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# Growth and Characterization of ZnN doped KClx Br1-x Mixed Crystals

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**Abstract:** ***Mixed crystals of ZnN doped KClx Br1-x crystals have been successfully grown from aqueous solution by slow evaporation technique. Doped crystals were grown in a period of three weeks. The lattice parameters were determined by PXRD analysis. FTIR analysis was used to confirm the presence of various functional groups in the grown crystals. Optical properties studies by UV analysis. From the SEM studies determined the particle size of grown crystals in various micrometer region.***

**Keywords:** ZnN, PXRD, FTIR, lattice parameter, UV, SEM

## 1. Introduction

A crystal assembles itself out of its own constituent disarray the puzzle puts itself together, each piece falling as through by chance into its correct location[1].

Crystal growth is a relatively small but important area of materials science. lt is clearly more difficult to prepare single crystals than polycrystalline material and the extra effort is justified only if single crystals have outsta Single crystals are also advantageous when excellent optical quality is required, as in lasers, nonlinear optic, and optical modulator applications.nding advantages[2].

The growth methods depend on growth kinetics, crystal size, shape and nature of the application of the crystals. The method should also be economically feasible and the crystal formed should be free from defects[3].

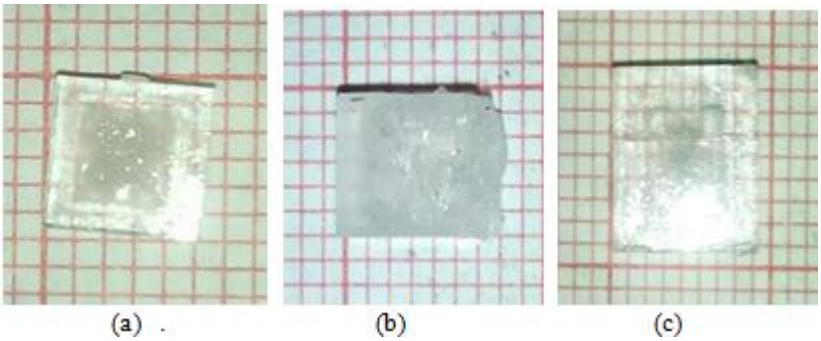
Crystallography or the science of crystals is today of crucial importance in many, often unrecognised ways. Understanding the nature of crystals, especially their atomic structure, is vital for many practising scientists and for industry. A particular drug is patent – protected by supplying a powder diffraction pattern, or occasionally a full crystal structure determination. Crystallography is one of the most interdisciplinary subjects in science [4].

## 2. Materials and Methods

Analar grade of KCl and KBr and dissolved in distilled water were take for the 250 ml beaker. Super saturated solutions of KClxBr1-x were prepared for various molar concentrations.. The KCl and KBr doped mixed crystals were grown by a desired molecular ratio and 0.05 mol % of ZnN taken in a beaker dissolved in the magnetic stirrer using the slow evaporation method. The beaker is closely tightly with polythene paper, and the small holes are made for the perfect evaporation. The period of crystal growth was 4 or 5 weeks. After completion of growth, the crystals were harvested. A large size crystals were selected for the experiments.

The formed crystals were carefully harvested from the beakers. The crystals are dried using filter paper. The grown crystals are characterized by powder XRD, FTIR, UV and SEM analysis.

