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Research Paper

EDAX, Thermal, UV-Vis and SHG studies of pure and creatinine doped KDP crystals

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Abstract— In the current article, we explore the growth and diverse characterizations, encompassing elemental, thermal, UV-Vis, and SHG properties of pure and creatinine doped KDP crystals. The doping of creatinine was varied from 0.2wt% to 0.8wt% and its effect on various properties of pure KDP crystal is analyzed and discussed in this article. For crystal growth, undoped and creatinine-doped KDP crystals were cultivated at room temperature utilizing the solution growth technique. Elemental analysis was conducted to confirm the presence of dopant atoms. The thermo-grams confirmed the increased thermal stability of 0.2wt% and 0.4wt% creatinine doped KDP crystals without a phase change. The presence of creatinine increased the decomposition temperature and mass loss of pure KDP. The DTA curves showed the corresponding endothermic peaks and shifting of the peaks after doping of creatinine. UV-Vis analysis showcased the exceptional transparency of all crystals across the entire visible spectrum without any change in transmittance and shifting of cut-off wavelength towards higher wavelength side after doping of creatinine. The energy bandgap values of all the crystals were evaluated by using Tauc's method and observed reduction in the energy bandgap after doping of creatinine. The SHG analysis showed increased NLO efficiency of creatinine doped KDP crystals. The results are discussed accordingly.

Keywords— Pure and creatinine doped KDP crystals, EDAX, TGA, DTA, UV-Vis, SHG

1. Introduction

The crystals of potassium dihydrogen phosphate (KH_2PO_4), is a renowned non-linear optical (NLO) crystal for its wide range transparency and applicability in industrial laser facilities as a frequency multiplier, parametric amplifier, and electro-optical shutters [1]. It belongs to scalenohedral class of tetragonal crystal system [2]. In the current study, solution-grown KDP crystals were produced at room temperature with and without creatinine doping. Creatinine has a number of functional groups and substituents as well as imino and amino tautomeric forms, e.g CH_3, CH_2, NH_2 and C=O [3]. During the doping of creatinine, the proton gaining or losing property of the functional groups should be expressed as of either improvement or damping in SHG efficiency of creatinine doped KDP crystals. The results are discussed elaborately.

2. Related Work

The KDP crystal was subject to numerous attempts to be altered, either by altering the growth conditions or by adding other impurities with an aim to improve its thermal and NLO properties, particularly the second harmonic generation (SHG) efficiency [1, 4-7]. These studies have consistently demonstrated the significant role played by tiny amount impurities in influencing development pattern and physical characteristics properties of the crystals [8].

3. Experimental

The solution growth approach was used to develop pure KDP crystals as well as crystals doped with 0.2wt%, 0.4wt%, 0.6wt%, and 0.8wt% creatinine at room temperature. First, KDP powder was dissolved in the appropriate quantities of distilled water to create a 250 ml saturated solution. It was equally divided into five beakers. First beaker was left for growing pure KDP crystal. In remaining beakers, 0.2 gm, 0.4 gm, 0.6 gm and 0.8 gm creatinine was added for growing four dopants i.e., 0.2wt% to 0.8wt% creatinine doped KDP crystals. The beakers were all kept in a dust-free, disturbance-free environment and shielded with filter paper that had some pinholes in it. After a time period of nearly one month, all the crystals were grown and harvested for the various characterizations. The photographs of the crystals are shown in the figure 1.

