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Structural and Dielectric Properties of Nanocrystalline Barium Rare-Earth Antimonates Ba_2RESbO_6 (RE = La, Ho, Yb, Tm) Perovskites

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Abstract— Barium rare-earth antimony oxides (Ba_2RESbO_6) are materials of fundamental and technological importance due to versatility in physical and chemical properties. Nanocrystals of Ba_2RESbO_6 (RE = La, Ho, Yb, Tm) perovskites is synthesized using an optimized combustion process, their crystal structure, and dielectric properties are summarized in this article. Crystal structures are analyzed from calculated and experimental IR spectra as well as Rietveld analysis of X-ray diffraction measurements. The IR spectrum is calculated using the B3LYP/LANL2DZ level of density functional theory (DFT). The studies show that La has crystallized in the monoclinic whereas the others in cubic structures. The dielectric constant and loss factors of the materials were studied up to 5 MHz at room temperature. Loss factors of some of these materials are the lowest reported for a double perovskites ceramic compound at 5 MHz frequency at room temperature, the origin of which is possibly due to the chemical purity of the materials because of the atomic level mixing achieved through a combustion process.

Index Terms—complex perovskites, combustion synthesis, modeling, dielectrics, superconductivity

I. INTRODUCTION

Due to their wide array of physical properties and flexibility to generate a large number of materials, complex perovskites have been extensively investigated [1-6]. Ideal perovskites, with stoichiometry ABO_3 , where A is a 12-fold oxygen coordinated cation and B is a 6-fold oxygen coordinated cation, are cubic and their charges are aligned. The BO_6 octahedral net works, which constitute the extended perovskite structure, are perfect with 90° angles and with six equal B–O bonds. i.e, each B ion is surrounded by equidistant anions. Reducing from ideal cubic symmetry, the unit cell structures of majority of perovskites consists of non-ideal octahedra depending upon the relative sizes of the A, B, and O ions. In general, the wide variety of properties exhibited by perovskite family originates from the deviation of the symmetry of the octahedra. In the perovskite structure, A and B sites can accommodate two or more cations, either homovalent or aliovalent, maintaining the electrical neutrality of the crystal. Such compounds are called complex perovskites, represented by $(AA')(BB')O_6$.

The quaternary double perovskites based on lanthanides (rare-earths), antimony, and barium with a general stoichiometry Ba_2RESbO_6 have attracted considerable interest due to a number of fundamental and technological importance [7]-[10]. The Ba_2RESbO_6 Perovskites combine an alkali metal (Ba), a lanthanide (RE), a semi-metal (Sb), and a non-metal (O) – a combination of them would have interesting physical properties. A number of analogues for these double perovskites with general formula Ba_2REMO_6 , where M represent Nb, Ta, Mo, Ir, Ru, and Bi characterized by a range of crystal structure and properties[11]-[12] have been reported. The Ba_2RESbO_6 has been known since 1965 when Blasse synthesized Ba_2GdSbO_6 [13]-[14]. Later, Casedo et al. synthesized nine other compounds of this group using rare-earths from Pr to Ho excluding Tm [15]. These authors pointed out that Ba_2RESbO_6 have high melting points; therefore, they could be used as refractory materials. They have moderate dielectric constant (ϵ') and low loss tangent ($\tan\delta$) making them attractive dielectrics in microwave electronic circuits [16]. The Ba_2RESbO_6 perovskites are shown to be chemically stable with $YBa_2Cu_3O_{7-\delta}$ (YBCO) superconductors. This property enable Ba_2RESbO_6 as substrates for thin films of YBCO, active superconductor-insulator-superconductor interfaces, superconductor-insulator composites, and also as crucibles for single crystal growth of YBCO [17]. Recently Karunadasa et al showed that Ba_2RESbO_6 materials are model system to study geometric frustration of magnetism in double perovskites [9].

