**INTRODUCTION**

Hydroxyapatite (HAP) ceramic crystallizes in hexagonal crystal structure and presence of c-axis oriented tunnel type arrangement of Ca, P, O, and H atoms opens this material for multifunctional applications. Reinforcement material, drug delivery, biomaterial, biosensor, catalysts, and gas sensing are some of the areas where synthesized hydroxyapatite is explored by the researchers. Hydroxyapatite, collagen, and fluids are major components of bone. Selective use of dielectric and electric properties of HAp can play a very important role to improve (a) hydroxyapatite's bioactivity with electric poling (b) bone growth with electrical stimulation for bone grafts in spinal fusion (c) healing and repairing of bone fractures with electric field and (d) osteonecrosis and osteoarthritis treatment.

Hydroxyapatite \([\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2]\) crystallize in \(P\ 6_3/m\) space group. It has OH ions and these are located at the center of \(\text{Ca}^2+\) triangles situated along the c-axis in hexagonal unit cell of hydroxyapatite. Hydroxyapatite is also known...
to show ionic conduction due to the presence of Ca$^{2+}$ ions, OH$^-$ ions, and PO$_4$ ions in hexagonal lattice. Distance between two OH ions has been found more than the distance between OH and PO$_4$ groups, so transfer of ions occurs via PO$_4$ groups in hydroxyapatite.$^{1,2}$ Presence of several P-OH bonds on the surface of hydroxyapatite may affect its electronic/conductivity properties in presence of interacting gaseous environment. However, these properties are dominated by the presence of protons and vacancies in hydroxyapatite lattice, and type of atoms in gas. Hydroxyapatite composites and thin films have been explored by various researchers for its sensing capabilities in the presence of volatile organic compounds (VOCs) by impedance and conductivity.$^{11-16}$

Acetone is a very common volatile organic compound. It is used as primary chemical in various laboratory works and pharmaceuticals industries. Constant inhalation of acetone may cause severe headache and narcosis which can critically damage the nervous system. So the detection of acetone and its effect on the properties of bone material is very important for the safety of human being. Methanol is also a volatile organic compound having variety of applications in industries. It is also having toxicity effects in breathing if its quantity exceeded in environment. Sensing of methanol, ethanol, acetone, and ammonia is reported in literature by HAP and measured by electrical properties.$^{11-15}$ Sensing of 1000 ppm CO and CO$_2$ gases using Fe doped HAp thick films have been reported by Mene et al. with 91% and 112% response at temperature 140°C and 155°C, respectively.$^{10}$ Anjum et al. have reported the sensing of alcohols using nanocomposite thick film of graphite and HAp with response 465% (for 100 ppm methanol) at 30°C.$^{16}$ HAP is reported to show the change in conductivity and impedance by doping K, Ag, and Mg in its lattice.$^{17-15}$ Magnetic transition metals are essential needed for biological functions in living organisms and doping of these metals in hydroxyapatite at Ca site is less explored.$^{19}$ Hydroxyapatite shows diamagnetic behavior and their magnetic moment is not effected in presence of magnetic field. On the other hand, magnetic metals (Fe, Co, and Ni) contain unpaired electrons in its d orbitals. Magnetic transition metal-doped hydroxyapatite can be used for hyperthermia applications, cell separation, drug delivery, and magnetic resonance imaging.$^{20}$ Recent studies explored the cytocompatibility property of Fe co-doped calcium oxide materials.$^{21}$ Dielectric properties of Fe doped hydroxyapatite samples prepared by sol–gel method has been reported in literature.$^{22}$ Recently, we had reported the effect of substitution of Fe in the lattice of hydroxyapatite on the dielectric constant of HAP and magnetic field-dependent dielectric properties of Fe doped hydroxyapatite material.$^{23}$ In this manuscript, we have documented (a) Fe K-edge spectra of Fe doped hydroxyapatite samples and (b) studies on the change in impedance of Fe doped hydroxyapatite in presence of methanol and acetone environment.

