Structural, Optical, and Dielectric Studies of Eco-Engineered Eu^{3+}-Activated Hydroxyapatite from Fish Bone Waste for Multifunctional Applications

M Krishnam Raju[1*], Krunal Patel[1], Raj Kumar Samudrala[2], Neelima Kamjula[3], P Abdul Azeem[4]
[1]Faculty of Engineering & Technology, PIET, Parul University, Gujarat, India 391760.
[2]Department of Physics, SR University, Hasanparthy, Telangana, India 50637.
[3]Faculty of commerce & Management, Sigma University, Vadodara, Gujarat, India,390019.
[4]Department of Physics, National Institute of Technology, Warangal, Telangana, India 506004.
KRISHNAMRAJUEC@gmail.com

Abstract. In the present work, hydroxyapatite (HAp) was sustainably extracted from fish-bone biowaste and converted into a functional ceramic material through appropriate thermal treatment. The thermal behavior of the biogenic precursor was investigated using thermogravimetric and differential thermal analysis (TG–DTA) to determine suitable calcination conditions for the removal of organic matter and formation of a thermally stable phase. Phase evolution and structural optimization were studied by X-ray diffraction (XRD), which confirmed the formation of phase-pure and well-crystallized hydroxyapatite, with optimal crystallinity obtained in the temperature range of 1100–1200 °C. Europium (Eu^{3+}) ions were successfully incorporated into the hydroxyapatite lattice to introduce optical functionality, and the photoluminescence studies revealed intense and spectrally pure red emission arising from characteristic Eu^{3+} transitions. In addition, finite element simulation using COMSOL Multiphysics was employed to examine the electric potential and electric-field distribution in a hydroxyapatite-based capacitor structure, which showed stable and uniform field profiles, indicating good dielectric behavior. Overall, this study demonstrates a simple and sustainable route to convert fish-bone biowaste into a multifunctional hydroxyapatite material with promising optical and dielectric potential for photonic and capacitor-related applications.

Keywords: Hydroxyapatite, Fish bone, Europium, Photoluminescence, Dielectric, COMSOL

1 Introduction

In recent years, the development of eco-friendly functional materials from biowaste resources has received significant attention due to increasing environmental concerns and the growing demand for sustainable technologies. Among various biogenic wastes, fish-bone waste is generated in large quantities by seafood processing industries and is often discarded without proper utilization, leading to environmental and hygienic issues. Fish bones are naturally rich in calcium phosphate phases, making them an attractive and low-cost precursor for the synthesis of hydroxyapatite-based ceramic materials [1,2]. Hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) is a well-known bioceramics because of its

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excellent chemical stability, structural similarity to natural bone mineral, and good biocompatibility [3]. In addition to biomedical applications, nano-sized hydroxyapatite has recently attracted attention for functional applications in optics, dielectrics, and electronics due to its wide band gap and defect-related properties [4, 5] However, the functional performance of hydroxyapatite strongly depends on its crystallinity, phase purity, particle size, and defect concentration, which are in turn controlled by the synthesis route and post-heat-treatment conditions [6, 7] For hydroxyapatite derived from biogenic sources, thermal treatment is particularly important because it not only improves crystallinity but also removes residual organic matter present in the raw biological precursor . Thermogravimetric and differential thermal analyses (TG–DTA) are therefore essential for understanding the decomposition behavior of fish-bone-derived material and for identifying suitable calcination temperatures [6,8,9]. Proper optimization of the heat-treatment conditions helps in obtaining phase-pure hydroxyapatite and suppressing the formation of unwanted secondary phases such as β-tricalcium phosphate, which may adversely affect optical and dielectric properties [10, 11]. One effective approach to introduce optical functionality into hydroxyapatite is doping with rare-earth ions. Among them, trivalent europium (Eu^{3+}) is particularly attractive because of its intense red emission, sharp 4f–4f transitions, long luminescence lifetime, and high color purity [12]. When incorporated into the hydroxyapatite lattice, Eu^{3+} ions can partially substitute Ca^{2+} sites, leading to local lattice distortion and modified optical behavior [13,14] , Eu^{3+}-activated hydroxyapatite has therefore been explored for applications in phosphors and bio-related optical devices.

