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Synthesis, Growth, Optical, Thermal and Dielectric studies of Lead Boro Glutamate (PbBG)

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Abstract: A new semi organic non-linear crystal Lead boro glutamate (PbBG) was grown by slow evaporation technique at low temperature. PbBG has been characterized by UV-Visible, FTIR and thermal studies. Dielectric studies were also carried out to identify the electro optic nature. A positive SHG response was established by NLO studies.
Keywords: Lead boro glutamate; UV-Visible; FTIR; TGA/DSC; Dielectric studies.

1. Introduction:

Crystalline materials are the backbone for many of the current technologies applied in various fields such as electronics, photonics, fiber optics. Hence the synthesis and crystal growth of many technologically important crystals has been pursued with great vigor worldwide. The semiorganic crystals are an important class of compound which have guided prominence on account of their potential utility in microelectronic gadgets, medical equipments, radar systems, communication systems etc[1,2].The use of amino acids for the synthesis of semi organic crystals is interesting on account of its multi functional nature and dipolar character which imparts special characteristics to the compound[3]. In the current work, PbBG was synthesised by a low temperature solution growth technique. The compound was characterized by UV-Visible, FTIR, TGA/DSC and dielectric studies. The SHG response has been determined.

2. Experimental method:

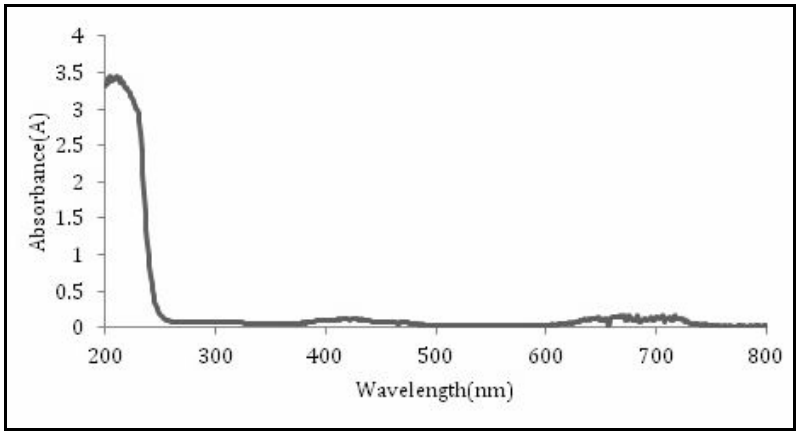
2.1 Synthesis & crystal growth:

Lead boro glutamate (PbBG) was synthesized by low temperature solution growth technique followed by slow vaporization method. Lead nitrate, boric acid and L-Glutamic acid in the ratio 0.5:1:2 was dissolved in double distilled water and stirred well for about 6 hours in a temperature controlled magnetic agitator and evaporated to dryness. Recrystallization was done by using a 1:1 ethanol: water mixture to get good quality of crystals.

3. Results and discussion:

3.1. UV-VIS Spectral analysis:

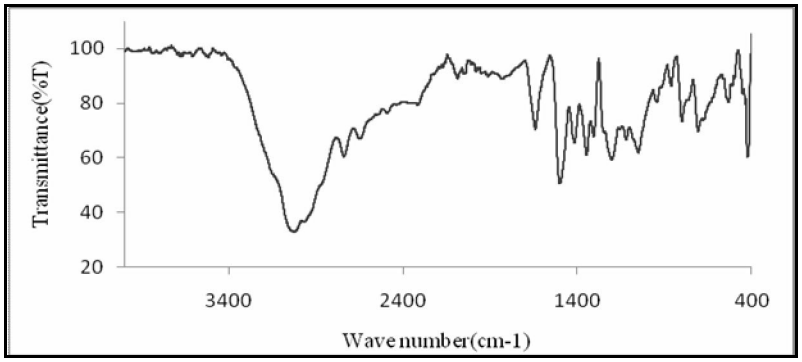
The UV-Vis absorption spectrum was recorded in the range of 200nm to 800nm using a lambda 25 uv-visible spectrophotometer at a rate 120nm/min. The graph shown in Fig.1.



The compound shows very low absorbance in the entire visible region and has an ample clearness of about 90% with a lower cut off wavelength of 250 nm. Thus the optical transparency of the compound from 250 nm to 800 nm is an important characteristics for many of the optical application and utilization in optical devices. The band gap of PbBG was calculated using the formula E_g= 1240/λ(nm) in eV. Where λ is the lower cut off wavelength(250 nm). The band gap of PbBG crystal is found to be 4.96ev.

3.2. FTIR analysis:

Functional groups present in PbBG has been identified using FTIR analysis. FT/IR-6300 type- A Spectrometer was used and the resultant spectrum shows in fig.2.

