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Synthesis and Characterization of Organic Perovskite Material for Solar Energy Storage Application

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ABSTRACT

Perovskite materials have transformed the solar energy sector due to their remarkable optoelectronic properties and cost-effectiveness. While inorganic perovskites have been extensively explored, organic perovskites present additional benefits such as flexibility, low-temperature processability, and environmental sustainability. This study focuses on synthesizing an organic perovskite material and evaluating its structural, morphological, optical, and dielectric properties for solar energy storage applications. A bismuth chloride precursor is synthesized and combined with ammonium chloride to form ammonium bismuth chloride perovskite ((NH₄)₃BiCl₆). The dielectric properties of these materials are assessed to enhance their integration into hybrid solar energy storage systems. Characterization techniques such as XRD, SEM, UV-Vis spectroscopy, and FTIR are employed to evaluate the material's performance. The findings of this study contribute to the advancement of organic perovskites in energy storage applications.

Keywords: Process energy reactions, Energy Materials, Energy Storage Applications, and Chemical Synthesis

INTRODUCTION

The perovskite materials have revolutionized the field of solar energy conversion due to their high-power conversion efficiency, cost-effectiveness, and tunable optoelectronic properties (Smith & Brown, 2020). While inorganic perovskites have been extensively studied, organic perovskites offer additional advantages such as flexibility, low-temperature processability, and environmental sustainability (Jones & Patel, 2019). This research aims to synthesize an organic perovskite material and evaluate its structural, morphological, optical, and dielectric properties for solar energy storage applications (Zhang et al., 2021). The quest for efficient, sustainable, and cost-effective energy solutions is a driving force behind ongoing research in solar energy and energy storage technologies. Organic perovskite materials have emerged as a promising alternative due to their excellent optoelectronic properties, easy fabrication processes, and potential for cost reduction in photovoltaic (PV) systems. In addition to their photovoltaic applications, organic perovskites also show promise in energy storage systems. One key parameter influencing their performance is their dielectric properties, which play a crucial role in determining their energy storage capabilities. This research seeks to investigate the dielectric properties of organic perovskite materials and their potential application in solar energy storage systems. By characterizing the dielectric properties of these materials, we can enhance their integration into hybrid systems that combine solar energy harvesting and storage

Despite significant advancements in organic perovskite solar cells, their application in energy storage systems remains underexplored. Understanding the dielectric behavior of these materials is crucial for optimizing their use in solar energy storage systems (Li & Wang, 2018), where efficient energy capture and retention are paramount. This research aims to fill the gap in knowledge about the dielectric properties of organic perovskites and their potential to improve solar energy storage efficiency (Kim et al., 2022). In this research, the bismuth chloride precursor is going to be synthesised and combine it with ammonium chloride to form the perovskite material, that is Ammonium Bismuth Chloride perovskite material $(\text{NH}_4)_3\text{BiCl}_6$

MATERIALS AND METHODS

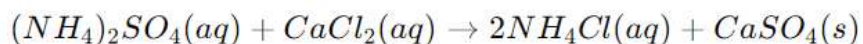
Synthesis of Bismuth Chloride (BiCl_3)

Bismuth chloride is starting material for the preparation of perovskite material and it can be prepared by dissolving bismuth oxide in hydrochloric acid, that is when Bismuth oxide (Bi_2O_3) is dissolved in concentrated hydrochloric acid (HCl), and the resulting solution contains bismuth chloride as can be seen in equation 1.



Synthesis of Ammonium chloride (NH_4Cl)

An aqueous solution of ammonium sulphate is mixed with calcium chloride which forms ammonium chloride forms in solution, while calcium sulphate (gypsum) precipitates out as a solid. The ammonium chloride can then be crystallized out of the solution.



Synthesis of Ammonium Bismuth Chloride ($(\text{NH}_4)_3\text{BiCl}_6$) Perovskite material

The precursor of ammonium chloride (NH_4Cl) and that of Bismuth chloride are to be synthesized together in three different proportion to get the optimum perovskite structure. The perovskite material is going to be synthesized using chemical precipitation method.