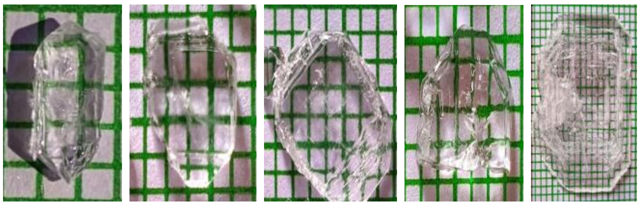

Figure 1: Growth of crystals

These crystals were characterized by EDAX, thermal analysis, UV-Vis and SHG. The Philips XL-30 instrument setup was used to conduct the EDAX. The TGA was conducted on NETZSCH STA-2500 Regulus Simultaneous Thermal Analysis set up from room temperature to 800oC using Al2O3 pan at heating rate 10^oC/min in the air atmosphere. The UV-Vis data were acquired using the Shimadzu UV-1700 Phamaspec spectrophotometer, employing the dissolution method with HPLC grade water as the internal standard. The transmittance values were recorded, and the maximum transmission and absorbance (expressed as a percentage) were determined by directly injecting the samples into the UV spectrometer. The UV chromatogram was obtained using the Shimadzu UV prob 2.6 software. For the second harmonic generation (SHG) analysis, the Kurtz powder test was conducted. This involved using a Q-switched high-energy Nd:YAG laser with modulated radiation at a wavelength of 1064 nm. The laser had a repetition rate of 10 Hz and a pulse width of 6 ns.

4. Results and Discussion

EDAX Analysis: The elemental analysis was completed in order to determine elemental composition of all crystals. The result of analysis is shown in the Table 1.

Table 1. EDAX Result

Sample No.	Sample	Atomic Weight (%)				
		C	K	O	P	N
1	Pure KDP	-------	34.35	48.29	22.03	-------
2	0.2wt% creatinine doped KDP	4.33	26.80	40.93	19.06	4.22
3	0.4wt% creatinine doped KDP	5.03	32.66	37.66	21.13	8.19
4	0.6wt% creatinine doped KDP	6.37	27.00	39.58	18.82	8.23
5	0.8wt% creatinine doped KDP	10.2	25.15	36.98	17.71	9.94

The chemical formula of potassium dihydrogen phosphate (KDP) is KH2PO4. The formula suggests that KDP contains potassium (K), phosphorous (P) and oxygen (O). Hence, Pure KDP is found to contain potassium (K), phosphorous (P), and oxygen (O) according to an EDAX study. The chemical formula of dopant creatinine is C4H7N3O. Therefore, when creatinine is doped into pure KDP, its EDAX analysis shows the presence of carbon (C) and nitrogen (N), which confirms the presence of dopant atoms in KDP crystal. From the table 1, it is observed that wt% of carbon and nitrogen along with creatinine is simultaneously increased in KDP, which confirms the successful doping of creatinine in KDP.

Thermal Analysis: Thermal study is a key component of practical device production since it reveals details regarding the stability and thermal disintegration of produced crystals. Figure 2 shows the curves of thermogravimetry of pure and different wt% creatinine doped KDP crystals, while the result of analysis is given in the table 2.