In parallel, the dielectric behavior of hydroxyapatite and related ceramic materials has also attracted growing interest for applications such as capacitors and insulating layers in electronic devices. The frequency-dependent dielectric constant and dielectric loss provide valuable information about polarization mechanisms, charge carrier dynamics, and microstructural effects such as grain boundaries and defects [15].

Although several studies have reported the synthesis and characterization of hydroxyapatite from natural sources, comprehensive investigations that combine biowaste-derived synthesis, thermal optimization, rare-earth activation, and dielectric evaluation in a single material system are still scarce. In particular, the combined effect of thermal treatment and Eu^{3+} doping on both luminescent and dielectric properties of fish-bone-derived hydroxyapatite has not been explored in detail [16].

This work is unique because it integrates sustainability, photonics and dielectric application in one platform. In the present work, hydroxyapatite is sustainably extracted from fish-bone waste and thermally optimized to obtain high crystallinity and phase purity. The optimized material is then activated with Eu^{3+} ions to introduce red luminescence while preserving the apatite structure. Detailed structural, thermal, morphological, optical, and dielectric (via finite element simulation) studies are carried out to establish clear structure–property relationships. This study demonstrates a simple and effective route to convert fish-bone biowaste into a multifunctional material platform suitable for both photonic and capacitor-related applications.

2 Materials and methods

In accordance with methods frequently documented for the fabrication of bio-derived hydroxyapatite, fish-bone waste was gathered from a nearby source and extensively cleaned to eliminate any leftover meat and surface contaminants. After being repeatedly cleaned with tap water and deionized water, the bones were dried overnight at 100 °C in a hot air oven. A mortar and pestle were used to smash the bones into a fine powder after they had dried. In order to remove organic components and produce hydroxyapatite, the powdered fish bone was thermally processed in a muffle furnace. According to previous research on biologically generated apatite, calcination was done at various temperatures between 900 and 1300 °C with a heating rate of 5 °C min^{-1} and a holding time of 2 hours. Following calcination, the powders were gently milled to a consistent particle size after the furnace was allowed to naturally cool to ambient temperature. The sample obtained at the optimal temperature was chosen for additional analysis based on phase purity and crystallinity.

The optimized hydroxyapatite was treated with 1 mol% Eu^{3+} for europium activation. After dissolving the necessary quantity of europium precursor in deionized water, the hydroxyapatite powder was combined with it while being constantly stirred. To guarantee that Eu^{3+} ions were properly incorporated into the hydroxyapatite lattice, the mixture was dried, powdered once more, and then heat-treated at the same ideal temperature.

3 Results and discussions

3.1 TG–DTA Discussion

Fig.1. shows, Thermogravimetric (TG) and differential thermal analysis (DTA) were carried out to investigate the thermal stability, decomposition behavior, and phase evolution of fish-bone-derived hydroxyapatite (HAp, Sample A) and europium-doped hydroxyapatite (Eu:HAp, Sample B). Such thermal analysis is essential for biogenic precursors, as they usually contain physically adsorbed water, organic matter, and volatile species that must be eliminated to obtain phase-pure and thermally stable hydroxyapatite [17]. For pure HAp (Sample A), the TG curve shows an initial weight loss below about 200 °C, which is mainly attributed to the removal of physically adsorbed water and residual moisture present on the surface and within the porous structure of the powder. This stage is accompanied by a weak endothermic feature in the DTA curve, confirming that this process is related to dehydration rather than any structural transformation .

A second and more pronounced weight loss is observed in the temperature range of approximately 200–600 °C. This region corresponds to the thermal decomposition of organic components inherited from the fish-bone source, such as collagen and other residual biomolecules. The associated exothermic peak in the DTA curve indicates oxidative decomposition of these organic species. Similar multistep thermal behavior has

been widely reported for hydroxyapatite obtained from various biogenic and animal waste sources.

Beyond about 600 °C, the TG curve becomes nearly flat, indicating that most volatile and organic components have been completely removed and a thermally stable calcium phosphate phase has been formed. A weak exothermic feature observed in the DTA curve between about 700 and 900 °C can be attributed to further lattice ordering and improvement in crystallinity of the hydroxyapatite phase [4].

