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# *Synthesis and Characterization of Noval $ABX_3$ type crystal usingBenzyltriethylammonium chloride and Metalhalide ($BaCl_2$) by SlowEvaporation method*

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**Abstract -**A noval $ABX_3$ type crystal using benzyltriethylammonium chloride and metal halide ($BaCl_2$) were synthesized by using slow solvent evaporation technique at room temperature. Crystals with high quality were grown within a weeks. Further the quality of the crystals were confirmed by elemental analysis (CHN). The crystalline nature and cell parameters were analyzed using X-ray diffraction (XRD) study. The presence of various functional groups and modes of vibrations were identified by FT-IR spectral analysis. From the UV-Vis-near IR transmittance spectrum, the good transparency is revealed from 200 nm to 1100 nm. Thermal behavior of the crystals were obdurate using TGA studies which reveals the crystals are stable to 850ºC.

***Key Words***:UV- Visible, X-Ray diffraction, TGA.

## 1.INTRODUCTION

Crystals are well defined by its regularity, periodicity and orderly arranged molecular structure called a lattice. Various types of crystals are grown to bridge the important gaps in conventional production in engineering. Diverse properties of crystals paved a way for its increase in industrial and scientific applications and also in development of technologies in the form of lasers, semiconductors, nuclear fusion, medical diagnostics, display, high and low energy particles in physics and thermal imaging

The method used here is slow evaporation. Basically evaporation is used to separate solutes from solvents by evaporating off the solvent, leaving solute alone. Evaporation can be done fast with heat or slowly with cooler temperatures. Slow evaporation method is considered one of the easiest method to grow crystals. It is also a cost efficient method when considered to other methods, all it requires is the supersaturated solution of the compound. Nearly all the solute which are added at the beginning are collected, this is considered as the most important advantage of this method. Experimental setup need for this method is simple. With the help slow evaporation method the combination of organic – inorganic metal halide crystal (benzyltriethylammonium chloride – $Bacl_2$) was grown and characterized using various techniques. In order to evaluate the crystal quality many techniques have been developed for analysing crystal perfection [1].

## 2. MATERIALS AND METHODS

*Crystal growth*

$ABX_3$ type Barium complex crystal were prepared by mixing Benzyltriethylammoniumchloride and Barium chloride ($BaCl_2$) in 1:1 molar ratio respectively using triple distilled water. The two solutions were mixed together thoroughly in acidic medium by using 1ml of HCl. Then the resulting solution was filtered through whatmann42 filter paper. The solution was kept at room temperature for preparation of solids by slow evaporation method. A colorless transparent crystalline product was obtained. Photograph of grown crystal was shown in.

$$[C_6H_5CH_2N(C_2H_5)_3]Cl + BaCl_2 \rightarrow$$

$$[C_6H_5CH_2N(C_2H_5)_3]BaCl_3$$

*Solubility*

Solubility of $ABX_3$ type Barium Complex crystals in water, ether and DMSO is checked. The crystal $ABX_3$ type Barium Complex crystals is soluble in water.

*Elemental Analysis*

Quality of the compounds in a crystal lattice can determined by elemental analysis study. It measures the weight percentage of different elements present in a compound. CHN analysis gives the calculated values of weight percentage of carbon, hydrogen and nitrogen [2][3].

*UV – Visible Spectroscopic studies*

Optical properties of crystals can be mainly analyzed by UV spectroscopy. UV spectroscopy is one of the best study to detect the impurities that cause defects in organic compounds [4]. It deals with the absorption of light in the UV Visible part in the spectrum of 210 – 900 nm. The transition results in the absorption of electromagnetic radiation in this region of the spectrum are transition between electronic energy levels.

*FT-IR studies*

FT-IR is an analytical technique used to identify organic materials and sometimes inorganic materials. FT-IR is a main technique to identify the types of chemical bond in an organic molecule. It can identify variation and determine the amount of component in the mixture. It is also used to identify the purity of crystal.

*X-Ray Diffraction studies*

The clear analysis of crystal structure and orientation of crystal for the successful use of single crystals in their technological applications was done using XRD. The various data on space group and unit cell parameters, data collection and processing, structure solution refinement etc. can be gained.

*Crystallinity study*

% Crystallinity of the crystal can be calculated using Hinrichsen's formula, (XRD deconvolution method) where the amorphous and crystalline contributions to the diffraction spectrum were separated. Crystallinity can be calculated from the ratio of the integrated area of all crystalline peaks to the total integrated area under the XRD peaks.

$$\% \text{ Crystallinity} = I_c / (I_c + I_a) * 100$$

Where,

$I_a$ &$I_c$ are the integrated intensities corresponding to the amorphous and crystalline phases, respectively.