The Ba_2RESbO_6 materials possess an array of crystal structures due to the differences in the relative sizes of RE and Sb ions. The Clausius – Mossotti equation [18],



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$$\epsilon_r = \frac{3V_m + 8\pi\alpha}{3V_m - 4\pi\alpha} \quad (1)$$

Where V_m is the molar volume and α is the total dielectric polarizability, predicts that a variation in crystal structure and molar volume thereby inevitably bring changes in their dielectric properties also. The total polarizability of the Ba_2RESbO_6 materials calculated by considering RE^{3+} and Sb^{5+} clearly shows the effect of their crystal structure on dielectric properties (Figure 1). The dielectric properties of the Ba_2RESbO_6 for materials other than when $RE = Ho, Tm,$ and Yb are reported in the open literature. To the best of our knowledge, little study was undertaken either on crystal structure or on dielectric properties for nanostructured Ba_2RESbO_6 ($RE = La, Ho, Tm,$ [4] and Yb) as well as correlating their dielectric properties and crystal structure. Purpose of the present study is to systematically analyze the crystal structure and dielectric properties of the above complex perovskites.

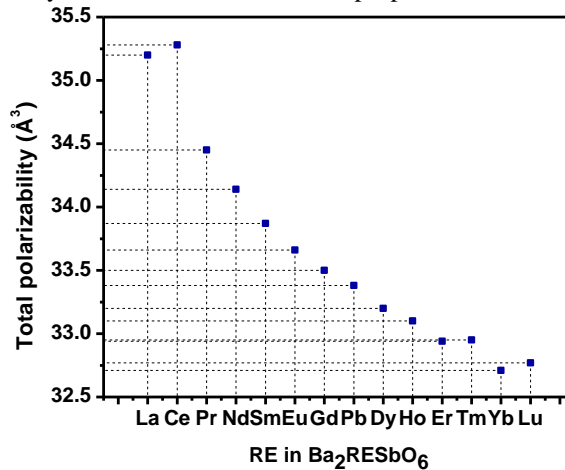


Fig 1: Variation of polarizability of lanthanides with respect to atomic number

II. EXPERIMENTAL DETAILS

An optimized combustion method was employed to synthesize the materials [5]. Briefly, aqueous solution containing ions of rare-earth (La, Ho, Yb, and Tm), Ba, and Sb were prepared from high purity RE_2O_3 (99.9%, Alfa Aesar U.S.A), $Ba(NO_3)_2$ (99.9%, CDH, India) and Sb_2O_3 (99.9, Merck) using the procedure described elsewhere. The RE_2O_3 and Sb_2O_3 were dissolved in nitric and tartaric acids, respectively. The amount of citric acid was calculated based on the two-third of the total valence of the cations as reported recently. The calculated amount of citric acid was added to the solution containing the metal ions to get a precursor complex. The oxidant fuel ratio of the system was adjusted by using nitric acid and ammonium hydroxide; and the ratio was kept at unity. The solution containing the complex precursor mixture at a pH of ~ 7.0 was heated using a hot plate at $\sim 250^\circ C$ in a ventilated fume hood. The solution boils on heating and undergoes dehydration and decomposition leading to a smooth deflation and foams. The foam then ignites by itself on persistent heating giving voluminous and fluffy product of combustion. The details of powder characteristics are in our previous paper [5].

The as-prepared Ba_2RESbO_6 were mixed with 5% polyvinyl alcohol, dried, and pressed in the form of cylindrical pellet of ~ 14 mm diameter and ~ 2 mm thickness at a pressure ~ 350 MPa. The pellets were then sintered at $1450^\circ C$ for 4 h. Microstructure of the materials were studied by scanning electron microscopy. Crystal structure of the materials were studied by combining ab-initio quantum chemical simulations of cluster models, Fourier-transform infrared spectroscopy (FTIR) (Thermo-Nicolet Avatar 370) in the range $400-4000\text{ cm}^{-1}$ using KBr pellet method, and X-ray diffraction (XRD) technique (Bruker D-8, Netherlands; Nickel filtered CuK_α radiation).

Dielectric properties such as dielectric constant (ϵ_r) and loss factor ($\tan\delta$) of the sintered Ba_2RESbO_6 at low (radio) frequencies were estimated using simple using an LCR meter (HIOKI3532-50) and the dielectric constant was calculated from the capacitance of the parallel plate capacitor formed by the sample. Sintered circular discs of Ba_2RESbO_6 with diameter 10 mm and radius 1mm was used for dielectric measurements. Silver electrodes were pasted on both sides to measure the capacitance measurements.

