iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 4 **Issue:** IX **Month of publication:** September 2016

DOI:

www.ijraset.com

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Volume 4 Issue IX, September 2016
ISSN: 2321-9653
International Journal for Research in Applied Science & Engineering Technology (IJRASET)

Optical and Mechanical Properties of KDP - ADP Mixed Crystals

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Abstract-KDP and ADP single and mixed crystals have been grown by slow evaporation solution technique. The solubility of data KDP and ADP were determined and structures, optical, mechanicals properties of the crystals have been investigated. The X-rays diffraction study shows that the crystalline perfection of are doped crystal is good. Fourier transform infrared studies confirm the functional groups of the crystals UV-vis studies shows that the optical transparency is increased much by the mixed crystals.

Keywords- FTIR, UV, XRD, KDP, ADP.

I. INTRODUCTION

Potassium di hydrogen phosphate (KDP) is a model system for non-linear optical device application. KDP is an efficient tuned crystals on di electric medium for optical harmonic generation in and near the visible region. This material offers high transmission throughout the visible spectrum and meets the requirement for optical birefringence, large enough to bracket its refractive index for even extreme wavelength range over which it is transparent. Among non-linear optical phenomena, frequency mixing and electro-optic are important in the field of optical image storage and optical communication [1-3].Ammonium di hydrogen phosphate (ADP), a hydrogen bonded compound, belongs to amorphous series of phosphates and arsenates that present a strong piezoelectric activity. These molecular crystals exhibit low temperature order- disorder phase transitions. ADP and KDP are non-linear optical materials that have been used as optical modulation, Q-switch, quantum electronics and frequency converters particularly, optical crystals with lower impurity and higher damage threshold are required for inertial confinement fusion [4,5]. In the past few decades ADP and KDP crystals have attracted much attention because of their excellent properties for ultraviolet (UV) non-linear optics (NLO).

II. EXPERIMENTAL PROCEDURE

A supersaturated solution of KDP and ADP powders were prepared in distilled deionized water. The amount of KDP and ADP salt to be separately dissolved is determined from its solubility and was at an average temperature of 35^{O}C. The solution was stirred long enough to ensure complete dissolution of the solute, and filtered using what man 100 filter paper to remove residual microscopic particles. Pure KDP and ADP crystals were grown from aqueous solution by slow evaporation method. The crystallization took place within 15-25 days and high quality transparent crystals were harvested form the aqueous growth medium. The same method was followed for mixed crystals of KDP - ADP. Saturated solutions of different proportions are 0.1, 0.25, 0.33, 0.5, 0.66, 0.75 and 0.9 of KDP, ADP (O.1 i.e. 1g of ADP, 9g of KDP) as shown in Table.1. The solubility of KDP and ADP in the solvents was determined. The solubility of KDP was 25.2g/100ml at 35^{O}C and ADP was 31.2g/100ml at 35^{O}C.

Table1. Nucleation Kinetics

Solution 1	KDP	Dissolved in 25ml of distilled water
Solution 2	ADP	Dissolved in 28ml of distilled water
Solution 3	(0.1 KAMC) 1g of ADP,9g of KDP	Dissolved in 27ml of distilled water
Solution 4	(0.25 KAMC) 2.5g of ADP, 7.5g of KDP	Dissolved in 29ml of distilled water
Solution 5	(0.33 KAMC) 5g of ADP,10g KDP	Dissolved in 30ml of distilled water
Solution 6	(0.5 KAMC) 5g each of ADP, KDP	Dissolved in 35ml of distilled water
Solution 7	(0.66 KAEMC) 7.5g of ADP,2.5g of KDP	Dissolved in 50ml of distilled water
Solution 8	(0.75 KAMC) 1g of ADP,, 9g of KDP	Dissolved in 55ml of distilled water.
Solution 9	(0.9 KAMC) 9g of ADP, 1g of KDP	Dissolved in 58ml of distilled water.

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Volume 4 Issue IX, September 2016
ISSN: 2321-9653
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III.CHARACTERIZATION STUDIES OF PURE AND MIXED CRYSTALS

Numbers of methods are available for the characterization of chemical compounds. The choice of the technique depends upon the requirement, state of the sample, interest of the investigator, accuracy of the results and finally, the availability of the instrument, application oriented and also nature of the crystal. The important techniques which have been used in this study and their underlying principles are discussed in the following paragraphs. The grown crystals of KDP-ADP mixed crystals with different proportions are shown in fig.1.

