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# NaCl, KCl and SrCl<sub>2</sub> Doping Effect on Linear and Nonlinear Optical Properties of KDP Crystal

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#### Authors' contributions

This work was carried out in collaboration between all authors. Author PSA designed the study, performed the initial experimental work and wrote the first draft of the manuscript in cooperation with author SKD. Authors ABG and GGM managed the analyses of the study and finalised the manuscript. All authors read and approved the final manuscript.

#### Article Information

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# ABSTRACT

The effect of NaCl, KCl and SrCl<sub>2</sub> doping on the crystal structure, optical transmission, and second harmonic generation (SHG) efficiency of potassium dihydrogen phosphate (KDP) crystals is investigated. Single crystals of pure NaCl, KCl and SrCl<sub>2</sub> doped KDP were grown by solution growth technique from aqueous solutions. The crystal structure

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was studied by powder X-ray diffraction. Doped crystals possess higher optical transparency than pure KDP crystals. The SHG efficiency of doped crystals was found more than pure KDP crystal. The grown crystals were subjected to the photoluminescence, atomic absorption and FT-IR spectroscopic study.

Keywords: Crystal growth; X-ray diffraction; nonlinear optics; FT-IR spectroscopy; optical transmission; second harmonic generation.

## 1. INTRODUCTION

Potassium dihydrogen phosphate (KDP) is an important inorganic hydrogen bonded nonlinear optical (NLO) crystal that exhibits unique NLO and ferroelectric properties. It is widely used in frequency conversion and electro-optic switching applications, and extensively investigated for the effect of changes in growth conditions and dopants on its properties like growth rate, optical transparency, thermal and mechanical stabilities, electrical properties and second harmonic generation (SHG) efficiency [1-7]. NLO applications demand crystals of large SHG efficiency, good thermal and mechanical stability, high laser damage threshold, and easy crystal growth process [8]. The effect of urea and KCI doping on the crystal structure of KDP has been studied [9]. In the study, the increase in lattice constants in urea doped KDP, while decrease in case of KCI doping has been reported. Ananda Kumari and Chanrdamani [10] have studied the effect of KCI and NaCI doping on dielectric properties and electrical conductivities of KDP. They have confirmed that the electrical conductivity is due to the movement of anions and it increases with increasing concentrations of KCI and NaCI. The increases in dielectric constants has also been reported in same communication but have not studied the linear and nonlinear optical properties. Probably for a first time, SrCl<sub>2</sub> is being used as a dopant to study its effects on the properties of the KDP crystal. The effect of titanium dioxide (anatase) nanocrystals on growth process, optical and structural properties of KDP crystal have been studied by Pritula et al. [11]. The trivalent impurity Cr(III) increases the mean growth rate along the [001] direction [12]. Owczarek and Sangwal have reported same observations in case of Fe(III) and Cr(III) doping [13]. The effect on different properties of KDP using amino acids Lalanine, L-arginine [14-16], L-glutamic acid, L-valine, L-histidine [17] and L-lysine [18] as a dopant have been studied. As amino acids possess high optical nonlinearity, it increases SHG efficiencies and optical transparencies of the KDP crystal. Cerium [19], N'N dimethyl urea [20], and urea phosphate [21] doping modifies the growth habit and optical properties. Potassium acetate (CH<sub>3</sub>COOK), potassium citrate (K<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>) [22] and potassium carbonate (K<sub>2</sub>CO<sub>3</sub>) [23], potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) [24] and urea [25] added into KDP results in lowering dielectric constants of the crystal. The dopants viz. potassium acetate (CH<sub>3</sub>COOK), potassium citrate (K<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub>) [22] and potassium carbonate (K<sub>2</sub>CO<sub>3</sub>) [23] improve optical transparency and SHG efficiency also. The effect of addition of potassium thiocyanate on dielectric, crystalline perfection, SHG efficiency, and hardness of KDP crystals has been reported [26]. DC and AC electrical measurements carried out at various temperatures along a- and c- directions indicate an increase of the electrical parameters with the increase of temperature, which can be attributed mainly to the increase of thermally generated hydrogen bond vacancies (L- defects). L-arginine addition leads to reduction of electrical parameters of KDP and ADP single crystals, which can be attributed mainly to the decrease of L-defects due to creation of additional hydrogen bonds by the impurity in random directions [27]. NaCl and NaBr dopants have been tried to modify various properties of KDP [28,29]. Urea and thiourea [30]; NH<sub>4</sub>CI, NH<sub>4</sub>NO<sub>3</sub>, NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> [31,32] added KDP single crystals

have been grown by the gel method using silica gels and electrical conductivity measurement has been carried out to study lattice variation and thermal parameters like Debye-Waller factor, mean-square amplitude of vibration, Debye temperature and Debye frequency.