IR spectroscopy of Ba₂RESbO₆

The Ba₂RESbO₆ could be viewed as a network of corner sharing REO₆ and SbO₆ octahedra; the dodecahedral interstice thus formed is filled by the Ba atom. Therefore, the stretching and bending modes of the REO₆ and SbO₆ octahedra of the Ba₂REMO₆ unit cell usually dominate the IR spectra of complex double perovskites perovskites [19]-[20]. In contrast, Sopracase et al. [21] shown that, based on a comparative study of the phonon modes in two types ABO₃ unit cells, most of phonon modes in these types of unit cells correspond to complex atomic vibrations and significantly different from one another which cannot be assigned only to a given type of vibration, i.e., vibrational, bending, or stretching modes.

In this report, the structural characteristics of the Ba₂RESbO₆ perovskites were studied by calculating the IR spectra of a basic molecular unit using b3lyp/lanl2dz level of DFT using Gaussian program packages [19]. Due to unavailability of the basis sets for rare-earths other than La and similarity of the structural units, i.e., LaO₆ and REO₆, Ba₂LaSbO₆ was used as a model system. Figure 2 shows the optimized structures of LaO₆ and SbO₆ octahedra as well as the complete structural element of Ba₂LaSbO₆.

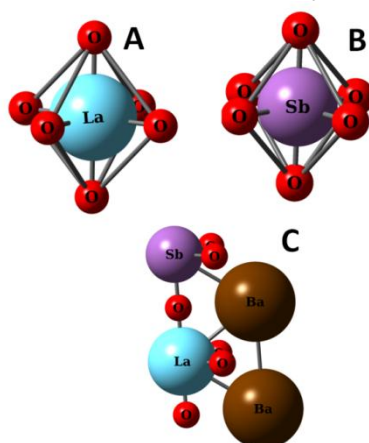


Fig 2: Optimized structures of (A) LaO₆ and (B) SbO₆ octahedra; and (C) a single molecular unit showing the basic structural unit composing the Ba₂RESbO₆ perovskite.

Figure 3 compares the calculated and experimental IR spectrum of the Ba₂LaSbO₆ perovskite and the respective vibrations are summarized in Table 1. The calculations showed symmetrical stretching or bending in the higher wave number region whereas the lower wave number side of the spectrum consists of complex vibrations involving all the atoms in the structure concerned as pointed out by Sopracase et al.[21] Generally frequencies determined from the calculations shifts compared to the experimental one because of the ideality of the structure concerned during the calculation. The band centred at ~470 cm⁻¹ is assigned to the asymmetric bending mode ν₄ (T_{1u}) of the SbO₆ (REO₆) octahedra. The intense band centred at ~620 cm⁻¹ is assigned to the asymmetric stretching mode ν₃ (T_{1u}) of the SbO₆ (REO₆) octahedra. All these four bands merged into a single Gaussian profile indicating the similarity of bending/stretching frequencies of the SbO₆ and REO₆ octahedra. Similar spectral pattern with two strong and well-defined IR bands in the 400-650 cm⁻¹ region has been found in a number of Ba₂RESbO₆ perovskite type materials [14],[22]-[24]. The weak band centred at 1053 cm⁻¹ is assigned to the symmetric stretching mode ν₁ (A_{1g}) of SbO₆ (REO₆) octahedra. The broad band centred at 1430 cm⁻¹ arises from water vapour adsorbed due to ultrafine particle size of the combustion product.

Table 1: Calculated IR data of the structure shown in the Figure 4.2 (C) and assigned modes based on the visual inspection using the Gauss View software.

No.	Frequency	Oscillator Strength	Mode
1	791.03	64.2783	O-RE-O symmetric stretching
2	782.46	30.5803	Symmetric REO ₆ stretching
3	750.32	175.6089	REO ₆ in-plane rotation
4	634.01	340.5842	Symmetric REO ₆ elongation along the corner sharing edges

5	527.38	3.6132	Symmetric SbO ₆ elongation along the corner sharing edges
6	427.85	2.7232	Asymmetric bending and/or stretching of SbO ₆ and REO ₆ octahedra
7	322.08	24.5788	SbO ₆ in-plane rotation
8	257.62	16.8169	Symmetric bending of SbO ₆ and REO ₆ octahedra
9	227.90	48.3305	Complex vibration involving all the atoms in structure
10	195.59	16.1439	Asymmetric bending of SbO ₆ and REO ₆ octahedra