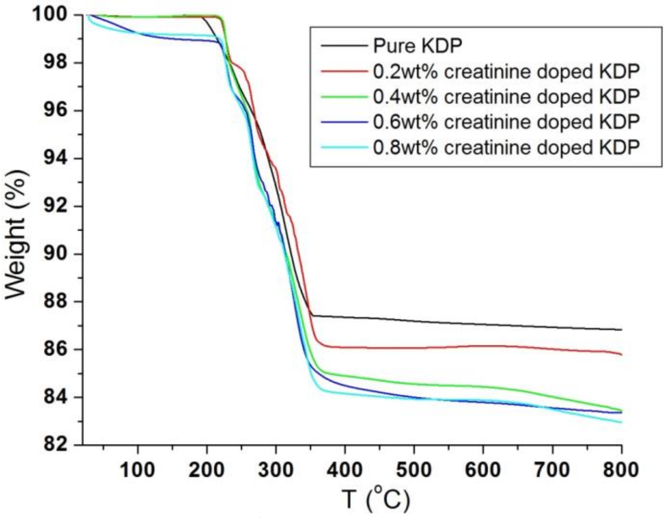

Figure 2. TG curves

The careful comparison of TG curve of pure KDP with different wt% creatinine doped KDP crystal indicates that pure KDP crystal remains stable up to 185^oC temperature, while 0.2wt% and 0.4wt% creatinine doped KDP crystal remains stable up to 219^oC and 225^oC temperature, respectively. This affirms that there is no surface absorbed water molecules in the grown crystals [9]. As the weight% of creatinine increases up to 0.6wt% and 0.8wt%, the crystal starts to loss the weight from the very beginning and then remains stable. Both 0.6wt% and 0.8wt% creatinine doped crystals loss nearly 1% weight within room temperature to 150^oC and 222^oC, respectively. This minor amount of weight loss in the initial stage might be due to water, which served as the crystallization's solvent, has a chance of being included in the crystal lattice. It can be concluded that 0.2wt% and 0.4wt% doping of creatinine in KDP increases the thermal stability of pure KDP crystals, demonstrated by their ability to withstand elevated temperatures without undergoing a phase change, makes them potentially suitable for device applications operating within the specified temperature range.

After the stable region, pure as well as GA doped KDP crystals show one step decomposition process. The one step decomposition process of pure and doped KDP is in well agreement with the reported in literature [1]. Pure KDP decompose within temperature range 185 to 350 °C with mass loss 12.6%, while 0.2wt% creatinine doped KDP decompose within temperature 219 to 367 °C with mass loss 13.87%, 0.4wt% creatinine doped KDP decompose within temperature range 225 to 365°C with mass loss 15%, 0.6wt% creatinine doped KDP decompose within temperature range 216 to 355°C with mass loss 15.35% and 0.8wt% creatinine doped KDP decompose within temperature range 222 to 360 °C with mass loss 15.76%. This indicates that the presence of dopant creatinine increases the decomposition temperature of pure KDP from 350°C and also increases the mass loss from 12.6% to 15%. The increase in the decomposition temperature of creatinine doped KDP indicates increase in the bond energy due to the presence of dopant creatinine. Further, the residual mass in the crucible after decomposition up to 800°C temperature is 85%, which indicates that pure as well as creatinine doped crystals are highly stable.

Figure 3 shows the DTA curves of pure and different wt% creatinine doped KDP crystals.

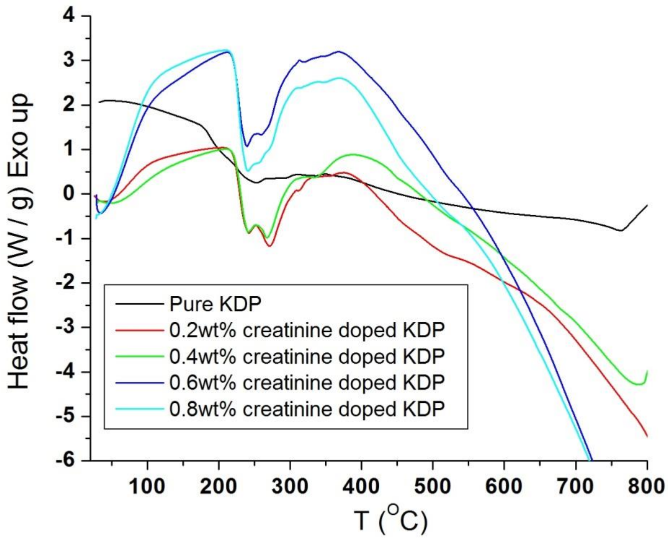

Figure 3. DTA curves

The DTA curve of pure and creatinine doped KDP shows endothermic peaks corresponding to the decomposition of pure and creatinine doped KDP crystals. The pure KDP shows endothermic peak at temperature 253 oC, while 0.2wt%, 0.4wt%, 0.6wt% and 0.8wt% creatinine doped KDP shows endothermic peak at temperature 270 °C, 270 °C, 240 °C and 240 °C, respectively. Hence, 0.2wt% and 0.4wt% doping of creatinine shifts the endothermic peak towards higher temperature side, while 0.6wt% and 0.8wt% doping of creatinine shifts the endothermic peak towards lower temperature side. The thermogram results are shown in the table 2.