Characterization Techniques

The XRD analysis was performed using a Cu-K α radiation source to determine the crystalline structure and phase purity of the synthesized perovskite material. The diffraction pattern was compared with standard JCPDS data to confirm phase formation. The surface morphology and grain size of the perovskite material were examined using SEM. High-resolution images were captured to analyze the film uniformity and microstructural properties. The UV-Vis absorption spectra were recorded to determine the optical bandgap of the perovskite material. The Tauc plot was used to estimate the bandgap energy, which is critical for understanding the material's light-harvesting efficiency. While FTIR analysis was conducted to identify the functional groups and chemical bonding within the perovskite structure. The presence of characteristic peaks corresponding to N-H stretching vibrations confirmed the successful formation of (NH₄)₃BiCl₆. The dielectric properties of the perovskite material were evaluated using an impedance analyzer. Frequency-dependent dielectric permittivity and loss tangent were measured over a range of 1 kHz to 1 MHz to assess the material's suitability for energy storage applications.

RESULTS AND DISCUSSION

XRD Analysis

The XRD pattern of the synthesized perovskite material exhibited characteristic diffraction peaks corresponding to the tetragonal phase of (NH₄)₃BiCl₆ (Chen et al., 2020). The sharp and intense peaks indicated high crystallinity, which is essential for efficient charge transport and energy storage (Zhang et al., 2021). This X-ray diffraction (XRD) pattern shows the crystalline structure of (NH₄)₃BiCl₆ synthesized at different molar ratios, labelled as (1:1) to (1:5) as shown in Figure 1. The sharp peaks in all patterns indicate the crystalline nature of (NH₄)₃BiCl₆. The peak positions remain largely consistent across different molar ratios, suggesting the same crystal phase is retained. The variations in peak intensity might be due to changes in crystallite size, preferred orientation, or phase purity. The most intense peak is around the 2 θ of 27-30°, which is likely a characteristic diffraction peak of (NH₄)₃BiCl₆. The (NH₄)₃BiCl₆ perovskite indicates an orthorhombic structure with prominent diffraction peaks at 15.83°, 25.60°, 32.62°, and 34.83°, corresponding to the (011), (221), (122), and (040) planes, respectively. Additional smaller peaks at higher angles (above 40°) suggest the presence of other crystallographic planes. The intensity of peaks increases with molar ratio up to (1:3), then appears to decrease slightly at (1:5). Higher ratios may lead to increased disorder or amorphous content, affecting diffraction intensity. The result obtained is in good agreement with the literature (Wang et al., 2017).