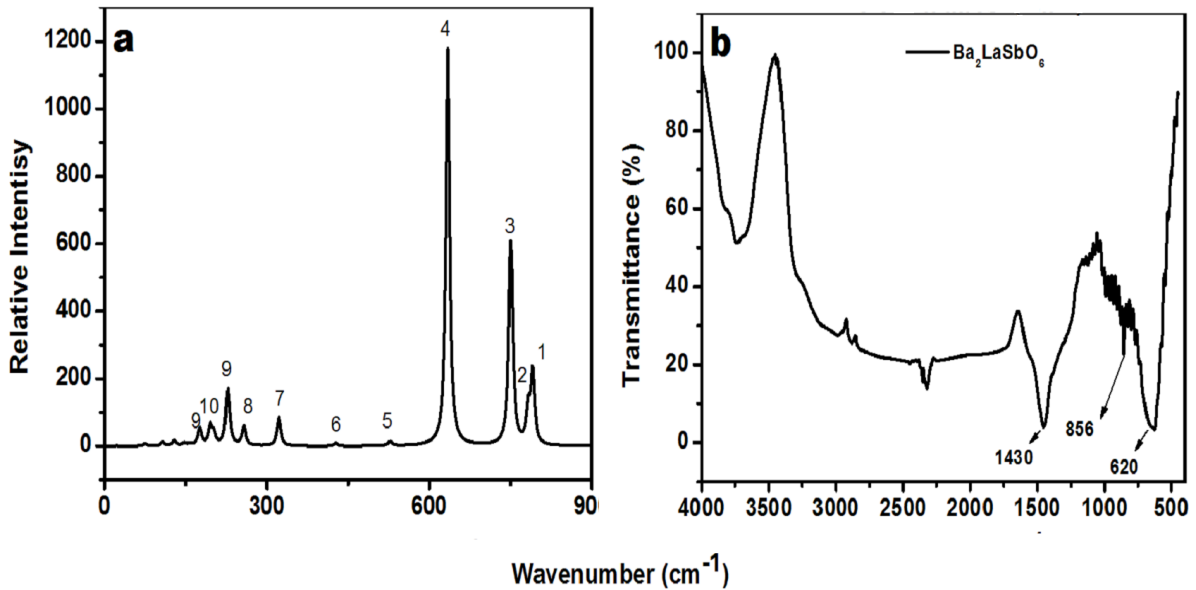


Fig 3: (a) Calculated IR data of the structure of a single molecular unit showing the basic structural unit composing the Ba₂LaSbO₆ perovskite; (b) experimental IR spectra of Ba₂LaSbO₆ perovskite.

Crystal structure of Ba₂RESbO₆ from Rietveld Analysis of XRD

The crystal structure of the materials was studied using sintered samples. Figure 4 shows the SEM images of a typical sintered Ba₂LaSbO₆. The samples were consisted of well formed cuboidal grains having size ~700 nm.

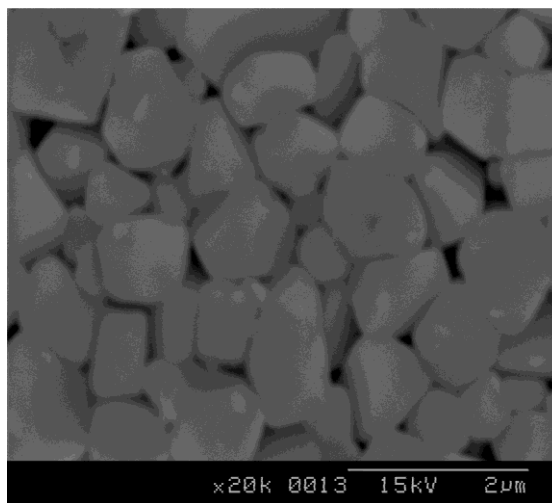


Fig 4: Scanning Electron Microscopic image of sintered Ba₂LaSbO₆



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Table 2: Refined structural model of Ba₂LaSbO₆ and its crystallographic parameters

