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Doping effect of carboxylic acids on optical, electrical, mechanical and thermal traits of KDP crystal

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ABSTRACT

Single crystals of maleic acid (MA)- and oxalic acid (OA)-doped potassium dihydrogen phosphate (KDP) materials have been grown by slow evaporation solution technique. The structural parameters of the grown crystals have been determined using the single crystal X-ray diffraction technique. Fourier transform infrared analysis has been done to identify the functional groups of grown crystals. The modifications in optical transmittance, optical band gap and different optical constants of KDP crystal due to addition of MA and OA have been investigated by means of UV–Visible spectral analysis. The Vicker's microhardness studies have been carried out to examine the mechanical behaviour of pure and doped KDP crystals. The influence of dopants on thermal stability of KDP crystal has been investigated using the thermogravimetric and differential thermal analysis. The dielectric constant, dielectric loss, ac resistivity and ac conductivity of pure and doped KDP crystals were evaluated under dielectric studies.

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KEYWORDS
Growth from solution; dielectric studies; optical studies; thermal studies

1. Introduction

Since past decade, the interest of researchers has been sustained in upgrading the properties of potassium dihydrogen phosphate (KDP) crystal as it is largely demanded for fabricating the devices for SHG systems, industrial photonics, optical parametric oscillators, electro-optic modulators, telecommunication and large laser facility systems [1]. The KDP crystal owing to high optical homogeneity, good SHG coefficient (d_{36} = 0.39 pm/V) and excellent cubic susceptibility (χ^3) of order 10^{-14} esu realises huge technological impetus for blue–green laser systems and all-ultrafast optical synchronisation and signal processing devices [2,3]. The need of high quality non-linear optical (NLO) material for hi-tech components has enforced many researchers to modify the intrinsic properties of KDP crystal by inclusion of Li(I), Ca(II), Ce(IV), V (V), amaranth-, rhodamine-, methyl orange-dye, urea and bis-thiourea zinc chloride additives [4–7]. Recent studies have shown the significant role of organic additives particularly carboxylic acids in uplifting the qualities of ADP and KDP crystals which hold importance for device fabrication. The impact of increasing molar ratio of DL-malic acid has successively enhanced the optical, electrical, mechanical and SHG response of ADP crystal [8]. The addition of oxalic acid (1 mole) has promoted the SHG efficiency, mechanical and electrical performance of ADP crystal [9]. In our last report large enhancement in optical, SHG efficiency, third order NLO and electrical performance of KDP crystal has been achieved by doping different mole% of formic acid [1,10]. The large enhancement in SHG efficiency and third order NLO performance of KDP crystal has been observed due to incorporation of oxalic acid (OA) and maleic acid (MA) [11]. To subject a material for particular device fabrication, the details on properties of material are of prime importance. Hence, in this manuscript, we firstly report the impact of OA and MA on structural, optical, mechanical, thermal and electrical properties of KDP crystal which plays key role in tuning the NLO devices.

2. Experimental procedure

The high purity Merck make KDP salt was gradually dissolved in double-distilled water to obtain the supersaturated solution of KDP at room temperature. The precisely measured quantity of 1mole of MA and OA was separately introduced to the supersaturated solution of KDP material taken in two beakers. The MA and OA added KDP solutions were allowed to stir for 6 h to achieve the homogeneous doping throughout the solution. These solutions were filtered in rinsed beakers using the micro filter paper and the solutions were crystallised by slow solution evaporation technique in a constant temperature bath of accuracy ±0.01 °C. The single crystals

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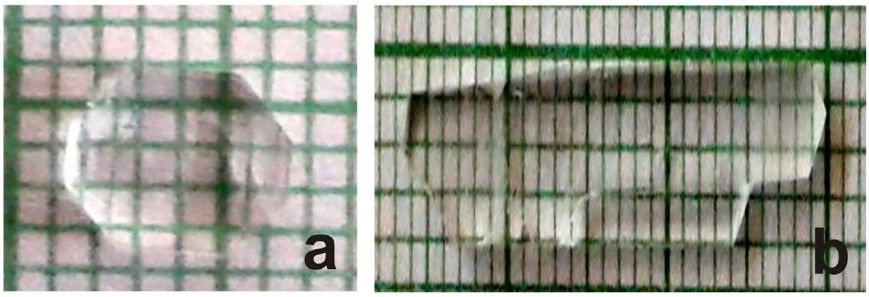

Figure 1. Crystal of (a) KDPOA, (b) KDPMA.