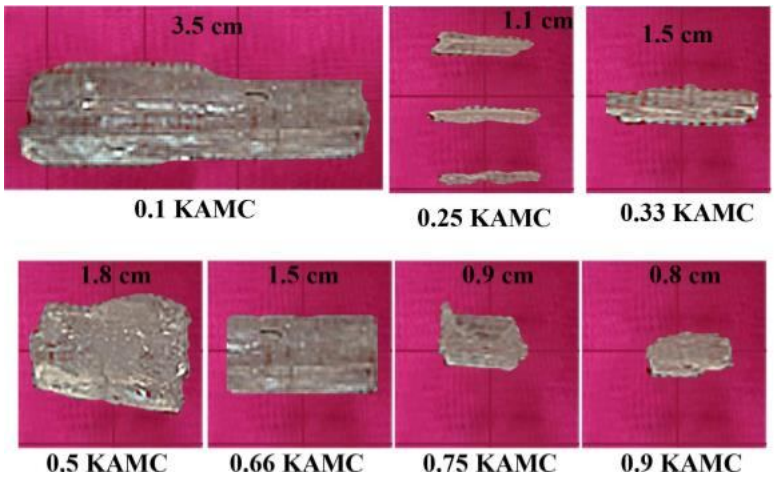
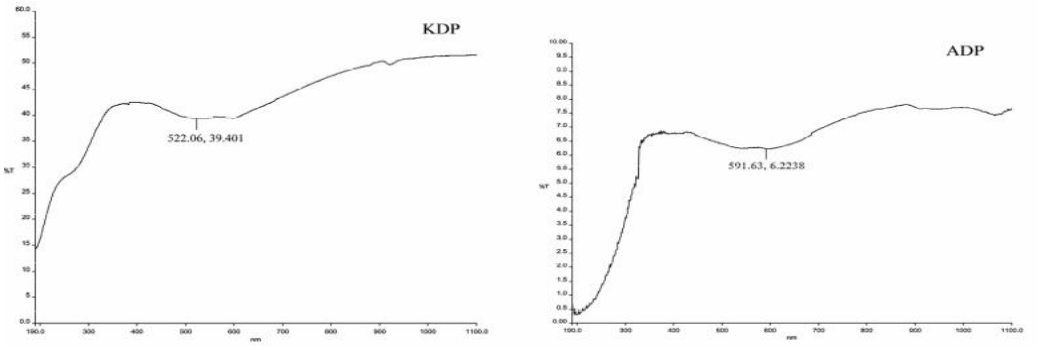


Fig.1. KDP-ADP mixed crystals

A. UV-Visible Spectra Of KDP-ADP Mixed Crystals

Optical transmission spectrum has been recorded for pure and mixed crystals in the range between 200 and 1100 nanometer. Crystal plates of pure and mixed KDP and ADP were cut polished at face (100) without any coating for optical measurements. In both the pure and mixed crystals, the dimensions of the used crystals are 10x10x5 mm^3. It is observed from the Fig 2 that the pure and different molar ratios of mixed crystals are more than 80% of transmittance. In order to confirm the reproducibility, several times the beam was passed through the various regions of the crystals and the same results were observed. The ADP has 65% transmittance [4, 6, 7]. The large transmission in the entire visible region enables it to be a good candidate for electro - optic and NLO applications [5, 8]. ADP and KDP crystals grown from declaration show more than 80% of transparency in the entire visible region [9,10]. The grown mixed crystal by the slow evaporation method also shows approximately the same transparency and indicates that the crystals have higher crystalline perfection and is suitable for device fabrications.



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Volume 4 Issue IX, September 2016
ISSN: 2321-9653
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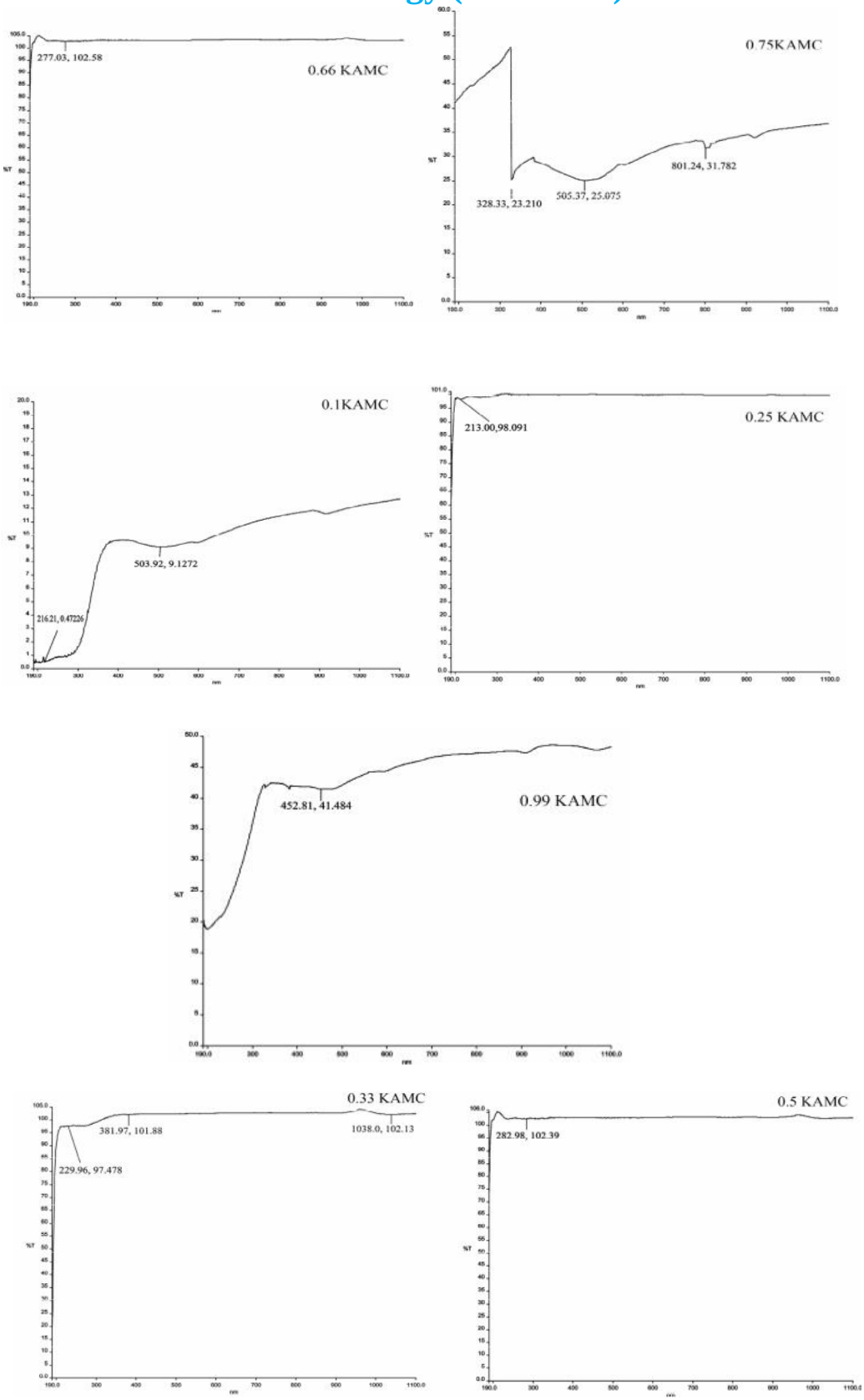