Table 2. Thermal Analysis Result

Sample	Temperature range (°C)	Remaining weight (%)
Pure KDP	From RT to 185	100
	From 185 to 350	87.4
0.2 wt% creatinine doped KDP	From RT to 219	100
	From 219 to 367	86.13
0.4 wt% creatinine doped KDP	From RT to 225	100
	From 225 to 365	85
0.6 wt% creatinine doped KDP	From RT to 150	99
	From 150 to 216	stable
	From 216 to 355	84.65
0.8 wt% creatinine doped KDP	From RT to 90	99
	From to 90 to 222	Stable
	From 222 to 360	84.24

UV-Vis spectroscopy Analysis: In the case of NLO crystals, crucial parameters include minimal optical absorption of light in the UV-Vis region and a broader transparency window. These characteristics are significant for efficient nonlinear optical processes and overall optical performance. Whether, the grown crystals fulfill such requirements or not, UV-Vis analysis is carried out. Figure 4 shows optical transmittance spectra of pure and creatinine doped KDP crystals.

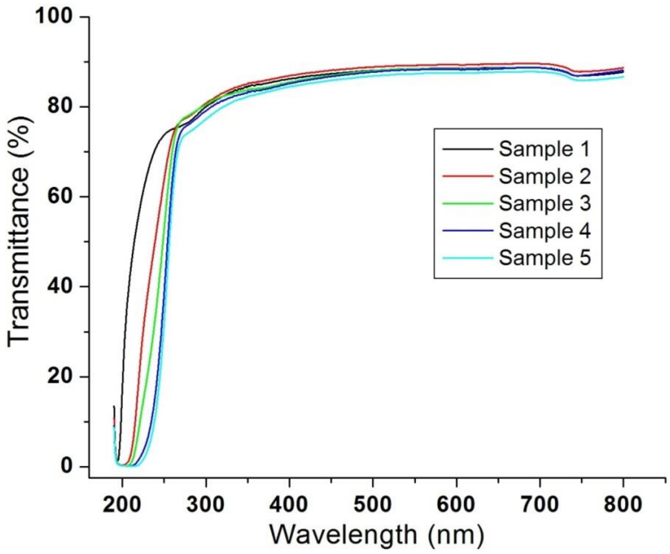

Figure 4. Transmission spectra

All the spectra demonstrate exceptional transparency throughout the entire visible region, with no noticeable alteration in transmittance, which is a critical requirement for crystals possessing nonlinear optical characteristics. In other words, the absence of additional absorption in the studied region is the well desired property for NLO [10]. It is well known that any crystal having high transmittance and low cut-off wavelength in the range of 200 to 400 nm can be used for optical device fabrication [11]. The crystal used in the setup ensures a good transmission throughout the entire visible region, making it highly suitable for second harmonic generation. [12]. The optical transmittance is decreased very slightly when creatinine is doped into KDP. The cut off wavelength of 195 nm is observed for pure KDP crystal, while it is observed to shift towards higher wavelength side after doping of creatinine.

For all the crystals, the transmittance% within optical region along with cut off wavelength is shown in the table 3.

Optical energy bandgap determination: The optical energy band gap (E_g) is evaluated for the pure and creatinine doped KDP crystals by using the Tauc relation [13]:

$\alpha h\nu = A(h\nu - E_g)^n$,

Where, h is Planck constant, ν is the frequency of incident photon, A is constant, Eg is the average band gap of the material and n is the index that depends on the types of transition. The value of the index n = 1/2, 3, 2 and 3/2 denotes allowed direct transition, forbidden indirect transition, indirect allowed transition and forbidden direct transition, respectively [14]. As the electromagnetic radiation passes through a given material, its intensity decreases. A measure of the rate of decrease in the intensity is known as optical absorption coefficient (α). The dependence of (α) on the photon energy (hν) is helpful for studying the electronic transition that occur in the material [15]. From the transmittance data, the value of α is calculated by using the standard formula given in the literature [16]:

$$\alpha = \frac{2.303 \log\left(\frac{1}{T}\right)}{d}$$

Figure 5 shows Tauc's plot for pure and creatinine doped KDP crystals.

