



Investigation of morphological and dielectric properties of HA-based biomaterials

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Dielectric properties is a measure of the interaction of materials with electromagnetic fields. When people are exposed to non-ionizing radiation (near ultraviolet rays, visible light, infrared, microwave, radio waves, low frequency and radio frequency (long wave)), such interactions take place in the human body at various levels of organization and can initiate biological responses. These biological effects may or may not be desirable, but they need to be well understood in order to benefit from their full potential and beneficial effects for the purposes and protection of human health. Dielectric spectroscopy, or the study of the frequency dependence of dielectric properties, helps to elucidate such interaction mechanisms at the cellular and molecular level. Therefore, in this study, HAp-based Sr-doped powder samples, which are biomaterials compatible with the human body, will be prepared with the help of the hydrothermal method, turned into pellets, and their dielectric properties will be examined. The changes in the dielectric properties of pure and 3% Sr-doped HAp powders depending on the 10-hour reaction time were examined.

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1. Introduction

Biomaterials are of great importance for humanity because they are used to replace diseased biological structures. Hydroxyapatite (HA) is often used in biomedical applications because they are biocompatible materials. HA is a calcium salt found in the structure of bone and tooth enamel. HA is widely used in orthopedic applications because it prevents harmful interaction between the implant and tissue and adapts to the tissue. [1-3].

Biomaterials are materials that are designed to interact with biological systems for a medical purpose. Any matter, surface, or construct that interacts with biological systems. Biomaterials can be derived from nature or synthesized in the laboratory using metallic components, polymers, ceramics, or composite materials. The modern field of

biomaterials combines medicine, biology, physics, and chemistry, and more recent influences from tissue engineering and materials science.

Biodielectric is the electrical polarization that occurs in living cells. The ability to be polarized is related to the biological reactions that occur in living cells. Biodielectric polarization occurs in living things as a result of different biological processes. Bio-dielectric behavior regulates the basic properties of biological systems, and any morphological and functional changes caused by a diseased physiological state lead to an electrically/electronically traceable variation in this property.

This study, a biomaterial-based capacitive bioelectric sensor that determines various factors in the human body will be developed.

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2. Experimental

Calcium nitrate tetrahydrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ di-Ammonium hydrogen phosphate $\text{H}_9\text{N}_2\text{O}_4\text{P}$ was used to synthesize HA. $\text{N}_2\text{O}_6\text{Sr}$ Strontium nitrate was used to create calcium deficiency and observe the change. 120 ml $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and 80 ml $\text{H}_9\text{N}_2\text{O}_4\text{P}$ were stirred in deionized water for 30 minutes. $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ solution and $\text{H}_9\text{N}_2\text{O}_4\text{P}$ solution were placed in a container and stirred at 600 rpm for 1 hour at room temperature. $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ mole fraction of 0% and 3% $\text{N}_2\text{O}_6\text{Sr}$ (Strontium nitrate= Sr) was added to the solution and stirred at 600 rpm for 1 hour at room temperature. HA-based biomaterials with 3% Sr dop HAP were demonstrated at 10h growth time. $n \text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O} / n \text{H}_9\text{N}_2\text{O}_4\text{P} = 1.67$ ($n \text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O} + n\text{Zn}$) / $n \text{H}_9\text{N}_2\text{O}_4\text{P}$ ratio was reduced to provide a ratio of 1.67, while Sr's mole ratio was increased at the same rate and the same result was obtained. After the solutions were prepared, they were mixed in an ultrasonic bath for 10 minutes. Then the ph was set to 11. The solution was transferred to a teflon container to be placed in the FYTRONIX FYHD-8000 hydrothermal system in Fig.1 and the hydrothermal system was set to 180 °C, 10h a'clock and the system was started. At the end of this period, the solution taken from the hydrothermal vessel was filtered and dried on a hot plate at 50 °C for 3 days.