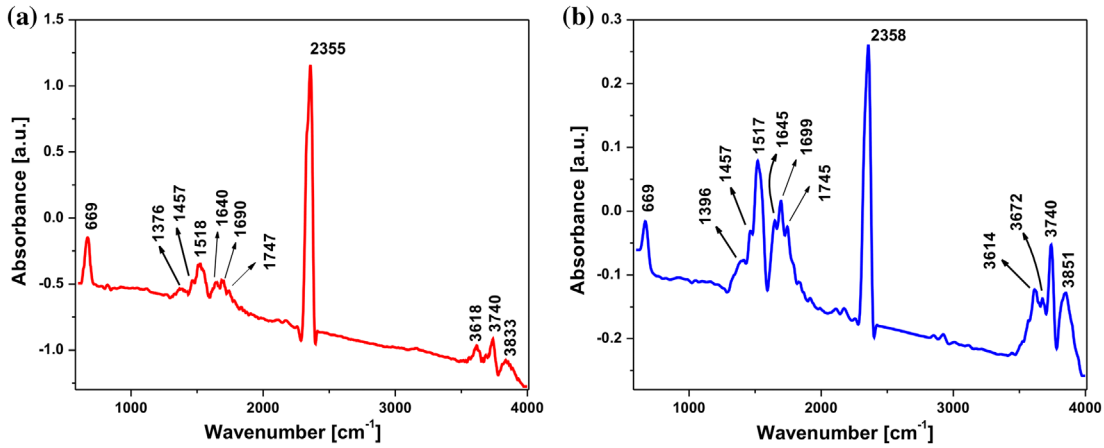

Figure 2. FTIR spectrum of (a) KDPMA, (b) KDPOA crystal.

Table 1. Crystallographic data.

Crystal	Cell parameters (Å)	Volume (Å)^3	Crystal system	Space group
KDP	a = b = 7.44, c = 6.94	384	Tetragonal	I-42d
KDPOA	a = b = 7.45, c = 6.97	387	Tetragonal	I-42d
KDPMA	a = b = 7.45, c = 6.99	388	Tetragonal	I-42d

of OA-doped KDP (KDPOA) and MA-doped KDP (KDPMA) materials are shown in Figure 1(a) and (b), respectively. The purity of grown crystals has been achieved by repetitive recrystallisation process.

3. Results and discussion

3.1. Single crystal XRD analysis

The crystallographic parameters of grown crystals were experimentally determined by means of X-ray diffraction (XRD) technique using the ENRAF NONIUS CAD4 single crystal X-ray diffractometer. The grown crystals were confirmed to have tetragonal crystal symmetry and the structural parameters of pure and doped KDP crystals are discussed in Table 1. The small changes observed in lattice parameters of doped KDP crystals indicate the influence of dopants in lattice sites of KDP crystal. It is notable that the volume of KDP crystal increases with addition of dopants, while the space group and crystal system remain unaltered.

