

VIBRATIONAL SPECTROSCOPIC STUDIES OF KDP , PURE AND DOPED

WITH NH₄Br

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ABSTRACT

KDP is well known organic crystal. It has an interesting property such as NLO, wide frequency conversion, high damage threshold against power laser and good UV transmission. In the present study single crystal of pure KDP and doped with different concentration (0.1 mol, 0.2 mol, 0.3 mol) of ammonium bromide crystal has been successfully grown by slow evaporation method. The influence of doping on grown crystals has been studied. The grown KDP doped with ammonium bromide crystals has been studied by raman spectra analysis.

Keywords : NLO, ammonium bromide, ammonium bromide, wide frequency conversion, organic crystal.

1. POTASSIUM DIHYDROGEN PHOSPHATE (KDP)

Monopotassium phosphate or potassium dihydrogen phosphate, KDP (KH₂PO₄) is a soluble salt which is used as a fertilizer[1-6], a food additive and fungicide. It is a source of phosphorus and potassium. It is also a buffering agent.

Potassium dihydrogen phosphate (KDP) isomorphous crystals, deuterated KDP known as KD*P or DKDP (KD₂PO₄) are widely used in frequency doubling, frequency tripling and frequency quadrupling of Nd-doped laser systems, as well as the electro-optical modulators. DKDP is mainly used in pockels cells [1].

Potassium dihydrogen phosphate is well known for their good piezoelectric and nonlinear optical properties [2, 3].

Non linear optics is a very useful technology because it extends the usefulness of lasers by increasing the number of wavelengths available. Non linear optics has emerged as one of the most attractive field of current research in view of its vital applications in the area of optical modulation, optical switching, optical logic, frequency shifting, high speed information processing and different auto electric applications [4-7]

They have attracted interest of many theoretical and experimental researchers probably because of their comparatively simple structure and very fascinating properties associated with a hydrogen bond system. Involving a large isotope effect, broad transparency range, a high optical damage threshold and relatively low production cost [8-13].

On account of various applications, doping on pure KDP crystals by some additive is increasingly interesting because of fact that dopant may change its properties [20-24].

In the present paper, some KDP crystals doped with NH_4Br prepared using solution method, and their Raman Spectra are studied.[14-18]

2.EXPERIMENTAL

The crystals for the present study (pure KDP, KDP doped with NH_4Br in three different concentrations (0.1 mol, 0.2 mol and 0.3 mol) are prepared using solution growth method and gel method.[19-23]

In a beaker 100 ml of distilled water was taken. The saturated solution of KDP (33 gm) was prepared. After 5 days the seed crystals were found. From that good quality

transparent crystals were collected. These were hung in the solution by a nylon thread. After 16 days the crystals were harvested and washed with triple distilled water.

Ammonium bromide were added as dopants with KDP. According to the molarity of ammonium bromide, they were taken in three different ratios and added with KDP[25-30].

In a beaker, 100 ml of distilled water was taken. A saturated solution of KDP (33gms) was prepared. For 0.1 mol/l molarity of ammonium bromide, 1g of it was added to the saturated solution of KDP. After 10 days seed crystals were found, which were later harvested and washed with triply distilled water.

Similarly, for 0.2 mol/l molarity, 2g of ammonium bromide was added with the saturated solution of KDP, and for 0.3 mol/l, 3g of ammonium bromide was added with KDP. After a week seed crystals were found which were later harvested.

3.RESULTS AND DISCUSSION

The Raman spectra of pure KDP, and KDP doped with NH_4Br , in different concentrations, were recorded[31-35].

Raman spectra of pure and doped KDP are shown in Figs 1 to 4. The observed Raman peaks, and their assignments to different modes are given in table 1 respectively.

