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Growth, structural, functional and thermal properties of L-asparagine doped ammonium pentaborate crystals

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***Abstract**: Ammonium pentaborate (APB) and different molar percentage L-asparagine doped APB have been synthesized by constant temperature and evaporation technique. The structure symmetry of the grown crystals have confirmed by power XRD, which is orthorhombic. The crystals are characterised by FTIR spectroscopy. The APB and L-asparagine doped APB crystals have been subjected to thermal studies. By TGA and DTA studies investigated the decomposition of APB and doped APB crystals. To get the information of successive doping of dopant., EDAX analysis was done.*

***Keywords:** Ammonium Pentaborate, Doping, Optical Property, Complex Impedance Study, Second Harmonic Generation.*

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I. Introduction:

Generally the boron atom is coordinates with three or five oxygen atoms forming $[BO_3]^{3-}$ or $[BO_4]^{5-}$ groups. Accordingly, the electronic orbital of boron atoms are hybridized forming SP^2 structures with trigonal symmetry or SP^3 structure exhibiting tetrahedral symmetry. Borate anions can be found in many structural types, allowing one to choose an appropriate material for specific applications [1]. Because of comparatively good transparency and high resistance in case of laser induced damage in the UV region, NLO crystals with B-O bonds are very useful for UV light generation with high power.

Ammonium penta borate is a biaxial crystal of the orthorhombic system. The properties of pure ABP like piezoelectric crystallographic and elastic have been reported [2]. The qualitative study have made on α and β phase in ammonium penta borte [3]. APB has twelve stress and twelve strain optical constants [4]. The large difference in the electro-negativity of boron and oxygen is believed to be responsible for transmission of shorter wavelength light. The conjugated π orbital and high anisotropic distribution of electrons in the planar borate triangles is useful for large second order susceptibility and birefringence. A detailed review is given recently by Yang et al [5]. Pentaborotes crystals are known for their polar symmetry structure, which is known as the family of hydrated pentaborates. The APB crystal forms the twin crystal morphology and it consists of double ring structure, which is the main structural unit of pentaborate group [6].

Some of the amino acid doped KDP crystals are reported [7-9]. A few works are reported based on doping in APB crystals such as amino acids[10,11] and nanoparticles like ZnS nano-particles[12].

The novelty of the present communication are the UV-Visible transmittance, the refractive index dispersion below the energy bandgap, energy band gap, thermal decomposition intermediates, the complex impedance, and modulus spectroscopy, etc.

II. Materials And Methods:

Pure ammonium pentaborate and different molar percentage L-asparagine doped APB are grown by constant temperature and evaporation technique with constant temperature. The present authors have doped L-asparagine with different molar percentages (1M%, 3M%, and 5M%) in the APB. The said crystals are given in figure 1. The crystal have undefined morphology. The description of such a method is given elsewhere [12].

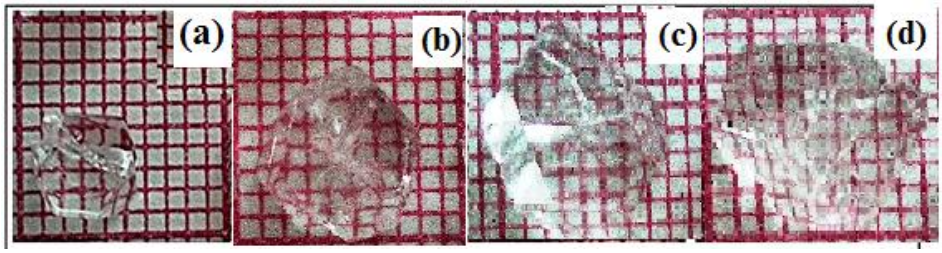

Figure 1:(a) Pure APB, (b) 1M% L-asparagine doped APB, (c) 3M% L-asparagine doped APB and (d) 5M% L-asparagine doped APB crystals

III. Result and Discussion:

3.1 Powder XRD:

The PXRD of APB crystal indicated that it has orthorhombic crystal system and the space group was Aba_2. In the table 1 the unit cell parameters of APB and L-asparagine doped APB crystals are mentioned. The figure 2 is the PXRD pattern which indicates, the present work was given the confirmation with reported work. [11,12]. The lattice strain calculated by WH formula . From the figure 3 one can observe slight shifting of (1 2 2) which indicates the presence of APB on account of L-asparagine doping. Such behaviour is attributed to the dearth experienced by lattice of APB [13-15].