3.2. Fourier transform infrared (FTIR) analysis

The qualitative analysis of doped KDP crystals has been done using the FTIR analysis using the Bruker-ATR spectrophotometer operative in the range of 600–4000 cm^{-1}. The recorded FTIR spectrum of MA- and OA-doped KDP crystal is shown in Figure 2(a) and (b), respectively. The assigned wavenumber for identified functional groups is shown in Table 2. The absorption peak at 669 cm^{-1} corresponds to O–H bond bending vibration. The mild absorption peaks observed at 1376 and 1396 cm^{-1} are contributed due to C–H bond deformation of respective carboxylic acid. In doped KDP crystals, the P–C bond stretching vibration is evident at wavenumber 1457 cm^{-1}. The C–H bending vibration is attributed at 1518 and 1517 cm^{-1}. The symmetric stretching of O=P=OH bond is evident at 1640 and 1645 cm^{-1}. The characteristic C=O stretching of carbonyl group is evident at 1690 and 1699 cm^{-1}. The aliphatic C=O stretching vibrations are associated at 1747 and 1745 cm^{-1}. The P–H bond stretching of phosphate group in KDP is evident at 2355 and 2358 cm^{-1}. The peaks observed at 3618 and 3614 cm^{-1} correspond to the O–H bond stretching vibration. The CH_2 stretching vibration is evident at 3740 cm^{-1} [10,12]. These spectral analyses confirm the incorporation of OA and MA in KDP crystal.

Table 2. Functional groups of doped KDP crystals.

Wavenumber (cm^{-1})		
KDPMA	KDPOA	Assigned functional group
669	669	O–H bending
1376	1396	C–H deformation
1457	1457	P–C stretching
1518	1517	C–H bending
1640	1645	O=P=OH stretching
1690	1699	C=O stretching
2355	2358	P–H stretching
3618	3614	O–H stretching
3740	3740	CH_2 stretching

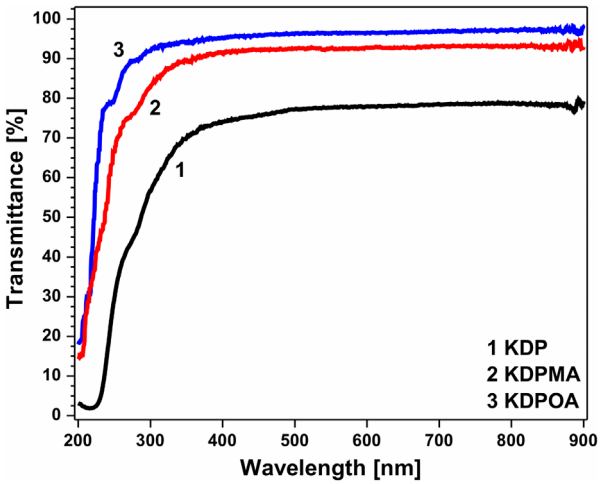

Figure 3. UV–Visible transmittance spectrum.

3.3. UV–visible spectral analysis

The material with excellent optical performance in visible region fetches large demand for devices assistive in UV-tunable lasers and laser frequency conversion systems [13]. The UV–visible transmittance spectrum (Figure 3) of pure and doped KDP crystals was recorded in the range 200–900 nm using the Shimadzu UV-2450 spectrophotometer. The spectrum reveals that the optical transparency of doped KDP crystals is significantly higher than KDP in entire visible region. The transmittance (T) of KDP crystal is found to be 78% in absence of dopant, 92% with dopant MA and 96% with dopant OA. The increased transparency of doped KDP crystals is superior parameter desirable for efficient transmission of SHG radiation of Nd:YAG laser and NLO devices [14]. The tauc's plot has been obtained using the equation $(\alpha hv)^2 = A\ (hv - E_g)$ which conveys the dependence of optical band gap (E_g) on absorption coefficient (α) of the material. The analysis of Figure 4(a) and (b) revealed that the optical band gap of pure, MA- and OA-doped KDP crystal is found to be 5.1, 5.24 and 5.5 eV, respectively. It is observed that the band gap value of KDP crystal has been enhanced due to addition of dopant MA and OA, respectively. The crystals having larger E_g value exhibit high transmittance over wide range of wavelength [15], which stands true in case of doped KDP crystals in present study. The doped KDP crystals with

