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Synthesis, Spectral and Antibacterial Studies of Semi-Organic Single Crystal: Barium Thiourea Chloride.

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ABSTRACT

The semi-organic single crystal of Barium Thiourea Chloride (BTC) was grown by slow evaporation method. The structure of the grown BTC single crystal was confirmed using powder x-ray and single crystal x-ray diffraction. The presence of different functional groups was identified by FT-IR spectroscopy and it agrees well with the calculated and reported values. The optical absorption studies revealed low absorption in the UV -Visible regions and the cut off wave length was found to be 290 nm and the grown crystals were highly transparent in the visible region. Antibacterial activity confirmed that the grown crystals were inhibiting the growth of pseudomonas organism and not the Ecoli. Good transparency, thermal stability and organism specific activity suggests Barium Thiourea Chloride single crystals can be used for optical and antibacterial applications.

Keywords: Optical materials, Crystal Growth, Antibacterial studies, X-ray diffraction

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INTRODUCTION

The advancement of laser technology and the use of lasers in the modern day devices need very high quality single crystals. In recent year's nonlinear optical (NLO) effect have found increasing interest. NLO materials grown by low temperature solution growth are used widely because of the low cost in production and relative ease with which large volume of optically transparent and homogenous single crystals can be grown. The organic materials will have high Second Harmonic Generation (SHG) efficiency but it is least stable as most of the organic molecules are formed by weak van der walls and hydrogen bonds with conjugated π electrons while the inorganic compounds are highly stable but it has less SHG efficiency. This creates interest in semi organic compounds, which are the hybrids of good stability and good efficiency. Thiourea is an interesting molecule which possesses centro symmetry but when co-ordinated with metal complexes it becomes non-centro symmetric molecule [1-2]. Based on the transmission analysis of an electron in a molecule it has been reported that small organic molecules like =C=N- ; =C=O- were selected as SHG active units [3]. Structure and characteristics of thiourea metal complexes have been reported [4-13]. We tested the grown crystals for antibacterial activity of some common organisms like Ecoli and pseudomonas. Since the optical behavior of the material is important to determine its usage in opto-electronic devices, the UV-Visible transmission analysis also done. Along with these studies we are reporting single crystal structure analysis, FT-IR spectral analysis.

EXPERIMENTAL

In our present work barium thiourea chloride single crystals have been grown by the slow evaporation method. Saturated solutions of thiourea with barium chloride in triple distilled water in the molar ratio of 1:1 is prepared by the equation

$$BaCl_2 + CS[NH_2]_2 \rightarrow Ba[CS\,[NH_2]_2]Cl_2$$

By repeated recrystallization process the purity of the BTC salt was further increased and the super saturated solution of BTC was kept undisturbed at room temperature. Optically transparent defect free crystal was obtained within the period of 15 days. Single crystal XRD and Powder X-ray diffraction analyses were used to estimate the lattice parameters and crystalline quality of the crystal by using Bruker Nonious CAD-4/MACH 3 single X-ray diffractometer and Brucker AXSCAD4 diffractometer respectively. The IR spectroscopy is effectively used to identify the functional groups of the grown crystals and the FTIR spectrum of the sample was recorded by JASCO FTIR 410 spectrometer. The optical transmittance spectrum of BTC crystals was recorded with Perkin-Elmer Lambda 35 Spectrophotometer in the range of 190–1100 nm.

RESULTS AND DISCUSSION

X-ray diffraction studies

Single crystal x-ray diffraction is a non destructive analytical technique which provides detailed information about the lattice of a crystal. The crystallographic characteristics measured using single crystal x-ray is shown in Table 1. The powder X-ray diffraction was also recorded and the diffractogram is shown in the Figure 1. All the peaks in the x-ray diffractogram were indexed and lattice parameter calculated agrees well with the single crystal values. The prominent sharp peaks obtained from X-ray diffraction confirm the crystalline quality of the grown crystal.

FT-IR Spectral analysis

FT-IR spectrum for the BTC has been recorded in the range 400- 4000 cm^{-1} and is shown in Figure 2. The spectrum shows the presence of functional groups in the BTC crystal. The FTIR spectrum of BTC crystal is compared with the spectrum of thiourea [14] in Table 2,with the calculated and already reported spectrum of BTC as shown in Table 3. When it is compared with the spectrum of thiourea few peaks were found to be shifted. The absorption band observed at 3317 cm^{-1} in the spectrum of BTC corresponds to that of thiourea of about the same frequency 3376 cm^{-1} and can be assigned to the NH_2 symmetric bending vibration. The absorption band corresponds to 1634 cm^{-1} in the spectrum of BTC corresponds to the thiourea of about the same frequency at 1627 cm^{-1} and can be assigned to the NH_2 bending vibration. The absorption band at 1371

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cm-^{1} corresponds to 1417 cm-^{1} of thiourea and can be assigned to the C=S asymmetric stretching vibration. The absorption band at 690 cm-^{1} corresponds to 740 cm-^{1} of thiourea and can be assigned C=S symmetric stretching vibration. The absorption observed at 570 cm-^{1} in the spectrum of BTC corresponds to 497 cm-^{1} absorption of thiourea.