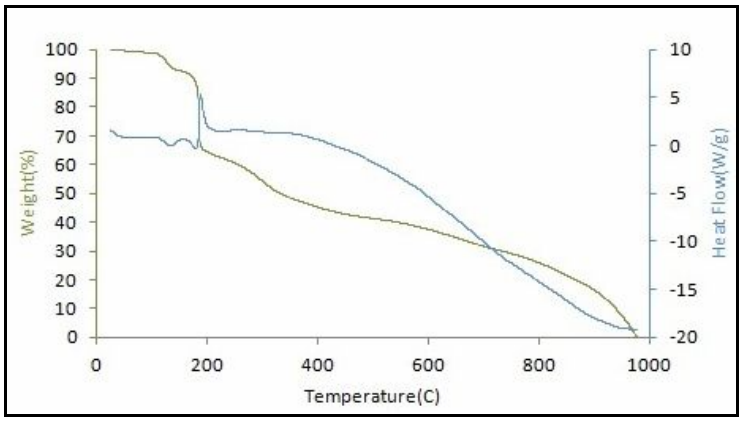


The stretching and bending vibrations corresponding to the different functional groups and bonds present in PbBG has been assigned as shown in the table below.

Frequency in wave number (cm-1)	Assignment of functional group
3027.69cm^{-1}	Asymmetric stretching(NH_3^+)
2646.82cm^{-1}	Symmetric stretching(NH_3)
1641.13cm^{-1}	Bending vibration(NH_3^+)
1501.31cm^{-1}	Bending vibration(CH_3)
1414.53cm^{-1}	Symmetric stretching(COO^-)
1347.03cm^{-1}	Asymmetric stretching(B-O)
1305.57cm^{-1}	Asymmetric stretching(B-O)
1203.36cm^{-1}	Stretching vibrations(CH_3)
1119.48cm^{-1}	Rocking vibrations(NH_3^+)
1050.05cm^{-1}	Rocking vibrations(CH_3^+)
860.09cm^{-1}	Symmetric stretching(B-O)
798.39cm^{-1}	Symmetric stretching(B-O)
532.25cm^{-1}	Bending vibration(OH)
497.54cm^{-1}	Rocking vibrations(COO^-)
422.33cm^{-1}	Stretching vibration of Pb-O

3.3.Thermal analysis:

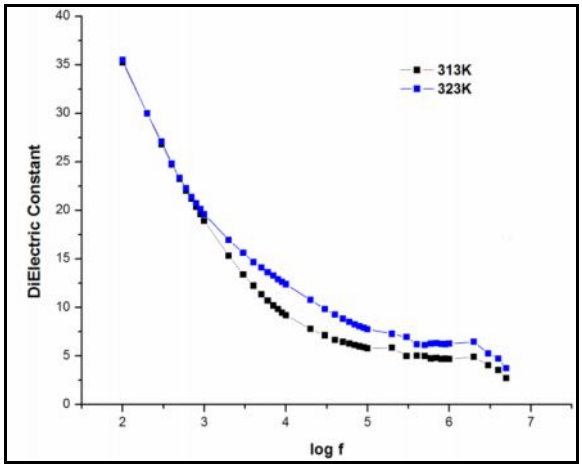
The thermal stability of PbBG has been investigated by thermo gravimetric analysis(TGA) and differential scanning calorimetry(DSC) studies concurrently. The equipment SDT Q600 V20.9 Build 20 analyzer was used in a nitrogen environment in the temperature range of 36 ^{o}C and 1000^{o}C with 10 ^{o}C/min rate. The mass of the sample was used for the analysis was 5.6110mg. TGA-DSC curves are illustrated in fig.3.



The thermo gravimetric analysis shows that the material has very good thermal stability up to 100 ^{o}C. From the TG curve it is inferred that the decomposition of the PbBG compound takes place in two stage weight loss. The first decomposition commences at 100 ^{o}C and ends at 140 ^{o}C and second decomposition at 200 ^{o}C with the elimination of 99.4908 % material into gaseous products. The various gaseous products evolved are CO_2, OH, and hydrocarbon gases. The residual mass of 0.5092 % (0.02857 mg) which is left out in the crucible maybe carbon mass present after all the decomposition processes. It is further observed that the DSC curve shows that first endothermic peak at around 100°C is assigned to melting point of the title compound and a second and third endothermic peak at 120°C and 200°C are due to the-removal of the hydroxyl and borate groups expelled from the compound respectively[4-5].