Atom	site	x	y	z	Numerical criteria ^a	Lattice parameter
Ba ₂ LaSbO ₆						
Ba	4e	-0.00121	0.00014	0.25132	R _p = 0.1525 R _{wp} = 0.2347 S = 1.4	a = 6.0498 Å
La	2d	0.50000	0.00000	0.00000		b = 6.0545 Å
Sb	2c	0.50000	0.00000	0.50000		c = 8.5743 Å
O1	4e	0.25215	0.26492	0.00442		□ □ = 90°
O2	4e	0.30715	-0.26642	-0.03585		□ □ □ □ □ □ □ □
O3	4e	-0.02880	0.54596	0.21642		□ □ □ □ □ □ □ □
Ba ₂ HoSbO ₆						
Ba	8c	0.2500	0.2500	0.2500	R _p = 0.1505 R _{wp} = 0.2172 S = 1.41	a = 8.5818 Å
Ho	4a	0.0000	0.0000	0.0000		
Sb	4b	0.5000	0.0000	0.0000		
O	24e	0.2658	0.0000	0.0000		
Ba ₂ TmSbO ₆						
Ba	8c	0.2500	0.2500	0.2500	R _p = 0.1438 R _{wp} = 0.2014 S = 1.4	a = 8.5318 Å
Tm	4a	0.0000	0.0000	0.0000		
Sb	4b	0.0000	0.0000	0.0000		
O	24e	0.2613	0.0000	0.0000		
Ba ₂ YbSbO ₆						
Ba	8c	0.2500	0.2500	0.2500	R _p = 0.144 R _{wp} = 0.201 S = 1.4	a = 8.4618 Å
Yb	4a	0.0000	0.0000	0.0000		
Sb	4b	0.5000	0.0000	0.0000		
O	24e	0.2625	0.0000	0.0000		

^a The goodness-of-fit indicator S, is $S = R_{wp} / R_e$; $R_e = 'R - expected' = [(N - P) / \sum w_i y_{oi}^2]^{1/2}$

$$R_{wp} = \left[\frac{\sum_k w_k [I_k(obs) - I_k(calc)]^2}{\sum_k w_k I_k(obs)^2} \right]^{1/2}; R_p = \frac{\sum_k |I_k(obs) - I_k(calc)|}{\sum_k I_k(obs)}$$

The tolerance factors (*t*) of Ba₂RESbO₆ with an atomic number larger than that of Ho are ≥ 0.99; and therefore, they adopt the cubic perovskite structure whereas that of the La analogue is 0.95. Rhombohedral and monoclinic structures are proposed for the Ba₂LaSbO₆ material.[3]-[4],[12] Figures 5 shows XRD patterns of the Ba₂RESbO₆ (RE = La, Ho, Yb, Tm) and refined using the Powder Cell 2.3 program [25]. Table 2 summarizes the lattice parameters, atomic parameters and reliability parameters. Figure 6 shows the crystal structure of a typical Ba₂HoSbO₆ viewed at different crystallographic directions. The lattice parameter of the sintered Ba₂RESbO₆ was similar to that calculated from the XRD pattern of the as-prepared nanocrystals. The Ba₂LaSbO₆ was fitted to a monoclinic system (P2₁/n) and the others to cubic unit cell with space group Fm $\bar{3}$ m (No. 225). These space groups also allow two crystallographically distinct octahedral sites in the Ba₂RESbO₆ material, thus permitting 1:1 positional ordering between B site ions (RE³⁺ and Sb⁵⁺). The ions are arranged alternatively and have a rock salt sub lattice. The alternative arrangement of RE³⁺ and Sb⁵⁺ ions results in a superstructure. The Sb and RE cations arranged randomly in equivalent positions of the crystal structure in a substitutional solid solution. If upon suitable heat treatment the random solid solution rearranged into a structure in which the Sb and RE cations occupy the same set of positions but in a regular way, the structure is described as a superstructure. Presence of (odd, odd, odd) reflections such as (311) and (511) in the XRD patterns indicate the formation of the superstructure which lead to multiplication of the basic perovskite ABO₃ unit cell. Note that these reflections are also used to characterize the antiphase tilting occurs when 0.964 < *t* < 0.985.[26] However, *t* of the Ba₂RESbO₆ (Ho, Yb, Tm) is ≥ 0.99 which is expected to be an untilted system; and therefore, those reflections denote the super structural

doubling of the basic perovskite unit cell. On the other hand, for La, the (odd, odd, odd) reflections could be related to the antiphase tilting also.