Optical transmission studies

The optical property of the BTC crystal was assessed by using UV-Vis spectrophotometer. The UV-Visible spectrum gives limited information about the structure of the molecule because; the absorption of UV-Vis light involves promotion of the electron in the σ and π orbital from the ground stable to higher energy state [15, 16]. Fig.3 shows the transmission curve in which the lower cutoff wave length is 290 nm. The optical band gap of the grown crystal is calculated to be ~4.06 eV, showing it might be a good dielectric material.

Antibacterial activity

The developed crystal is tested for its therapeutic efficacy through microbiological technique. The crystal is made as fine powder and dissolved in distilled water. The prepared sample is tested using plate method to investigate inhibition on microbiological growth. Organisms were cultured on agar plate to assess the efficacy of the stock culture as a control study. The micro organisms such as *Eschreichia coli* and *Pseudomonas aeroginosa* were selected to assess the antibacterial effect of the crystal material discovered. Special attention was given on *Pseudomonas* culture because of its high pathogenisity by causing diseases like pneumonia, septic shock, urinary tract infection, Gastro intestinal infections, skin and soft tissue infections. [17], [18]. The culture plates were inoculated with the drug of different concentration such as 5, 10 and 15 µl. The cultures were incubated scientifically following all reported procedures of Prescot *et al*. for two days. [19] On the third day the cultures were observed for inhibition by the sample. The *E. coli* plate did not shown any significant result whereas *Pseudomonas* plate has shown remarkable inhibition on microbial culture. When we compare the results we observed dose dependent inhibition as per table 4. And plate No 1 (figure 4) .Maximum inhibition was observed in 15 µl concentration which was around 6 mm. The results indicate that the sample is quite specific in controlling the growth of specific microbe, *Pseudomonas*. Hence this result provides a strong platform for further researchers to probe and develop organism specific antibiotic.

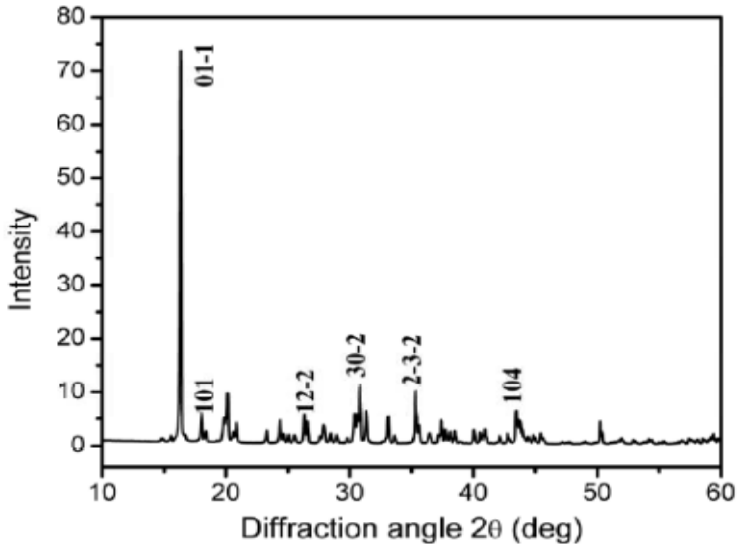

Figure 1

Fig. 1. Powder Xray pattern of BTC crystals

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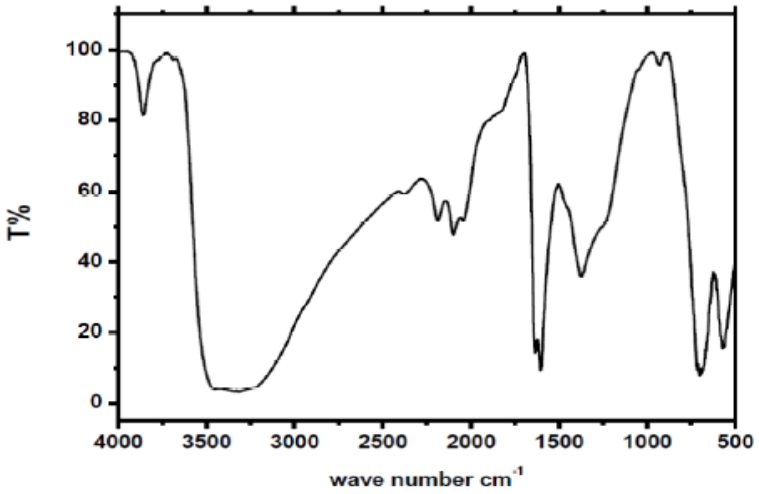