Fig 2. UV-visible spectra of KDP- ADP mixed crystals

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Volume 4 Issue IX, September 2016
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The above result indicates that the addition of KDP increased the transmittance for pure KDP and ADP the cut off frequencies also lies very much closure to the visible frequency region as shows in the Table 2. But increasing the concentration of KDP shifts the cut to a lower frequency region. The addition of KDP increases the cut off transparency also in the UV region.

Table.2 Calculated cutoff frequency for mixed crystals of KDP and ADP

System	Cut off wave length in nano meter	Percentage of transmittance
KH2PO4	340	41
N H4H2PO4	360	6.7
0.1 KAMC	390	9.5
0.25 KAMC	220	98
0.33 KAMC	210	97
0.5 KAMC	210	100
0.66 KAMC	200	100
0.75 KAMC	320	53
0.9 KAMC	340	42

B. Energy Band Gap

The optical band gap of mixed KDP and ADP crystals were determined from the UV-Vis spectra. The relation between the optical absorption and coefficient α was determined from the transmittance T using the relation $\alpha=2.303/t \times \log(1/T)$ where t is the sample thickness and T is the transmittance. To study the band gap (Eg) nature and to estimate the value of the energy gap Eg, the following equation was used $\alpha h\gamma = A(h\gamma - Eg)n$ where n is an index found by a nature of the electron transition during the absorption and A is a constant nearly independent of photon energy known as the disorder parameter [11]. Optical band gap was determined to be 2.1ev, 1.6 ev etc., for pure and mixed crystals of KDP and ADP respectively. It is shown in the Table 3. The photon energy for mixed crystals is given in Figure 3.