In the present study the effect of NaCl, KCl and SrCl<sub>2</sub> doping on the crystal structure, optical transmission, photoluminescence and second harmonic generation efficiency of KDP crystal has been investigated. The single crystals of pure NaCl, KCl and SrCl<sub>2</sub> doped KDP were grown from solution at low temperature by evaporating water solvent at a constant temperature. The crystal structure was studied by powder X-ray diffraction (XRD) and the data was analysed using software PowderX [33]. In case of KCl doped crystals, the decrease in the lattice constants has been reported while for other doped crystals, all the lattice constants have been increased. The doped crystals have higher optical transparency than pure KDP crystals. SHG efficiency of doped crystals is more than pure KDP crystal. The maximum SHG efficiency of NaCl and KCl doped crystals has been observed for 2mol% doping and in case of SrCl<sub>2</sub> doped crystals, it is observed for 1mol% doping.

## 2. MATERIALS AND METHODS

#### 2.1 Sample Preparation and Crystal Growth

Analytical reagents grade KDP, NaCl, KCl and SrCl<sub>2</sub> (S D Fine-Chem Ltd. India) chemicals were used for the preparation of sample solutions. One solution of pure KDP and nine solutions of 1, 2 and 4mol% NaCl, KCl and SrCl<sub>2</sub> doped KDP were prepared in 50ml of double distilled water by taking appropriate amount of KDP, NaCl, KCl and SrCl<sub>2</sub> chemicals. The solutions were stirred continuously for four hours using magnetic stirrer at 40°C, filtered and then kept for crystallisation at a constant temperature 35°C to get seed crystals. The harvested seed crystals were used to grow good quality crystals of pure and doped KDP. An abbreviation; KDP is used to represent pure KDP crystal, while other doped crystals are abbreviated as first letter K that represents KDP, second character is a number representing doping percentage and third and fourth characters represent the compound used as a dopant. NC, KC and SC are used to represent NaCl, KCl and SrCl<sub>2</sub> respectively. For example, K1NC is an abbreviation for the crystal of KDP doped with 1mol% NaCl. Photographs of a few grown crystals are shown in Fig. 1.



Fig. 1. Photographs of grown crystals

# 2.2 Characterizations

FT-IR spectra of all samples were recorded on instrument FT-IR spectrometer (Schimadzu, Japan). UV-visible-NIR transmission spectra of the grown crystals were recorded with UV-visible spectrophotometer (Black-Comet-SR, Stellarnet Inc. USA) over wavelength range 190-1083nm.The powder XRD patterns of pure and doped KDP crystals were recorded on Bruker D8-Advance X-ray diffractometer (Germany) in the range  $2\theta$ =20 to  $80^{\circ}$  using CuK $\alpha$  radiation of wavelength 1.5406Å at room temperature. The powder XRD data was then analysed by using software PowderX [33]. The SHG efficiency of pure and doped KDP was measured by Kurtz and Perry method [34]. Photoluminescence study was carried out on the instrument fluorescence spectrophotometer (F-7000, Hitachi, Japan).

#### **3. RESULTS AND DISCUSSION**

## 3.1 Solubility

Solubility measurement of pure and doped KDP crystals was carried out gravimetrically in aqueous solutions at different temperatures, as it is important for deciding the crystal growth temperature and the crystal growth method. Pure and doped KDP compounds have moderate solubility and it increases with increasing temperature. Moreover, there is an enhancement in the solubility of doped crystals. This increase is attributed to the presence of ionized species, which polarizes water molecule, enhances interaction between water molecule and KDP molecule. It is desirable to have increased solubility of the KDP to grow good quality big size crystals with fast growth rate. Thus, with addition of dopants NaCl, KCl and SrCl<sub>2</sub>, solubility can be enhanced.