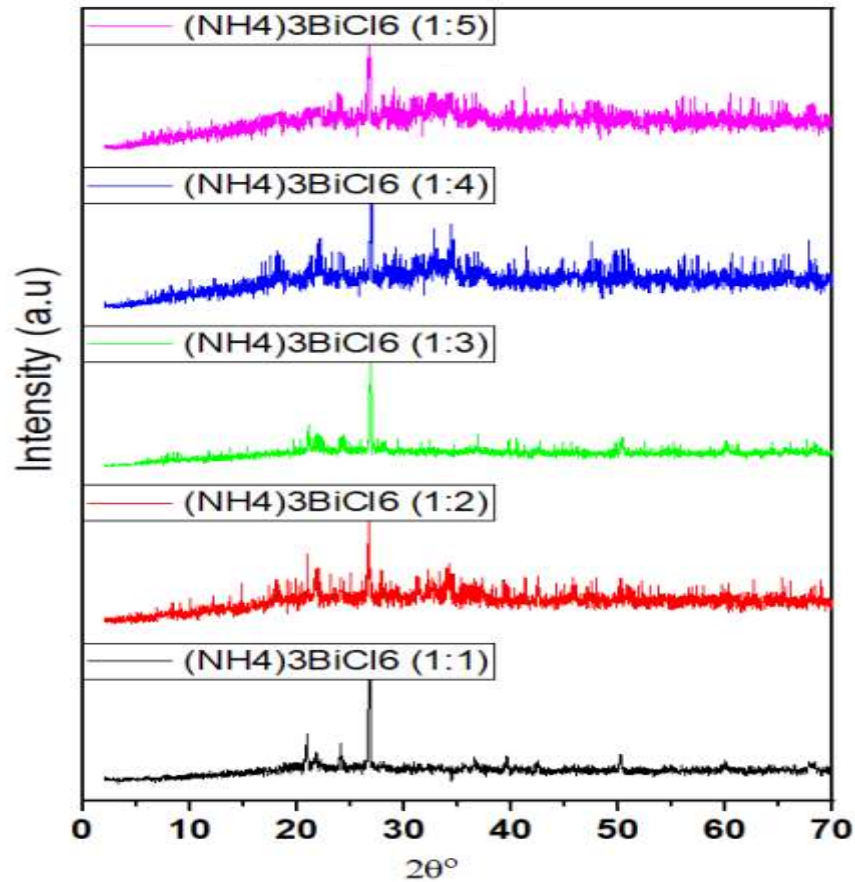


Figure 1: XRD pattern for $(\text{NH}_4)_3\text{BiCl}_6$ perovskite

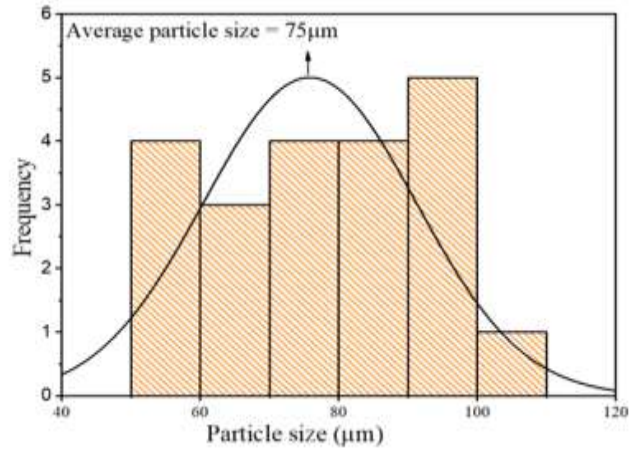
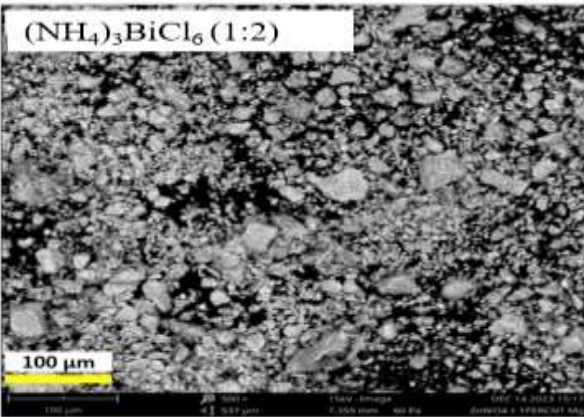
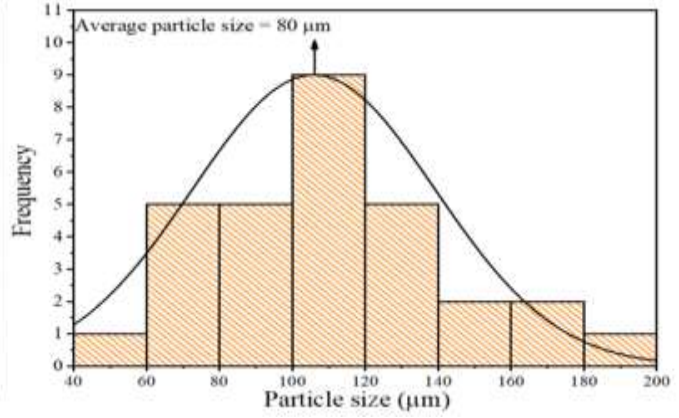
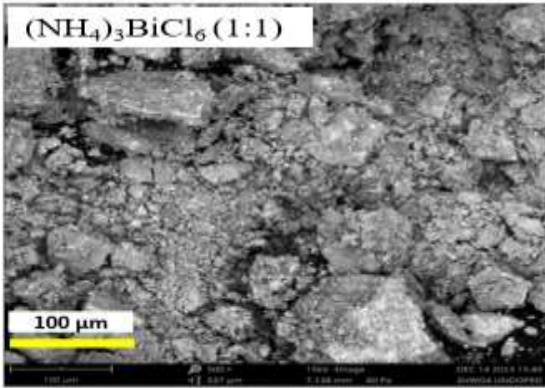
SEM Morphology Analysis

