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Growth and Characterization of Sodium Penta Borate Crystal

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Abstract: Sodium Penta Borate Na($H_4B_5O_{10}$) crystal has been grown by slow evaporation technique using deionized water as solvent. The grown crystal was confirmed by powder and single crystal X-ray diffraction studies. FTIR spectrum reveals the presence of functional groups present in the grown crystal. UV-Vis spectral studies performed the cut off wavelength of the grown crystal as 202 nm in the UV region. The thermal properties of the grown crystal were studied by TGA-DTA and the melting point has been found out as 198°C. The dielectric behaviour of the grown crystal has been investigated with different frequencies at different temperatures.

Keywords: Growth and Characterization of Sodium Penta Borate Crystal.

1. Introduction

Inorganic Borates exist in numerous structural types. Sodium, Potassium and Ammonium Penta Borates are excellent non-linear optical (NLO) materials particularly in UV region. These Borate crystals generally possesses chemical stability, high damage threshold and high optical quality as well as wide range of transparency in the ultraviolet region on account of the rather large difference in the electro negativity of the B and O atoms [1]. The experiment conducted by Becker on NLO materials proved that Borate compound materials are superior to other commonly used material for UV applications [2]. The characterization of different Borate crystals such as Potassium Penta Borate [3-8], Ammonium Penta Borate [9-10] and Barium Penta Borate [11] have been already reported. Sodium Penta Borate crystal has been grown by the conventional slow evaporation method. Attention has been given to characterize this crystalline material through powder and single crystal techniques along with FTIR, UV, Thermal, and Dielectric studies.

2. Crystal Growth

The starting material was synthesized by taking Sodium Carbonate and Boric acid in the appropriate ratio and are dissolved in deionized water at room temperature. The chemical equation governing the reaction is,

$$Na_{2}CO_{3}+10H_{3}BO_{3} \longrightarrow 2[Na(H_{4}B_{5}O_{10}).2H_{2}O] +9H_{2}O+CO_{2}\uparrow$$
(1)

The solution was agitated with a magnetic stirrer for 8 hours continuously and filtered. The filtered solution has been taken in the beaker covered with porous paper and kept in a dust free atmosphere. After the period of 32 days, colourless, transparent crystals were obtained and is depicted in Fig. 1.



Fig. 1 Photograph of the grown Sodium Penta Borate crystal

3. Characterization

The X-ray diffraction pattern (XRD) of the powdered sample was obtained by using Philips X' Pert Pro X-ray automatic diffractometer in the range of 10-70[°] with CuK α radiation of wavelength 1.54056 Å. The single crystal X-ray diffraction studies of the grown crystal has been carried out using ENRAF NONIUS CAD4 single crystal X-ray diffractometer with MoK α (λ =0.717 Å) radiation. The FTIR spectrum was recorded in the range of 4000-400 cm⁻¹ using Perkin Elmer RX1 spectrometer. The UV visible studies have been carried out by using Perkin Elmer lambda 35 spectrometer in the region 190-1200 nm. DTA and TGA spectra are recorded for the range of temperature 0°C to 736°C using the instrument NET SZCH STA 490C. The dielectric studies have been carried out by using Agilent 4284A 20Hz-1MHz Precision LCR meter.

4. **Results and Discussion**

4.1 XRD Analysis

The observed powder XRD spectrum of the grown Sodium Penta Borate crystal is shown in Fig. 2. Sharp peaks of XRD pattern indicate high degree of crystal nature of the grown crystal. From the single crystal XRD experiments, it has been found that the given crystal crystallized in monoclinic system with lattice parameters $a = 11.103 \pm 4$ Å, $b = 16.438 \pm 3$ Å, $c = 13.563 \pm 2$ Å, $\alpha = \gamma = 90^{\circ}(0^{\circ})$, $\beta = 101.98(4^{\circ})$ and volume = 171.7Å³. It is in close agreement with the values already reported, thus confirms the identity of the grown crystal [13].



Fig. 2 Powder XRD pattern of the grown Sodium Penta Borate crystal

4.2 FTIR Analysis

The crust powder of Sodium Penta Borate crystals were mixed separately with KBr and pelletized using a Hydraulic press and subjected to the FTIR studies. The FTIR spectrum of the title compound is shown in Fig. 4.3. The strong peak appears at 3451cm⁻¹ is due to the O-H stretching frequency [14]. The peak appears at 1651cm⁻¹ has been attributed to the bending vibration of O-H bond. This confirmed the presence of water molecules in the present compound. The band at 3385cm⁻¹ has been attributed to N-H asymmetric stretching [15]. The narrow band absorbed at 1432cm⁻¹ has been assigned to B-O terminal asymmetric stretching. The intense peak appears at 1356 and 1248cm⁻¹ are assignable to B-O asymmetric stretching. The B-O terminal symmetric stretching vibrations appear at 1099 and 1024cm⁻¹[16]. The B-O ring stretching vibrations are found at 922cm⁻¹ with strong intensity. The medium intensity bands appearing at 783 and 692cm⁻¹ are attributed to O-B-O ring asymmetric bending. The bands designated at 589cm⁻¹ has been assigned to O-B-O terminal bending. It is noticed that the bands appear at 505 and 454cm⁻¹ has been attributed to O-B-O ring symmetric bending. FTIR Assignments are listed in Table.1.