## 3. Results and Discussion



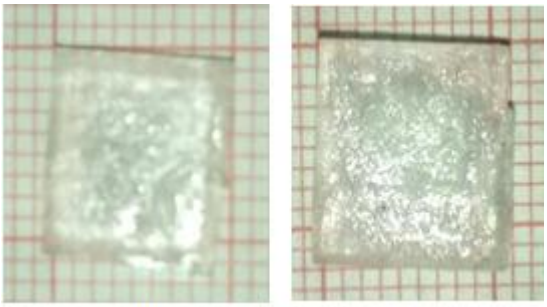



**Figure 1:** Photograph of all the grown crystals

**Table 1:** PXRD Intensity for different 2θ peaks for pure KClcrystal

| For pure KCl | | | | |
|---|---|---|---|---|
| Intensity % | Angle in degrees (2θ) | d spacing (Å) | Miller indices (hkl) | Lattice parameters (Å) |
| 100 | 28.2938 | 3.15429 | (200) | a = 6.308 |
| 34.79 | 40. 4845 | 2.2282 | (220) | |
| 8.4 | 66.3216 | 1.40942 | (420) | |
| 4.99 | 58.5604 | 1.5763 | (400) | |
| 4.37 | 50.1714 | 1.81836 | (222) | |

**Table 2:** PXRD Intensity for different 2θ peaks for pure KBr crystal

| For pure KBr | | | | |
|---|---|---|---|---|
| Intensity % | Angle in degrees (2θ) | d spacing (Å) | Miller indices (hkl) | Lattice parameters (Å) |
| 100 | 27.1796 | 3.28101 | (200) | a = 6.4859 |
| 36.86 | 38.7385 | 2.32452 | (220) | |
| 6.76 | 69.8940 | 1.34587 | (420) | |
| 7.02 | 55.8248 | 11.64687 | (400) | |
| 10.86 | 47.8801 | 1.89989 | (222) | |

**Table 3:** PXRD Intensity for different 2θ peaks for mixed crystal

| For $KCl_{0.2}$ $KBr_{0.8}$ | | | | |
|---|---|---|---|---|
| Intensity % | Angle in degrees (2θ) | d spacing (Å) | Miller indices (hkl) | Lattice parameters (Å) |
| 100.00 | 27.2213 | 3.27608 | (200) | a = 6.552 |
| 23.62 | 38.9050 | 2.31495 | (220) | |
| 6.81 | 63.5215 | 1.46462 | (331) | |
| 5.73 | 48.1477 | 1.88995 | (222) | |
| 5.44 | 56.1916 | 1.63699 | (400) | |
| 5.31 | 23.5039 | 3.78514 | (111) | |
| 4.26 | 70.4416 | 1.33674 | (420) | |
| 3.02 | 45.9573 | 1.97480 | (311) | |

**Table 4:** PXRD Intensity for different 2θ peaks for mixed crystal

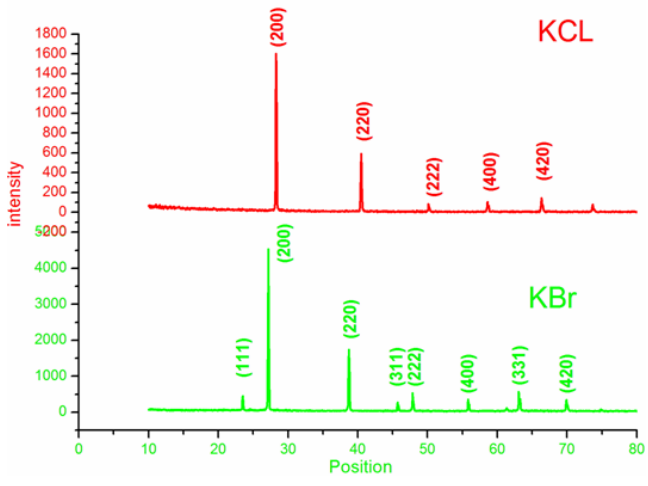
| For **$KCl_{0.8}$ $KBr_{0.2}$** | | | | |
|---|---|---|---|---|
| Intensity % | Angle in degrees (2θ) | d spacing (Å) | Miller indices (hkl) | Lattice parameters (Å) |
| 100 | 28.2055 | 3.16397 | (200) | a = 6.32794 |
| 9.91 | 40.1271 | 2.24722 | (220) | |
| 7.08 | 58.4234 | 1.57967 | (400) | |
| 3.48 | 66.1174 | 1.41327 | (420) | |
| 2.97 | 49.9623 | 1.82548 | (222) | |