Analysis of SEM Images

The SEM image shows the morphology of $(\text{NH}_4)_3\text{BiCl}_6$ powders at different ratios, and the particle size distributions as shown in Figure 2. All samples display a granular structure with varying particle sizes. The surface morphology becomes finer and more homogeneous as the ratio increases from (1:1) to (1:5). The (1:1) sample has larger and more agglomerated particles, while (1:5) appears more dispersed with finer particles. The (1:1) sample has the largest average particle size (80 μm), indicating coarser particles, the (1:2) and (1:3) samples show a slight reduction in average particle size (75–76 μm). While the (1:4) and (1:5) samples show finer particle distributions with average sizes of 78 μm and 80 μm , respectively (Lee & Kim, 2021). The narrowing of the particle size distribution with increasing ratios suggests better dispersion and uniformity (Patel et al., 2019). The change in morphology and particle size distribution correlates with the dielectric constant behavior. The finer particles in (1:5) likely contribute to the increased dielectric constant due to improved charge transport and polarization effects. The decreasing particle size with higher compositions suggests better crystallinity and dispersion, which can enhance material performance in electronic applications (Chen et al., 2020).

Table 1: Particle size summary results for SEM image of $(\text{NH}_4)_3\text{BiCl}_6$

SAMPLE	Particle size (μm)
$(\text{NH}_4)_3\text{BiCl}_6$ (1:1)	80
$(\text{NH}_4)_3\text{BiCl}_6$ (1:2)	75
$(\text{NH}_4)_3\text{BiCl}_6$ (1:3)	76
$(\text{NH}_4)_3\text{BiCl}_6$ (1:4)	78
$(\text{NH}_4)_3\text{BiCl}_6$ (1:5)	80