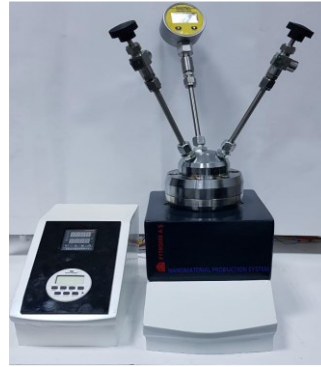


Figure 1: Fytronix FY8000 hydrothermal device

After the dried biomaterials were turned into round pellets under 5 bar pressure with the help of fytrox pellet holder, their dielectric properties were investigated with the help of fytrox dielectric analyzer holder and solarphystech controls in Figure 2.

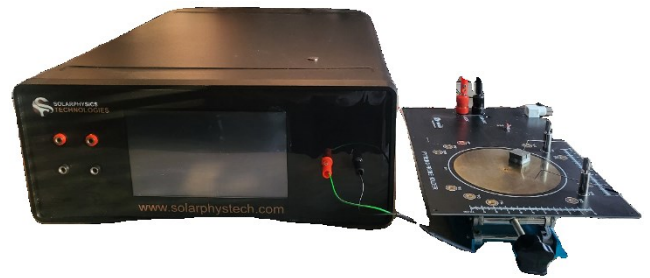


Figure 2: Fytronix sample holder

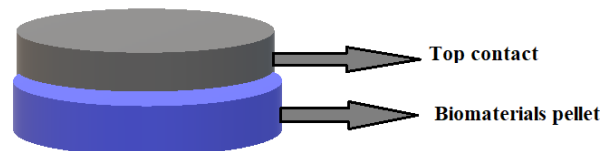
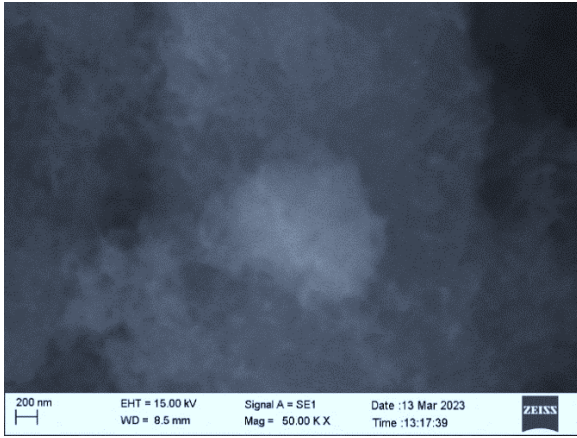


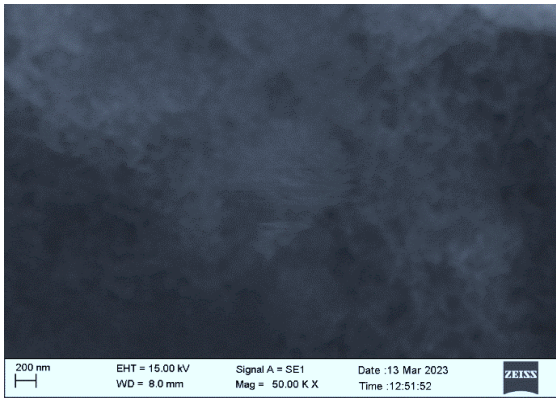
Figure 3: Biomaterial based pellets

3. Results

ATR-FTIR and SEM analysis of the produced pure HA and 3% Sr doped HA powders were performed in powder form. Nanostructured HA and nHA/Sr powders using hydrothermal method. SEM images of the powdered biomaterials shown in Figure 4. were taken at the same temperature (10 hours). As seen in the figures, SEM images show that the grain distribution and surface roughness of the surface structures of the samples have changed, although not significantly. Since properties such as particle distribution and surface roughness in materials will change the electrical conductivity transport properties of materials, this will cause changes in dielectric properties.