## 3.2 Atomic Absorption Spectroscopy

The amount of K, Na and Sr elements present in the doped KDP crystal was determined by atomic absorption spectroscopy. 0.1g powder sample of each doped crystals was dissolved separately in 10ml double distilled water and used for the measurement. The instrument was firstly calibrated using reference solutions of K, Na and Sr. The estimated amount of K, Na and Sr elements in solutions, added during crystal growth, and in crystals are given in Table 1. The concentration of Na and Sr in the crystals is found to be low as compared to the concentration in solution during crystal growth. It is expected that Na and Sr can replace some K positions and enter in to the interstitial positions. The possibility of entering Sr atoms at interstitial position as compared to the Na atoms is less. It may be the reason behind less concentration of Sr in doped crystals. In case of KCI doping, the variation in concentration of K in doped crystals can be assigned to entering K atoms at interstitial positions.

#### 3.3 FT-IR Spectroscopy

The FT-IR spectra of pure and doped KDP crystals were recorded in 400-4000 cm<sup>-1</sup> wave number range. The powder samples of all crystals mixed with KBr placed in sample holder were used for the measurement. The absorption peaks observed in all the samples, at around 2731 cm<sup>-1</sup> corresponds to the P-OH stretching, 2422 cm<sup>-1</sup> to O-H and P-OH stretching, 1651 and 1550 cm<sup>-1</sup> to the P-O-H bending, and 1296 cm<sup>-1</sup> to O-H deformation and P=O stretching. The peak around 1095 cm<sup>-1</sup> is attributed to P-OH stretching and HO–P-OH bending. In doped crystals, these peaks are displaced from the position as in pure KDP. Moreover, a few more peaks are apparent in the 3000-3400 cm<sup>-1</sup> wave number range in doped crystals, which correspond to the hydrogen bonds formed with oxygen and chlorine atoms. The appearance of more peaks in this region and displacement of peaks of pure KDP in case of doped crystals confirms qualitatively the inclusion of dopant in the KDP host crystal. The absorption band at 1621 cm<sup>-1</sup> in KDP corresponding to the P-OH bending found to shifted on higher frequency side in FT-IR spectrum of K4KC (appears at 1652 cm<sup>-1</sup>) and lower frequency side in spectra of K4NC and K4SC (appear at 1620 and 1618 cm<sup>-1</sup>, respectively). Another peak 1296 cm<sup>-1</sup> in KDP spectrum corresponding to the P=O stretching and O-H deformation found to appear at 1304 cm<sup>-1</sup> in K4KC spectrum. The band centered at 928 cm<sup>-1</sup> representing OH stretching in KDP appears at 903 cm<sup>-1</sup> in K4NC and K4SC. The shifting in absorption bands on higher frequency side in K4KC and lower frequency side in K4NC and K4SC.

Crystal	Quantity present (mg/1000ml)				Weight %		
	In solution				In crysta	inclusion	
	Na	К	Sr	Na	ĸ	Sr	
K1NC	16.9			4.15			24.56
K2NC	33.7			6.32			18.75
K4NC	67.3			7.25			10.77
K1KC		2901.8			2878.1		99.18 (17.71) <sup>@</sup>
K2KC		2930.5			2880.3		98.29 (12.70) <sup>@</sup>
K4KC		2988.0			2881.1		96.42 (7.04) <sup>@</sup>
K1SC			64.3			7.01	10.90
K2SC			128.5			8.12	6.32
K4SC			256.5			8.82	3.44

# Table 1. Amount of K, Na and Sr elements present in growth solutions and grown crystals

<sup>@</sup>The numbers in parenthesis represents weight percent inclusion of K atoms as a dopant

