Applied Sciences Journal
ISSN: 2997-6243|
Volume 13 Issue 2, April-June, 2025
Journal Homepage: https://ethanpublication.com/journals/E9
Official Journal of Ethan Publication

IMPACT OF METAL ION INCORPORATION ON GROWTH HABIT AND MORPHOLOGY OF POTASSIUM HYDROGEN PHTHALATE CRYSTALS

Anantharaman, M. Lokesh
Department of Physics, Sri Theagaraya College, Chennai, India
DOI: https://doi.org/10.5281/zenodo.17416721

Abstract
Optically transparent single crystals of doped metal ion KAP crystals were grown in aqueous solution by slow evaporation technique at room temperature. Optical absorption studies revealed that doped crystals possess in the visible region. Transparency of the doped crystal is suitable for (NLO) application and UV is compared with pure KAP crystal. The doped crystals present an absorption band at nearly 325nm and 340nm.the other properties of KAP such as refractive index was analyzed.

Keywords: OTA Solubility, Growth from solutions, Single crystal growth, Metal organic, nonlinear optic materials

Introduction

The crystals of potassium hydrogen phthalate or potassium acid phthalate (KAP) were studied intensively since they started to be used as X-Ray monochromators and X-ray analyzers [1]. Semi organic crystals have attracted considerable interest due to their large nonlinear optical coefficients, high resistance to laser induced damage, low angular sensitivity and excellent mechanical hardness.

Potassium hydrogen phthalate is a semi organic salt that belongs to the orthorhombic class of alkali acid phthalate series, with the chemical formula of K (C6 H4COOH-COO). There are four such chemical units in a unit cell. The crystal structure of KAP is ionic consisting of potassium ions and alkali phthalate ions and it was assigned to the Pca21 [2] space group. It has platelet morphology with a perfect cleavage along (010) plane. Recently KAP crystals were used as substrate for epitaxial growth of oriented polymers [3, 4] and for hierarchical growth of organized materials [5].

KAP crystals are playing an important role in the field of nonlinear optical materials, they are known second harmonic generating materials that have long stability in devices due to their electro- optical properties [6] and exhibit interesting piezoelectric, pyro electric and elastic properties that are useful in many application[7,8].

Numerous authors report the relation between growth conditions and morphology for pure [9, 10] and doped KAP [11, 12] crystals .Theoretical studies were dedicated to the growth kinetics from aqueous solutions. Recently new growth techniques were developed [13].The properties of KAP crystals doped with different impurities were extensively studied [14-17].

Applied Sciences Journal
ISSN: 2997-6243|
Volume 13 Issue 2, April-June, 2025
Journal Homepage: https://ethanpublication.com/journals/E9
Official Journal of Ethan Publication

This report presents the studies regarding the influence of the doping with NaCl, LiCl impurities on the structural, morphological and optical properties of potassium hydrogen phthalate crystals. The results obtained from powder X –ray diffraction, optical transmission are reported.

Experimental Details

KAP was prepared by successive recrystallization process. Metal ions doped crystals were grown by slow evaporation technique. A saturated solution of KAP (100ml) at room temperature was performed and good quality crystals were selected for the growth. The mother solution was prepared using the solubility relation c (T) =9.283-0.059T+0.0058T^{2} ^{of} KAP in water [18]. Where c (t) is the solubility of KAP in water and T is the temperature (°C). A Super saturated solution of pure KAP and1mole% of NaCl and LiCl doped at room temperature was obtained by constant stirrer up to 5 hours and then filtered. After 24 hours the good quality seeds were suspended in respective beakers using the nylon thread. After completion of growth run crystals were harvested as shown in Fig .1.The growth was carried out in a constant temperature bath. The grown crystals were characterized with powder XRD for structural properties. X-ray diffract meter with CuKα radiation (λ=1.5405A°) was used.

a) b)

Fig.1. (a) Grown crystal of KAP doped with NaCl ;(b) Grown crystal of KAP doped with LiCl

Characterization

The grown impurities doped KAP crystal was subjected to various characterizations viz; powder X-ray diffraction, UV-visible spectral study and the results were compared with that of pure KAP.

3.1 Result and discussion

The powder X-Ray diffraction analysis on NaCl doped KAP and LiCl doped KAP crystals was recorded using CuKα diffractometer. This analysis revealed that powder crystals of doped KAP crystallize in orthorhombic system with space group Pca21. The calculated lattice parameters for doped crystals were compared with that of pure KAP [19]. The xrd results have confirmed that the incorporation of metal ions in the KAP crystal lattice does not change the crystal structure though there is a small change in the lattice parameter.