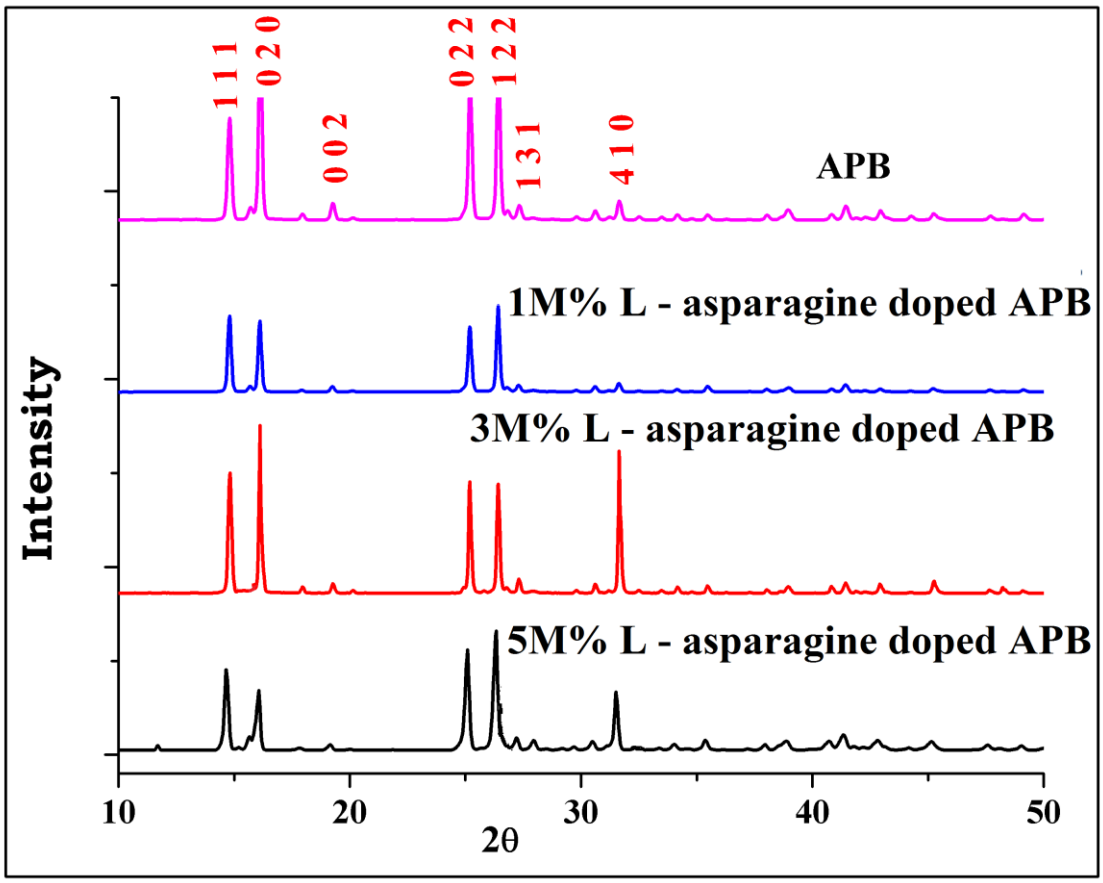

Figure. 2: PXRD patterns of pure and L-asparagine doped APB crystals.