#### 3.4 UV-Visible-NIR Transmission Study

Optical transparency of all grown crystals was studied by UV-visible-NIR spectroscopy. In the measurement, polished thin samples of thickness 2mm were used to study the transmission over wavelength range 190-1083nm. The UV-visible-NIR spectra are shown in Fig. 2. The magnified portions of transmission spectra over 80-86% transmission range are shown in inset to visualize the change in the optical transparency. All the crystals show high transmission over wide range of wavelengths. Lower cutoff for all the crystals has been found at around wavelength 195nm. 2mol% NaCl and KCl doped crystals have highest optical transparency among their respective group. Lowering transparency with higher doping level may be due to the insertion of impurity levels below conduction band, which is confirmed from photoluminescence study for 2mol% KCl doping. The full width at half maximum (FWHM) of emission peaks of the sample K2KC found to increased as compare to KDP (Table 4). In SrCl<sub>2</sub> doped crystals, 4mol% doped KDP crystal has maximum transparency. NaCl, KCl and SrCl<sub>2</sub> are ionic compounds and according to Kumari and Chanrdamani [10] the electrical conductivity is due to anions. At room temperature also, the anions can move inside the crystal due to the presence of vacancies, leaving behind cations, separated anions, and polarized molecules forms hydrogen bonds with KDP molecules and distort it in some extent. The distortion of KDP molecules due to the doping may be the reason behind increasing SHG efficiency, lattice parameters and optical transparency.

The optical absorption coefficients ( $\alpha$ ) of all crystals for wavelength range 190-1083nm were calculated by using equation;

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$$\alpha = \frac{2.303}{d} \log \frac{1}{T};$$

Where, 'T' is the transmittance and 'd' is the thickness of the crystal.

The plots of variation of  $(\alpha hv)^2$  verses photon energies (hv) are shown in Fig. 2. The values of band gap of all the crystals calculated as per procedure discussed elsewhere [35] are given in Table 3. The values of band gap are found to be more for all doped crystals and it may be because of reduction in width of absorption edge near cut-off wavelength.



Fig. 2. UV-visible-NIR transmission spectra of pure and doped KDP crystals and plots of variation of photon energies (hv) vs. (αhv)<sup>2</sup>

#### 3.5 Powder XRD study

The recorded powder XRD patterns of pure and doped KDP crystals are shown in Fig. 3 with assigned 'hkl' values to the observed peaks. PowderX software [33] was used to analyze powder XRD data and to calculate lattice parameters. Calculated lattice parameters are given in Table 2. The increase in lattice constants of NaCl and SrCl<sub>2</sub> doped crystals and decrease in lattice constants of KCl doped crystals observed, while the crystal system and space symmetries remain same. This change in the lattice constants may be because of replacement of K by Na and Sr, which leads to the increase in lattice constants of KCl doped KDP crystal lattice at interstitial positions leading to the distortion in the crystal. The observation of decrease in lattice constants of KCl doped KDP crystals, may be because of bond tightening, confirm the report of Kumari and Chanrdamani [10].



Fig. 3. Powder XRD patterns of pure and doped KDP crystals

Table 2. Calculated lattice parameters of pure and doped KDP crystals

Crystal	Unit cell parameters
KDP	a= b=7.4494Å, c=6.9773Å
	$\alpha = \beta = \gamma = 90^{\circ}$
	V=387.1964Å <sup>3</sup>
K4NC	a=b=7.4529Å, c=6.9785Å
	$\alpha = \beta = \gamma = 90^{\circ}$
	V=387.6203Å <sup>3</sup>
K4KC	a=b=7.4470Å, c=6.9730Å
	$\alpha = \beta = \gamma = 90^{\circ}$
	V=387.1220Å <sup>3</sup>
K4SC	a=b=7.4540Å, c=6.9985Å
	$\alpha = \beta = \gamma = 90^{\circ}$
	V=388.8540Å <sup>3</sup>

#### 3.6 SHG Efficiency Measurement

The SHG efficiency of powder samples was measured by Kurtz and Perry method [34] with reference to the pure KDP. Q-switched, mode locked Nd: YAG laser of wavelength 1064nm of peak power 7.1mJ, pulse duration 8 ns, beam diameter 6mm and repetition rate 10Hz was used in the SHG measurement. The power of second harmonics of 1064nm at wavelength 532nm signal was measured at the output. The SHG efficiencies are tabulated in the Table 3. The SHG efficiency of 1mol% NaCl doped KDP is found less as compared to