XRD analysis.

The powder X-ray diffractogram of doped KAP presented has been recorded up to 2θ =80° and at a scan rate 10.13 min prove that both crystals have same structure. X-ray powder pattern of the crystal was recorded on a XPERT-PRO diffractometer using CuKα (1.5406A˚) radiation. The recorded X-ray spectra and the corresponding data obtained for the grown crystal in the present study fig.2, reveal that diffraction

Applied Sciences Journal
ISSN: 2997-6243|
Volume 13 Issue 2, April-June, 2025
Journal Homepage: https://ethanpublication.com/journals/E9
Official Journal of Ethan Publication

peaks for both the doped crystals were compared with the values available in the literature and the peaks match very well compared WITH the JCPDS data of pure KAP and the h k l value s were noted. From the Table 1.It is reported that the pure KAP as values were close in arrangement. From the XRD spectrum of doped crystals, it also reveals that there are more additional peaks, but only change in the intensity of the peaks. This shows the presence of additional phase due to doping

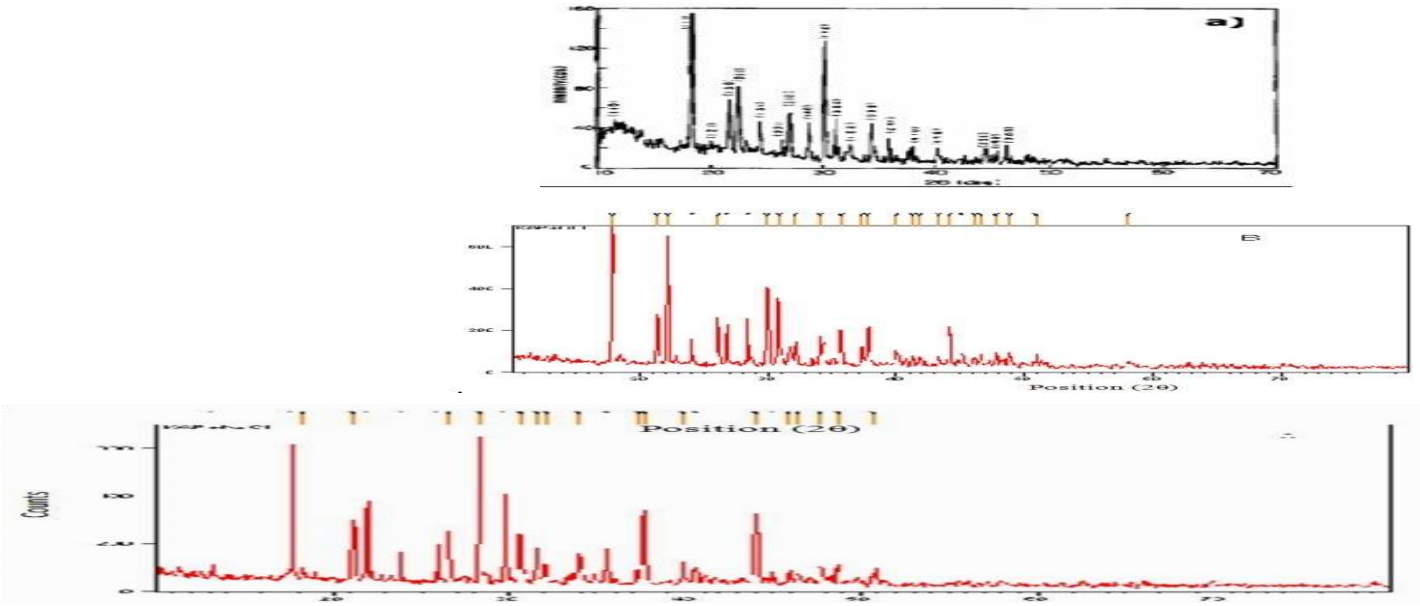

Fig.2. (a) XRD spectra for pure KAP ;(b) XRD spectra for LiCl doped KAP ;(c) XRD Spectra for NaCl doped

Table: 1 COMPARISION OF DOPED KAP CRYSTAL WITH JCPDS VALUE

2θ IN DEGREES		d(A°)LiCl		d(A°)NaCl		h	k	L
Observed	JCPDS	Observed	JCPDS	Observed	JCPDS			
17.799	17.8866	4.97932	4.95505	4.97932	5.00003	1	1	1
21.235	21.3247	4.18071	4.16330	4.03298	4.04196	1	2	1
22.022	22.1294	4.03298	4.01369	3.14860	3.15017	1	3	0
26.1116	26.010	3.40991	3.42296	2.98897	2.99666	1	3	1
28.4159	28.354	3.13842	3.14514	2.91276	2.91849	0	1	2
37.3461	37.268	2.40593	2.41077	2.38787	2.38964	3	3	1
44.2096	44.169	2.04702	2.04882	2.02584	2.05354	3	5	0