### EXPERIMENTAL

Ca$_{1-x}$Fe$_x$(PO$_4$)$_6$(OH)$_2$ (0 ≤ x ≤ 0.3) samples were synthesized by wet chemical method. Stoichiometric amount of Ca(NO$_3$)$_2$.4H$_2$O, (NH$_4$)$_2$HPO$_4$, and Fe(NO$_3$)$_3$.9H$_2$O precursors were used with ammonia solution to synthesize these compositions. In preparation process, 0.5M solutions of Ca(NO$_3$)$_2$.4H$_2$O and Fe(NO$_3$)$_3$.9H$_2$O were mixed with 0.5M solution of (NH$_4$)$_2$HPO$_4$ (keeping the Ca/P ratio 1.67) at temperature ~80°C. Mixture was continuously stirred at 80°C for 2 hours with maintained pH 10 using ammonia. The products were collected and kept for drying. After drying, all the compositions were grounded and calcined at 800°C for 2 hrs.

Powders were pelletized in 20 mm dia pellets and sintered at 1200°C for 2 hrs in air. These conditions for the preparation of parent and Fe doped hydroxyapatite were optimized for retaining OH group and hexagonal crystal structure of hydroxyapatite. Powder X-ray diffraction method was used to examine the phase and crystal structure of polycrystalline parent and Fe doped hydroxyapatites with Cu K$_{α1}$ radiation ($λ$ = 1.5405 Å). Data were collected from 10 to 90° with data separation 0.02. The Fe K-edge XANES spectra were recorded at BL-9, Scanning extended edge X-rays absorption fine structure (EXAFS) Beamline of Indus-2, RRCAT, Indore, India. The Fe K-edge XANES measurements of Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ and Ca$_{9.7}$Fe$_{0.3}$(PO$_4$)$_6$(OH)$_2$ samples were done in fluorescence mode using Vortex detector. The beamline consists of Rh/Pt coated meridional cylindrical mirror for collimation and Si (111) based double crystal monochromator to select excitation energy. The energy range of XANES spectra was calibrated using Fe metal foil at 7112 eV. The data normalization and LCF analysis were done using ATHENA software.$^{24}$ Fourier transform infrared (FTIR) spectra of powder samples were recorded using the KBr pellet technique from 400 cm$^{-1}$ to 4000 cm$^{-1}$ on perkin elmer spectrometer at room temperature. Sintered pellets were used to record the electrical properties in methanol and acetone gas environment. We have used a glass chamber equipped with vacuum pump and facilities to insert vaporize methanol/acetone gas into it (Figure 1).

Temperature of glass chamber was kept ~50°C and vacuum maintained inside it while inserting methanol/acetone gas. Methanol and acetone were converted into gaseous state from liquid state. Following formulas were used to calculate the ppm concentration of gas in chamber: acetone (μL) = [(2.367 × PPM × dilution)/0.790] and for methanol (μL) = [(1.3077 × PPM × dilution)/0.790]. Measurements of change in impedance in presence of methanol/acetone were done by calculating the change in real part of impedance (at frequency 1 kHz) data using novaccontrol make Impedance analyzer by keeping ~5 mm gap between two probe on the sample pellet. The change in impedance [Response (R%)] calculated by the respective formula (Z$_{a}$ − Z$_{g}$) × 100/Z$_{g}$ where
Zg is the impedance in presence of methanol/acetone gas and Za is the impedance in vacuum.

3 | RESULTS AND DISCUSSION

Figure 2 shows the X-ray diffraction (XRD) patterns of parent and Fe doped hydroxyapatite Ca_{10-x}Fe_{x}(PO_4)_6(OH)_2 [x = 0.0, 0.1, and 0.3] samples. XRD patterns show the crystallization of all the samples in hexagonal symmetry. There is no other impurity phase present in it. All the peaks are indexed for space group P 6_3/m. These peaks are matching with the JCPDF data (73-0294) reported by Kay et al.1