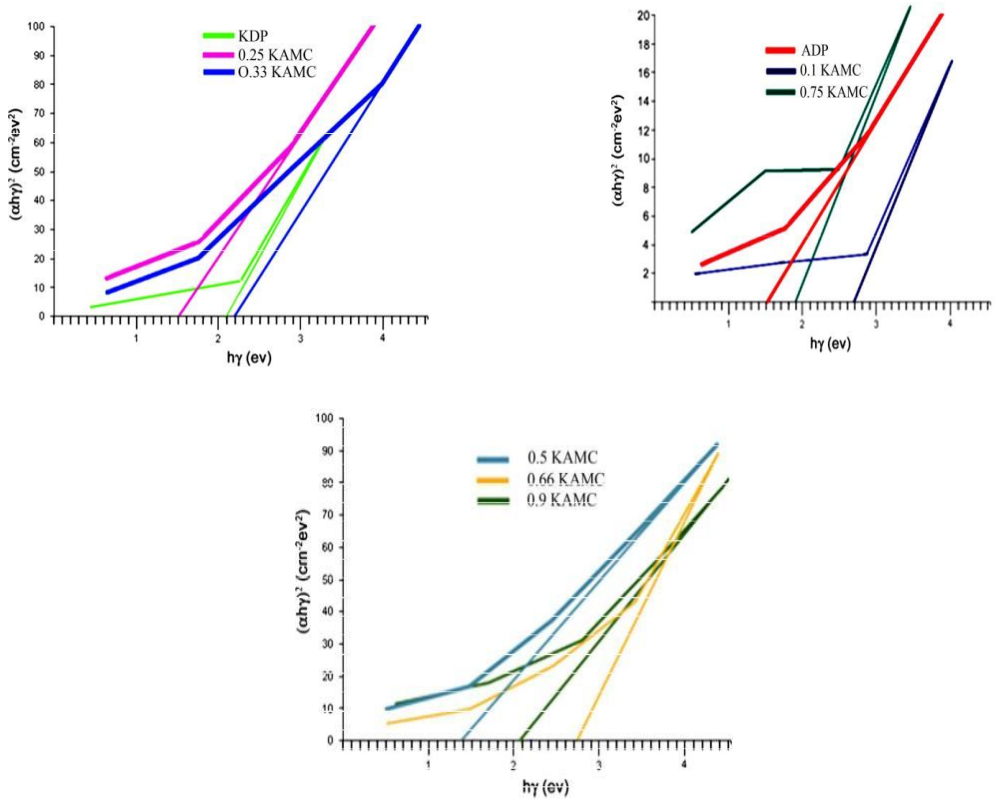

Fig. 3 Band gap measurement of KDP, ADP, KAMC

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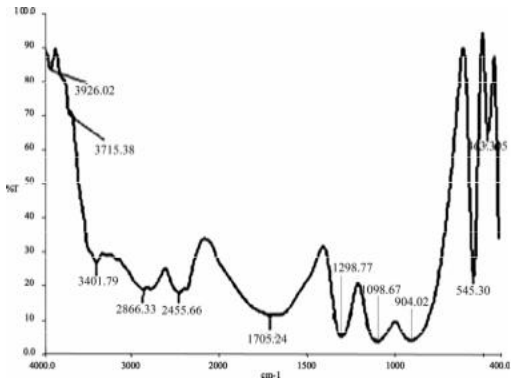
Volume 4 Issue IX, September 2016
ISSN: 2321-9653
International Journal for Research in Applied Science & Engineering Technology (IJRASET)

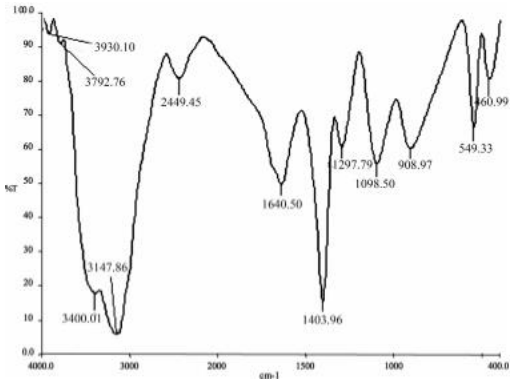
Table. 3 Calculated optical energy gap Eg for KDP – ADP mixed crystal

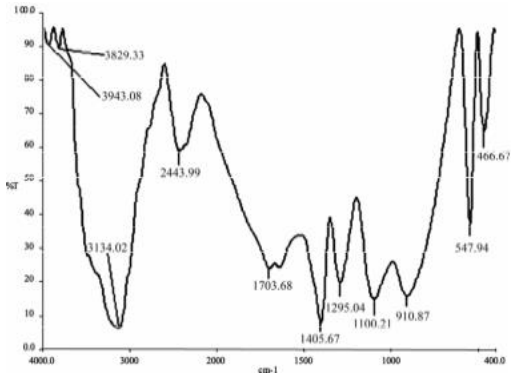
System	Eg (ev)
KH2PO	2.1
N H4H2PO4	1.6
0.1 KAMC	2.7
0.25 KAMC	1.5
0.33 KAMC	2.2
0.5 KAMC	1.4
0.66 KAMC	2.75
0.75 KAMC	1.9
0.9 KAMC	2.1

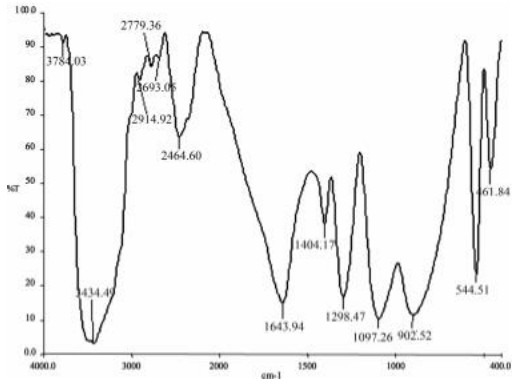
C. FTIR Spectral Studies Of KDP-ADP Mixed Crystals

The vibrational frequencies of O-H appear in the range of 3731-3836 cm-1. There is a broad strong absorption between 1400 and 1410 cm-1 resulting from hydrogen bonded N-H stretching bonds. Absorption in this region is characterized by fine structure on the lower wave number side of the band. The bands which appeared at 3715 cm-1 in pure KDP, 3792 cm-1 in ADP, in the various respective molar ratios of KDP and ADP were assigned to free O-H stretching. The broad absorption band appeared at 3401, 3400, 3429, 3434, 3268, 3268, 3134, 3142, 3424 and 3240 cm-1 were assigned to hydrogen bonded O-H stretching frequencies in pure KDP, ADP and 0.1, 0.25, 0.33, 0.5, 0.66, 0.75 & 0.9 proportions of KAMC respectively. It is shown in Figure 4.