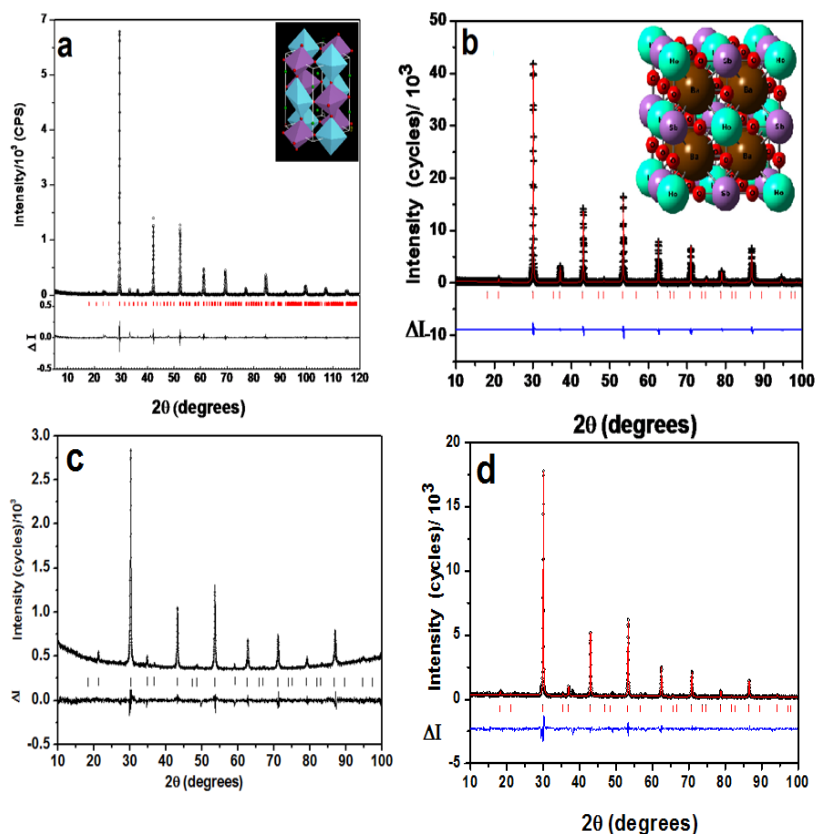


Fig 4: Rietveld refinement plot for sintered (a) Ba_2LaSbO_6 ; (b) Ba_2HoSbO_6 ; (c) Ba_2TmSbO_6 ; and (d) Ba_2YbSbO_6 . The experimental and simulated intensity data (I_e and I_s) are plotted as plus (+) or circles (o) and solid lines, respectively; and the difference between the experimental and calculated pattern (ΔI) = $I_e - I_s$ below. The small vertical lines are the positions of all Bragg reflections corresponding to the compound.

Dielectric properties of Nanostructured Ba_2RESbO_6 ($RE = La, Ho, Tm, Yb$)

As shown in Figure 1, the polarizability followed an exponential lowering with respect to the atomic number of the rare-earth atom in the Ba_2RESbO_6 perovskites. The dielectric constants of the present materials are in Figure 7. Despite the systematic lowering of dielectric polarizability in the Ba_2RESbO_6 materials, the reported dielectric constants showed monotonous variation probably because of the differences in their crystal structures. For example, the unit cell volume of the Ba_2LaSbO_6 is $\sim 636.056 \text{ \AA}^3$, if it is assumed cubic perovskite with lattice parameter $\sim 8.6 \text{ \AA}$. [27] However, the Ba_2LaSbO_6 is monoclinic with cell volume 297.401 \AA^3 thereby accounting the irregular variation in dielectric constant of Ba_2RESbO_6 .

The variation of dielectric constant (ϵ_r) and loss factor ($\tan \delta$) of Ba_2RESbO_6 with frequency at room temperature in the frequency range 50 Hz – 5 MHz is shown in Figure 8. At 5 MHz and room temperature, the sintered Ba_2RESbO_6 gave dielectric constant $\sim 12 - 18.5$ and $\tan \delta \sim 0.9 - 3 \times 10^{-3}$. Some of these values are the lowest loss factor reported for a double perovskites ceramic compound at 5 MHz frequency at room temperature. This low value of dielectric loss could be assigned to the chemical purity of the materials because of the atomic level mixing achieved through a combustion process. The low values of ϵ_r and loss factor ($\tan \delta$) fulfils one of the most important requirements of a material to be used as substrate for the fabrication of high T_c superconducting films.