Samples	a(Å)	b(Å)	c(Å)	Unit cell volume(Å^3)	Lattice strain x10^{-4}
APB	11.65	11.01	9.20	1180.05	0.15
1M% L-asparagine doped APB	11.65	10.92	9.18	1167.86	0.35
3M% L-asparagine doped APB	11.64	11.01	9.15	1172.63	6.88
5M% L-asparagine doped APB	11.652	11.02	9.22	1183.89	7.98

Table 1: XRD unit cell parameters pure and L-asparagine doped APB crystals.

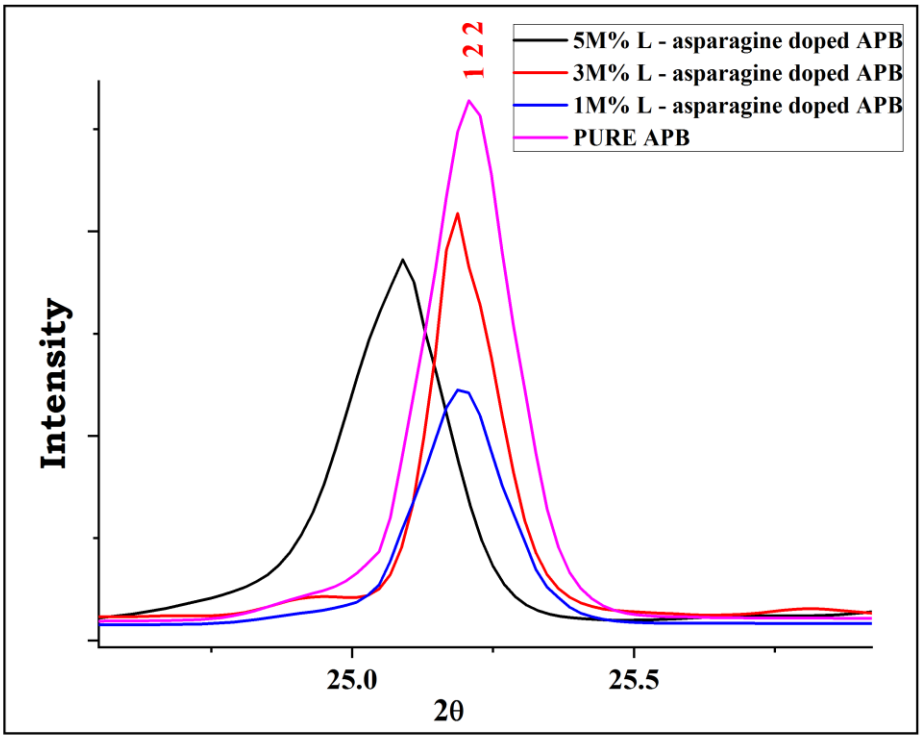

Figure. 3: Shifting of (122) reflex in APB crystals grown from the solutions with different L-asparagine concentrations.