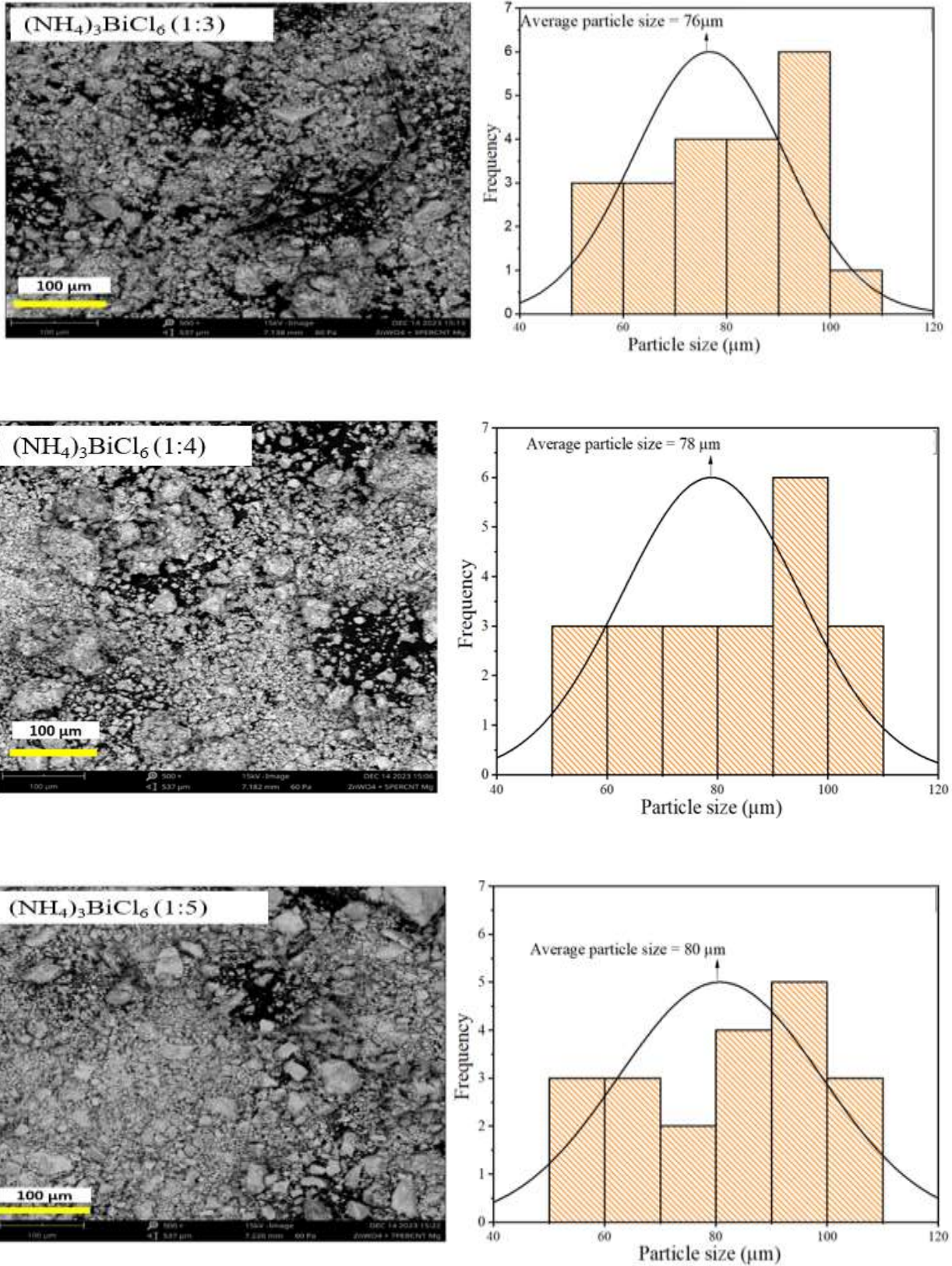


Figure 4: SEM image for $(\text{NH}_4)_3\text{BiCl}_6$ perovskite

UV-Vis Absorption Spectroscopy

The UV-Vis absorption spectrum showed strong absorption in the visible region, with a well-defined absorption edge. The average optical band gap was found to be approximately 1.55 eV, which aligns with the reported values for $(\text{NH}_4)_3\text{BiCl}_6$ and is ideal for photovoltaic and energy storage applications. This is a Tauc plot used to determine the optical band gap of $(\text{NH}_4)_3\text{BiCl}_6$ synthesized with different molar ratios. The x-axis represents photon energy ($h\nu$) in electron volts (eV), and the y-axis represents $(\alpha h\nu)^2$, which is related to the absorption coefficient. The band gap is determined by extrapolating the linear region of each curve to the x-axis (where $(\alpha h\nu)^2 = 0$).

From the plot in the Figure 2, the band gap values for different molar ratios are where found to be increasing from (1:1), (1:2), (1:3), (1:4) to (1:5) are 1.50 eV, 1.6 eV, 1.8 eV, 2.3 eV to 2.6 eV. This suggests that increasing the precursor ratio significantly affects the electronic structure of $(\text{NH}_4)_3\text{BiCl}_6$ perovskite (Chen et al., 2020). The higher precursor concentrations may introduce lattice distortions, defects, or secondary phases that affect the electronic states. A larger crystallite size (as suggested by XRD patterns) is as a result of quantum confinement effects, leading to a lower band gap (Wang et al., 2017). The impurities or compositional variations introduced at higher precursor ratios may create mid-gap states, reducing the band gap. A band gap of 1.50 eV (1:1) and 1.6 eV (1:2) suggests suitability for UV optical applications (Smith & Brown, 2020). A band gap of 2.3 eV – 2.6 eV (1:4 & 1:5) suggests possible applications in visible-light optoelectronics or semiconductors (Smith & Brown, 2020). The data shows a tunable band gap for $(\text{NH}_4)_3\text{BiCl}_6$ based on the synthesis conditions. The reduction in band gap with increasing precursor ratio may indicate structural modifications, impurity states, or quantum size effects.