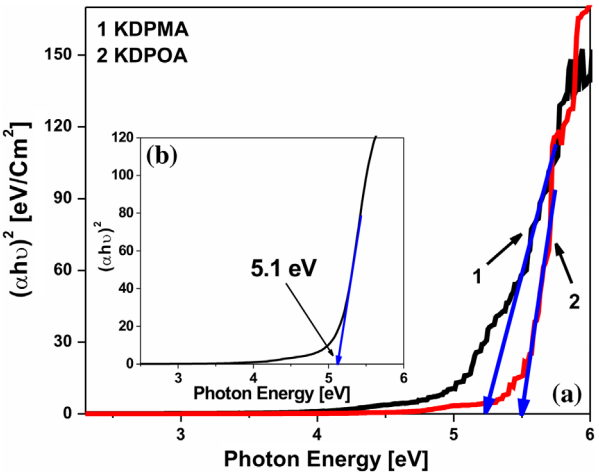

Figure 4. Tauc's plot.

high transmittance and enhanced band gap value are thus highly suitable for optoelectronics devices [16].

The optical density and loss of optical energy in crystal medium due to reflectivity, absorption and scattering effect can be understood by evaluating various optical constants such as, optical conductivity, extinction coefficient, refractive index and reflectance of the material medium. The optical conductivity of doped KDP crystals increases with increasing photon energy as depicted in Figure 5(a). The increasing magnitude of optical conductivity and lower extinction coefficient (Figure 5(b)) suggests the prime utility of doped KDP crystals for optical information processing and computing devices [17]. The nature of refractive index and reflectance of pure and doped KDP crystals in visible region is shown in Figure 6(a) and (b), respectively. The refractive index and reflectance of doped KDP crystals are impressively lower than KDP crystal which is desirable quality for anti-reflection coating for solar thermal devices [18,19]. The doped KDP crystals with lower refractive index find its prominence for calibrating the merit of photonic devices such as optical filters, resonators and reflectors [20]. The high optical transparency, lower refractive index and lower reflectance indicate that the dopants have largely suppressed the activity of point and line defects in KDP crystal [21]. The superlative optical homogeneity of OA-doped KDP crystal is the key requirement for designing electro-optic modulators [22].

3.4. Vicker's microhardness studies

In order to study the mechanical stability of the crystal material, the microhardness analysis of pure and doped KDP crystals has been performed along ⟨0 0 1⟩ plane of crystals at 25, 50 and 100 g using the Shimadzu HMV-2T microhardness analyzer. To calculate the Vicker's hardness number (H_v), several indentations were made on the crystal surface for each load and the corresponding diagonal length of indentation was recorded. The hardness number was calculated using the relation,

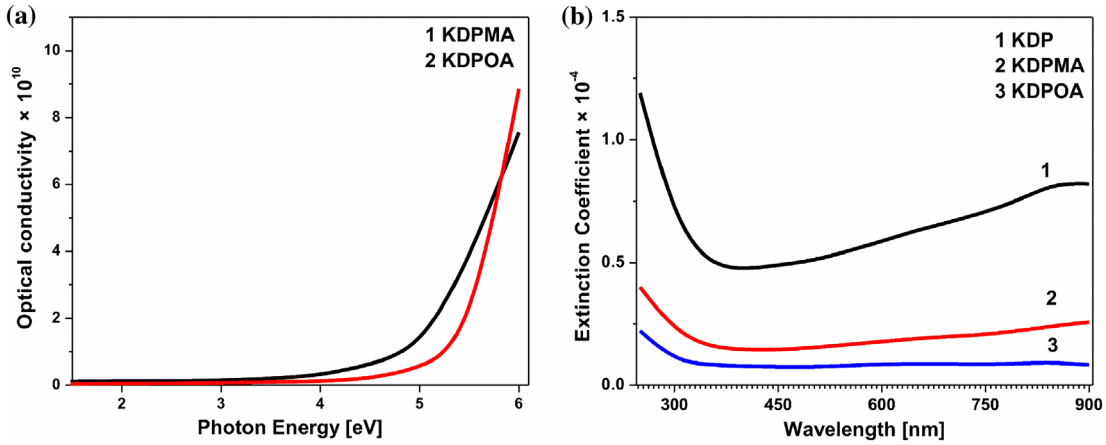

Figure 5. (a) Plot of optical conductivity. (b) Extinction coefficient as a function of wavelength.

