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# Growth and characterization of pure and Magnesium Sulphate doped Potassium Hydrogen Phthalate crystals

**Muthukumaran.B, Santhanam.V, P. Paraimala**

Asst.Professor, Department of Chemistry, SCSVMV (Deemed to be University), Kanchipuram, India
Asst.Professor, Department of Chemistry, SCSVMV (Deemed to be University), Kanchipuram, India

**ABSTRACT**: Single crystals of $MgSO_4$ doped Potassium Hydrogen Phthalate were grown successfully by slow evaporation method. The dopant concentration was varied from 3 mmol to 7 mmol in the mother solution. The Fourier Transform infrared spectroscopy (FTIR) study confirms the incorporation of $MgSO_4$ into Potassium Hydrogen Phthalate crystal. The thermal study indicates the dissociating nature of the crystal. The nonlinear optical property of the grown crystal has been confirmed by Kurtz powder method. The dopant of 7m mol shows higher second harmonic generation result than pure Potassium Hydrogen Phthalate. Results are discussed.

**KEYWORDS**: KHP, FTIR, Slow evaporation, NLO

## I. INTRODUCTION

In the past few decades there has been a growing interest in crystal growth process particularly in view of the increasing demand for materials for technological applications [1-3].New materials are not usually discovered by device engineers or solid state theorists; they are mostly grown by crystal growers. More attention is paid on the growth of single crystals which is a vital and fundamental part of materialistic science and engineering, as single crystals of suitable size and perfection are required for lasers, optical communication, detectors, integrated circuits and data storage technology [4]. The search for new materials has identified novel semi organic systems of considerable potential and high performance. Potassium Hydrogen Phthalate, often called simply KHP (also known as potassium acid phthalate) is an interesting NLO material as an analyzer material in X-ray spectroscopy [5,6].

## II. SIGNIFICANCE OF THE SYSTEM

The semi-organic KHP crystallizes from its mother solution in the orthorhombic crystal system with a space group Pac21. Its higher chemical stability and economic feasibility with good kinetic growth properties have made to pay attention on it in the past decades. The aim of the present work is to grow and to explain the effect of incorporation of $MgSO_4$ in the thermal and mechanical properties of KHP.

## III. METHODOLOGY

### A. Preparation

Potassium hydrogen phthalate (KHP) crystal was grown from high purity salt in the aqueous solution. Solubility of KHP was maintained in the temperature range 30.c to 35.c and the growth solution was prepared according to the solubility data. A dopant magnesium sulphate in various concentrations (3 mmol, 5 mmol and 7 mmol was added to the parent crystal (KHP). The solution was stirred at 30°C and slow evaporation method was used for the growth of perfect crystal from aqueous solution. The FT-IR spectrum was recorded on BRUCKER ALPHA-T FTIR spectrometer in the range from 400- 4000 $cm^{-1}$. Powdered sample in KBr medium was used to record the FTIR spectrum. Powder X-ray diffraction analysis was performed to confirm the quality of the crystals and to indentify the cell dimensions using Bruker A X3D8 PERT-PRO, advance model powder diffractometer with Cuka radiations (I=1.5405984).