The Eu-doped HAp sample (Sample B) exhibits a similar overall thermal behavior, but with noticeable differences in both TG and DTA curves. The initial weight loss below 200 °C is slightly smaller than that of pure HAp, suggesting lower moisture retention, possibly due to lattice modification induced by Eu^{3+} incorporation [18]. In the intermediate temperature region (200–600 °C), the total mass loss is also marginally reduced for the doped sample, indicating that the presence of Eu^{3+} ions influence the decomposition behavior of residual organic matter. The corresponding exothermic peak in the DTA curve appears slightly shifted and broadened, reflecting changes in decomposition and crystallization kinetics caused by dopant–lattice interactions.

At higher temperatures, the DTA curve of Eu-doped HAp shows a more pronounced exothermic feature compared to that of pure HAp, which can be attributed to enhanced crystallization and phase stabilization induced by Eu^{3+} substitution at Ca^{2+} sites [13,14]. The ionic radius mismatch between Eu^{3+} and Ca^{2+} introduces local lattice distortion, which promotes atomic rearrangement and improves thermal stability of the apatite lattice. Importantly, no significant mass loss is observed above about 800 °C for either sample, indicating that no further decomposition or formation of secondary phases occurs in this temperature range.

The TG–DTA results clearly demonstrate that Eu-doped hydroxyapatite exhibits slightly improved thermal stability and reduced total mass loss compared to pure HAp. The presence of Eu^{3+} ions influences dehydration, organic decomposition, and crystallization processes, which is beneficial for high-temperature processing and functional applications of this material system.

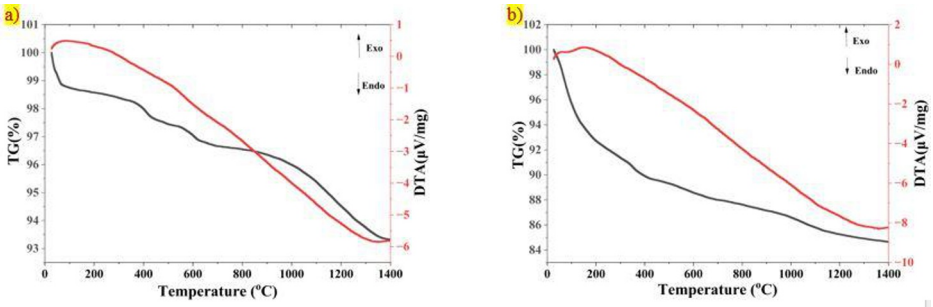

Fig. 1. TG-DTA curves (a) pure hydroxyapatite and (b) hydroxyapatite doped with europium made from fish bone

3.2. Structural studies

In Fig. 2. X-ray diffraction (XRD) analysis was carried out to examine the phase evolution, crystallinity, and thermal stability of fish-bone-derived hydroxyapatite (HAp) subjected to heat treatment in the temperature range of 900–1300 °C. The diffraction pattern of the sample calcined at 900 °C confirms the formation of crystalline hydroxyapatite with characteristic reflections corresponding to the hexagonal apatite structure (JCPDS No. 09-0432). However, the diffraction peaks at this temperature are relatively broad and of lower intensity, indicating small crystallite size, limited crystallinity, and the presence of lattice disorder, which are commonly observed in biogenic apatites due to residual carbonate substitution and trace impurities [19]. When the calcination temperature is increased to 1000 and 1100 °C, the HAp reflections become noticeably sharper and more intense, clearly indicating a significant improvement in crystallinity and grain growth. This behavior can be attributed to enhanced atomic diffusion and progressive elimination of residual volatile species. The gradual reduction in peak broadening suggests a decrease in macrostrain and an increase in crystallite size, reflecting improved structural ordering. Importantly, no additional diffraction peaks are detected in this temperature range, confirming that the fish-bone-derived precursor yields phase-pure hydroxyapatite under these conditions [19]. At 1200 °C, the diffraction peaks become very sharp and well defined, indicating near-optimal crystallization of the hydroxyapatite phase. At this temperature, the apatite lattice appears to be highly ordered and thermally stable, with no detectable evidence of secondary phases. Such a high degree of structural refinement is particularly desirable for functional applications, especially where good dielectric stability and optical performance are required. However, when the calcination temperature is further increased to 1300 °C, subtle changes in the diffraction pattern can be observed, such as the appearance of weak additional reflections and slight peak splitting. These features are typically associated with the partial thermal decomposition of hydroxyapatite into secondary calcium phosphate phases, especially β-tricalcium phosphate (β-TCP), which occurs due to dehydroxylation and structural instability at very high temperatures. This indicates that, although crystallite growth continues at 1300 °C, excessive heat treatment begins to compromise phase purity. Based on the XRD results, the temperature range of 1100–1200 °C is identified as optimal for the preparation of fish-bone-derived hydroxyapatite, as it provides the best compromise between high crystallinity and phase stability. Therefore, this temperature range was selected for further functional studies and Eu^{3+} activation.