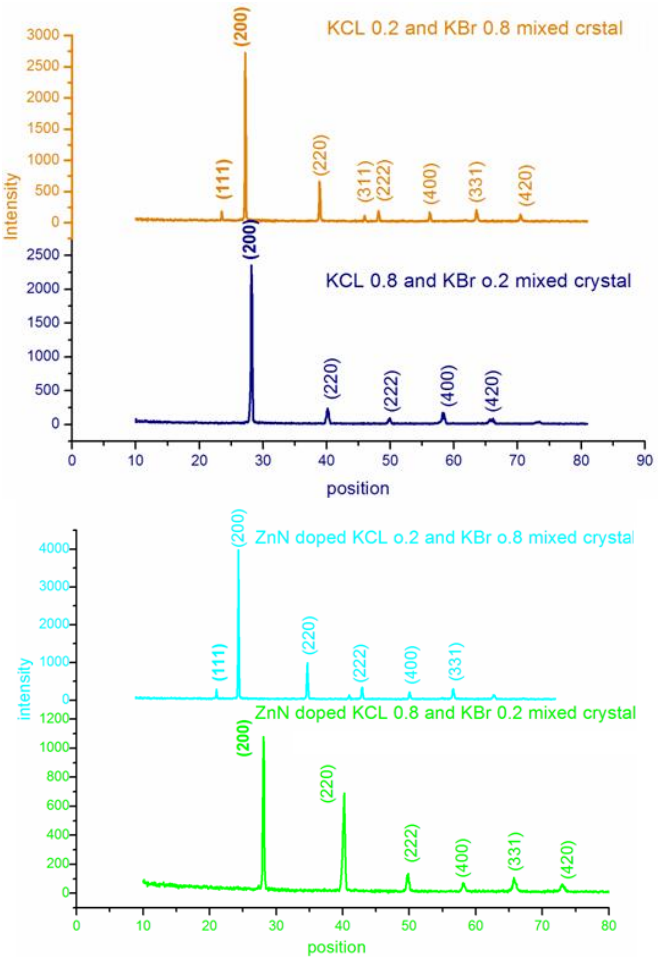
**Table 5:** PXRD Intensity for different 2θ peaks for ZnN doped mixed crystal

| **For $KCl_{0.2}$ $KBr_{0.8}$ Zinc nitrate** | | | | |
|---|---|---|---|---|
| Intensity % | Angle in degrees (2θ) | d spacing (Å) | Miller indices (hkl) | Lattice parameters (Å) |
| 100.00 | 27.1589 | 3.28347 | (200) | a = 6.56694 |
| 70.25 | 38.7873 | 2.3217 | (220) | |
| 16.07 | 63.374 | 1.46767 | (331) | |
| 15.36 | 47.9887 | 1.89584 | (222) | |
| 13.64 | 23.4353 | 3.79606 | (111) | |
| 7.49 | 56.0438 | 1.64096 | (400) | |
| 6.96 | 70.274 | 1.33952 | (420) | |
| .92 | 75.3102 | 1.26196 | (311) | |

**Table 6:** PXRD Intensity for different 2θ peaks for ZnN doped mixed crystal

| **For $KCl_{0.8}$ $KBr_{0.2}$ Zinc nitrate** | | | | |
|---|---|---|---|---|
| Intensity % | Angle in degrees (2θ) | d spacing (Å) | Miller indices (hkl) | Lattice parameters (Å) |
| 100.00 | 28.1709 | 3.16778 | (200) | a = 6.335 |
| 59.70 | 40.2503 | 2.24063 | (220) | |
| 22.35 | 66.0910 | 1.41377 | (331) | |
| 15.56 | 49.8618 | 1.82892 | (222) | |
| 6.35 | 58.3555 | 1.58134 | (400) | |
| 505 | 73.3291 | 1.29107 | (420) | |