3.2 FT-IR Spectroscopy:

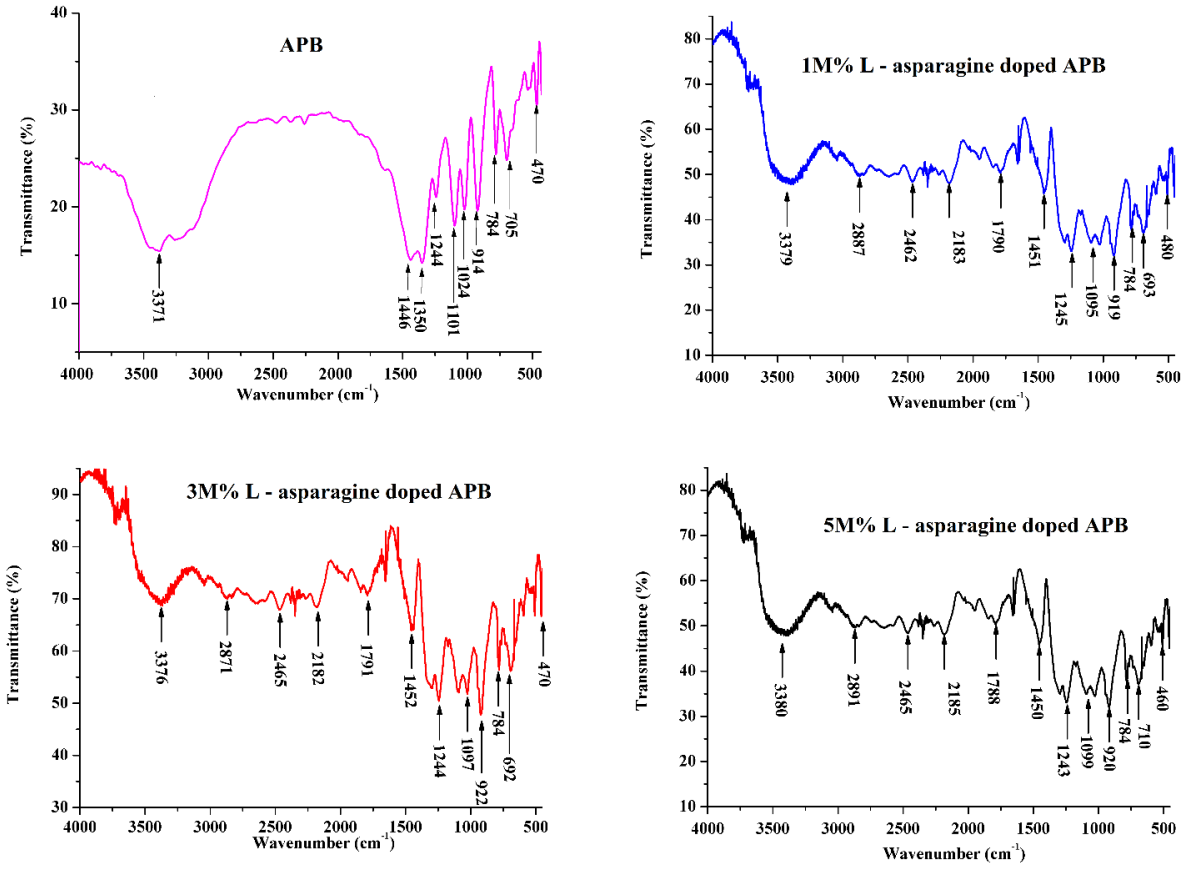

Figure. 4: The FT-IR spectra of APB and L-asparagine doped APB crystals.

The FT-IR was done to characterize the pure and L-asparagine doped APB crystals. The different functional groups of APB and L-asparagine doped APB listed in figure 4. The presence of L-asparagine shifted all the absorption bands of APB which confirm the doping of L-asparagine in APB crystal, as mentioned in Table 2.

Wave numbers (cm^{-1})				Assignments
APB	1M% L-asparagine doped APB	3M% L-asparagine doped APB	5M% L-asparagine doped APB	
3371	3379	3376	3380	N-H asymmetric stretching, O-H asymmetric stretching
-	2887	2871	2891	C-H symmetric stretching
-	2462	2465	2465	C-N stretching
-	2183	2182	2185	C-C stretching
-	1790	1791	1788	O-H bending group, C=O stretching of COOH
1446	1451	1452	1450	N-H vibration of NH4, C-O stretching
1244	1245	1244	1243	B-O asymmetric stretching
1101	1095	1097	1099	B-O symmetric stretching, C-O stretching
914	919	922	920	B-O ring asymmetric bending, C-H stretching
784	784	784	784	O-B-O ring asymmetric bending
705	693	692	710	O-B-O ring asymmetric bending
470	480	470	460	O-B-O ring symmetric bending

Table 2: Wavenumbers of absorption lines in FTIR spectra and their assignment for pure and L-asparagine doped APB crystal