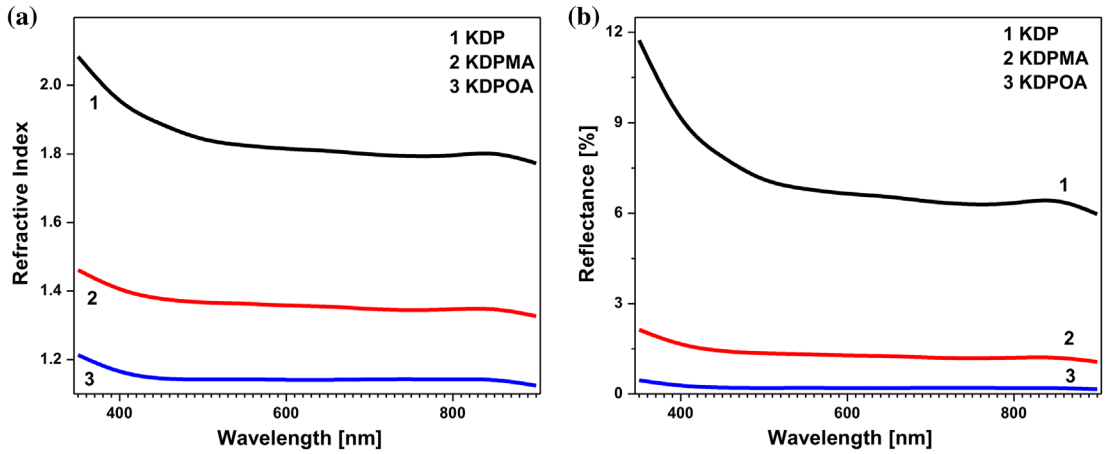

Figure 6. Wavelength dependent profile of (a) Refractive index. (b) Reflectance.

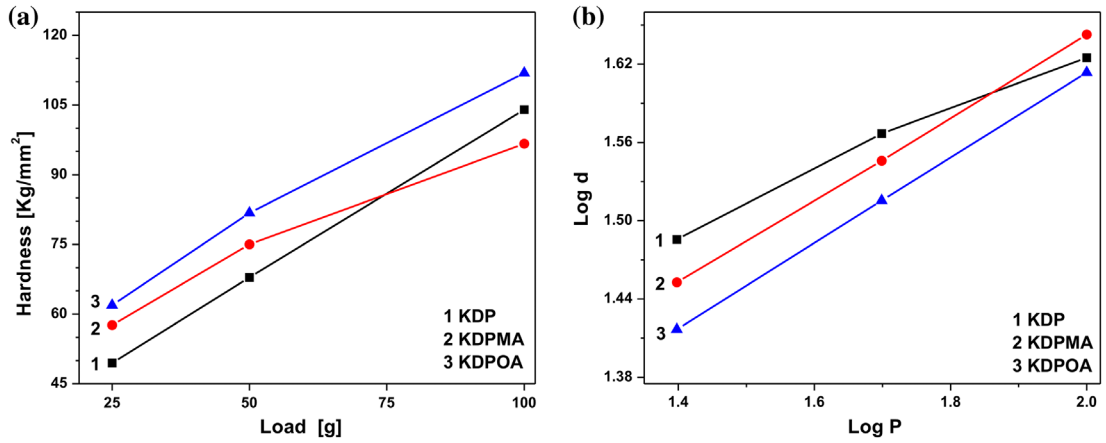

Figure 7. (a) Load dependent hardness (b) log *P* vs. log *d*.