The powder method is used to determine the value of the lattice parameters accurately. The numerous sharp peaks found in the PXRD patterns give a clear cut proof of the crystalline nature of all the grown crystals. The phases have been clearly indexed doping of Zinc nitrate in 0.05 mol% concentration produce a slight shift in the Bragg angle. The peak intensity of pure crystals is decreased / increased with increasing concentration of Zinc nitrate in the host lattice the observed sharp peaks and low full-width at half maximum confirm that the crystalline nature of the grown crystals. This may be due to the absorption or substitution of doped atom in lattice sight. There is slight variation in the lattice parameters depending upon the impurity addition and at the same time, the total crystal structure is not affected.

**Table 7:** Inter planar spacing for mixed crystals

| Composition | Intensity % | Miller indices (hkl) | d spacing (Å) |
|---|---|---|---|
| Pure KCl | 100 | (200) | 3.15429 |
| Pure KBr | | | 3.13514 |
| $KCl_{0.2}KBr_{0.8}$ mixed crystal | | | 3.27608 |
| $KCl_{0.8}$ $KBr_{0.2}$ mixed crystal | | | 3.16397 |
| ZnN doped $KCl_{0.2}KBr_{0.8}$ mixed crystal | | | 3.25936 |
| ZnN doped $KCl_{0.8}$ $KBr_{0.2}$ mixed crystal | | | 3.17541 |