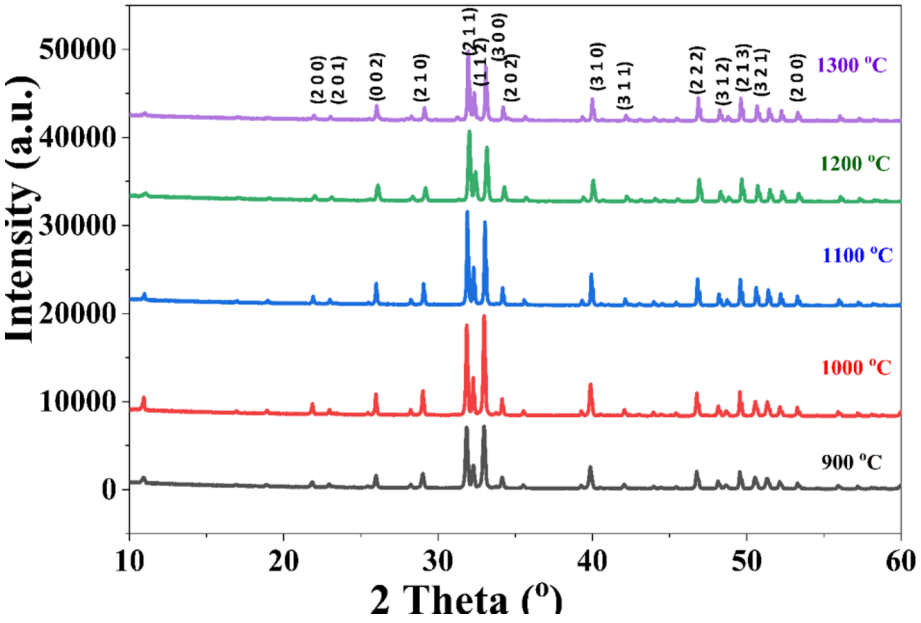

Fig. 2. X-ray diffraction (XRD) patterns of hydroxyapatite derived from fish bone after heat treatment at 900 °C, 1000 °C, 1100 °C, 1200 °C, and 1300 °C

3.3 Photoluminescent Studies

The photoluminescence (PL) properties of Eu^{3+}-doped hydroxyapatite (Eu:HAp) were investigated in detail in order to evaluate the optical activation, excitation behavior, and emission characteristics of the phosphor material. Rare-earth ions, especially Eu^{3+}, are widely used as luminescent activators due to their sharp 4f–4f transitions, long excited-state lifetime, and high color purity, which make them suitable for photonic and display-related applications. Fig. 3(b). shows the excitation spectrum of Eu: HAp monitored at the characteristic red emission wavelength 613 nm. The spectrum consists of a broad band in the near-ultraviolet region, which can be attributed to charge-transfer transitions between O^{2-} and Eu^{3+} ions, along with several sharp peaks corresponding to the f–f transitions of Eu^{3+} ions. Crystallite size is 40–80 nm.The presence of strong absorption in the near-UV region indicates that the material can be efficiently excited using commercially available near-UV light sources, which is advantageous for practical phosphor applications. The emission spectrum of Eu: HAp under near-UV excitation is presented in Fig. 3(a). Several sharp emission lines are observed in the visible region, which are characteristic of the intra-4f transitions of Eu^{3+} ions. The dominant peaks can be assigned to the transitions from the excited 5D0 level to the 7FJ (J = 1–4) levels. Among these, the 5D0 → $7F_2$ transition in the red region exhibits the highest intensity, indicating that Eu^{3+} ions occupy lattice sites without inversion symmetry in the hydroxyapatite host, which enhances the electric-dipole transition probability. The intensity ratio ($^5D_0 \rightarrow {}^7F_2$ / $^5D_0 \rightarrow {}^7F_1$) confirms non-centrosymmetric environment. The