$H_v = 1.8544 \times P/d^2$ kg/mm^2 where, P is the applied load in kg and d is the diagonal length in mm. The variation of hardness with applied load is shown in Figure 7(a) and it reveals that the hardness of pure and doped KDP crystals increases with increasing load. The hardness of doped KDP crystals is found to be greater than KDP crystal at all loads except the hardness of MA-doped KDP crystal is low at 100 g. In MA-doped KDP crystal, the cracks were developed at 100 g which is attributed to liberation of internal stress generated by indentation [23]. It is notable that OA offered exclusively higher hardness to KDP crystal, this implies that dopant has significantly minimised the defect centres and solvent inclusions reinforcing the enhancement in threshold load for lattice dislocation. The Meyer's relation, $P = ad^n$ explains the effect of applied load on work hardening

index (n) of the crystal. It is calculated from the plot of log P vs. log d as displayed in Figure 7(b). The value for n is found to be 4.3, 3.1 and 3 for pure, MA- and OA-doped KDP crystals, respectively. In accordance to Onitsch and Hannemen study, as the value of n exceeds 1.6 the material belongs to the category of soft nature. However, in present analysis the value of n of KDP successively decreases with dopant MA and OA which is imperative quality for reducing the lattice dislocation [24]. The improved mechanical properties are necessary to avoid loss of material while polishing and processing for device fabrication [23], for which the hardness reliant properties such as yield strength ($\sigma_y = (0.1)^{n-2} H_v/3$) and elastic stiffness coefficient (given by Wooster's empirical relation, $C_{11} = H_v^{7/4}$) have been calculated. The higher magnitude of σ_y and C_{11} confirms the constructive impact of dopants in enhancing the intermolecular bond strength [25]. The analysis of data (shown in Table 3) confirms excellent mechanical behaviour of KDP crystal in presence of dopant OA.

3.5. Thermal analysis

The thermal behaviour of grown crystal samples have been investigated in the homogeneous nitrogen atmosphere by means of thermogravimetric and differential thermal analysis (TGA–DTA) within the temperature range of 30–450 °C at a heating rate of 20 °C/min. The recorded TGA curve of pure and doped KDP crystals is shown in Figure 8(a). The nature of recorded TGA curves indicate that the dopants have significantly reduced the thermal stability of KDP crystal which might have been occurred due to the weak hydrogen bonding associated with carboxylic acid. The DTA curve (Figure 8(b)) helps to determine the effect of dopants on melting point of KDP crystal. The doped KDP crystals exhibited endothermic peaks at lower temperatures as compared to KDP which is attributed to the lower thermal sustainability of carboxylic acid in high temperature range. It is noticeable that the OA-doped KDP crystal displays a mild endothermic peak around 100 °C attributed due to the liberation of solvent impurity within the crystal, which is absent in pure and MA-doped KDP crystal. The obtained thermal data of grown crystals are shown in Table 4. The preliminary melting of doped KDP crystals is found to be appreciably higher as compared to urea phosphate [26], L-alanine and L-arginine [27] doped KDP crystals.

3.6. Electrical studies

The dielectric studies of pure and doped KDP crystals have been investigated within 10–100 kHz using the Gwinstek-819 LCR cubemeter. The flat crystal (2.5 mm thickness) samples were smoothly layered by silver paste so as to conduct the measurements with high accuracy. The recorded dielectric constant at different frequencies is shown in Figure 9(a). In a crystal medium, the dielectric constant is contributed by electronic, ionic, dipolar and space charge polarisation effects which are active at lower frequencies and less responsive in high frequency domain [28]. It is observed that the dielectric constant of all the crystals decreases with increase in frequency however, the magnitude of dielectric constant of KDP crystal successively decreases with addition of OA and MA, respectively. The Miller rule and Phillips-Van Vechten-Levine-Xue bond theory successfully explained the fact that lower dielectric constant of material is most desirable parameter for enhancing the SHG coefficient

Table 3. Hardness parameters.

Crystal	H_v (kg/mm^2)	n	σ_y (MPa)	C_{11} (GPa)
KDP	73.85	4.3	11.31	33.89×10^5
KDPMA	76.41	3.1	16.89	34.91×10^5
KDPOA	85.17	3.05	24.36	42.54×10^5

Table 4. Thermal analysis data.