Figure 3 shows the close look of X-ray diffraction patterns of parent and Ca_{9.9}Fe_{0.1}(PO_4)_6(OH)_2 compositions from 21 degree to 43 degree. We have found the position of (002) peak for both compositions at the same position. Difference in the position of peaks (200) and (202) for parent and Ca_{9.9}Fe_{0.1}(PO_4)_6(OH)_2 is same. It shows that Fe doping does not affect the length of c-axis of hydroxyapatite crystal. After comparing the positions of peaks (200), (300), and (310) for parent and Ca_{9.9}Fe_{0.1}(PO_4)_6(OH)_2 compositions, we have found that the 2 times difference in the positions for (200) peaks, 3 times for (300) peaks, and 4 times for (310) peaks. These findings show that the Fe doping effect only a-axis and b-axis lattice parameters while lattice along the z-axis is not affected much.23 To find out the valence states of Fe in Fe doped hydroxyapatites, we have recorded Fe K-edge X-ray absorption near edge structure (XANES) spectra for x = 0.1 and x = 0.3 [Ca_{10-x}Fe_{x}(PO_4)_6(OH)_2] samples.

Figure 4 shows the normalized Fe K-edge XANES spectra of Ca_{9.9}Fe_{0.1}(PO_4)_6(OH)_2, Ca_{9.7}Fe_{0.3}(PO_4)_6(OH)_2 samples along with the Fe metal foil, FeO (Fe^{2+}), and Fe_2O_3(Fe^{3+}) standard references. From the Fe K-edge XANES spectra (Figure 4), it has been observed that Ca_{9.9}Fe_{0.1}(PO_4)_6(OH)_2 sample absorption edge matches with Fe^{3+} while Ca_{9.9}Fe_{0.3}(PO_4)_6(OH)_2 sample shows mixed-valence states Fe^{2+} and Fe^{3+}. To quantify the amount of Fe^{2+} and Fe^{3+} valence state in Ca_{9.9}Fe_{0.1}(PO_4)_6(OH)_2 sample, Fe K-edge XANES spectra were fitted with linear combination fitting (LCF) using Athena software within an energy range of −20 eV below to +20 eV above the edge (Figure 5).24 The LCF of Ca_{9.9}Fe_{0.1}(PO_4)_6(OH)_2 sample was done using a combination of Fe^{2+} and Fe^{3+} standard spectra and goodness

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**FIGURE 1** Instrument set-up for measuring impedance in controlled atmosphere

**FIGURE 2** X-ray diffraction patterns of parent and Fe doped Hydroxyapatites. All the compositions are crystallized in hexagonal symmetry
of fit parameters (R-factor and reduced $\chi^2$) along with the percent that each contributes to the fit. The obtained fitting parameters for best fit are $R = 0.001639$ and $\chi^2 = 0.00141$ for Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ sample. The LCF fitting of Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ sample indicates the presence of Fe valence states [Fe$^{2+}\sim58.3\%$ (±4%) and Fe$^{3+}\sim41.7\%$ (±4%)]. The combined results of X-ray diffraction and XANES show the presence of Fe$^{2+}$ valence state is present at Ca site in Fe doped Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ hydroxyapatite. To explore about the lattice properties and the presence of OH group in hydroxyapatite, we have recorded infra-red spectroscopic data for these samples.

Figure 6 shows the FTIR spectra of (a) parent Ca$_{10}$(PO$_4$)$_6$(OH)$_2$ and (b) Fe doped hydroxyapatite Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ from 400 cm$^{-1}$ to 4000 cm$^{-1}$. Symmetric stretching mode $\nu_1$ is observed at 959 cm$^{-1}$ for parent while Fe doped composition shows peak at 960 cm$^{-1}$. Symmetric bending modes $\nu_2$ are observed at 570, 601, and 633 cm$^{-1}$ for both compositions. Asymmetric stretching modes $\nu_3$ are observed at 1048 and 1088 cm$^{-1}$ for both compositions. Asymmetric bending modes $\nu_4$ are observed at 434 and 474 cm$^{-1}$ for both compositions. Vibrational mode of OH$^-$ is observed at 3568 cm$^{-1}$ and liberation mode at 634 cm$^{-1}$ for parent Ca$_{10}$(PO$_4$)$_6$(OH)$_2$ composition. Vibrational
mode of OH\(^{-}\) is observed at 3575 cm\(^{-1}\) and liberation mode at 632 cm\(^{-1}\) for Fe doped Ca\(_{9.9}\)Fe\(_{0.1}\)(PO\(_4\))\(_6\)(OH)\(_2\) composition. Most of the observed vibrational modes for Ca\(_{9.9}\)Fe\(_{0.1}\)(PO\(_4\))\(_6\)(OH)\(_2\) composition are at the same positions with Ca\(_{10}\)(PO\(_4\))\(_6\)(OH)\(_2\), this is due to the combined contribution effect of the small ionic radius and high atomic number of doped Fe\(^{2+}\) in comparison with Ca\(^{2+}\) in apatite lattice. Presence of OH vibrational modes in both compositions and change in lattice parameters show the effective substitution of Fe\(^{2+}\) at Ca\(^{2+}\) in hydroxyapatite.

