



NANO-TiO₂ DOPED HYDROXYAPATITE NANOCERAMICS: DEVELOPMENT OF AMMONIA SENSOR

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ABSTRACT

Hydroxyapatite (HAp) is a nanoceramic material having high porosity and better thermal stability. The modified Hydroxyapatite (HAp) material, to be used as sensor, is prepared by blending different weight concentration of TiO₂ in it. The phase purity and crystallinity for HAp, TiO₂ and composites are determined by XRD. The TGA is carried out to show the thermal stability of the materials. FTIR is performed to confirm functional groups. Surface morphological studies are carried out by SEM. The sensitivity, often called as gas response, is determined by noting resistance change in air and in gas/vapors, at various temperatures in the range of 30^oC-350^oC. The operating temperature of the sensor is determined by means of gas response at various temperatures. The study involves gas sensing parameters like gas response, response/recovery time, and saturation limit which provides assessment of sensor material for its device application. In order to improve the gas sensing properties, HAp is blended with TiO₂ so as to sense ammonia vapors in meager quantities in the surrounding air. The results showed that, compared to commercial HAp, the sensitivities of the commercial TiO₂ and composites are found to be more. Hence TiO₂ blended HAp nanoceramic can be used as an ideal substrates for ammonia detection at lower concentration and at room temperature.

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INTRODUCTION

Hydroxyapatite [Ca₁₀(PO₄)₆(OH)₂], abbreviated as HAp, is of considerable interest owing to its potential usefulness as biomaterials¹, adsorbents²⁻⁶, ion-exchangers⁷ and gas sensors^{8,13}. Gas sensors are used in environmental monitoring, industrial production, personal safety, medical, and military applications¹⁴. Conductometric gas sensors based on semiconducting metal oxides are actually one of the most investigated groups of gas sensors. They have attracted the attention of industries interested in gas sensing devices under atmospheric conditions, due to low cost and flexibility associated to their production and simplicity of their use. It has advantages that large number of detectable gases can be detected¹⁵.

Titanium dioxide (TiO₂) has also found itself to be commercially viable in photo catalytic products, air cleaners and air conditioners and self-cleaning surfaces¹⁶. TiO₂ has been of immense use in photo and electrochemical systems in photo catalysis and in solar cells.

Attempts have been made to study TiO₂ for the detection of gases, including, NH₃, CO, H₂, H₂S, vapors of alcohols, humidity and others¹⁷. There is a need to detect low concentration of ammonia (NH₃) in many fields, such as food technology, chemical engineering, medical diagnosis, environmental protection, monitoring of car interiors¹⁸. Most NH₃ gas sensors are based on metal oxide films, which have a limited maximum sensitivity and need a high working temperature¹⁹. The present work elucidates to explore the possibility of using TiO₂-modified HAp as a potential sensor material for ammonia detection at lower temperature and also at its lower concentrations.

MATERIAL AND METHODS

Nano-ceramic (HAp) was prepared using wet chemical process. Calcium nitrate tetra hydrate (Ca (NO₃)₂.4H₂O) and diammonium hydrogen phosphate ((NH₄)₂HPO₄) was used to prepare HAp using wet chemical precipitation at pH values between 10 and 12, while taking appropriate molar ratios so as to get theoretical Ca/P ratio close to 1.66¹. Addition of diammonium hydrogen phosphate solution in to calcium nitrate solution was followed by continuous stirring for about 3 hours at a temperature 80^oC. The pH of the reaction was adjusted by the addition of NH₄OH. The resulting white precipitate was

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washed thoroughly with double distilled water and dried in air oven at 100°C and finally sintered at 1000°C for 2 hours²⁰. TiO₂-HAp blending is done by mechanical mixing of 0.5wt%, 1 wt%, 1.5 wt% and 2 wt% TiO₂ in HAp powder. Thick films of such blended materials are prepared by standard screen printing method.

Instrumentation

The XRD patterns of sensor materials are recorded by (Miniflex) model, in the range of 10 to 60 degree. Thermal stability is seen by TGA Instrument (Shimadzu) by heating the sensor materials in temperature range 40-1000°C. FTIR was used in the range of 4000 cm⁻¹ to 400 cm⁻¹ to identify functional groups of materials. The surface morphology of materials was evaluated by SEM.