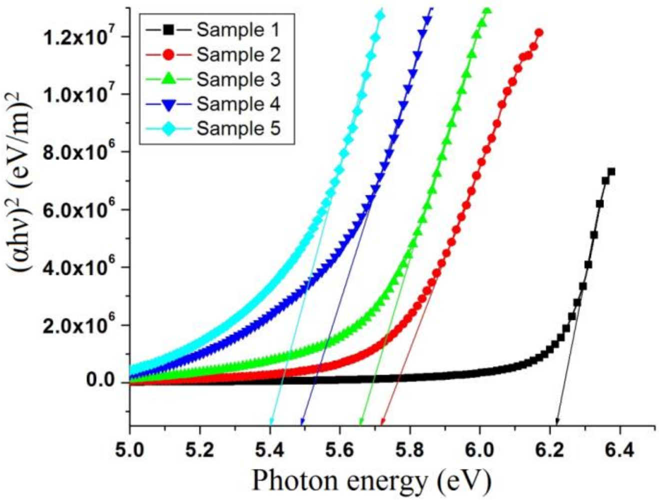

Figure 5. Tauc's plot of sample 1: pure KDP and samples 2 to 4: 0.2wt%, 0.4wt%, 0.6wt% and 0.8wt% creatinine doped KDP crystals

The plotted curves display the relationship between energy (hν) on the X-axis and $(\alpha h\nu)^2$ on the Y-axis. By extending the tangent of the linear portion of the graph to the X-axis, specifically at $(\alpha h\nu)^2 = 0$, we can determine the optical energy band gap (E_g) of the grown crystals. The optical energy band gap (E_g) values for both pure and creatinine-doped KDP crystals are provided in Table 3.

Table 3. Transmittance and optical energy band gap (E_g) values of pure and creatinine doped KDP crystals

Sample	Transmittance% within visible range	Bandgap energy (eV) from		Cut off wavelength λ(nm)
		the plot	calculation	
Pure KDP	85.59% to 88.72%	6.19	6.36	195
0.2 wt% creatinine doped KDP	86.94% to 89.6%	5.72	6.14	202
0.4 wt% creatinine doped KDP	85.52% to 88.76%	5.66	5.96	208
0.6 wt% creatinine doped KDP	85.22% to 88.67%	5.49	5.32	233
0.8 wt% creatinine doped KDP	84.49% to 87.80%	5.40	5.25	236

The results mentioned in table 3 indicate that the creatinine doped KDP crystals show there is a gradual decrease observed in the energy band gap value. Despite the decrease in the energy band gap value in creatinine-doped KDP crystals, the reasonably good energy band gap value suggests that the grown crystals are still suitable for the production of optoelectronic devices [17]. The obtained wide band gap energy of all the grown crystals confirms its large transmittance in the entire visible region [18,19]. In nonlinear optical applications, the requirement for a large band gap crystal arises from the fact that the energy band gap of a crystal indicates its ability to be polarized when subjected to intense radiation. In this context, the determined energy band gap (Eg) values for pure KDP and 0.2wt% and 0.4wt% creatinine-doped KDP crystals are found to be notably superior compared to other amino acid-doped ADP crystals, such as L-threonine, L-alanine, L-valine, L-cystine, L-lysine, and L-glycine [16,20-24]. The reduction of energy bandgap of creatinine doped KDP crystals compared to pure KDP indicates the formation of recombination centre in the form of lattice imperfection. These energy levels, positioned within the forbidden band of the material, have the capability to recombine with conduction electrons and holes [25].

The band gap is theoretically calculated using the following relationship:

$$E_g = \frac{hc}{\lambda_c}$$

Where c is the velocity of light and λc is cut-off wavelength. The calculated theoretical band gap is listed in the table 3 and it is closely related to the value of the band gap obtained from the Tauc's plot.