### 3.1 Impedance study in presence of methanol environment

Figure 7 shows the impedance behavior of Ca\(_{9.9}\)Fe\(_{0.1}\)(PO\(_4\))\(_6\)(OH)\(_2\) pellet sample in presence of (A) 50 ppm, (B) 100 ppm, and (C) 500 ppm concentration of methanol in chamber (Figure 1). Figure 7 (D) shows the change in impedance of Ca\(_{10}\)(PO\(_4\))\(_6\)(OH)\(_2\) by exposing the pellet sample in 500 ppm methanol concentration. Ca\(_{9.9}\)Fe\(_{0.1}\)(PO\(_4\))\(_6\)(OH)\(_2\) pellet sample shows the huge decrease in impedance when methanol gas is inserted in chamber and fast increase in impedance when gas is sucked out from chamber. Ca\(_{10}\)(PO\(_4\))\(_6\)(OH)\(_2\) pellet sample does not show much change in impedance after inserting methanol in chamber. Response of Ca\(_{9.9}\)Fe\(_{0.1}\)(PO\(_4\))\(_6\)(OH)\(_2\) pellet sample has been found ~80% for 50 ppm, ~109% for 100 ppm, and ~238% ppm for 500 ppm methanol concentration. While Ca\(_{10}\)(PO\(_4\))\(_6\)(OH)\(_2\) pellet shows only ~5% response for 500 ppm concentration of methanol and shows negligible response for 50 ppm concentration of methanol. All the impedance measurements in controlled environment were done in the apparatus shown in Figure 1.

In Figure 8, response of Ca\(_{9.9}\)Fe\(_{0.1}\)(PO\(_4\))\(_6\)(OH)\(_2\) pellet sample with the different concentration of methanol has been shown. Minimum response ~80% has been found for 50 ppm concentration of methanol and 238% response has been found for 500 ppm concentration of methanol. To find out the reason behind the higher response of Fe doped hydroxyapatite Ca\(_{9.9}\)Fe\(_{0.1}\)(PO\(_4\))\(_6\)(OH)\(_2\) toward methanol in comparison with Ca\(_{10}\)(PO\(_4\))\(_6\)(OH)\(_2\), we have fitted experimentally observed response data with the Freundlich adsorption isotherm equation R = αC\(^{β}\), where R is the response, α is the proportionality factor, C is the concentration of gas and β is the exponential factor (which confirms the adsorption of gas is due to physisorption or chemisorption). Insets of Figure 8 show the fitted response data in Log R = Log α + βlog C equation. Value of β is calculated from the linear fitted data.

![Figure 6](image_url)

**Figure 6** Infra-red (IR) spectroscopic spectra of (A) parent and (B) Fe doped Hydroxyapatite

![Figure 7](image_url)

**Figure 7** Change in impedance of Ca\(_{9.9}\)Fe\(_{0.1}\)(PO\(_4\))\(_6\)(OH)\(_2\) by exposing the pellet sample in (A) 50 ppm, (B) 100 ppm, and (C) 500 ppm methanol concentration. (D) shows the change in impedance of Ca\(_{10}\)(PO\(_4\))\(_6\)(OH)\(_2\) by exposing the pellet sample in 500 ppm methanol concentration. “In” indicate the time of insertion of gas and “out” shows the time of taking out of gas from chamber.
between log R and log C. We have found the value of β is 0.63 for 50 to 200 ppm concentration of methanol gas (inset A of Figure 8) and 2.16 for 300 to 500 ppm concentration of methanol gas (inset B of Figure 8). It shows that the adsorption of methanol gas is dominated up to 200 ppm concentration by physical sorption. Beyond 200 ppm concentration of methanol gas, adsorption is dominated by chemical sorption on Ca₉Fe₀.₁(PO₄)₆(OH)₂ pellet sample.