presence of narrow and well-resolved emission lines further confirms the good crystalline quality of the host lattice and the successful incorporation of Eu^{3+} ions into the apatite structure. The choice of 1 mol% Eu^{3+} concentration was based on commonly reported optimized values in literature, where concentration quenching is minimized while maintaining strong emission intensity. Higher concentrations may lead to concentration quenching

The energy-level scheme illustrating the excitation and emission processes of Eu^{3+} ions in the hydroxyapatite host is shown in Fig.3. (c). Upon near-UV excitation, electrons are promoted to higher excited states or charge-transfer states and subsequently relax non-radiatively to the 5D0 level. Radiative transitions from the 5D0 level to the lower-lying 7FJ levels then give rise to the observed characteristic red emissions. This energy-transfer and relaxation mechanism is consistent with the typical luminescence behavior of Eu^{3+}-activated phosphor materials.

To evaluate the color purity of the emitted light, the Commission Internationale de l'Éclairage (CIE) chromaticity coordinates were calculated from the emission spectrum and are shown in Fig. 3(d). The calculated coordinates lie in the red region of the CIE chromaticity diagram, close to the standard red-emission zone, indicating good color purity of the Eu:HAp phosphor. This confirms that the present material exhibits stable and spectrally pure red emission, which is desirable for lighting and display applications [20].

The PL results clearly demonstrate that Eu^{3+}-activated hydroxyapatite derived from fish-bone waste exhibits strong red emission, good spectral purity, and efficient excitation in the near-UV region. The combination of good crystalline quality and effective Eu^{3+} incorporation makes this biowaste-derived material a promising candidate for phosphor and other photonic applications, in agreement with previous reports on rare-earth-doped hydroxyapatite systems [13,14].

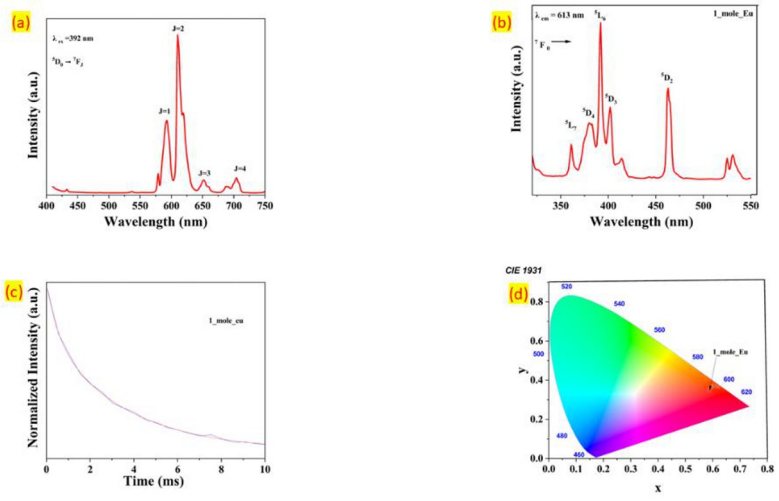

Fig. 3 (a-d). PL excitation, emission, decay characteristics, and CIE chromaticity coordinates of Eu^{3+}-activated fish-bone-derived hydroxyapatite phosphor.