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Volume 4 Issue IX, September 2016
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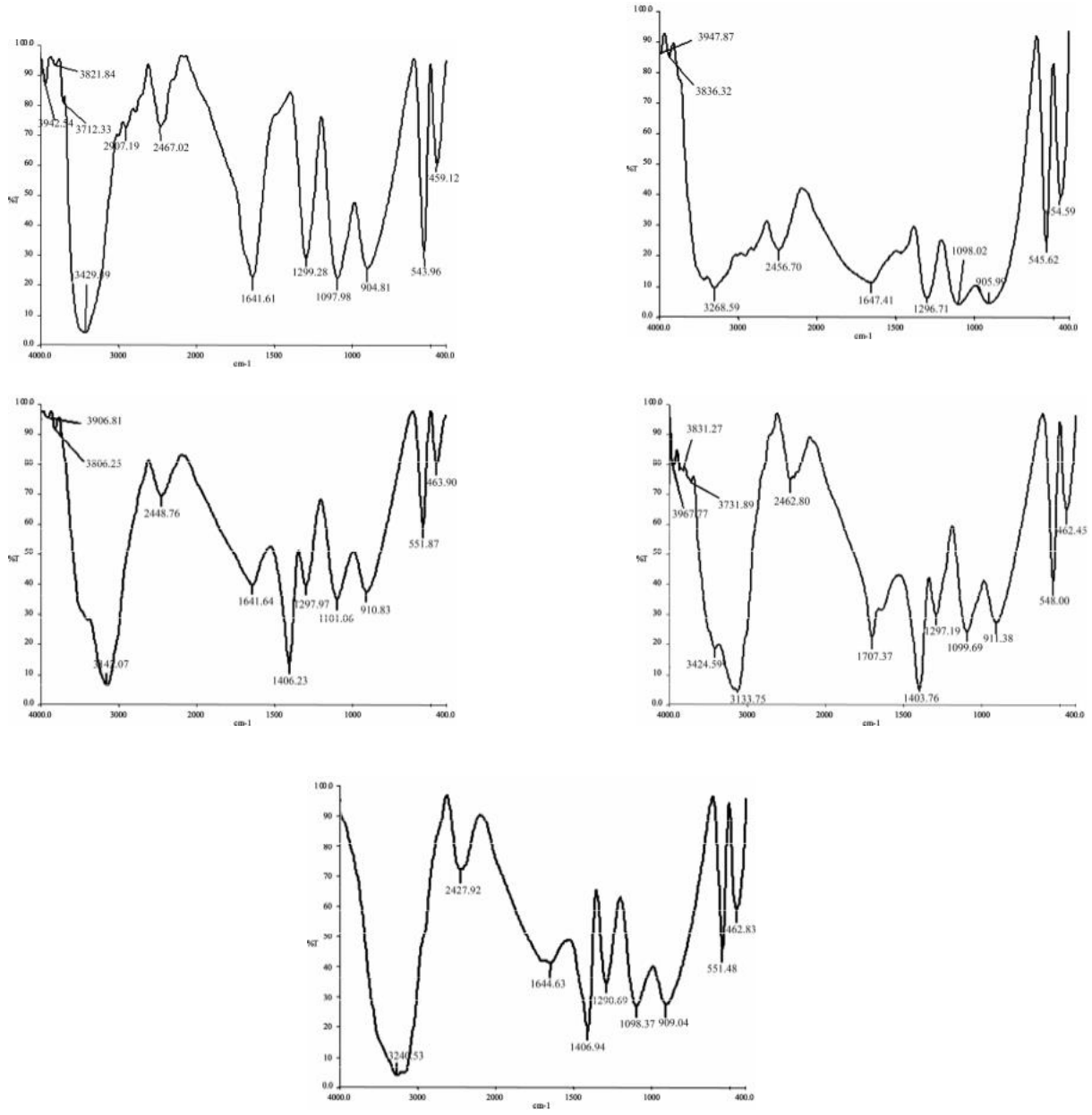

Fig. 4 FT-IR Spectrum of KDP, ADP, KAMC

The observed frequency at 3333 cm-1 of O-H stretching was in close agreement with the experimentally obtained pure KDP and ADP. Its deviation from pure KDP to lower frequency at 0.33, 0.5, 0.66, 0.75 and 0.9 indicate clearly the interactions of dopants with P -O - H group of KDP and in weakening the strength of the bond between oxygen and hydrogen (Table 4). This leads to the decrease in the frequency of O - H stretching and confirmed the Hydrogen bonding changes of pure and mixed KDP and ADP crystals at these sites in the crystal lattice. This property is also reflected in the P=O, P-O, P-OH stretching and HO-P-O-H bending vibrations. The present FT-IR study on pure and mixed KDP clearly indicates the effect of dopants on the crystal structure of pure KDP, which leads to change in the absorption of FTIR frequencies and the non-linear optical property of both the crystals. The study also confirmed that the dopants had entered the lattice sites of tetragonal KDP. ADP mixed with KDP crystals which also change optical properties.