Factor group analysis for KDP was done for the KDP crystal which belongs to D_{2d} , forming space group $I4_2d$. The cell dimensions are $a = 0.74532(3)$ nm and $c = 0.69742$ nm. The primitive cell contains 4 formula units [36-39]. Using the

Bhagavantham and Venkatarayadu method of factor group analysis, the irreducible representations of the lattice vibration of the KDP crystal are as follows:

$$11 A_1 + 11 A_2 + 13 B_1 + 13 B_2 + 24 E.$$

For the D_{2d} group the Raman and infrared active optical phonon modes at zero wave vector are:

$$11 A_1 (\text{Raman}) + 13 B_1 (\text{Raman}) + 12 B_2 (\text{Raman, IR}) + 23 E (\text{Raman, IR}).$$

The A_2 modes are neither active in Raman nor active in IR. This means that only 82 Raman and 35 Infrared peaks are expected theoretically. From Tables it can be seen that there are 98 Raman lines observed in the case of pure KDP, It can be seen from the figures that the Raman lines are of much higher intensity for the sample doped with NH_4Br doped at 0.1 M concentration[40-45].

The lines at less than 100 cm^{-1} are assigned to lattice modes. The number of lines are almost the same in all the samples. The main difference is in the case of the sample doped with 0.1 M NH_4Br . The intensity of the line at 85 cm^{-1} is more than double in intensity, compared to other samples[46-50].

There is a Raman shift occur at 115.34 cm^{-1} . Only the band corresponds to KDP doped with NH_4Br (0.3 M) shows a shift at 119.2 cm^{-1} . Other two bands for KDP doped with NH_4Br (0.1 M, 0.2 M) shows a peak at 115.3 cm^{-1} .

The lines between 300 to 600 nm is assigned to the PO_2 stretching mode. Similarly, the lines between 600 and 1000 nm are assigned to the POP asymmetric stretching. The bands between 1000 and 1300 nm are assigned to the PO_3 modes. The lines above 1400 nm are mainly due to NH_4 vibrations, the assignment of which are shown in the table. Some lines observed in this region for the pure KDP are probably due

to some combination bands. Most the ammonium lines are observed in all the doped samples. The most intense line at 915 cm^{-1} is due to this band. There is slight variation in the intensity of the lines. The intensity of all the lines are much higher in the case of the sample doped with $0.1\text{ M NH}_4\text{Br}$.

At 362 cm^{-1} the band of KDP doped with NH_4Br shows a Raman shift (360 cm^{-1}). But the other two bands do not show any bands.

Another Raman shift occurs at 529 cm^{-1} . The corresponding peak of KDP doped with NH_4Br (0.1 M) is at 528 cm^{-1} . For KDP doped with NH_4Br (0.2 M) is at 531 cm^{-1} . At the same time, the peak for KDP doped with NH_4Br (0.3 M) is at 526 cm^{-1} .

At 641.8 cm^{-1} and 655 cm^{-1} the band corresponding to pure crystal shows a small increase in intensity while comparing with other three bands. But at 680 cm^{-1} the intensity of band corresponding to KDP doped with NH_4Br (0.2 M) is higher than that of other three bands.

At 915 cm^{-1} , all the four bands show the maximum intensity. The intensity is high for KDP doped with NH_4Br (0.1 M) which is almost double the intensity of the pure KDP crystal. KDP doped with NH_4Br (0.2 M) shows a decrease in intensity compared with pure KDP. The band corresponding to KDP doped with NH_4Br (0.3 M) has almost same intensity as that of pure KDP crystal.

At 1206 cm^{-1} the intensity of band of KDP doped with NH_4Br (0.3 M) shows a high value than that of pure KDP. At 1243 cm^{-1} the intensity of band corresponding to KDP doped with NH_4Br (0.2 M) is higher than all the other bands. At 1266.64 cm^{-1} the intensity of band of pure KDP is higher than that of other three bands. At 1295 cm^{-1} , the intensity of the KDP doped with NH_4Br (0.3 M) is high.

At 1347.6cm^{-1} the intensity of band of KDP doped with NH_4Br (0.2 M) sharply decreases when compared with pure KDP. Also the Raman band corresponding to KDP doped with NH_4Br (0.1 M) shows a split at 1345 cm^{-1} and 1351 cm^{-1} .

At 1457 cm^{-1} the intensity of band corresponding to KDP doped with NH_4Br (0.3 M) is twice greater than that of pure KDP.

At 1496 cm^{-1} the intensity of pure KDP is decreases rapidly. At the same time, at 1494 cm^{-1} the intensity of band of KDP doped with NH_4Br (0.3 M) is very high. At 1692 cm^{-1} the intensity of KDP doped with NH_4Br (0.3 M) band is very high compared to pure KDP.