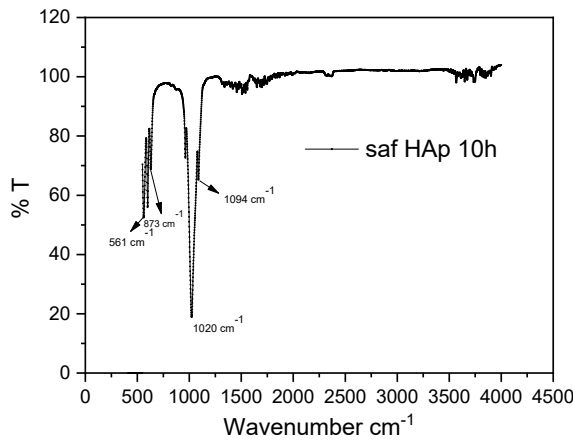
a)



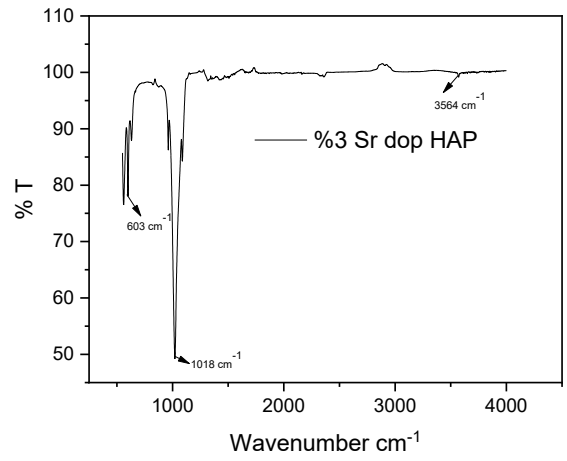
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Figure 4: SEM images of a) pure HA b) 3% Sr doped HA 10h (50000x)

As seen in Figure 5, very small changes were observed in the positions and intensities of the peaks depending on the amount of doping. This shows that the doped Sr settles in the crystal lattice of HA, that is, doping takes place. At the same time, the bands detected at 1020 and 561 cm⁻¹ in Fig.5 a belong to the vibrational modes of phosphate groups and these bands are in agreement with other data in the literature.



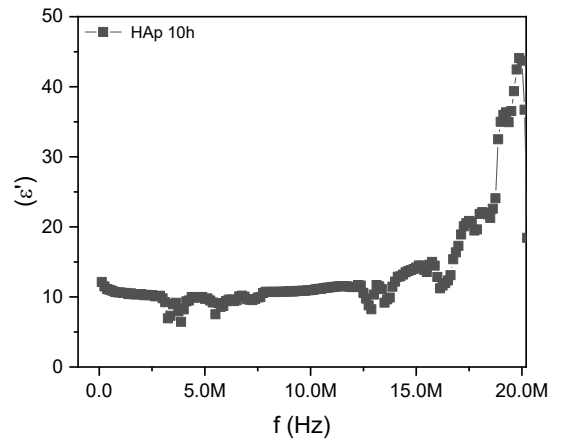
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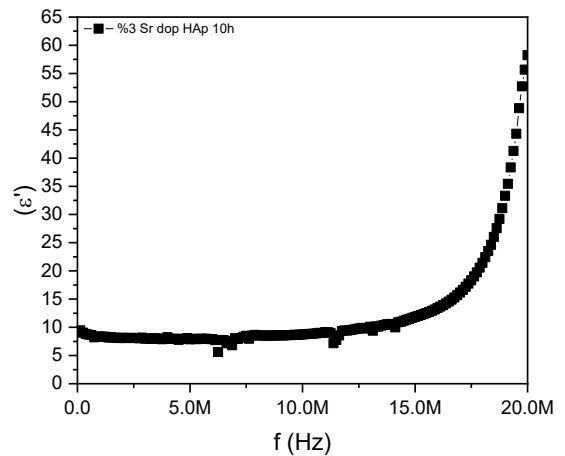
b)

Figure 5: FTIR analysis of a) pure HA and b) 3% Sr doped HA powder samples at 10h reaction time

Notably, all the representative bands that characterize the phosphate (561, 610, 873, and 102 cm⁻¹) and hydroxyl (3564 and 603 cm⁻¹) groups were still present in FTIR spectra after strontium substitution. It was also found that the OH bands in the range of 561–603 cm⁻¹ attributable to molecularly bonded OH are reduced after the substitution of Sr²⁺ ions.



a)



b)

Figure 6: Dielectric constant of pure HA and Sr-doped HA.