3.4.Dielectric studies:

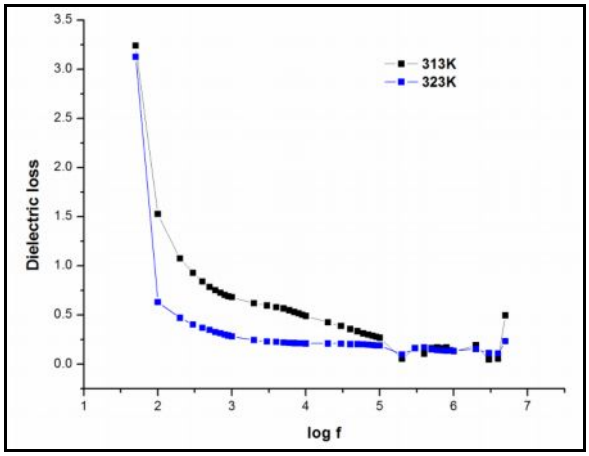
To study the electrical properties of the solids, dielectric constant can be used. The CPPC (conventional parallel plate capacitor method) can be used to study, measure the capacitance and dielectric loss with the frequency between 50 Hz to 5 MHz with HIOKI-LCR HITESTER 3535 at different temperature 343k and 353k. The studies carried out during the sample was cooling, the dielectric constant was analyzed and found by taking the average capacitance (Ccrys). Fig 4. depicts the difference of the dielectric constant using the process of frequency.



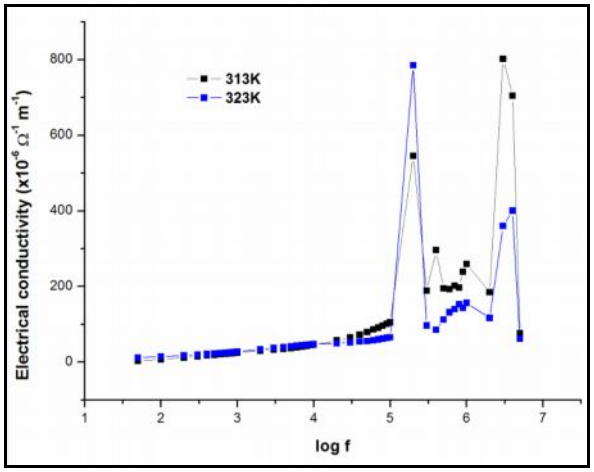
To study the PbBG crystals, high clearness rectangular pattern crystals were used. To achieve the best conductive exterior layer, each sample was layered with best quality of graphite. During the study it was identified through the graph that the dielectric constant value was more at lesser frequency and almost stable

during the greater frequencies. Based on the measure of divergence and charge dislocation the dielectric stability value may vary. Differences in dielectric stability at greater frequencies is ascribed to the lack of space charge divergence near the grain boundary interface[6-8].

Dielectric loss (tan δ) should be maintain as low as possible, for the material probable applicant of NLO application. To use the NLO applications at best, greater frequencies should be used which can reveal very low dielectric loss. This can be find in the graph Fig 5.



This crystal was focused with an separate electric field, after commonly a redistribution of charges founds and currents are triggered. By the use of formula[9] $\sigma ac = \varepsilon_0 \varepsilon r \omega \tan\delta$, the calculation was done for ac conductivity, in this ω is the angular frequency ($\omega = 2\pi\nu$). The PbBG crystals ac conductivity changes was showed in Fig 6. with frequency variations.



The higher the frequency the conductivity too get increases but its zero until 10KHz. The reason for the lesser effect in the mobility and the charge carriers ionic size is because of the less electric conductivity. This also tends to variation in the structure of electronic bonds. Notable ac conductivity variation will be seen at greater frequencies. It proofs the well found relation $\sigma = n_d e \mu e$, that electrical conductivity is relative to carrier concentrations and mobility. In this n_d is the number solidity of electron and μe is the mobility of electron. This shows the PbBG crystals optical conductivity will grows by increase in the energy which applies.

3.5. NLO Test:

Non-linear optical quality of PbBG was studied by Kurtz powder method. In this method an output of NdYAG laser having a wavelength of 1064 nm with 35ps duration of the pulse and the repetition speed of 10Hz was allowed to falls on the powdered material. The second harmonic generation signal in visible region at 532 nm(green light) is measured at different points on the powder material using a photomultiplier tube and gated integrator. The efficacy of frequency doubling is found to be 1.3 times more than that of KDP. This property of PbBG suggests potential use in communication systems.

4. Conclusion:

A new nonlinear (NLO) material PbBG has been synthesized by low temperature solution growth method. Optical absorption studies shows that the crystal shows lucid property and the UV cut off wavelength is identified at 250 nm. The lower cut off value of the optical transmittance shows that PbBG can be used for the propagation of lower wavelength lasers. These studies emphasize that PbBG can be considered for the tailoring of optoelectronic devices. TGA & DTA shows that the material is thermally stable up to 140^{o}C. From Dielectric studies PbBG is appropriate material to be apply in electro-optical devices. From Kurtz powder technique, the powder SHG efficacy of PbBG is comparable to that of KDP. Its efficacy is 1.3 times greater than that of KDP crystals.

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