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Volume 4 Issue IX, September 2016
ISSN: 2321-9653
International Journal for Research in Applied Science & Engineering Technology (IJRASET)

Table: 4 Observed FT-IR frequencies of pure and mixed crystals of KDP and ADP (cm-1)

KDP	ADP	0.1 KDMC	0.25 KDMC	0.33 KDMC	0.5 KDMC	0.66 KDMC	0.75 KDMC	0.9 KDMC	Assignment
3715	3793	3712	3784	3836	3829	3906	3731	-	Free O-H Stretching
3401	3400	3429	3434	3268	3134	3424	3240	-	N-H Stretching
2866	-	2904	2914	-	-	-	-	-	P – O – H symmetric stretching
2455	2449	2467	2464	2443	2443	2448	2462	2427	O ‖ P – OH Stretching
1705	1640	1641	1643	1647	1703	1641	1701	1644	O ‖ P – OH stretching
-	1403	-	1404	-	1405	1406	-	1406	P = O Stretching
1298	1297	1299	1298	1296	1295	1297	1297	1290	N = H bending of the dopant
1098	1098	1197	1097	1098	1100	1101	1099	1098	P = O Stretching
904	908	904	902	905	910	910	911	909	P – O – H Stretching
545	549	543	543	545	547	551	548	551	C – H out of plan bending
463	460	459	459	454	466	463	462	462	C – C out of plan bending

D. Single Crystal XRD Of KDP – ADP Mixed Crystal

The crystals composition of mixed crystal grown from equimolar mixture is determined as ADP and KDP by single crystal XRD analysis. It crystallizes in monoclinic system with space group P21/C. Slight changes are observed in the interatomic distances of the pure and mixed crystals can be calculated. The cell parameter values of all the mixed crystals agree well with the values calculated by Vegard's relation. Table.5 gives the values a=xa1+ (1-x)a2, b=xb1+(1-x)b2, c=xc1+(1-x)c2-- - - (5.1) a1, b1, and c1 are the lattice parameters of parent (NH4H2PO4) and a2, b2, and c2 are the lattice parameters of parent (KH2PO4). a, b, and c are the lattice parameters of KDP - ADP mixed crystals.

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Volume 4 Issue IX, September 2016
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Table.5 Cell parameter determined from XRD studies and derived from Vegard's relations.

system	Determination from single crystal XRD				Calculated using Vegard's relations		
	a(Aº)	b(Aº)	c(Aº)	V(Aº)	a(Aº)	b(Aº)	c(Aº)
KH_2PO_4	7.452	7.452	6.974	387.42	7.452	7.452	6.974
N $H_4H_2PO_4$	7.5024	7.5024	7.566	4.28.06	7.5024	7.5024	7.566
0.1 KAMC	7.451	7.45	6.978	387.5	7.451	7.451	7.0332
0.25 KAMC	7.42	7.42	6.973	383.6	7.4646	7.4646	7.122
0.33 KAMC	7.452	7.45	7.019	389.6	7.4686	7.4686	7.1693
0.55 KAMC	7.56	7.56	7.34	419.6	7.4772	7.4772	7.27
0.66 KAMC	7.463	7.46	7.305	406.9	7.4857	7.4857	7.3707
0.75 KAMC	7.44	7.44	7.31	404.6	7.4898	7.4898	7.418
0.9 KAMC	7.450	7.45	7.47	414.5	7.4973	7.4973	7.5068

E. Powder X-Ray Analysis Of KDP-ADP Mixed Crystals

The powdered pattern XRD Spectrum of KDP-ADP Mixed Crystal has been indexed using CHEKCELL software in Table 6 to14. The cell parameters are a= 7.452 Aº, b=7.452 Aº, c= 6.974 Aº, and α=90.00°, β = 90, γ = 90. The system belongs to TETRAGONAL. The CU K λ = 1.54060. The lowest experiment 2θ value and the highest experimental value respectively say. $2\theta_1$=17.5823 and $2\theta_2$=74.5665 have been taken to calculated the hkl values. The 2θ position and the d-spacing are also calculated. The observed prominent peaks of pure and mixed crystals of KDP and ADP are (020), (010), (121), (502), (113), (132), (110), (121), and (312). It was found that peak intensity at (200) plane increased by a large amount along with slight change in intensity of some other peaks.

Table.6 Calculated hkl values, 2θ and d spacing values for KDP

hkl value	2θ cal	2θ exp	d cal	d exp
011	17.4699	17.306	5.07229	5.1201
111	21.6527	21.028	4.10098	4.2214
020	23.7637	23.836	3.74123	3.7300
121	29.7397	29.607	3.00166	3.0148
112	30.6897	30.506	2.91091	2.9280
022	35.1845	35.023	2.54863	2.5600
301	38.5161	38.359	2.33549	2.2385
231	40.4295	40.287	2.22926	2.2385
231	45.7192	45.666	1.98288	1.9851
132	46.4021	46.292	1.95528	1.9597
040	55.1158	55.734	1.66499	1.6480
024	57.9737	57.887	1.58953	1.5917
332	58.8299	58.642	1.56842	1.5730
323	59.7103	59.517	1.54738	1.5519
501	63.6664	63.708	1.46043	1.4596
152	69.6336	69.525	1.34914	1.3510
350	74.1092	74.039	1.27834	1.2794
060	76.6223	76.566	1.24255	1.2433

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ISSN: 2321-9653
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Table.7 Calculated hkl values, 2θ and d spacing values for ADP

hkl value	2θ cal	2θ exp	d cal	d exp
010	16.8716	16.698	5.25081	5.3273
020	23.6601	23.700	3.75739	3.7512
121	29.1757	29.091	3.05837	3.0671
212	33.7760	33.742	2.65162	2.5102
231	45.1926	45.156	2.00476	2.0063
330	51.6173	51.648	1.76831	1.7683
240	54.8220	54.648	1.67322	1.7683
233	57.6446	57.362	1.59782	1.6776
242	60.2050	60.304	1.53584	1.5336