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## IV. EXPERIMENTAL RESULTS

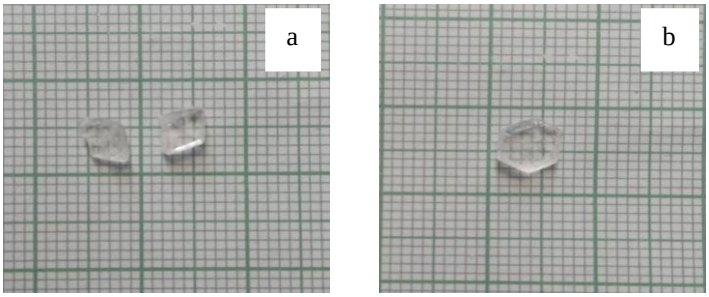


**Figure.1. a) Pure KHP b) $MgSO_4$ doped KHP crystals**

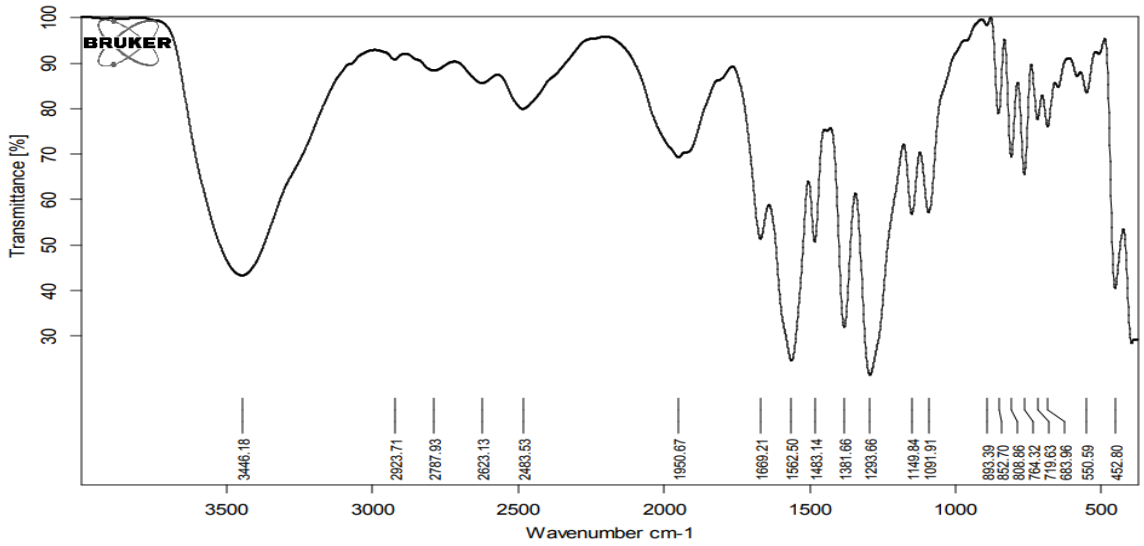


**Figure.2. FTIR spectrum of Pure KHP**

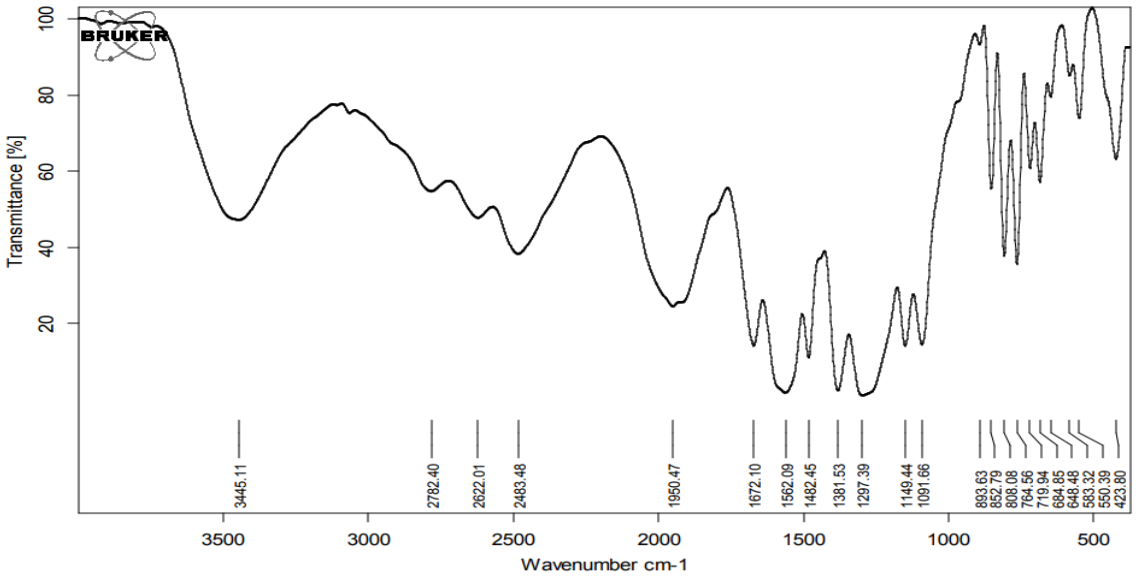


**Figure.3. FTIR spectrum of Pure KHP**

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| Pure KHP | Magnesium sulphate doped KHP | Assignments |
|---|---|---|
| 3550 | 3474 | O-H Stretching |
| 2924 | - | C-H Stretching |
| - | 2781 | C-H Stretching |
| - | 2620 | C-H Stretching |
| 2483 | 2482 | C-H Stretching |
| 1950 | 1949 | C=O,$O^{-}K^{+}$ Stretching |
| 1565 | 1561 | C=O Stretching |
| 1481 | 1482 | C-H Stretching |
| 1381 | 1381 | O-H bending |
| 852 | 852 | C-H out of plane bending,disubstituted aromatic stretching |