Crystal sample	KDP	KDPMA	KDPOA
Decomposition temperature	302 °C	291 °C	260 °C

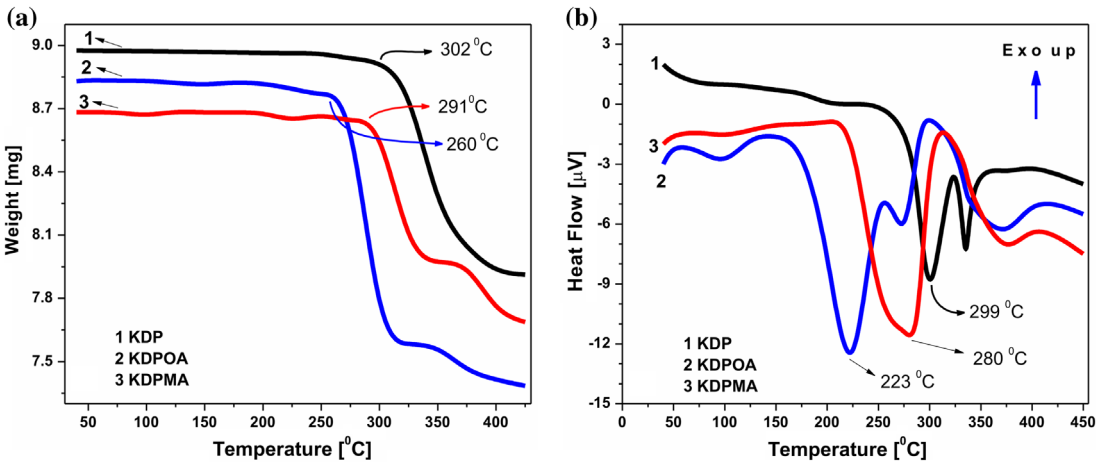

Figure 8. (a) TGA curve. (b) DTA curve.

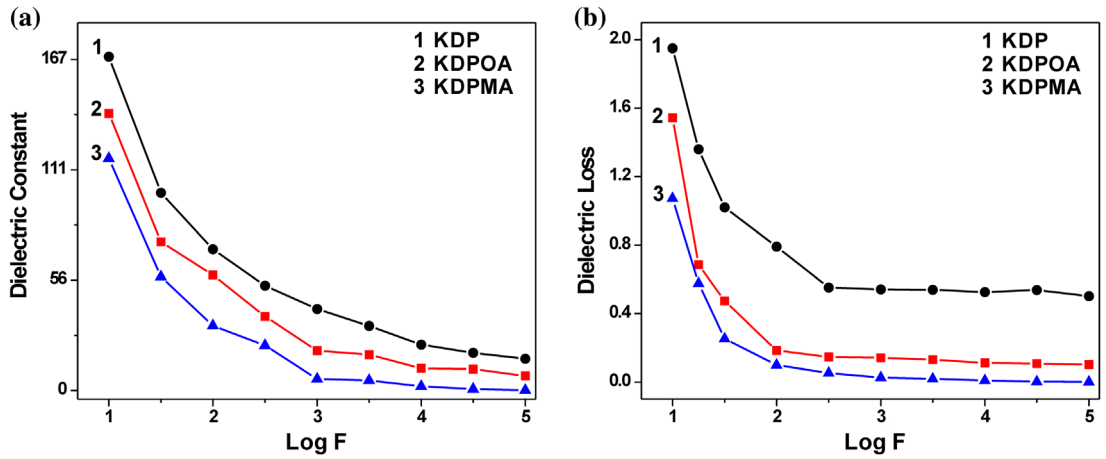

Figure 9. Frequency dependent. (a) Dielectric constant. (b) Dielectric loss.