## 4. FTIR Analysis

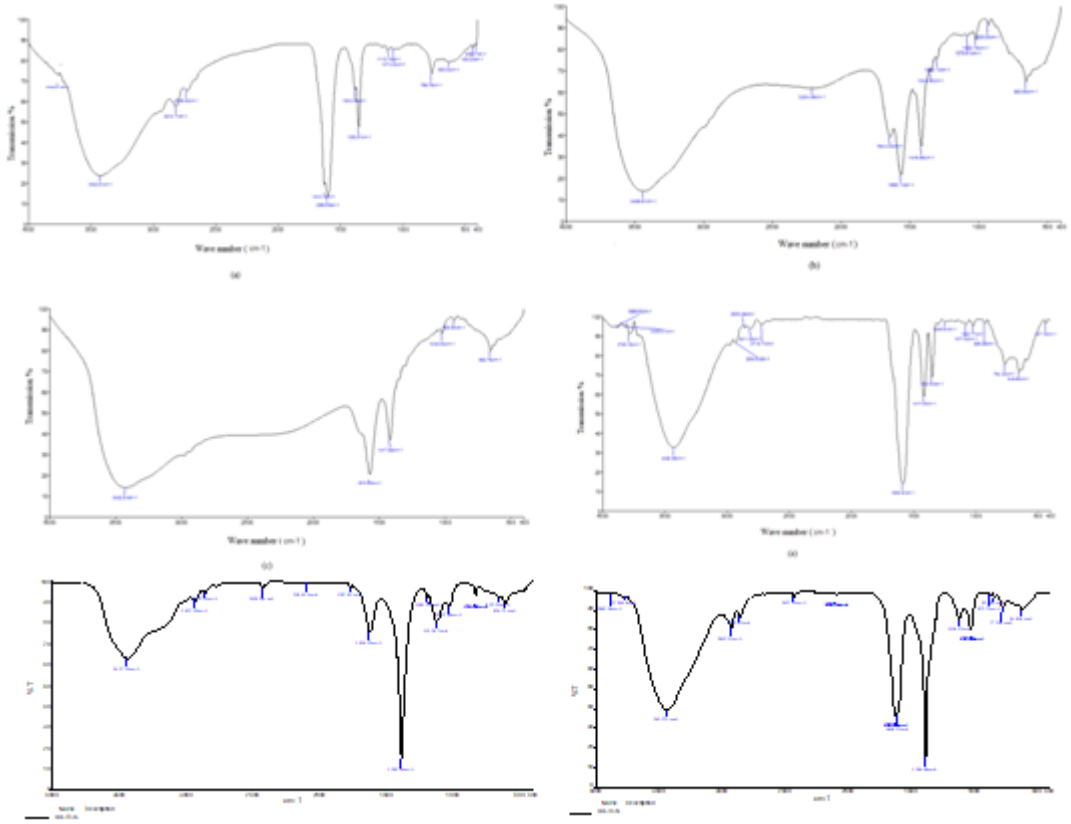


**Table 8:** Wave number and force constant of all the grown crystals

| System | Wave Number $cm^{-1}$ | Absorption maximum wave Number $cm^{-1}$ | Force Constant $X10^2$ N/cm |
|---|---|---|---|
| pure KCl crystal | 2921.83 | 3422.51 | 6.3444 |
| pure KBr crystal | 2909.84 | 3436.61 | 6.3464 |
| $KCl_{0.2}$ $KBr_{0.8}$ mixed crystal | 2921.8 | 3422.54 | 6.3454 |
| $KCl_{0.8}$ $KBr_{0.2}$ mixed crystal | 2918.01 | 3426.99 | 6.345 |
| ZnN doped $KCl_{0.2}$ $KBr_{0.8}$ mixed crystal | 2927.55 | 3437.22 | 6.3527 |
| ZnN doped $KCl_{0.8}$ $KBr_{0.2}$ mixed crystals | 2925.37 | 3435.71 | 6.3511 |

The fundamental bands of relatively heavy metal ions, torsional modes, and other low-frequency excitations are typically located in the far-IR. Strong fundamental vibrations of the aluminosilicate framework of minerals and glasses, as well as the principal vibrational modes of most molecular species (e.g., Si-O, C-O, S=O, and P-O) are located in the mid-IR.[30]

FTIR has made energy limited region more accessible. It has made the middle infrared (400-4,000$cm^{-1}$) also more useful. It is an important technique in organic chemistry. It is an easy way to identify the presence of certain functional groups in a molecule.[31]

FTIR spectra have been recorded by using IFS BRUKKER 66V spectrophotometer in the range of 400$cm^{-1}$-4000$cm^{-1}$ in order to find the presence of various functional groups. The FTIR spectrum are taken for all the samples to study the spectroscopic properties of the grown crystals.

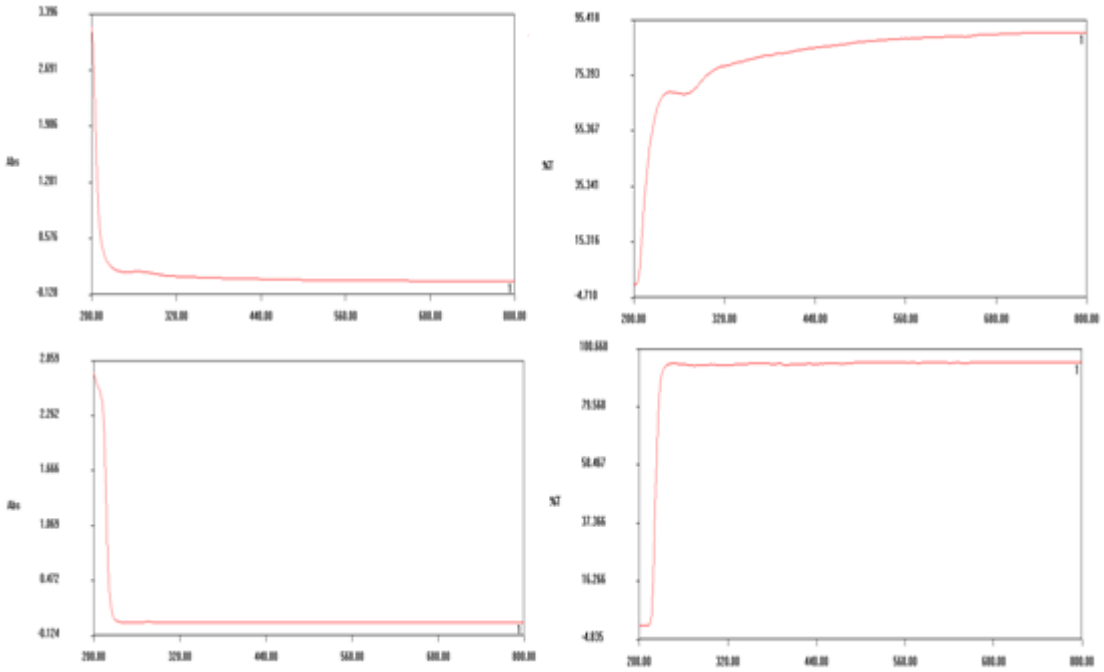
Force constant of the grown crystals were determined from the FTIR transmission data using the formula,