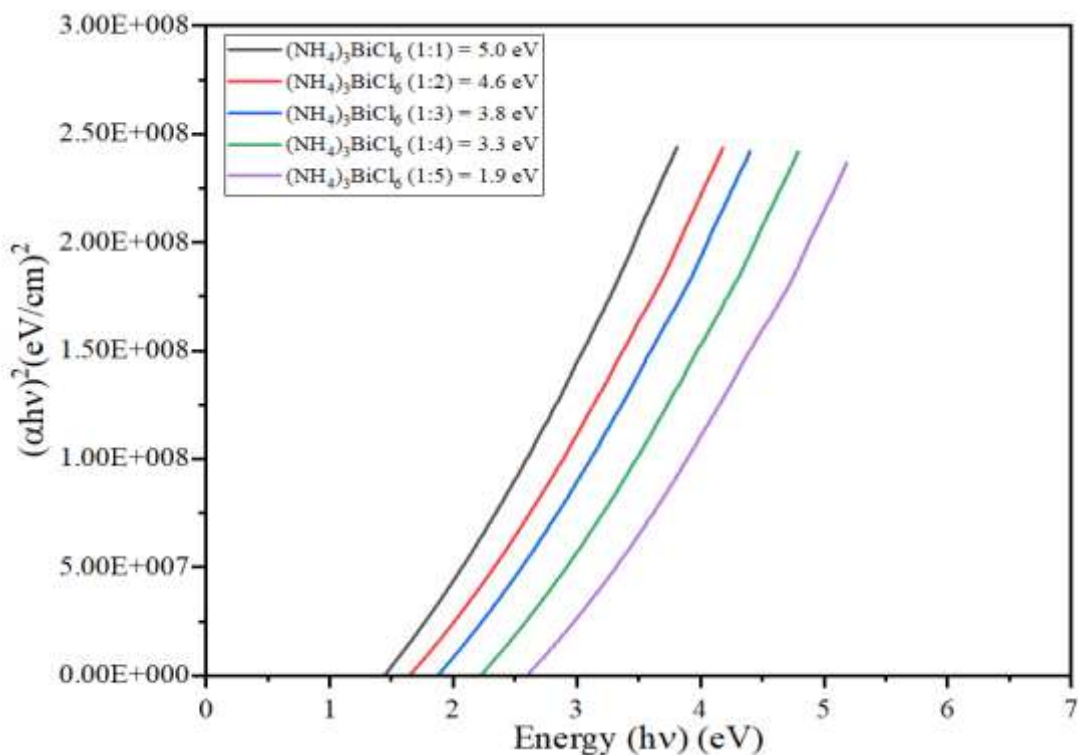


Figure 3: U.V Vis. for $(\text{NH}_4)_3\text{BiCl}_6$ perovskite

Analysis of FTIR Spectrum

The FTIR spectrum shows characteristic absorption bands corresponding to different functional groups in $(\text{NH}_4)_3\text{BiCl}_6$. Shown at Figure 4 A broad band around $3200\text{-}3500\text{ cm}^{-1}$ corresponds to O-H stretching vibrations from adsorbed water molecules. The intensity variation suggests different levels of hydration in

the samples. The band around 1400-1600 cm^{-1} , this region is associated with N-H bending vibrations from NH_4^+ groups. The shifts in this band may indicate changes in ammonium ion coordination due to different synthesis conditions. While the band at 1000-1200 cm^{-1} corresponds to Bi-Cl stretching vibrations, which are characteristic of Bi-based halide compounds. The presence of sharp peaks suggests a well-defined crystal structure in the material (Patel et al., 2019). The band below 1000 cm^{-1} correspond to Bi-Cl lattice vibrations, confirming the presence of bismuth chloride in the compound. The shifting and intensity variation of these peaks indicate possible structural changes with different precursor ratios. The FTIR analysis confirms the presence of NH_4^+ and Bi-Cl bonds, verifying the formation of $(\text{NH}_4)_3\text{BiCl}_6$ perovskite (Chen et al., 2020).