3.4. Dielectric Behavior and COMSOL Simulation

The simulation serves as a theoretical baseline, experimental HAp expected to behave similarly due to high crystallinity. In order to understand the electric-field distribution and capacitive behavior of hydroxyapatite (HAp), finite element simulation was carried out using COMSOL Multiphysics. The simulation was performed using the hydroxyapatite material available in the COMSOL material library and a parallel-plate capacitor geometry. Although the simulated material parameters correspond to standard HAp, this model provides a useful physical picture of how a hydroxyapatite-based dielectric layer behaves under an applied electric field and serves as a reference for evaluating the potential of the experimentally fabricated material. Fig.4. shows the simulated electric potential distribution across the capacitor structure with HAp as the dielectric layer. A smooth and continuous potential gradient is observed from one electrode to the other, indicating that the applied voltage drops uniformly across the dielectric region. The absence of any abrupt potential distortion or localized potential crowding suggests that hydroxyapatite behaves as a stable and homogeneous insulating medium in this configuration. Such a uniform potential distribution is a key requirement for dielectric materials used in capacitor applications, as it ensures reliable and predictable electrical behavior [21,22].

The corresponding electric-field norm distribution is shown in Fig 5. It can be clearly seen that the electric field is nearly uniform throughout the bulk of the HAp layer, with a slightly higher field intensity near the electrode–dielectric interfaces. This behavior is typical of parallel-plate capacitor structures and arises due to boundary effects at the interfaces. The overall homogeneous field distribution within the dielectric indicates that hydroxyapatite can sustain an applied electric field without significant field concentration or distortion, which is important for avoiding premature dielectric breakdown and ensuring long-term device stability [21–23]. The region of relatively higher electric-field intensity observed near the interfaces is physically reasonable and has been reported in several simulation and experimental studies of ceramic dielectrics and bioceramics-based insulating layers [21,22] . Such interfacial field enhancement is generally associated with discontinuities in material properties and electrode boundary conditions, rather than with any intrinsic instability of the dielectric itself.

Although the present simulation uses standard hydroxyapatite data from the COMSOL material library and does not directly incorporate the experimentally synthesized fish-bone-derived samples, the results provide a meaningful baseline for comparison. Since the fabricated HAp in the present work exhibits good crystallinity and phase purity, as confirmed by TG–DTA and XRD analyses, its dielectric response is expected to be comparable to or better than that of ideal hydroxyapatite reported in the literature [24]. In particular, improved crystallinity and reduced impurity content generally lead to more stable electric-field distribution and lower dielectric loss in ceramic dielectrics [25].

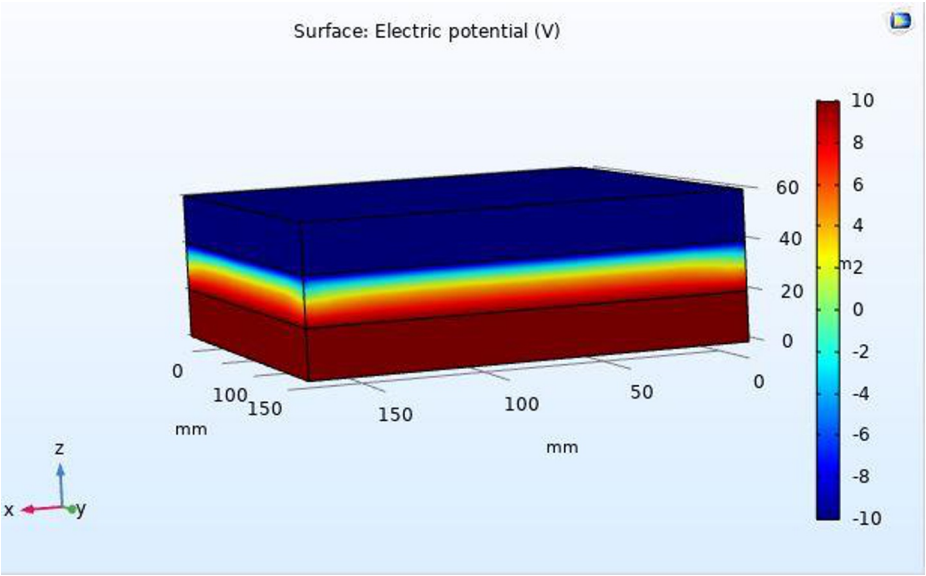

Fig. 4. Simulated 3D electric potential distribution in a parallel-plate capacitor using hydroxyapatite (HAp) as the dielectric, indicating a uniform potential gradient across the electrodes