$$\nu = 5.3 \times 10^{-2}\sqrt{kf}$$

Where ν is the wave number corresponding to the absorption maximum, kf is the force constant and μ is the reduced mass.

The force constant value depends on the absorption maximum wave number. The force constant increases, when the absorption maximum wave number increases. The force constant values are 6.3444$X10^2$ N/cm, 6.3464 $X10^2$ N/cm, 6.3454 $X10^2$ N/cm, 6.3446 $X10^2$ N/cm, 6.3450 $X10^2$ N/cm, 6.3463 $X10^2$ N/cm, 6.3527 $X10^2$ N/cm, 6.3511 $X10^2$ N/cm for pure, mixed and ZnA doped mixed crystals.

## 5. UV- Vis Spectroscopy



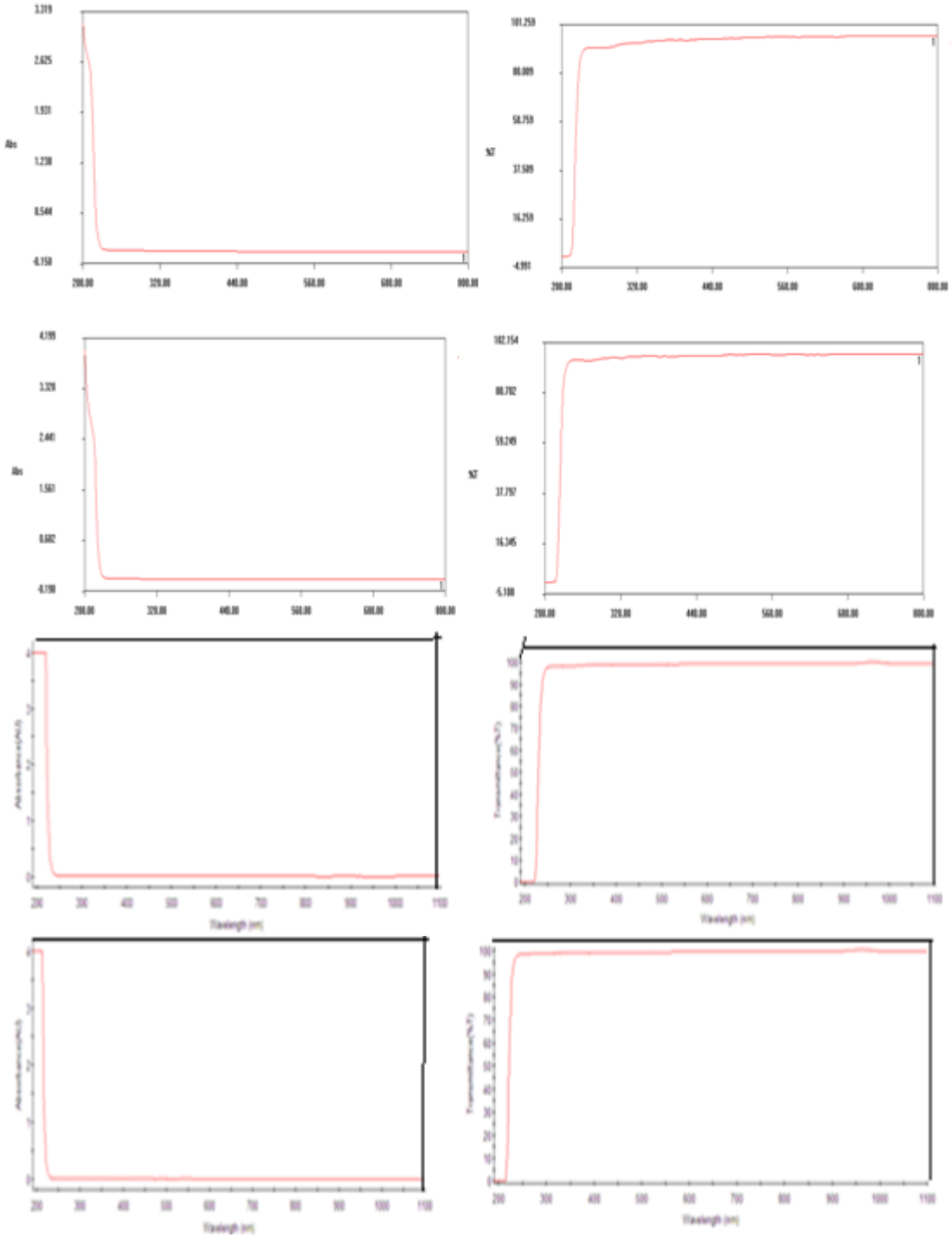
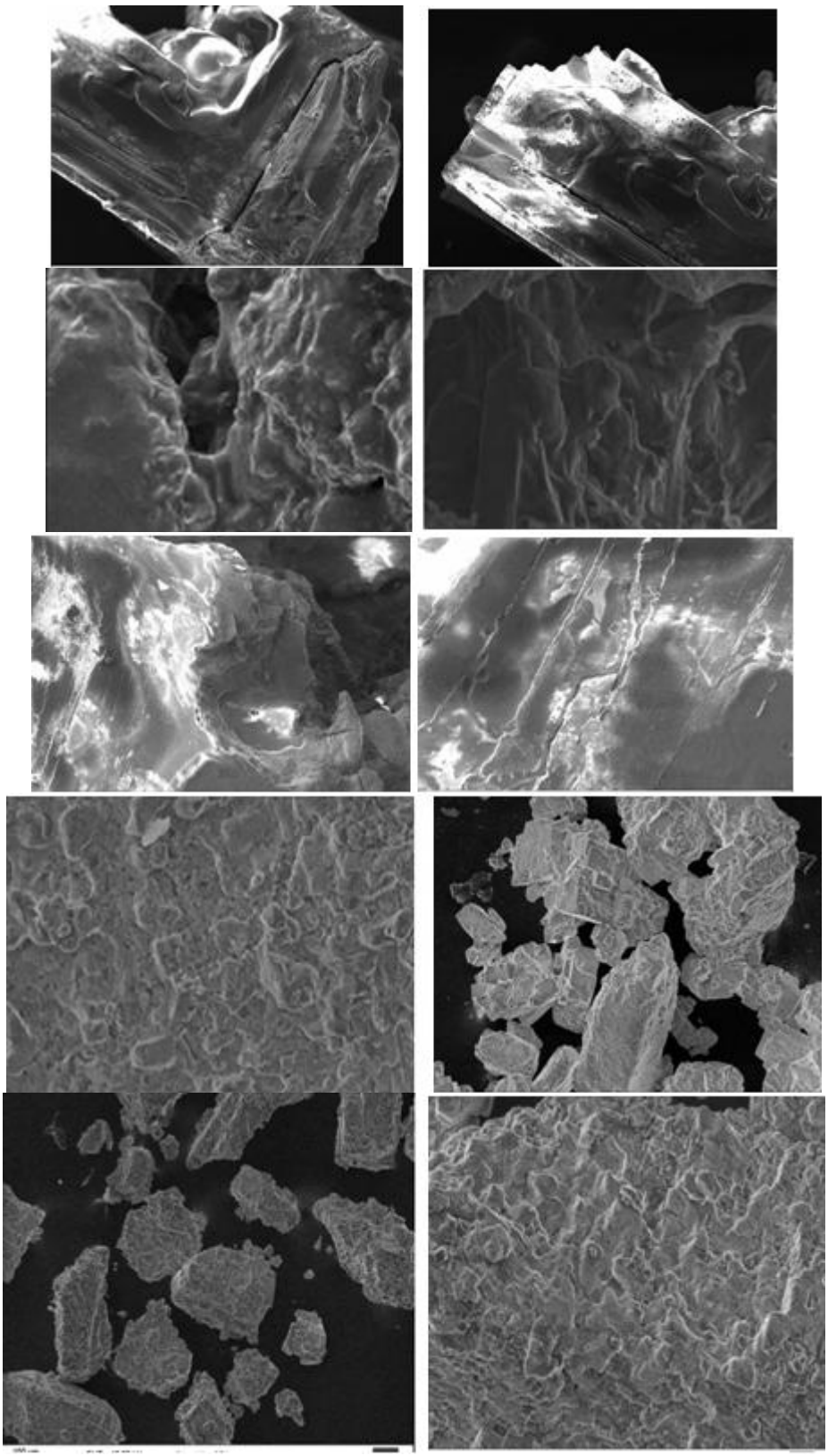