Fig. 3 The FTIR Spectrum of Sodium Penta Borate crystal

 Table.1
 FTIR Assignments of Sodium Penta Borate Crystal

Wave number (cm ⁻¹)	Assignments
3451	O-H Stretching
3385	N-H asymmetric Stretching
3072	N-H Symmetric Stretching
1651	O-H bending
1432	B-O terminal asymmetric stretching
1356	B-O asymmetric stretching
1248	B-O asymmetric stretching
1024	B-O terminal symmetric stretching
1099	B-O terminal symmetric stretching
922	B-O ring stretching
783	OBO ring asymmetric bending
692	OBO ring asymmetric bending
589	OBO terminal bending
505	OBO ring symmetric bending
454	OBO ring symmetric bending

4.3 UV-Visible Spectral Analysis

The optical transmission spectrum of Sodium Penta Borate crystal is shown in Fig 4.4. The grown Sodium Penta Borate crystal is highly transparent in the visible region (above 390nm) and in near UV region also. The cut off wave length for the grown crystal has been found out as 202 nm in the UV region.



Fig. 4 The optical Transmission spectrum of Sodium Penta Borate crystal

4.4 Thermal Analysis

A ceramic (Al₂O₃) crucible was used for heating and the analysis was performed in nitrogen atmosphere at heating rate of 20^{0} C/min. The DTA trace of Sodium Penta Borate crystal is shown in the Fig...5.



Fig. 5 DTA spectrum for Sodium Penta Borate crystal

In the first stage, it is found that a weight loss of 4.8% occurs upto 150° C, is mainly attributed to the decomposition of water molecules. The strong endothermic peak observed at 197.87° C is assigned to the melting point of the given compound. The second stage of decomposition was observed at the temperature range 150° C to 220° C, accompanied with a weight loss of 17 percentage, corresponds to the loss of Hydrogen

molecules. The third stage occurs from 220°C to 736°C, having a weight loss of 77%, due to the removal of Sodium Oxide and Borates.

4.5 Dielectric studies

The defect free crystals of Sodium Penta Borate have been cut in the appropriate orientation and is used for dielectric measurement. Rectangle specimen of thickness approximately 2.95 mm thickness and area of cross-section of 9.75mm² has been used for dielectric measurements. The capacitance (C_p) and dissipation factor (tan δ) of the given crystal were measured as the function of frequency in the range of 100 Hz to 1MHz with different temperatures (40^oC- 150^oC).



Fig.6 The variation of Dielectric constant with temperature at different frequencies for Sodium Penta Borate crystal

The variation of dielectric constant with temperature at different frequencies is shown in Fig. 6. It is found that the dielectric constant decreases exponentially with increasing frequency and then obtains almost a constant value in the high frequency region (above 10 KHz). The same trend is also observed in the case of dielectric loss versus frequency. The high value of dielectric constant at lower frequencies may be due to the space charge polarization due to lattice defects, depend on the purity and perfection of the material [17-18]. At higher frequencies the dipoles are not able to comply with the external electric field, due to that there is a considerable loss of significance of polarizations gradually occurs.



Fig.7 The variation of Dietectrituloss with temperature at different frequencies for Sodium Penta Borate crystal

The amount of power loss in a dielectric under the action of applied voltage is known as dielectric loss, shows higher values in the low frequency region is due to the loss associated with ionic mobility. The variation of dielectric loss with temperature at different frequencies is shown in Fig. 7. However, the low value of dielectric loss indicates that the Sodium Penta Borate crystal possesses good crystalline quality with lesser defects [19]. The AC conductivity has been determined using the relation $\sigma_{ac} = \varepsilon_0 \varepsilon_r 2\pi f \tan \delta$. Here, ε_0 is the permittivity of free space (8.85×10⁻¹² Farad/m), ε_r is the dielectric constant, tan δ is the dielectric loss and f is the frequency of the applied field. The variation of ac conductivity with temperature at different frequencies is shown is Fig.8



Fig. 8 The variation of AC Conductivity with temperature at different frequencies for Sodium Penta Borate crystal

Ac conductivity is noted to be slightly increased with increasing temperature in the lower frequencies. It fulfills an intensive rise with increasing temperature in high temperature and high frequency region, is mainly attributed to the thermally generated charge carriers [20]. The electrical conduction in dielectric is mainly a defect control process in the low temperature region. The presence of impurities and vacancies predominantly affect the conductivity in this region. The energy needed to form the defect is much larger than the energy needed for its drift.

5. Conclusion

Sodium Penta Borate Na($H_4B_5O_{10}$) crystal has been grown by slow evaporation technique using deionized water as solvent. The crystalline nature of the given crystal has been confirmed with the help of powder XRD technique. It is crystallized in monoclinic system. The lattice parameters has been found out by single crystal XRD technique. Functional groups were determined by FTIR analysis. The cut off wave length have been found out as 202 nm in the UV region. High degree of transparency is observed in the visible as well as UV region. Using DTA spectrum it has been found that the melting point of title compound is 197.87°C. Dielectric studies have been carried out for different temperatures at different frequencies and is analysed.

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