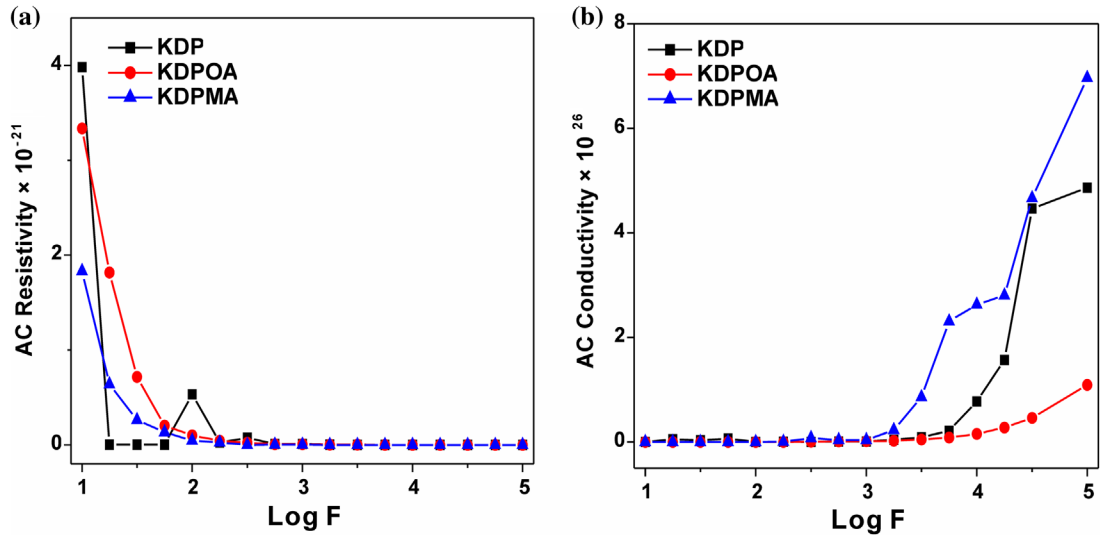

Figure 10. Frequency dependent (a) ac resistivity, (b) ac conductivity.

[23]. The lower dielectric constant of doped KDP crystals signifies the least power consumption which is essential requirement for fabricating devices for broadband electro-optic modulators (EOM), filed detectors, photonics and microelectronics applications [29]. Figure 9(b) shows the frequency dependent profile of dielectric loss. The doped KDP crystals show lower dielectric loss than KDP crystal. This confirms the occurrence of minimum electrically active defects which are responsible for enhancing the intrinsic quality of OA- and MA-doped KDP crystal [30,31]. For studied crystals, the measured ac resistivity decreases and ac conductivity increases with increase in frequency as depicted in Figure 10(a) and (b), respectively. The large value of ac conductivity at higher frequencies closely agrees with the small polaron hopping model theory [32].

4. Conclusion

Optically transparent OA- and MA-doped KDP crystals have been successfully grown by slow solution evaporation technique. The tetragonal crystal structure and slight change in cell parameters of doped KDP crystals have been confirmed by single crystal XRD analysis. The prominent peaks of carbon coordinated bonds were successfully identified in FTIR spectrum of MA- and OA-doped KDP crystals. The UV–visible analysis revealed the rise in optical transparency of KDP crystal by 14 and 18% due to inclusion of MA and OA, respectively. The corresponding E_g value of MA and OA doped KDP crystal is found to be 5.24 and 5.5 eV. The lower extinction coefficient, lower refractive index and lower reflectance demonstrate huge advantage of doped KDP crystals for applications of SHG, optoelectronics and solar thermal devices. The microhardness studies along ⟨0 0 1⟩ plane revealed that the hardness number of pure, MA- and OA-doped KDP crystal is found to be 73.85, 76.41 and 85.17 kg/mm^2, respectively. It is also confirmed that the mechanical properties of KDP crystal has been remarkably increased with addition of MA and OA. In TG/DTA analysis, the decomposition temperature of KDP crystal is found to be higher in absence of dopants MA and OA. The lower dielectric constant and dielectric loss of doped KDP crystals are vital parameters

for NLO applications. The nature of ac resistivity and ac conductivity of crystals has been studied. Interestingly, the optical and mechanical properties of KDP crystal are excellent with dopant OA while the dopant MA dominates to offer lower dielectrics to KDP crystal. Above studies infer that OA and MA played decisive role in enhancing the characteristic features of KDP crystal making it more promising material for fabricating NLO devices.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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