At 1648 cm^{-1} intensity is high for KDP doped with NH_4Br (0.3 M) .the intensity is lightly high for KDP doped with NH_4Br (0.1 M) shows a decrease in intensity of KDP doped with NH_4Br (0.2 M).

The intensity of band at 1717 cm^{-1} for the KDP doped with NH_4Br (0.1 M) is high the intensity of band at 1781 cm^{-1} KDP doped with NH_4Br (0.2 M) is very high compared to all the other bands.

At 1918 cm^{-1} the intensity of pure KDP crystal is very high. All the other bands have no prominent peaks. At 2011 cm^{-1} the intensity of band for KDP doped with NH_4Br (0.2 M) is high.

At 2221 cm^{-1} the band corresponding to KDP doped with NH_4Br (0.1 M) is very high compared to that of pure KDP. At the same time the band of KDP doped with NH_4Br (0.3 M) is less intense than that of pure KDP. Also the band for KDP doped with NH_4Br (0.2 M) shows very low intensity when compared with band of pure KDP.

At 2676 cm^{-1} the intensity of band of KDP doped with NH_4Br (0.2 M) is high intense when compared with that of other bands at same wave number.

At 2684 cm^{-1} the band of pure KDP is higher than that of the band corresponding to KDP doped with NH_4Br (0.1 M).

At 2709 cm^{-1} the bands corresponding to KDP doped with NH_4Br (0.1 M) and KDP doped with NH_4Br (0.2 M) overlap with each other.

At 1295 cm^{-1} , the intensity of KDP doped with NH_4Br (0.3 M) is high.

At 1347.6 cm^{-1} the intensity of band of KDP doped with NH_4Br (0.2 M) sharply decreases when compared with pure KDP. Also the band corresponding to KDP doped with NH_4Br (0.1 M) shows a split at 1345 cm^{-1} and 1351 cm^{-1} .

At 1457 cm^{-1} the intensity of band corresponding to KDP doped with NH_4Br (0.3 M) is twice greater than that of pure KDP.

At 1496 cm^{-1} the intensity of pure KDP is decreases rapidly. At the same time, at 1494 cm^{-1} the intensity of band of KDP doped with NH_4Br (0.3 M) is very high.

At 1692 cm^{-1} the intensity of KDP doped with NH_4Br (0.3 M) band is very high compared to pure KDP.

At 1648 cm^{-1} intensity is high for KDP doped with NH_4Br (0.3 M). The intensity is lightly high for KDP doped with NH_4Br (0.1 M) shows a decrease in intensity of KDP doped with NH_4Br (0.2 M)

The intensity of band at 1717 cm^{-1} for the KDP doped with NH_4Br (0.1 M) is high the intensity of band at 1781 cm^{-1} KDP doped with NH_4Br (0.2 M) is very high compared to all the other bands.

The band of pure KDP at 3212 cm^{-1} shows a shift. At 3332 cm^{-1} the intensity of band corresponding to pure KDP is high. All the other three bands have comparatively low intensity.

At 3337 cm^{-1} the bands corresponding to all doped crystals have peaks but band corresponding to pure KDP does not have any peak.

At 641.8 cm^{-1} only the pure KDP crystal shows maximum intensity, but all the other doped crystals do not show any peaks.

At 915 cm^{-1} , all the four bands show the maximum intensity. The KDP doped with NH_4Br (0.1 M) shows a decrease in intensity than that of the pure KDP crystal. The band corresponds to KDP doped with NH_4Br (0.2 M) shows almost same intensity as that of pure KDP. The band corresponding to KDP doped with NH_4Br a (0.3 M) show is intensity high as that of pure KDP crystal.

Another Raman shift is occurs at 1218.4 cm^{-1} . The band corresponds to KDP doped with NH_4Cl (0.2 M) shows a shift at 1220 cm^{-1} , at the same time the Raman shift for KDP doped with NH_4Cl (0.3 M) is at 1222.2 cm^{-1} . But the bands correspond to KDP doped with NH_4Cl (0.1 M) does not show any shift.

At 1255 cm^{-1} , the band of KDP doped with NH_4Cl (0.2 M) shows a prominent peak, but in other samples this band is absent.