3.3 Thermal analysis:

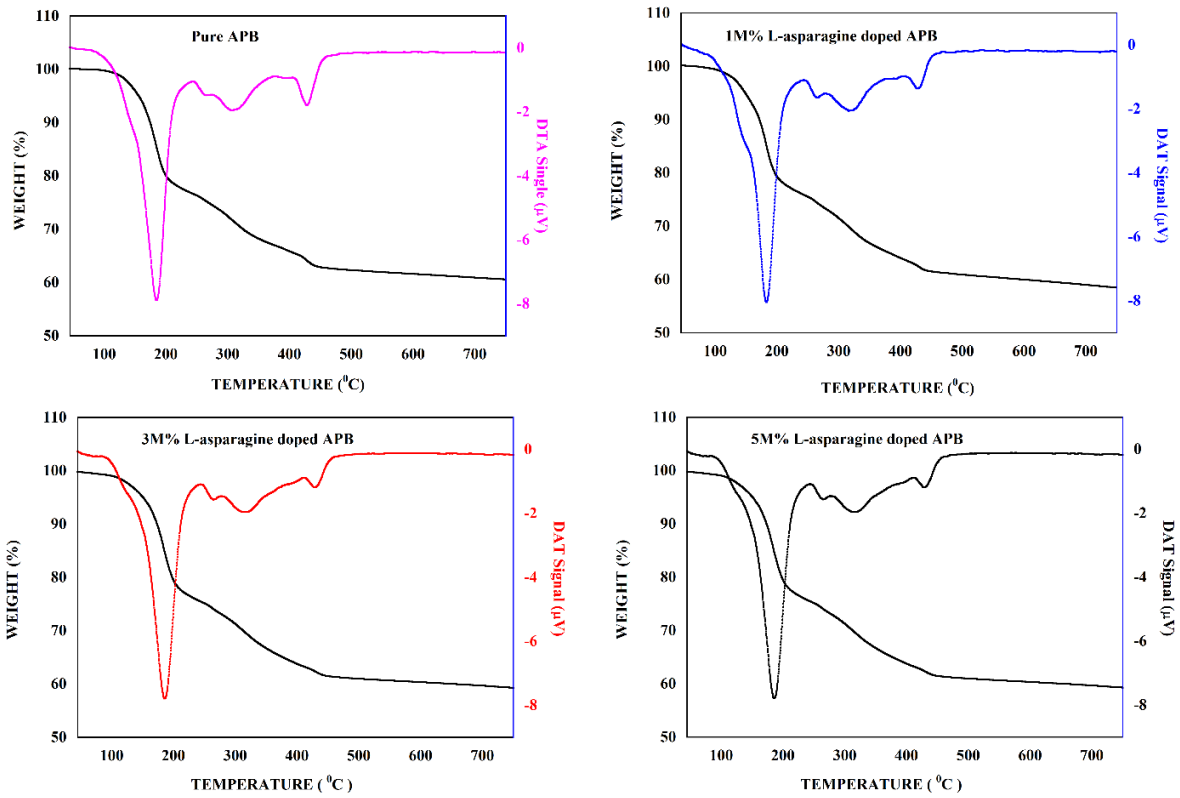

Figure 5: Thermograms of pure and L-asparagine doped APB crystals.

Figure 5 shows the thermograms of pure and L-asparagine doped crystals. Table 3 indicates the experimentally obtained weight in percentage from thermo-gram at different temperatures and theoretically

calculated weight in percentage along with the reaction taking place. From table 3, one can find that the three processes occurring as in the pure APB sample.
However, no major change in thermal stability is found in the broad sense with compare with pure and L-asparagine doped crystal.