Table 8 Calculated hkl values, 2θ and d spacing values for 0.1 KAMC

hkl value	2θ cal	2θ exp	d cal	d exp
502	23.9460	23.901	3.71316	3.7200
112	30.7799	30.750	2.90255	2.9053
031	38.3678	30.499	2.90255	2.9053
013	40.6495	40.649	2.21770	2.3365
231	45.7900	45.824	1.97998	1.9786
132	46.4497	46.536	1.95340	1.9500
040	48.9140	48.930	1.86058	1.8600
332	58.3701	58.910	1.57967	1.5665
243	69.7329	69.429	1.34762	1.3526
343	76.6957	75.867	1.24155	1.2530

Table 9 Calculated hkl values, 2θ and d spacing values for 0.25 KAMC

hkl value	2θ cal	2θ exp	d cal	d exp
101	29.1026	29.747	2.98919	3.0010
112	30.8931	30.700	2.89217	2.9099
220	34.2022	34.104	2.61955	6.6269
202	35.3365	35.228	2.53803	2.5456
301	38.5958	38.534	2.33085	2.3345
013	40.6751	40.544	2.21637	2.2232
023	45.9159	45.939	1.97485	1.9729
132	46.5742	46.538	1.94845	1.9499
114	55.2454	55.362	1.66139	1.6582
332	58.9619	58.942	1.56523	1.1565
224	63.8940	63.827	1.45577	1.4556
152	69.8101	69.883	1.34616	1.3449
252	74.3409	73.773	1.27493	1.2833

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Table.10 Calculated hkl values, 2θ and d spacing values for 0.33 KAMC

hkl value	2θ cal	2θ exp	d cal	d exp
011	17.4699	17.306	5.07229	5.1201
111	21.6527	21.028	4.10098	4.2214
020	23.7637	23.836	3.74123	3.7300
121	29.7397	29.607	3.00166	3.0148
112	30.6897	30.506	2.91091	2.9280
022	35.1845	35.023	2.54863	2.5600
301	38.5161	38.359	2.33549	2.2385
231	40.4295	40.287	2.22926	2.2385
231	45.7192	45.666	1.98288	1.9851
132	46.4021	46.292	1.95528	1.9597
040	55.1158	55.734	1.66499	1.6480
024	57.9737	57.887	1.58953	1.5917
332	58.8299	58.642	1.56842	1.5730
323	59.7103	59.517	1.54738	1.5519
501	63.6664	63.708	1.46043	1.4596
152	69.6336	69.525	1.34914	1.3510
350	74.1092	74.039	1.27834	1.2794
060	76.6223	76.566	1.24255	1.2433

Table.11 Calculated hkl values, 2θ and d spacing values for 0.5 KAMC

hkl value	2θ cal	2θ exp	d cal	d exp
101	17.1203	17.305	5.17509	5.1203
111	21.5459	21.101	4.12107	4.2069
121	29.4495	29.821	3.03057	2.9936
220	34.4507	34.342	2.60122	2.6092
130	38.0680	38.546	2.36195	2.3338
203	45.7669	45.447	1.98093	1.9941
331	54.8905	54.245	1.67129	1.6896
042	56.2183	56.119	1.63492	1.6376
050	62.0237	62.917	1.49510	1.4760
224	63.3812	63.251	1.46631	1.4690
052	68.9686	68.815	1.36052	1.3632
225	76.2997	76.184	1.24700	1.2486

Table .12 Calculated hkl values, 2θ and d spacing values for 0.66 KAMC

hkl value	2θ cal	2θ exp	d cal	d exp
101	17.1203	17.305	5.17509	5.1203
111	21.5459	21.101	4.12107	4.2069
121	29.4495	29.821	3.03057	2.9936
220	34.4507	34.342	2.60122	2.6092
130	38.0680	38.546	2.36195	2.3338
203	45.7669	45.447	1.98093	1.9941
331	54.8905	54.245	1.67129	1.6896
042	56.2183	56.119	1.63492	1.6376
050	62.0237	62.917	1.49510	1.4760
224	63.3812	63.251	1.46631	1.4690

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052	68.9686	68.815	1.36052	1.3632
225	76.2997	76.184	1.24700	1.2486

Table.13 Calculated hkl values, 2θ and d spacing values for 0.75 KAMC

hkl value	2θ cal	2θ exp	d cal	d exp
110	16.9569	16.885	5.22458	5.2467
020	23.9137	23.967	3.71811	3.7100
121	29.4438	29.532	3.03115	3.0223
130	38.1104	38.330	2.35941	2.3464
231	45.4707	45.762	1.99314	1.9811
223	51.9716	51.080	1.75808	1.7866
133	54.8607	54.188	1.67213	1.6913
124	57.9625	57.462	1.58981	1.6025
034	63.3536	63.151	1.46688	1.4711
052	68.6869	68.128	1.36541	1.3753
440	71.3321	71.926	1.32113	1.3117
513	76.6114	76.099	1.24270	1.2498