At 1266.6 cm^{-1} , only the pure KDP shows a prominent peak. At 1339.9 cm^{-1} the band of KDP doped with NH_4Cl (0.3 M) shows a prominent peak. But other three bands are absent at that point.

Another Raman shift is occurs at 1382.3 cm^{-1} . The band corresponds to KDP doped with NH_4Cl (0.1 M and 0.2 M) shows a shift at 1380.4 cm^{-1} . But the bands correspond to KDP doped with NH_4Cl (0.3 M) does not shows any shift.

There is a prominent peak at 1426.7 cm^{-1} , for pure KDP, but not in KDP doped with NH_4Cl (0.1 M). A weaker line is observed in KDP doped with NH_4Cl (0.2 M). At 1546.2 cm^{-1} , the band corresponds KDP doped with NH_4Cl (0.3 M) shows maximum intensity, and other bands are absent in that point.

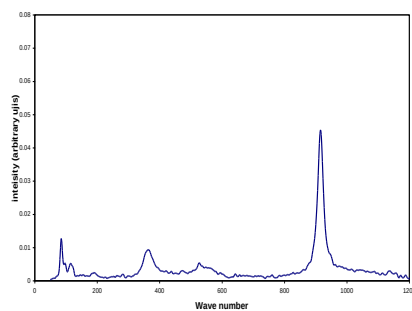
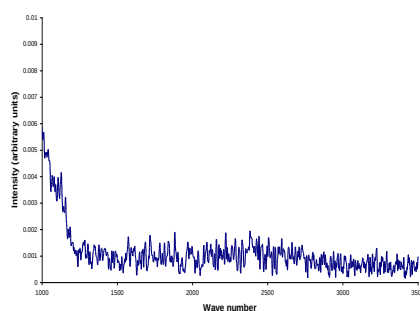
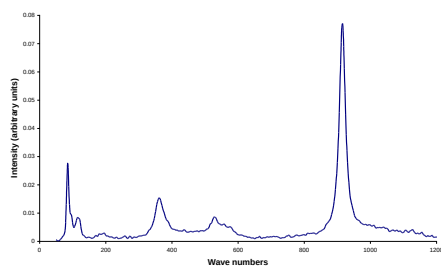
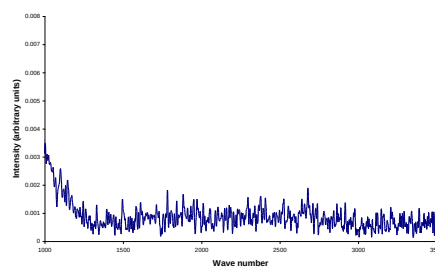
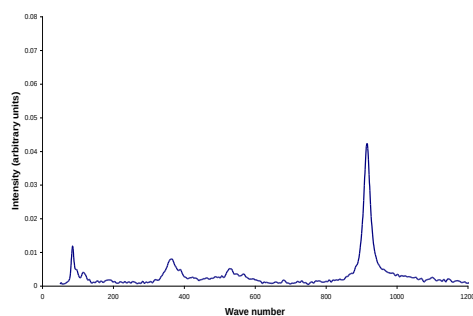
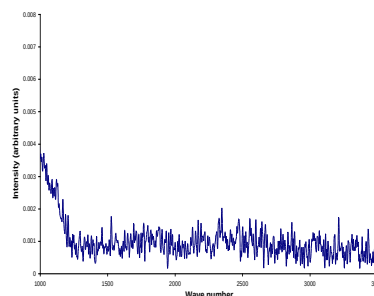
Except in the pure KDP, all the ammonium lines are observed. The assignments are shown in the Table.

CONCLUSIONS

The Raman spectra recorded are analysed. In the case of KDP doped with NH_4Br , the increase in intensity is seen for 0.1 M NH_4Br doped sample, but not as prominent as in the case of the 0.2 M, 0.3M NH_4Br doped sample.

In the case of Raman spectra of these samples, an increase in intensity of the Raman lines is observed in the case of KDP doped with 0.1 M NH_4Br . Such an increase is not observed in other cases. In fact doping with higher concentration of NH_4Br are reducing the intensities of Raman intensities. There is no considerable shift in the frequencies. A few shifts observed may be either chance of instrumental error.