SHG Analysis: To verify the nonlinear optical (NLO) property, the grown crystals of pure KDP and various weight percentages (wt%) of creatinine-doped KDP were subjected to a Kurtz powder test. This test involved using a Q-switched high-energy Nd:YAG laser with modulated radiation at a wavelength of 1064 nm, a repetition rate of 10 Hz, and a pulse width of 6 ns. The laser beam was directed onto the crystal powder, with a visible blocking filter in place, to maximize the powder's second harmonic generation (SHG). The emission of green radiation at a wavelength of 532 nm confirmed the generation of the second harmonic signal in the crystals, validating their NLO behavior. The NLO SHG efficiency was calculated by comparing it to the monocrystalline powder of KDP used as a reference material, employing the appropriate equation [26]:

SHG Efficiency = Output Energy (sample)/Output Energy (KDP)

The SHG efficiency of pure and creatinine doped KDP crystals are listed in the table 4.

Table 4. SHG efficiency

Sample	Output (mV)	SHG Efficiency
Pure KDP	23	1
0.2wt% creatinine doped KDP	25	1.08
0.4wt% creatinine doped KDP	28	1.21
0.6wt% creatinine doped KDP	29	1.26
0.8wt% creatinine doped KDP	39	1.69

From Table 4, it is evident that the second harmonic generation (SHG) efficiency, measured relative to KDP, exhibits a gradual increase with the addition of creatinine and an increase in its weight percentage. The increased efficiency of creatinine doped KDP can be hypothesized that the doping of creatinine into KDP, may find the interstitial position due to its large size and it might have been acted as a proton donor and hence donate the hydrogen to KDP, which combine with the unit cell of KDP, which ultimately increases the SHG efficiency of KDP. As the doping concentration is minute, this occurs with slight distortion in the KDP lattice. Further, the increased SHG efficiency of creatinine doped KDP also suggests that the dopant has not affected the non-centrosymmetric packing of the molecular crystal of KDP and results into favorable molecular alignment for nonlinearity.

5. Conclusion and Future Scope

Pure and creatinine doped KDP crystals were successfully grown at room temperature by solution growth technique. Transparent, colorless and almost rectangular shaped crystals were grown within a time period of nearly one month. The carbon and nitrogen from dopant atoms were present, according to the EDAX study and weight% was observed to increase as the weight% of dopant creatinine was increased. The thermogravimetry analysis showed increased thermal stability of 0.2wt% and 0.4wt% creatinine doped KDP crystals. The presence of dopant creatinine influenced the decomposition temperature and mass loss% of pure KDP. The decomposition temperature as well as mass loss% of pure KDP was observed to increase due to the effect of dopant creatinine. The DTA curves showed endothermic peaks corresponding to the decomposition temperatures of pure and creatinine doped KDP crystals. 0.2wt% and 0.4wt% doping of creatinine shifted the endothermic peak towards higher temperature side, while 0.6wt% and 0.8wt% doping of creatinine shifted the endothermic peak towards lower temperature side. Compared to pure KDP, the UV-Vis analysis showed the shifting of cut-off wavelength towards higher wavelength side and slight reduction in the transmission due to doping of creatinine. The energy bandgap of creatinine doped KDP was reduced compared to pure KDP. The SHG analysis showed increased SHG efficiency of creatinine doped KDP crystals up to 69%.

Data Availability
The raw data required to ongoing study; hence it cannot be shared.

Conflict of Interest
The authors declare that there are no competing interests.

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Authors' Contributions
Experimental, conceptional study, data collection: H.Bhuva; Analysis & interpretation of results : Dr.H.O.Jethva, H.Bhuva; Draft manuscript design : H. Bhuva; Supervising & editing manuscript : Dr.H.O.Jethva;

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Dr. H. O. Jethva is a post graduate from Saurashtra University, Rajkot, Gujarat and Doctorate from the same university. He has published thirty research papers in International Journals of repute and five books for the undergraduate students of the same university. He holds an experience of teaching Physics to undergraduate and postgraduate students for twenty-seven years. He is currently working as the Professor in the Department of Physics at the same university.

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