Temperature (^oC)	Reactions involved	Weight (%) Theoretical	Weight (%) Experimental
50 – 150	No decomposition	100	100
150-200	$(NH_4)_2.O.5B_2O_3.8H_2O \rightarrow (NH_4)_2.O.5B_2O_3.\ 6H_2O + 2H_2O$	93	91.3
200-310	$(NH_4)_2.O.5B_2O_3.6H_2O \rightarrow (NH_4)_2.O.5B_2O_3.\ 2H_2O + 4H_2O$	79	78.2
310-460	$(NH_4)_2.O.5B_2O_3.2H_2O \rightarrow (NH_4)_2.O.5B_2O_3 + 2H_2O$	71	71.4
	$(NH_4)_2.O.5B_2O_3 \rightarrow 5B_2O_3 + 2(NH_3) + H_2O$	61	60.7

Table 3: Theoretical and Experimental weight percent values and chemical reaction for pure APB

3.4 EDAX measurements:

The EDAX spectra of APB and L-asparagine doped APB crystals has been shown in figures 6 and Table 4 provide the information regarding the presence of constituent elements in APB and L-asparagine doped APB crystals. The successful doping of L-asparagine in APB is well expected based on such figures and tables.

Sample	Carbon (C)		Nitrogen (N)		Oxygen (O)	
	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %
Pure APB	0.63	0.85	7.83	9.01	92.25	90.14
1M% L-asparagine doped APB	5.15	6.1	7.25	8.28	87.6	85.62
3M% L-asparagine doped APB	5.26	6.69	9.85	10.21	84.89	83.1
5M% L-asparagine doped APB	6.02	7.68	12.05	12.89	81.93	79.43

Table 4: Value of Carbon, Nitrogen and Oxygen for pure and L-asparagine doped APB crystal.

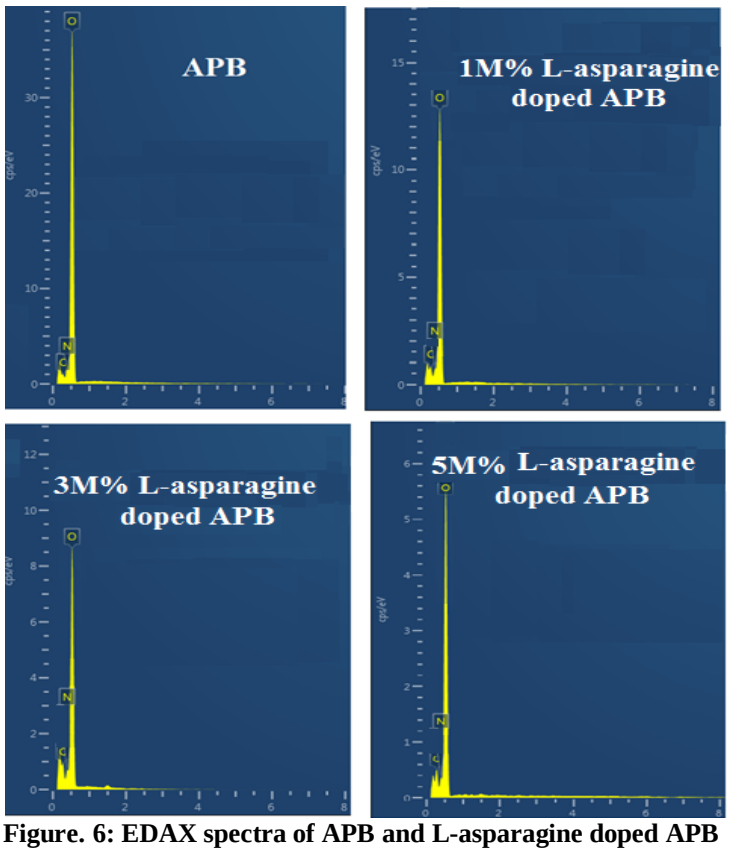

Figure. 6: EDAX spectra of APB and L-asparagine doped APB

IV. Conclusion:

The successful growth of single-phase pure and L-asparagine doped APB crystals was achieved using the low temperature solution growth technique. The presence of dopant like L-asparagine is successfully confirmed using the FT-IR and EDAX, respectively. The thermal stability was marginally affected by addition of L-asparagine in APB crystals.

Author Contribution Statement:
K.R.Rathod: Conceptualization, Formal Analysis, Investigation **M.J.Joshi:**Writing-Review & Editing, **K.D.Parikh:** Supervision.

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