It can be concluded that doping the KDP crystal with 0.1 M NH_4Br increases the bond strength. This is in agreement with the findings in [11].

**Fig. 1b Raman Spectrum of Pure KDP (0 to 1200 cm⁻¹)****Fig. 1a Raman Spectrum of pure KDP (1000 to 3500 cm⁻¹)****Fig. 2b Raman Spectrum of KDP + NH₄Br (0.1M) (0 to 1000 cm⁻¹)****Fig. 2a Raman Spectrum of KDP + NH₄Br (0.1M) (1000 to 3500 cm⁻¹)****Fig. 3b Raman Spectrum of KDP + NH₄Br (0.2M) (0 to 1000 cm⁻¹)****Fig. 3a Raman Spectrum of KDP + NH₄Br (0.2M) (1000 to 3500 cm⁻¹)**

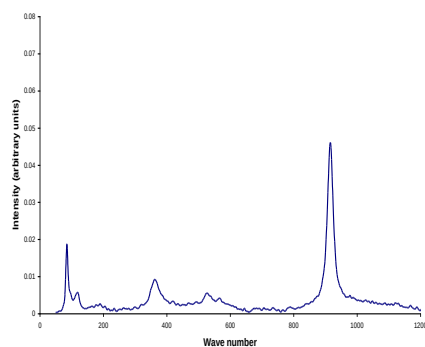
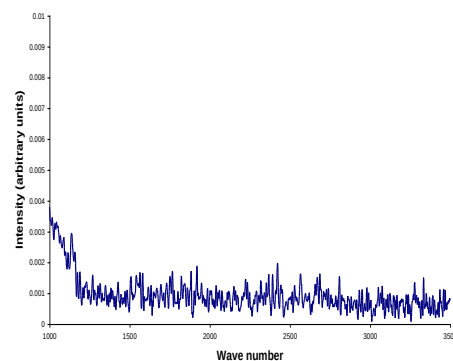
Fig.4b Raman Spectrum of KDP + NH₄Br (0.3M) (0 to 1000 cm⁻¹)Fig. 4a Raman Spectrum of KDP + NH₄Br (0.3M) (1000 to 3500 cm⁻¹)

Table 1 Observed Raman shifts and their assignments

Pure KDP	KDP+ NH ₄ Br 0.1 M	KDP+ NH ₄ Br 0.2 M	KDP+ NH ₄ Br 0.3M	Assignment
85 s 96 sh	84 s 94 sh	84 s	84 s	Lattice modes
113 m 123 sh	115 m	113 m	117 w	ν (O – H – O)
153 vw 136 vw 146 w 190 w 246 vw	169 w 194 w 231 vw	130 w 153 w 184 w(b) 217 vw	161 vw 188 vw 204 w 232 vw	External modes.
261 vw 279 vw 296 vw	258 w 271 vw 285 w	242 vw	261 w 296 w	ν (K ⁺ - O)
362 vs 437 w 472 w 507 sh 528 s 595 w	329 sh 360 vs 435 w 504 sh 528 s 555 w 572 w	317 w 362 vs 387 sh 421 w 462 w 506 w 526 s(b) 566 w	317 sh 362 vs 402 sh 420 w 433 w 468 w 493 w 526 s	δ PO ₂

		593 w	566 m	
641 w 655 vw 759 w 782 w 859 sh 915 vs 985 w	624 vw 664 w 751 m 798 w 811 w 828 w 915 vs	678 w 718 w 730 w 754 w 807 w 819 w 871sh 915 vs	616 w 643 w 682 w 705 w 717 vw 734 w 751 w 765 w 788 w 915 vs 977 w 987 w	$\nu_{\text{asy}}\text{POP}$
1014 w 1033 w 1087vw	1010 w 1068 w 1079 w 1089 vw	1002 w 1012 vw 1052 w 1068 w 1098 m	1027 w 1046 w 1069 vw 1087 w	$\nu^s\text{PO}_3$

Table1 Observed Raman shifts and their assignments (continued)