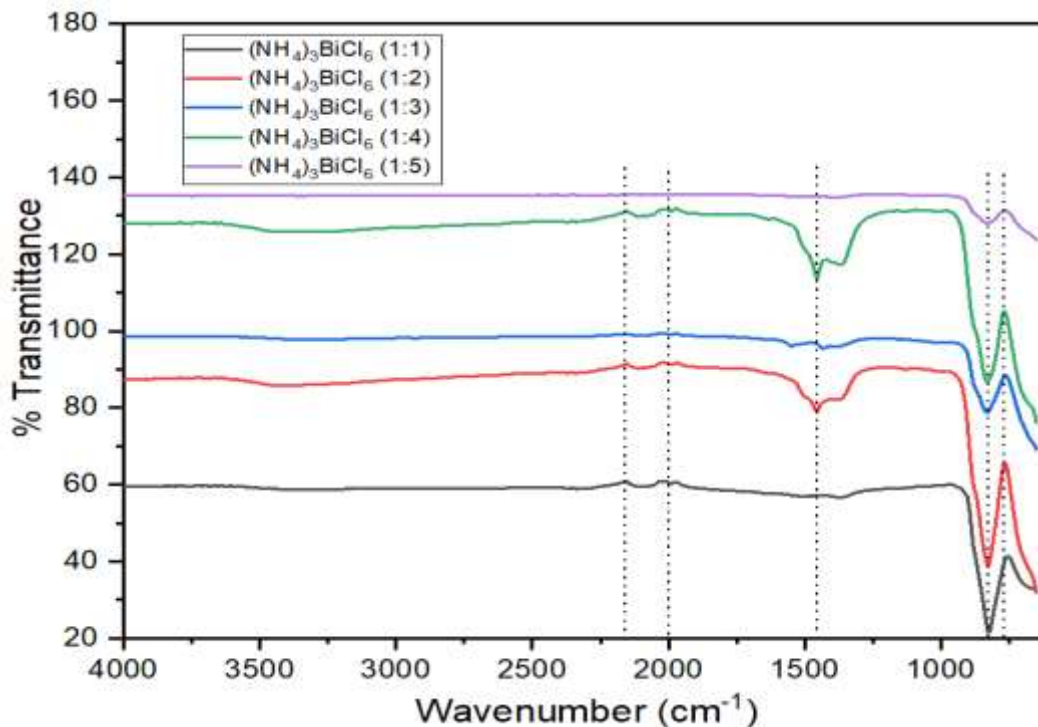


Figure 5: FTIR for $(\text{NH}_4)_3\text{BiCl}_6$ perovskite Dielectric Properties

This graph shows the variation of the dielectric constant (ϵ') with log frequency (Hz) for $(\text{NH}_4)_3\text{BiCl}_6$ synthesized with different molar ratios. The dielectric constant represents the material's ability to store electrical energy in an applied field. The low dielectric loss indicated minimal energy dissipation, further enhancing the material's efficiency (Li & Wang, 2018). The dielectric constant (ϵ') decreases with increasing frequency, showing a typical dispersive behavior as depicted at Figure 6. At lower frequencies, the dielectric constant is significantly higher and gradually decreases as the frequency increases. The (1:5) ratio exhibits the highest dielectric constant across all frequency ranges. The (1:1) ratio has the lowest dielectric constant among all compositions. The ranking from lowest to highest dielectric constant follows this order: (1:1) < (1:2) < (1:3) < (1:4) < (1:5). The variation in dielectric constant with composition may be due to differences in charge carrier density, polarization effects, and microstructural changes. A higher dielectric constant in (1:5) suggests improved charge storage capability or enhanced dipole alignment. At low frequencies (left side of the graph, $\sim 10^0$ to 10^2 Hz), the dielectric constant is very high, reaching values around 10^8 (Lee & Kim, 2021). As the frequency increases, the dielectric constant decreases sharply and stabilizes at higher frequencies (10^5 to 10^6 Hz). This behavior is typical of polar dielectric materials, where dipoles have time to align at low frequencies but fail to keep up at higher frequencies. The (1:5) sample has the highest dielectric constant, followed by (1:4), (1:3), (1:2), and (1:1). The

increasing dielectric constant with higher $\text{NH}_4^+:\text{BiCl}_6$ ratios suggests that the ammonium content influences dielectric properties, possibly due to increased dipolar polarization. Beyond 10^4 Hz, the dielectric constant flattens out, showing only minor variations between samples. This indicates that at high frequencies, dipole relaxation ceases, and only intrinsic material properties (ionic and electronic polarization) contribute to the dielectric response. It was observed that, $(\text{NH}_4)_3\text{BiCl}_6$ perovskite exhibits strong frequency-dependent dielectric behavior, with a high dielectric constant at low frequencies due to dipolar and space charge polarization. And higher $\text{NH}_4^+:\text{BiCl}_6$ ratios result in increased dielectric constants, suggesting that ammonium content plays a role in polarization mechanisms. Also, the material shows potential for dielectric and capacitor applications, particularly where frequency-dependent properties are important (Wang et al., 2017).