Previous experimental studies have reported that hydroxyapatite ceramics exhibit frequency-dependent dielectric behavior with relatively stable insulating characteristics, making them suitable for capacitor and bio-integrated electronic applications [21–23]. Therefore, the uniform potential and electric-field distributions observed in the present COMSOL simulation support the feasibility of using hydroxyapatite, including the fish-bone-derived material developed in this work, as a dielectric layer in simple capacitor-type device structures. The simulation results demonstrate that hydroxyapatite exhibits stable electric-field and potential distributions under an applied bias. When combined with the experimentally confirmed structural quality of the fabricated material, these results strengthen the argument that fish-bone-derived hydroxyapatite is not only optically active but also a promising candidate for dielectric and multifunctional electronic applications. Experimental validation will be part of future work

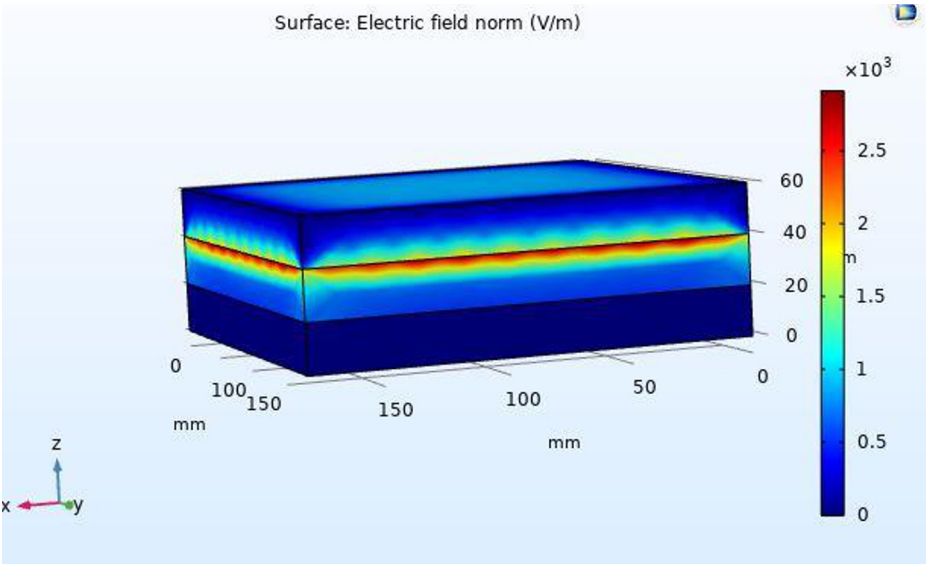

Fig. 5. Simulated 3D electric field intensity distribution in a parallel-plate capacitor using hydroxyapatite (HAp) as the dielectric, indicating homogeneous field confinement and stable dielectric behavior.

4. Conclusion

Hydroxyapatite was successfully extracted from fish-bone biowaste and converted into a functional ceramic material through appropriate thermal treatment. TG–DTA analysis helped to identify suitable heat-treatment conditions for complete removal of organic matter and formation of a thermally stable phase, while XRD results confirmed the development of phase-pure and well-crystallized hydroxyapatite, with optimal crystallinity achieved in the range of 1100–1200 °C. Eu^{3+} activation resulted in strong and spectrally pure red emission, confirming effective incorporation of the activator ions and good crystalline quality of the host lattice. In addition, COMSOL simulation of hydroxyapatite in a capacitor configuration showed uniform electric potential and electric-field distributions, indicating stable dielectric behavior. This work demonstrates a simple and sustainable route to convert fish-bone biowaste into a multifunctional material with both optical and dielectric potential, suitable for photonic and capacitor-related applications. This work is unique because it integrates sustainability , photonics and dielectric application in one platform.

Disclosure of Interests. The authors have no competing interests to declare that are relevant to the content of this article.

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