1133 s 1154 w 1172 w 1187 w 1187 w 1266 m	1106 m 1127 m 1154 w 1174 w 1185 w 1230 vw 1284 w	1131 w 1145 m 1172 w 1191 vw 1243 m 1260 w	1127 w 1168 w 1187 w 1206 vw 1218 vw 1295 w	$\nu^a\text{PO}_3$
1312 w 1345 w 1361vw 1370vw	1303 w 1316 w 1378 w	1330 m 1353 w 1393 m	1311 vw 1326 w 1341 vw 1378 w	$\nu_{\text{asy}}\text{PO}_2^{2-}$
1424 w 1561 m 1579 m	1445 vw 1464 w 1472 w 1519 w 1528 w 1555 w 1573 s	1407 w 1418 w 1438 w 1494 m 1532 w 1544 w 1567 w 1580 m 1596 w	1457 w 1486 w 1511 w 1528 vs 1542 vw 1559 m 1565 sh 1575 w 1590 w	$\nu_4\text{NH}_4$

1692 m	1615 m	2011 w	1602 w	$\nu_2\text{NH}_4$
1727 m	1636 w	2053 w	1615 w	
1748 m	1648 w	2088 w	1625 m	
1764 m	1685 m	2103 w	1648 m	
1881 m	1717 s	2126 vw	1671 m	
2221 s	1746 w	2146 w	1683 w	
2232 m	1783 m	2174 w	1692 s	
2365 s	1798 w	2203 vw	1702 w	
2389 s	1814 m	2252 vw	1748 m	
2421 s	1839 m	2294 m	1758 m	
2527 m	1881 s	2308 w	1766 s	
2564 m	2038 w	2338 w	1796 s	
2668 m	2074 w	2375 m	1806 w	
2684 m	2095 m	2392 m	1820 m	
	2120 m	2406 m	1862 m	
	2146 vw	2433 vw	2030 w	
	2159 m	2497 w	2039 vw	
	2174 w	2524 m	2117 w	
	2192 vw	2551 w	2128 w	
	2207 w	2562 w	2151 w	
	2221 s	2622 w	2171 m	
	2230 w	2637 m	2192 m	
	2240 vw	2678 s	2242 w	
	2261 w	2709 m	2281 vw	
	2284 m	2726 w	2308 w	
	2308 w	2738 m	2327 m	
	2329 m	2778 w	2346 s	
	2383 s		2369 vw	
	2425 m		2410 w	
	2443 m		2471 m	
	2483 vw		2500 m	
	2506 m		2512 w	
	2539 w		2552 m	
	2551 w		2578 w	
	2593 m		2599 m	
	2660 m		2633 w	
	2709 w		2643 m	
			2668 m	
			2728 w	
			2784 w	
	2848 m	2809 w	2813 m	$\nu_1\text{NH}_4$
	2857 m	2813 vw	2828 w	
	2869 w	2826 w	2836 s	
	2873 m	2842 m	2865 vs	
	2882 m	2861 s	2875 w	
	2900 w	2871 w	2884 m	
	2919 m	2878 w	2894 vw	
	2938 s	2886 m	2900 w	
	2948 w	2905 vw	2907 vw	
	2955 w	2915 vs	2923 m	
	2967 m	2928 w	2936 w	
	2984 m	2950 w	2948 w	
		2969 w	2969 m	
		2977 m		
		2986 vw		

3052 m	3019 w	3013 vw	u_3NH_4
3062 w	3033 vw	3031 w	
3071 w	3058 m	3038 w	
3083 w	3067 w	3069 m	
3098 w	3089 w	3075 w	
3131 m	3112 s	3081 sh	
3143 w	3119 s(b)	3090 vw	
3166 s	3144 s	3100 m	
3181 m	3162 m	3116 s	
3197 m	3183 w	3123 w	
3204 w	3206 vs	3133 m	
3224 s	3214 m	3152 w	
3245 w	3225 w	3168 vw	
3291 m	3264 w	3183 m	
3306 w	3272 w	3195 w	
3360 m	3291 s	3212 s	
3378 w	3303 vw	3270 w	
3405 m	3324 m	3310 m	
3436 m	3337 s	3347 m	
3455 w	3386 s	3386 s	
	3468 s	3416 m	
	3480 s	3432 vs	
		3467 w	

w: weak, vw: very weak, m: medium, s: strong, vs: very strong, b: broad, sh: shouler

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