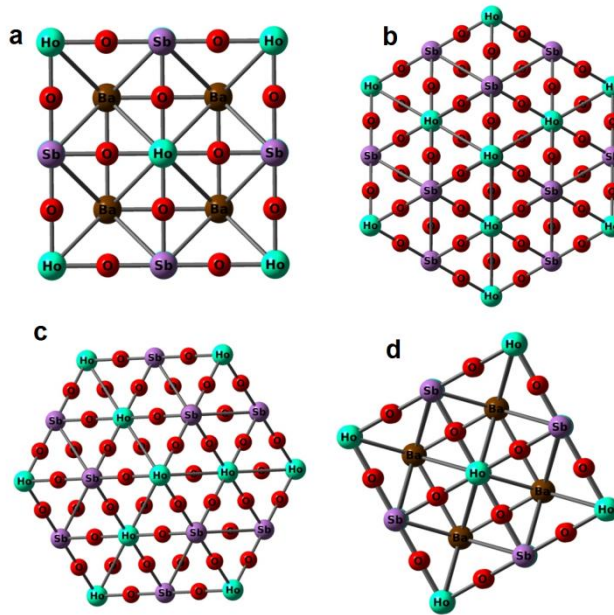


Fig 6: (A) Crystal structure of the Ba_2HoSbO_6 viewed through the (100) direction and (B) (111) direction. The diagram in (C) shows the Kagome lattice, which shows neighboring points in interlaced triangles. Two such interlaced triangles can be seen starting from the Sb atoms in the top and bottom of the structure shown, for example. (D) An alternate representation of the (100) plane shown in Figure (A).

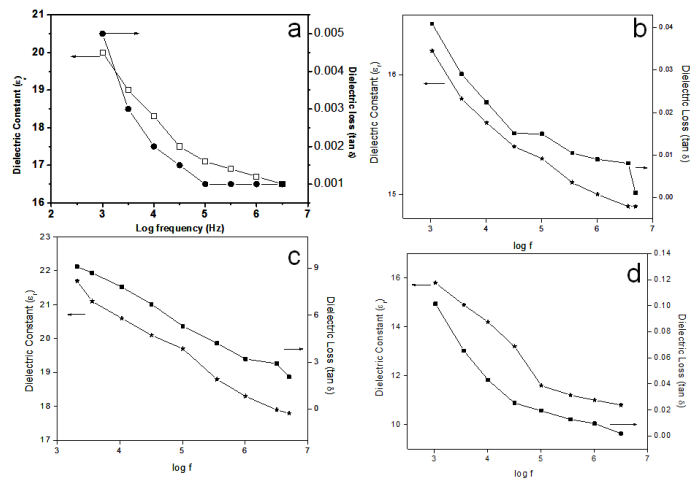


Fig 7: Variation of dielectric constant (ϵ_r) and loss factor ($\tan\delta$) with frequency for (a) Ba_2LaSbO_6 ; (b) Ba_2HoSbO_6 ; (c) Ba_2TmSbO_6 ; (d) Ba_2YbSbO_6 .

IV. CONCLUSIONS

In conclusion, nanocrystals of four complex perovskite materials, Ba_2RESbO_6 ($RE = La, Ho, Yb, Tm$) were synthesized and studied their crystal structure using IR and XRD spectra as well as measured their dielectric properties at low frequencies (≤ 5 MHz) and room temperature. The work provided quantum chemical proofs that IR spectra of complex perovskites have their symmetrical stretching or bending in the higher wave number region whereas the lower wave number side of the spectrum consists of complex vibrations involving all the atoms in the structure concerned. The studies show that La has crystallized in the monoclinic whereas the others in cubic structures. Loss factors of some of these materials are the lowest reported for a double perovskites ceramic



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compound at 5 MHz frequency at room temperature, the origin of which is possibly due to the chemical purity of the materials because of the atomic level mixing achieved through a combustion process.

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