Dielectric constant (ϵ' -frequency) curves of pure HA and HA based 3% Sr doped pelletized powder samples are shown in Figure 6. It is observed that the dielectric constant is constant up to a certain frequency value and then increases sharply after about 18MHz. Frequency is a critical factor affecting the behavior of dielectric materials, especially their polarization. The change in the dielectric constant is due to a change in the number of dielectric dipoles. Dielectric polarization causes a change in the dielectric constant of powder samples.

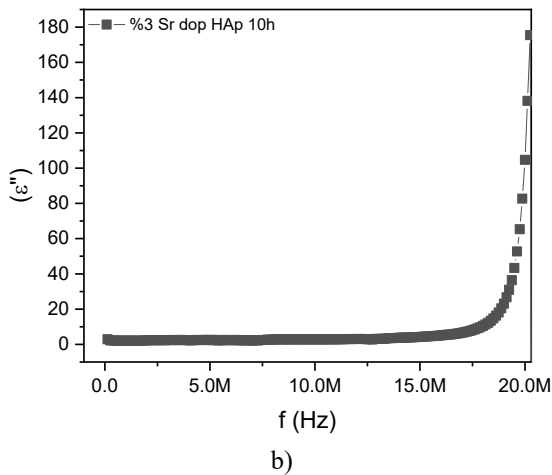
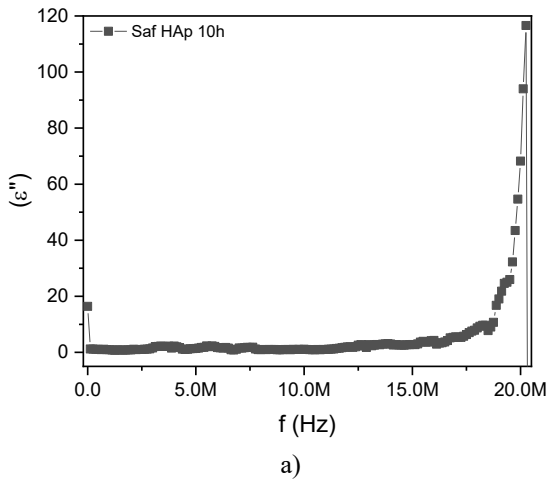


Figure 7: Dielectric loss factor of pure HA and Sr-doped HA.

The loss dielectric constant (ϵ'' -frequency) curves of pure HA and HA based 3% Sr doped pelletized powder samples are shown in Figure 7. The dielectric loss factor is also observed to be constant up to a certain frequency value, with a sudden increase after about 18MHz. Dielectric loss is defined as the phase shift between the periodic electric field and the out-of-phase electric displacement, which is the behavior of dielectric materials under the influence of a.c. voltage on the sample. It varies depending on the internal structure and crystal lattice of the samples. At the same time, dielectric loss is essentially a measure of how much energy is lost in the material due to the applied electric field.