**Table.1 FTIR frequency table**

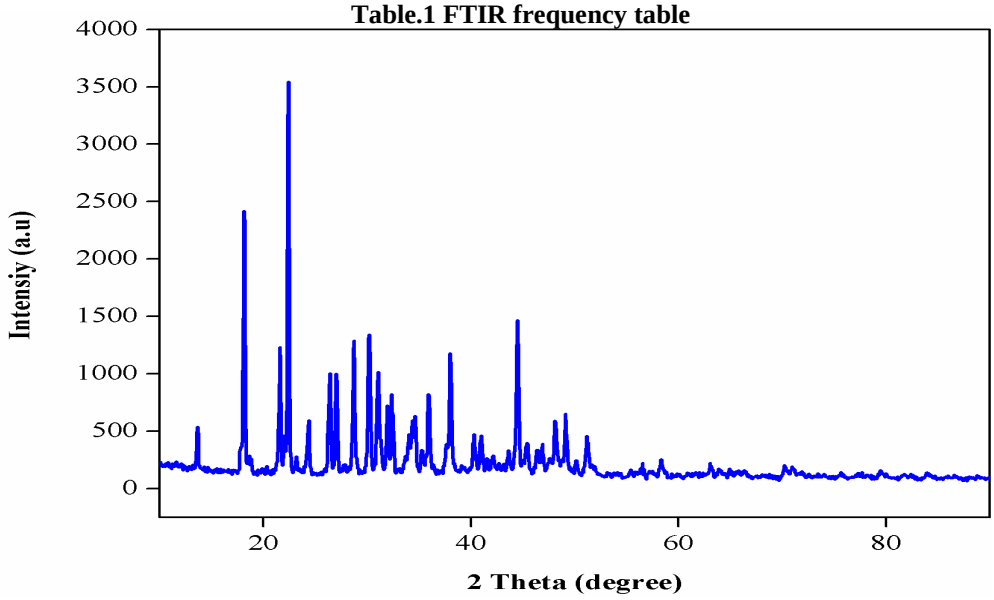


**Figure.4. XRD Pattern $MgSO_4$ doped with KHP**

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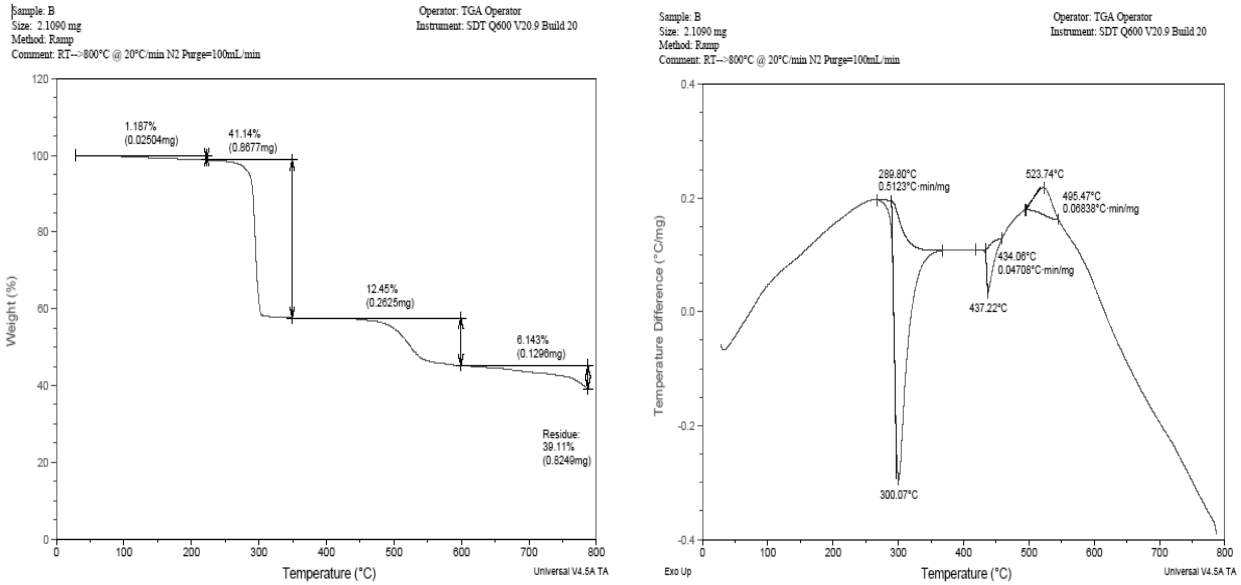