UV- visible spectrometer and refractive index

The UV-Vis-NIR spectral transmittance was studied using a Shimadzu UV-Vis spectrophotometer with a single crystal of 6 mm thickness in the range of 200 -1100 nm. The recorded spectrum is shown in Fig 3(a) and 3(b).The crystal has sufficient transmission in the entire region which is an important requirement for a material to be optically active. Pure KAP and doped KAP crystals were subjected to the spectral studies. The studies determine their percentage of transparency and absorbance. It is evident that

Applied Sciences Journal
ISSN: 2997-6243|
Volume 13 Issue 2, April-June, 2025
Journal Homepage: https://ethanpublication.com/journals/E9
Official Journal of Ethan Publication

KAP doped with LiCl has transparency with about 22% for NaCl transparency was about 75% .There is an absorption edge at 199nm.The refractive index is used to identify the particular substance, confirm its purity. The refractive index of the KAP doped LiCl crystal has μ= 1.3606(no unit) and for NaCl the refractive index of the KAP doped crystal has μ= 1.1856(no unit)

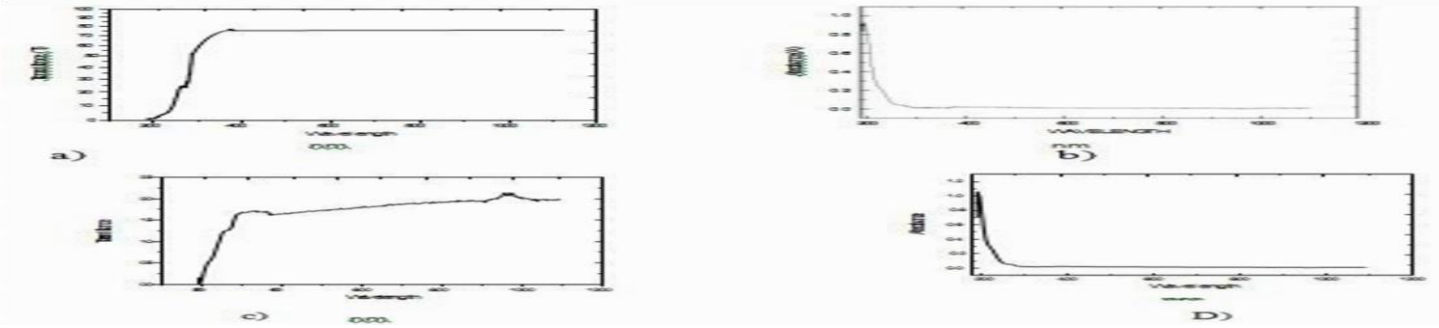

Fig.3. (a) Transmission spectra of NaCl doped KAP crystal ; (b) Absorbance spectra of NaCl doped KAP crystal ;(c) Transmission spectra of LiCl doped KAP crystal ;(d) Absorbance spectra of LiCl doped KAP crystal

Conclusion

Good quality single crystals of the pure and doped KAP crystal were grown by using the submerged-seed solution method. Lattice parameters were calculated from the XRD characterization to compare with pure KAP. The powder X-ray reveals stable lattice on doping with KAP. The optical transmission spectrum showed that doped KAP crystal has good transparency in the UV-Vis. The absorption bands at 280nm were analyzed for LiCl and absorption band at 325nm and 340nm were analyzed for NaCl.

Their refractive index was found as1.360 for LiCl and 1.8561 for NaCl. The dopants percent was very less so the transparency is also very less. In future, attempts will be made to grow bulk crystals of KAP with different impurity with different mole percentage and characterizations such as micro hardness, SHM, photoconductivity, dielectric studies will be carried out to study the perfection of the crystal.

References:

L.A.M.J.Jeten, B.Van der Hoek, W.J.P.Van Enckerort, J.Cryst.Growth 62 (1983) 603

G.R.Ester, P.J.Halfpenny, J.Cryst.Growth 187 (1998) 111

M.H.J.Hottenhis, C.B.Lucasius, J.Cryst.Growth 78 (1986} 379

M.H.J.Hottenhis, C.B.Lucasius, J.Cryst.Growth 91(1988} 623