Table.14 Calculated hkl values, 2θ and d spacing values for 0.9 KAMC

hkl value	2θ cal	2θ exp	d cal	d exp
011	16.9000	16.626	5.24204	5.3278
020	23.9105	23.740	3.71860	3.7450
121	29.3287	29.123	3.04278	3.0638
022	33.9386	33.616	2.63928	2.6639
312	45.4319	45.102	1.99475	2.0086
240	51.8113	51.703	1.76314	1.6748
024	54.8975	54.203	1.67109	1.6909
233	57.7585	57.377	1.59494	1.6046
242	60.5360	60.376	1.52823	1.5319
431	63.3764	63.224	1.46641	1.4696
521	68.6547	68.446	1.36597	1.3696
440	71.0857	71.151	1.32510	1.3241
334	73.7804	73.216	1.28383	1.2917
442	76.1633	76.086	1.24840	1.2500
253	78.6512	78.425	1.21551	1.2164

IV. CONCLUSION

Good quality single and mixed crystals ADP and KDP were grown and characterized using FT – IR , UV – Vis , TGA – DTA, Powder XRD and single crystal XRD studies. The functional groups present in the grown crystals have been confirmed by FT-IR spectral analysis. Frequency increases considerably from 3712 cm-1 to 3836 cm-1 due to the mixed crystals formation, which denotes the vibrational energy increases as per the formula E = hv. Hence, the frequency modulation denotes the semiconducting property which also can be modulated by the mixed crystals formulation as per our necessity. Lattice parameters of the mixed crystals agree well with the values calculated using Vegard's law. The UV – Visible spectrum reveals that the dopant enhanced the transparency.

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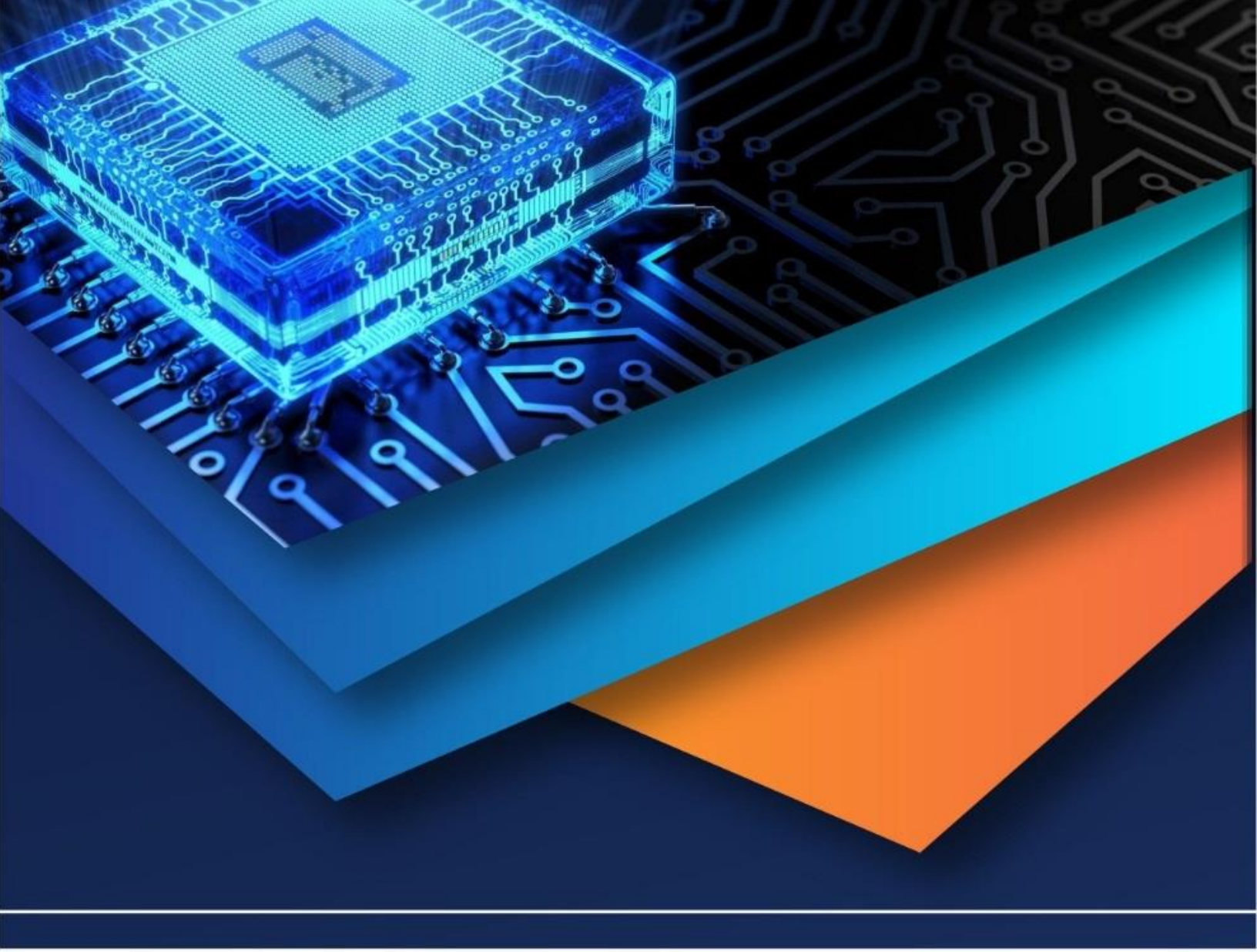

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