**Table 9:** Band gap energy of all the grown crystals

| System | $\lambda_{cut}$ nm | Eg eV |
|---|---|---|
| Pure KCl crystal | 218.5 | 5.6788 |
| Pure KBr crystal | 222 | 5.59622 |
| $KCl_{0.2}$ $KBr_{0.8}$ mixed crystal | 229 | 5.4252 |
| $KCl_{0.8}$ $KBr_{0.2}$ mixed crystal | 235 | 5.2867 |
| ZnN doped $KCl_{0.2}$ $KBr_{0.8}$ mixed crystal | 240 | 5.1765 |
| ZnN doped $KCl_{0.8}$ $KBr_{0.2}$ mixed crystals | 250 | 4.9695 |

From the spectra of lower cutoff wavelength lies nearly 218.5nm, 222nm, 229nm, 240nm, 250nm for pure and ZnS doped $KCl_xBr_{1-x}$ mixed crystals. It has low optical absorption and high transmittance (100%).The transmittance absorbance percentages of pure and mixed crystals are almost same. The absence of absorption in the visible region indicates that the grown crystals can be used for opto-electronic application.

## 6. Scanning Electron Microscope Analysis



The number of grains per square inch at 100X magnification

$$n=2^{G-1}$$

$$\log n=\log 2^{G-1}$$

$$\log n=(G-1)\log 2$$

$$G=(\log n/\log 2)+1$$

Where G is the American Society for Testing and Materials (ASTM grain size number.

## 7. Conclusion

Good optical quality of single crystals of pure and ZnN doped KClxBr1-x mixed crystal were grown by slow evaporation solution growth method at room temperature. The powder X-Ray diffraction study confirms the lattice parameter value. The lattice parameters have been found by single crystal X-ray diffraction technique. X-Ray diffraction

studies confirmed that pure and ZnS doped KClxBr1-x mixed crystalcrystals were crystallized in cubic system.

The FTIR spectrum reveals that the various functional groups present in the grown crystal. The FTIR studies assign vibrational frequencies. From the UV spectrum, the zinc nitrate crystal is found to be transparent in the UV region and it could be a useful candidate for optoelectronic applications in visible and infrared region. The morphology of all the grown samples were analysed through SEM images.

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