*Thermal analysis*

Thermal behavior of samples can be analyzed under various conditions. Amount of sample required for this analysis is small. According to international Confederation for Thermal Analysis and Calorimetry (ICTAC), thermal analysis is defined as a aggregation of techniques in which the property of sample is viewed against time or temperature under controlled environment [5].

## 3. RESULTS AND DISCUSSION

*Solubility*

The crystal $ABX_3$ type Barium complex crystals are soluble only in water while Ether and DMSO is also used as a solvent for testing its solubility. The results were tabulated in **Table -1.**

**Table -1: Solubility of $ABX_3$ type Barium complex crystals**

| Solvent | Solubility |
|---|---|
| Water | Soluble |
| Ether | Insoluble |
| DMSO | Insoluble |

*Elemental Analysis*

Composition of the grown crystal is verified by using results obtained from the elemental analysis (C, H, N). The percentage composition of carbon, hydrogen and nitrogen were also determined using the following data from the **Table -2.**

**Table -2: Elemental analysis of $ABX_3$ type Barium complex crystals**

| NAME | CARBON % | | HYDROGEN % | | NITROGEN % | |
|---|---|---|---|---|---|---|
| | EXP | THEO | EXP | THEO | EXP | THEO |
| [BTEATC – Ba] | 33.42 | 35.86 | 5.02 | 5.04 | 5.24 | 3.21 |

*UV-Visible Spectroscopic studies*

From the UV spectrum absence water is confirmed, since there is no peak is seen for water. This proves that the grown $ABX_3$ type Barium Complex crystals are highly crystalline in nature. The peak at 270 nm shows the presence of aromatic group. The UV spectrum of crystal $ABX_3$ type Barium Complex crystals is given in **Figure 1.**

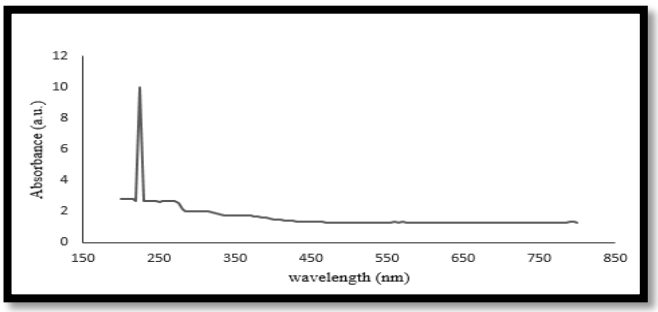


**Figure 1: UV spectrum of $ABX_3$ type Barium complex crystals**

*FT-IR studies*

The observed stretching frequency and the corresponding type of bonds are given in **Table -3**. The FT-IR spectrum of $ABX_3$ type Barium Complex crystals is shown in **Figure 2.**

**Table -3: FT-IR peak values of $ABX_3$ type Barium complex crystals**

| S.NO | ABSORPTION PEAK [$cm^{-1}$] | POSSIBLE GROUPS |
|---|---|---|
| 1. | 3448.72 | N-H Stretching |
| 2. | 2885.51 | C-H Alkyl |
| 3. | 1489.05 | C-C Stretch (aromatic) |
| 4. | 1211.30 | C-N Stretching |
| 5. | 1157.29 | C-N-C Stretching |
| 6. | 1002.98 | C-H Out of plane bonding |

| | | |
|---|---|---|
| 7. | 756.10 | C-N-C & C-C-N deformation |
| 8. | 702.09 | C-Cl Stretch |

The N-H stretching appears at 3448.72 $cm^{-1}$ is due to the presence of $NH^{4+}$ ion. The peak appears at 2885.51 $cm^{-1}$ indicate the presence of alkyl group. The peak at 1489.05 $cm^{-1}$ is due to C-C stretching of aromatic group. The peat at 1211.30 $cm^{-1}$ is due to C-N stretching. The peak at 1157.29 $cm^{-1}$ represents the asymmetric C-N-C stretching vibration. The C-H out of the plane bending occurs at 1002.98 $cm^{-1}$. The C-N-C and C-C-N deformation vibration occurs at 756.10 $cm^{-1}$. The C-Cl stretch occurs at 702.09 $cm^{-1}$ in the IR spectrum.

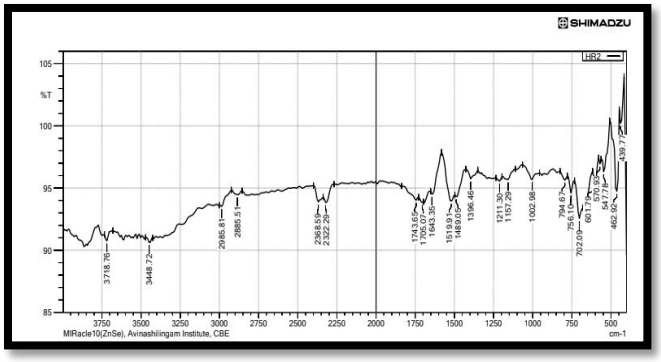


**Figure 2: FT-IR spectrum of $ABX_3$ type Barium complex crystals**

*XRD ANALYSIS*

X-ray powder diffraction pattern of the $ABX_3$ type Barium Complex crystals is shown in **Figure 3** Bragg's peaks of high intensity are obtained at specific 2Θ angles. This shows that the compound is crystalline in nature.

