDP crystal exhibits negligible weight loss in the region 40°C to 200°C as shown in Figure 6. The sharpness of the endothermic peak shows the good degree of crystallinity of the grown sample [12-13]. The decomposition of the crystal begins at 221°C and terminates at 305°C. The weight loss starts due to the liberation of volatile substances, probably water molecule of decomposed KDP. The TGA curve shows that there is a weight loss in the temperature range 221 °C – 305°C due to the release of volatile substances, probably carbon dioxide or ammonia. Prolonged heating up to 723°C does not produce any exothermic or endothermic peaks in the DTA curve whereas TGA shows almost complete weight loss and the residual weight obtained at 700 °C is 11.24 %.

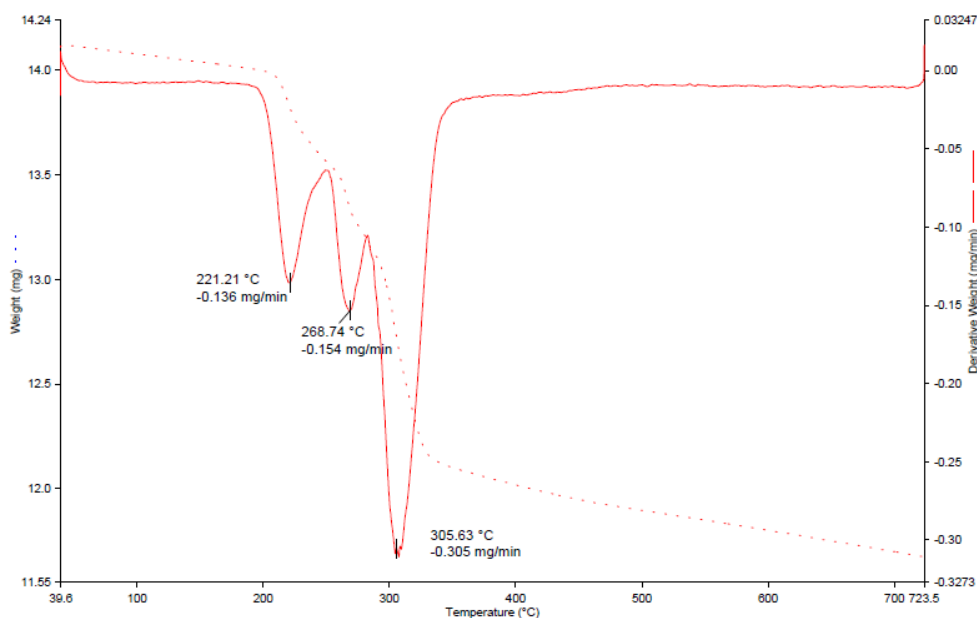


Figure 6. The TGA-DTA spectra of ginger extract doped KDP crystal is shown in.

#### IV. CONCLUSION

Optically good quality crystals of pure KDP and ginger extract doped KDP crystals have been grown by slow evaporation method. UV-Vis spectra showed that the grown crystal was optically transparent through 200-800 nm and hence suggests the suitability of this material for optical devices. Powder X-ray diffraction analysis confirms the crystalline nature of grown crystal. The FTIR spectral analysis confirms the presence of functional groups in the crystals. The SHG test proves that the grown crystals are potential candidates for nonlinear applications. The high optical transparency and SHG properties prove the optical quality and suitability of the grown KDP crystal doped with ginger extract for optoelectronic device applications.

#### ACKNOWLEDGEMENTS

The authors gratefully acknowledge the facilities offered by The Oxford College of Engineering, Bangalore, for doing this work and also place on record the support given by SAIF, Kochi, where the analyses were done.

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