**Figure.5. TGA and DTA of $MgSO_4$ doped with KHP**

## V. DISCUSSION

The O-H stretching vibration gives a broad band at about 3000 cm-$^{1}$. The fine structure is due to hydrogen bonding. In the higher wavelength region, the peak at 3445.11 cm$^{-1}$ is assigned O-H stretching vibration. The peak at 1666 cm$^{-1}$ indicating the C=O stretching mode of vibration. The C-H vibration gives peaks at 2981 and 2854.37cm$^{-1}$. The peak at 2854 cm$^{-1}$ is very much intense, as the less polar C-H bond gives much distortion in the electron cloud (polarizability). The C-H bending mode occurs at 1381 cm$^{-1}$. The broad peak at 1288 cm$^{-1}$ is presented as an inset view, owing to symmetric COO- vibrations. Hence this group interacts with other groups in the crystal. Because of its interaction this group could also be a good contributor to the piezoelectric property of the crystals. The C-O vibration gives its peak at 1092cm$^{-1}$ which is very sharp hence, less interaction with the other groups in the crystal. The C-C stretching mode of vibration. The spectra show absorption bands in the region of 1145 cm$^{-1}$ and 1092 cm$^{-1}$ which are due to in-plane C-H bending vibration (Figure.2, 3). The ring deformation occurs the peak at 852cm$^{-1}$and 807cm$^{-1}$C=O deformation is identified by the band at 826 cm$^{-1}$. C-H out-of plane bending peaks obtained at 682 cm$^{-1}$ and 718 cm$^{-1}$. Figure.4 shows the XRD analysis of Magnesium sulphate doped KHP crystals. The crystal belong to orthorhombic system and its cell parameters are a= 9.674 Å, b=13.291Å, c=6.516 Å, V=837.8086 Å3. The cell parameters were good agreement with the reported values [7, 8]. The TG/ DTA curves for $MgSO_4$ doped KHP crystal is shown in Figure 5. It is observed that there is a strong endothermic peak at 289.80 ˚C reveals the decomposition of doped KHP structure which is closer to the melting point of pure KHP (290.74˚C). The decomposition from 298.1 °C to 324.4 °C resulting in a mass reduction of 41.14%. The third stage of decomposition is from 504.6 °C to 550 °C resulting in the mass reduction of 12.45% [9].

## VI. CONCLUSION

The single crystals of pure and $MgSO_4$ doped KHP crystals were grown by slow evaporation solution growth method. The powder XRD confirms its structure and lattice parameters. The molecular structure of the pure and $MgSO_4$ doped crystals were confirmed by FTIR spectral analysis. Thermal stability of the crystals was investigated by TG/DTA analysis.

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## REFERENCES

[1]. Laudise RA. The growth of single crystals. Prentice Hall; 1970.
[2]. Brice JC. Crystal growth processes. Halsted Press; 1986.
[3]. Anandhi S, Shyju TS, Gopalakrishnan R. Studies on growth, thermal, optical, vibrational properties and hyperpolarizability of a complex orthonitroaniline with picric acid. Journal of crystal growth, vol.312 (22), pp.3292-9 . 2010.
[4]. Ganesh RB, Kannan V, Meera K, Rajesh NP, Ramasamy P. Synthesis, growth and characterization of a new nonlinear optical crystal sodium acid phthalate. Journal of crystal growth, Vol. 282 (3-4), pp.429-33, 2005.
[5]. Geetha SK, Perumal R, Moorthy Babu S, Anbarasan PM. Habit modification and improvement in properties of potassium hydrogen phthalate (KAP) crystals doped with metal ions. Crystal Research and Technology, Vol.1;41 (3), pp.221-4, 2006.
[6]. Madhurambal, G., Ramasamy, P., Anbusrinivasan, P. and Mojumdar, S., Thermal properties, induction period, interfacial energy and nucleation parameters of solution grown benzophenone. Journal of Thermal Analysis and Calorimetry, vol. 90(3), pp.673-679, 2007.
[7]. A. C. Sajikumar, S.Vinu and C. Krishnan. Int. Journal of E,ngineering Research and Applications ISSN : 2248-9622, Vol. 5,(2), pp. 50-52, 2015.
[8]. Muthukumaran.B, Ruthiramoorthy.K, Santhanam.V, Baby.P. (2017). Growth and characterisation of pure and thiourea doped rochelle salt crystals by slow evaporation method, International Journal of Materials Science ISSN 0973-4589, vol. (12), pp.325-334, 2017.
[9]. Thilagavathy, S. R., Rajesh, P., Ramasamy, P., & Ambujam, K. Growth and characterization of pure and doped KHP NLO single crystals. Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, vol. 127, pp. 248-255, 2014.