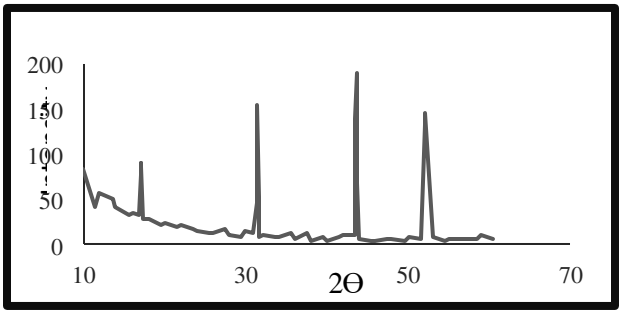


**Figure 3: XRD patter of $ABX_3$ type Barium complex crystals**

*Crystallinity study*

XRD data of $ABX_3$ type Barium Complex crystals was formulated using Hinrichsen's formula to compute the percentage crystallinity.

**Hinrichcen's Formula**

**% Crystallinity = $I_c$ / ($I_c$ + $I_a$) *100**

Where,

$I_a$ &$I_c$ are the integrated intensities corresponding to the amorphous and crystalline phases, respectively.

The % Crystallinity of Barium complex crystal was calculated as **93.82%**

*Thermal analysis*

The TGA curve shows the two stage weight loss when heated between the room temperature and 980ºC **Figure 4**. The first stage decomposition starts at 32.47 ºC and end at 206.08 ºC the weight loss noted at this temperature is 17.22 %. It is assumed that two Cl among three will get evolved first followed by the decomposition of hydrocarbons and nitrogen gas.

**$[C_6H_5CH_2N(C_2H_5)_3]BaCl_3$ →**

**$[C_6H_5CH_2N(C_2H_5)_3]Cl$ + $BaCl_2$**

When the complex undergoes decomposition as above, one mole of complex decomposed to one mole of benzyltriethylammoniumchloride and one mole of barium chloride. In the second stage, the decomposition of barium chloride starts at 565.60ºC and ends at 977.95ºC with the weight loss of 24.84% the weight loss can be accounted for formulating the following decomposition reaction [72].

**$BaCl_2$ → Ba + $Cl_2$ ↑**

The decomposition takes place between 32.47ºC and 977.95ºC, the total weight loss during this period is calculated as 42.06 %. It is clearly seems from the thermogram that the $ABX_3$ type Barium Complex crystals are thermally stable.

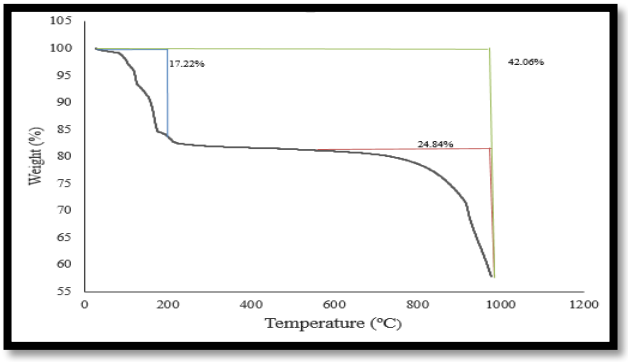


**Figure 4: TGA curve of $ABX_3$ type barium complex crystals**

## 4. CONCLUSION

The noval $ABX_3$ type Barium Complex crystals was grown by slow evaporation method at ambient temperature. Grown crystals were harvested within a week and subjected to various characterization studies. The crystals was soluble only in water, where ether and DMSO was also used for testing solubility. Similarly CHN analysis for two crystals gives the amount of carbon, hydrogen and nitrogen present in the crystals. UV-Visible spectrum confirms that the crystals are

free from impurities (no water). They are further aided by FT-IR data, there is no peak is obtained for water.

$ABX_3$ type Barium Complex crystals the functional group resent is also given by FT-IR spectrum. Highly intense peak of 189.8324 cps in XRD studies exhibit the crystallinity of the crystal. Hinrichsen's percentage crystallinity calculation gives the value as 93.82% which holds the XRD data. From TGA studies it is clear that the crystal is undergoing double stage decomposition and the $ABX_3$ type Barium Complex crystals is thermally stable up to 977.95 ℃.

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