Fig. 2. UV-Visible transmission spectra of BTC single crystals

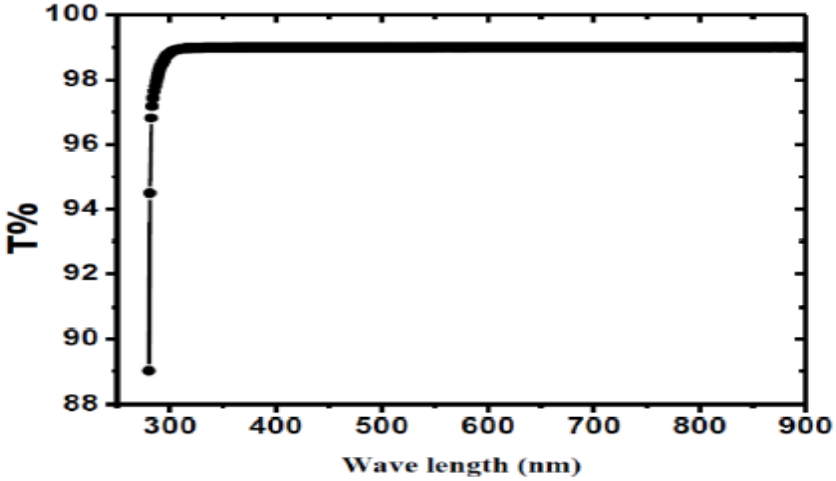

Fig. 3. FTIR spectra of BTC single crystals

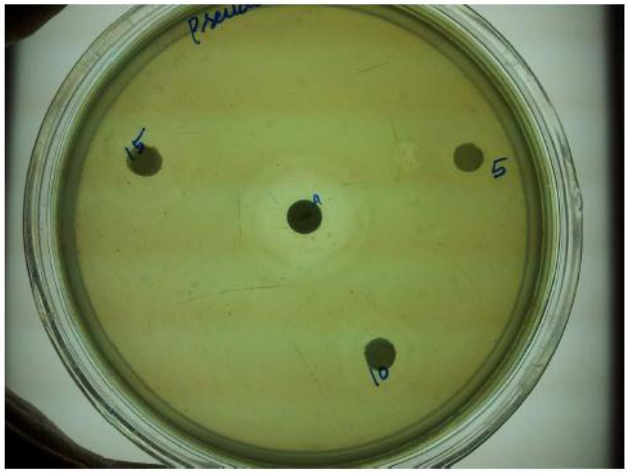

Fig.4. Plate no.1 of dose dependent inhibition

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Table 1.

Unit cell dimensions
a = 6.68 Å
b = 10.91 Å
c = 7.11 Å
α = 90.00°
β = 90.62°
γ = 90.00°
V = 518 A^3
System = monoclinic

Table 2.

Thiourea (cm^{-1})	BTC (cm^{-1})	Assignment (cm^{-1})
494	570	N-C-N Stretching
740	690	C=S Stretching
1089	929	C-N Stretching
1417	1371	C=S Stretching
1627	1634	NH2 Bending
3376	3317	NH2 Stretching

Table 3.

Calculated	Reported	observed	Assignment (cm^{-1})
3459	3330	3317	NH2 Symmetric Stretching
1652	1639	1634	NH2 Bending (Sci)
1604	1606	1605	NH2 Bending (Sci)
1366	1413	1371	NH2 Bending (rock)
855	730	715	NH2 Bending (wag)
660	684	690	C=S Stretching
605	560	570	NH2 Bending (twist)

Table 1. Crystal Data for Barium Thiourea Chloride crystals

Table 2. Comparison of FT-IR Assignments for thiourea [18] and BTC single crystal

Table 3. Comparison of FT-IR Assignments for calculated and reported [3] for BTC single crystal

Table 4

Trial	Name of the organism[*Pseudomonas aeroginosa*	Zone of inhibition in mm			
		5ul	10ul	15ul	Antibiotic
1	Plate 1	2	4	6	7
2	Plate 2	2	4	5	7
3	Plate 3	3	5	6	7
Mean		2.3	4.7	5.8	7

Table 4. Comparison of dose dependent inhibition

CONCLUSION

Transparent, good quality, colourless single crystal of BTC was grown using slow evaporation and slow. Single crystal and powder X-ray diffraction studies confirmed the crystal structure and quality of the

grown crystal. The presence of functional groups of the grown crystals was confirmed by FTIR spectral analysis.. UV-Vis transmittance studies reveal the transparency, cutoff wavelength and band gap energies of the grown crystals. The presence of NLO active functional groups, good transparency and high thermal stability suggest that materials like Barium Thiourea chloride and other thiourea metal chloride complex can be used for optical applications

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