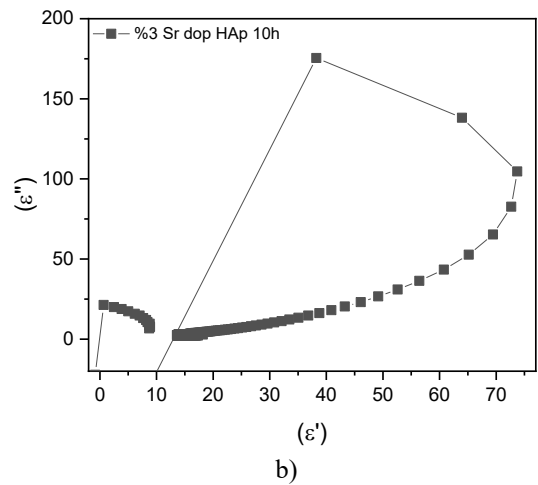
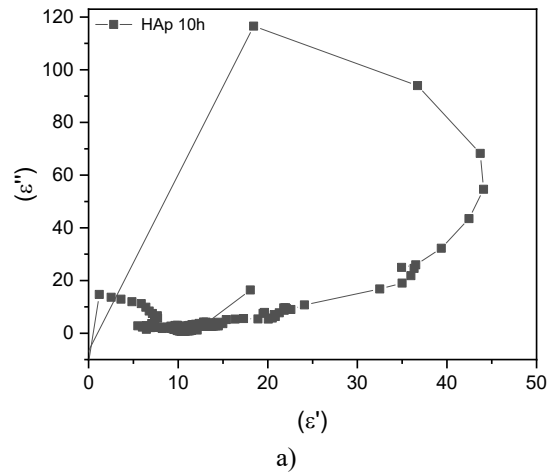
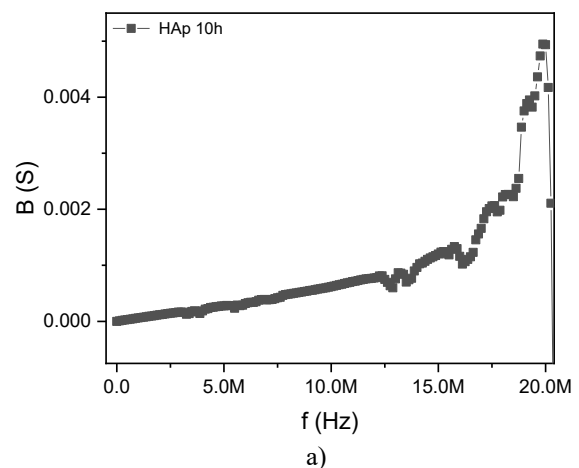


Figure 8: The cole-cole curve of pure HA and Sr-doped HA powder samples.

The cole-cole curves of pure HA and HA based 3% Sr doped pelletized powder samples are shown in Figure 8. In the Cole-Cole plots of the samples, the Cole cole curve means that the electric dipoles interact with each other and this causes a change in the dielectric dipole moment and hence a change in the relative permittivity and dielectric loss values. The values of ϵ' and ϵ'' are extremely important in defining the properties of the dielectric material.



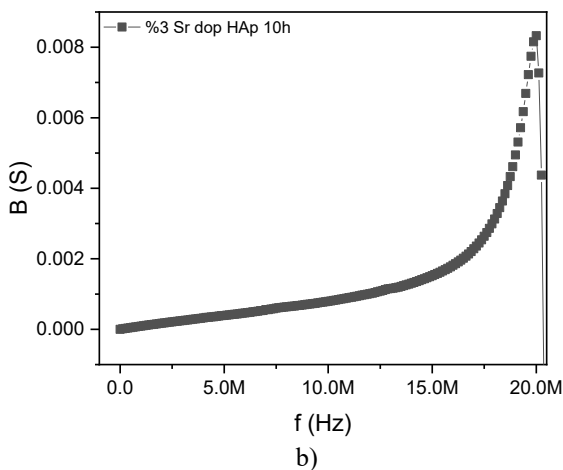


Figure 9: B(S)-frequency curves of pure HA and 3% Sr doped HA powder samples.

Figure 9 shows the B(S)-f curves of pure HA and HA based 3% Sr doped pelletized powder samples at 10h growth temperature. B (S) is the susceptance, the virtual part of the conductivity.

4. Conclusion

The intermolecular bonds of hydrothermally produced HA and %3 Strontium doped HA powder samples were analyzed by ATR-FTIR. As a result of the analysis, it was observed that Sr doping settled into the sample, that is, doping took place. As a result of SEM analysis, small changes in surface morphology due to doping were observed. Dielectric polarization was observed in the real and virtual dielectric constants curves of pure HA and HA based 3% Sr doped pelletized powder samples. Cole-Cole plots indicate that the electric dipoles interact with each other, resulting in a change in the dielectric dipole moment and hence in the relative permittivity and dielectric loss values.

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