### 3.2 Impedance study in presence of acetone environment

Figure 9 shows the impedance behavior of Ca₉Fe₀.₁(PO₄)₆(OH)₂ pellet sample in presence of (a) 100 ppm, (b) 200 ppm, (c) 400 ppm, and (d) 500 ppm concentration of acetone. Change in impedance is measured 126% for 100 ppm, 151% for 200 ppm, 349% for 400 ppm, and 537% for 500 ppm concentration of acetone. Figure 10 shows the change in impedance of Ca₁₀(PO₄)₆(OH)₂ and Ca₉Fe₀.₁(PO₄)₆(OH)₂ pellet samples in presence of 50 ppm acetone. Change in impedance is found ~92% for

**FIGURE 8** Response \([R = (Z_a - Z_g) \times 100/Z_g]\) of Ca₉Fe₀.₁(PO₄)₆(OH)₂ pellet sample with different concentration of methanol. Inset (A) shows the linear fitting on the data of response for 50 ppm to 200 ppm while inset (B) shows fitting on response data for 300 to 500 ppm concentration of methanol.

**FIGURE 9** Change in the impedance of Ca₉Fe₀.₁(PO₄)₆(OH)₂ by exposing the pellet sample in (A) 100 ppm, (B) 200 ppm, (C) 400 ppm, and (D) 500 ppm acetone concentration. “In” indicate the time of insertion of gas and “out” shows the time of taking out gas from chamber.

**FIGURE 10** Change in impedance of parent Ca₁₀(PO₄)₆(OH)₂ and Ca₉Fe₀.₁(PO₄)₆(OH)₂ by exposing in 50 ppm concentration of acetone. “In” indicate the time of insertion of gas and “out” shows the time of taking out gas from chamber.
Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ while ~1% has been measured for Ca$_{10}$(PO$_4$)$_6$(OH)$_2$ with 50 ppm concentration of acetone.

To find out the reason behind the higher response of Fe doped hydroxyapatite Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ toward acetone in comparison with Ca$_{10}$(PO$_4$)$_6$(OH)$_2$, we have fitted experimentally observed response data (Figure 11A) with the Freundlich adsorption isotherm equation $R = \alpha C^\beta$, where $R$ is the response, $\alpha$ is the proportionality factor, $C$ is the concentration of gas and $\beta$ is the exponential factor (which confirms the adsorption of gas is due to physisorption or chemisorption).$^{25}$ Figure 11B shows the fitted response data in Log $R = \log \alpha + \beta \log C$ equation. Value of $\beta$ is calculated from the linear fitted data between log $R$ and log $C$. We have found the value of $\beta$ is 0.71 for 50-500 ppm concentration of acetone, which is found less than 1. It shows that the adsorption of acetone gas is physical on Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ pellet sample. After the physical adsorption, acetone gas interacts with the OH group of Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$, which provides easy path for conduction through proton at room temperature. It results the decrease in impedance of sample.

Change in impedance in presence of acetone gaseous molecules can be explained based on the pictorial presentation shown in Figure 12.

4 | CONCLUSIONS

Parent and Fe doped HAp were synthesized by wet chemical method. Mixed-valence states of Fe$^{2+}$ and Fe$^{3+}$ are found in Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ while Ca$_{9.7}$Fe$_{0.3}$(PO$_4$)$_6$(OH)$_2$ contains only Fe$^{3+}$. Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ shows the ~80% response with 50 ppm, ~238% for 500 ppm, and ~616% response with 1000 ppm concentration of methanol. Ca$_{10}$(PO$_4$)$_6$(OH)$_2$ shows only ~5% response for 500 ppm concentration of methanol. Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ shows the ~92% response...
with 50 ppm and ~537% for 500 ppm concentration of acetone. Presence of Fe$^{2+}$ valence state in the lattice of Ca$_{9.9}$Fe$_{0.1}$(PO$_4$)$_6$(OH)$_2$ sample's hexagonal lattice increases the response for methanol and acetone.

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