KDP but for higher mole% doping, the SHG efficiency is more. Maximum SHG efficiency is found for 2mol% NaCl doping in KDP. In case of KCl doping, maximum SHG efficiency, 1.87 times KDP has been found for 2mol% doping. For all other KCl doping SHG efficiency is more than pure KDP. The SrCl<sub>2</sub> doping also enhances SHG efficiency of KDP. The maximum SHG efficiency has been found for 1mol% doping. As discussed above, the increase in SHG efficiency of doped crystals may be because of the distortion producesd in KDP crystal by dopant molecules. Nonlinear polarisation produced in the sample by fundamental wave results in SHG [36]. The dopants producing distortion in the crystal may enhance polarization, which may be in phase to the polarsation component responsible for SHG. The phase and extent of enhanced polarization may depend on nature of dopant and its concentration. The enhanced SHG efficiency of K2NC, K2KC, and K1SC may be due to the additional polarisation introduced by doping produced distortion is in phase to the polarisation responsible for SHG.

Crystal	SHG efficiency	Energy band gap (eV)
KDP	1	5.79
K1NC	0.85	5.83
K2NC	1.06	5.85
K4NC	1.03	5.82
K1KC	1.84	5.84
K2KC	1.87	5.82
K4KC	1.05	5.82
K1SC	1.29	5.80
K2SC	1.05	5.80
K4SC	1.10	5.82

Table 3. SHG efficiencies of pure and doped KDP crystals

#### 3.7 Photoluminescence Study

The emission spectra of pure and 2mol% NaCl, KCl and SrCl<sub>2</sub> doped KDP crystals recorded in spectral range 300-700nm using excitation wavelength 254nm are shown in Fig. 4. The curves are asymmetric; therefore, each curve was deconvoluted in to two curves (not shown in figure). The peaks of deconvoluted curves and the FWHM are given in Table 4. From Table 4, one can see that there is a red shift in the peak positions for NaCl and KCl doped crystal and blue shift for SrCl<sub>2</sub> doped crystals. FWHM is found to decrease for NaCl and SrCl<sub>2</sub> doped crystals, while it is increased for KCl doped crystal. The decrease in the FWHM may be attributed to the sharp emission enhanced by NaCl and SrCl<sub>2</sub> doping KCl doping in KDP may introduce impurity levels around edge of the conduction band to form impurity band, which may results in the broadening of emission band.

Table 4.	Details	of emi	ission	peaks
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Crystal	1 <sup>st</sup> Peak		2 <sup>nd</sup> Peak		
	Position (nm)	FWHM (nm)	Position (nm)	FWHM (nm)	
KDP	359.86	54.10	431.78	123.22	
K2NC	360.79	53.48	433.94	117.45	
K2KC	363.15	59.48	437.49	126.56	
K2SC	358.11	49.79	431.77	115.45	



Fig. 4. Emission spectra of pure and doped KDP crystals (Excitation wavelength  $\lambda_{ex}$ -254nm)

#### 4. CONCLUSION

Pure NaCl, KCl and SrCl<sub>2</sub> doped KDP crystals were grown by slow evaporation of solvent method at a low temperature from aqueous solution. UV-visible-NIR spectroscopy of grown crystals confirms increase in the optical transparency of doped crystals. The calculated band gaps of doped crystals are larger than pure KDP crystal. NaCl, KCl and SrCl<sub>2</sub> doping in KDP affects the absorption peak positions of characteristic bondings and functional groups in FT-IR spectra, which confirm qualitatively the doping in KDP crystal. NaCl and SrCl<sub>2</sub> doping results in increase in the lattice constants of doped KDP crystals, while decrease in lattice constants has been witnessed in case of KCl doping. The SHG efficiency study shows modifications in the efficiency of doped crystals. The maximum SHG efficiency has been found for 2mol% doping of NaCl and KCl in KDP crystals while SrCl<sub>2</sub> doped crystals has maximum efficiency for 1mol% doping. Photoluminescence study confirms the red-shift in emission peaks in NaCl and SrCl<sub>2</sub> doped crystals and blue-shift in KCl doped crystals.

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#### **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

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