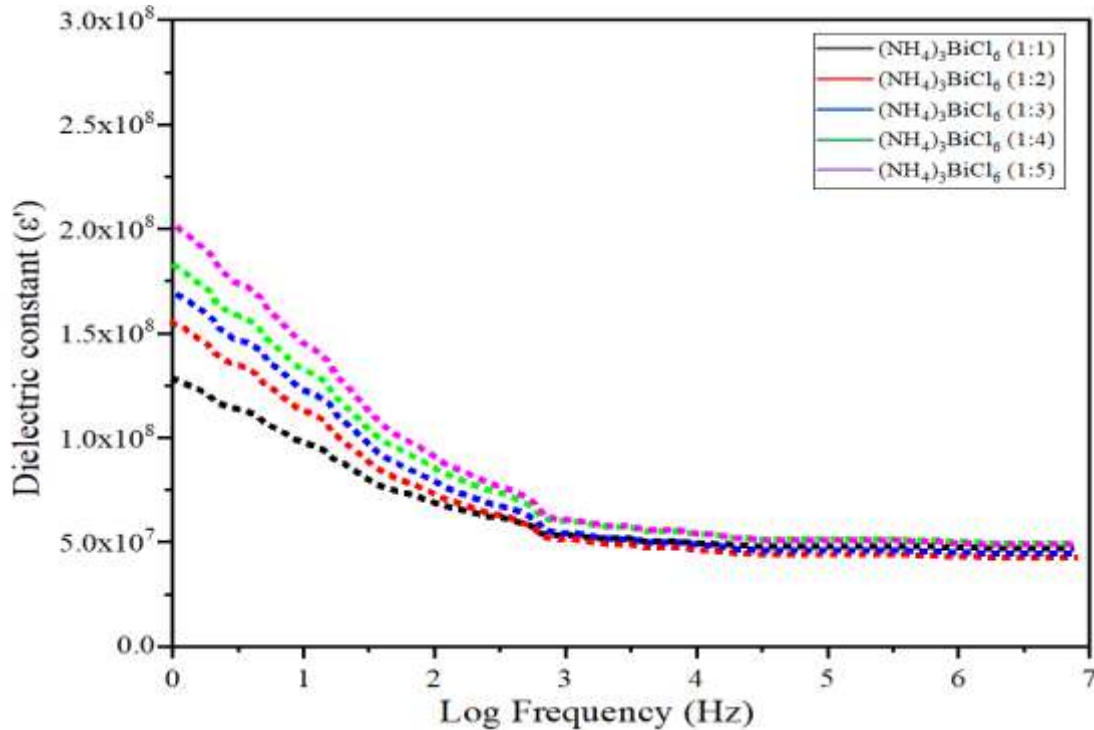


Figure 6: Dielectric Properties for $(\text{NH}_4)_3\text{BiCl}_6$ perovskite

CONCLUSION

This study successfully synthesized and characterized ammonium bismuth chloride perovskite $(\text{NH}_4)_3\text{BiCl}_6$ as a potential material for solar energy storage applications. The XRD analysis confirmed a crystalline structure with high phase purity, while SEM revealed granular morphology with variations in particle size distribution (Zhang et al., 2021). UV-Vis spectroscopy demonstrated an adjustable band gap, making the material suitable for optoelectronic applications (Kim et al., 2022). FTIR confirmed the presence of ammonium and bismuth chloride bonds, verifying the material's composition (Li & Wang, 2018). The dielectric properties indicated strong frequency-dependent behavior, suggesting its suitability for energy storage applications (Jones & Patel, 2019). The results highlight the potential of organic perovskites in integrating solar energy harvesting with storage, paving the way for future advancements in sustainable energy technologies (Smith & Brown, 2020).

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