Description of Sensor

The gas sensing characterization is carried out by means of two probe techniques described earlier^{7, 20}, to study sensing properties of commercial HAp, commercial TiO₂ and composites thick films. The resistance of thick films in air (R_a) and in gas (R_g) is determined. Thus the gas sensitivity or gas response is calculated from the following equation.

$$\text{Gas response or Sensitivity (\%)} = (R_{\text{gas}} - R_{\text{air}}) \times 100 / (R_{\text{air}})$$

The temperature of sensor substrate (thick film) is held in the temperature range of 350°C to 30°C for noting R_{gas} and R_{air} separately and subsequently calculated the sensitivity (%). Then a graph of sensitivity (%) as a function of temperature is drawn, which gives the maximum sensitivity at a particular temperature. This temperature, at which maximum sensitivity is seen, is called as the operating temperature of the sensor thick film²¹.

RESULT AND DISCUSSION

X-ray Diffraction

The XRD analysis is carried out for commercial HAp, TiO₂ and HAp blended TiO₂ materials. X-ray diffractometer (Miniflex) with CuK α ($\lambda = 1.54 \text{ \AA}$) incident radiation is used and the XRD peaks were recorded in the 2θ range of 10°–60°. Crystallite size (D) is obtained by Debye-Scherrer's formula given as follows.

$$D = \frac{k\lambda}{\beta \cos\theta}$$

Where;

D is the crystallite size,

λ is the wavelength of the X-ray radiation. ($\lambda = 1.54 \text{ \AA}$ for CuK α)

k is usually taken as 0.89; and

β is the line width at half-maximum height.

The XRD patterns of the HAp shows (211), (112), (300), (002), (222), (213) and (004) planes corresponds to 31.9°, 32.9°, 33.9°, 25.9°, 46.8°, 49.5° and 53.1° as shown in (Fig.1). The XRD pattern matches with those reported in literature¹⁸. Major peak at around 31.9° corresponds to (211) planes of HAp crystalline. The XRD pattern of commercial TiO₂ has preferential peaks oriented with planes (101), (200), (004), and (211) planes corresponds to 25.4°, 48.1°, 37.6° and 55.1° as indicated in (Fig.1). The XRD result for composite materials indicates that addition of TiO₂ does not bring any structural change or a new crystal phase of HAp or TiO₂. It is

also seen that XRD peaks of TiO₂ increases for higher TiO₂% in HAp matrix. The calculated grain size of commercial HAp, commercial TiO₂ and its composite viz; 0.5% TiO₂+HAp, 1%TiO₂+HAp and 2%TiO₂+HAp are 21.16 nm, 20.43 nm, 22.35 nm, 20.15 nm and 20.55 nm respectively.

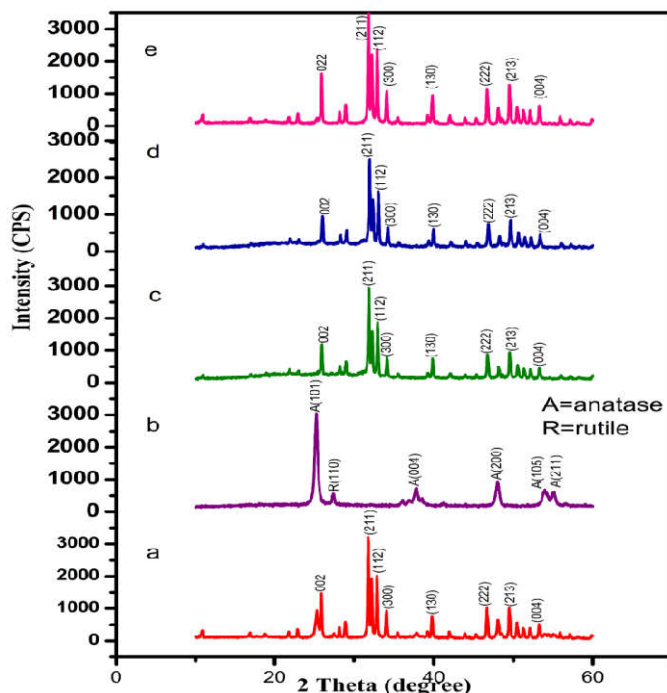


Fig 1. X-ray diffraction pattern of (a) commercial HAp, (b) commercial TiO₂, (c) 0.5% TiO₂ + HAp, (d) 1% TiO₂ + HAp, and (e) 2% TiO₂ + HAp.

TGA/DTA Analysis

The thermal properties of commercial HAp, TiO₂ and their composites are carried out by TGA by heating the material from 40°C to 1000°C. It shows no significant loss in weight with the associated exothermic peaks. This confirms the thermal stability of commercial HAp and TiO₂-HAp composite.

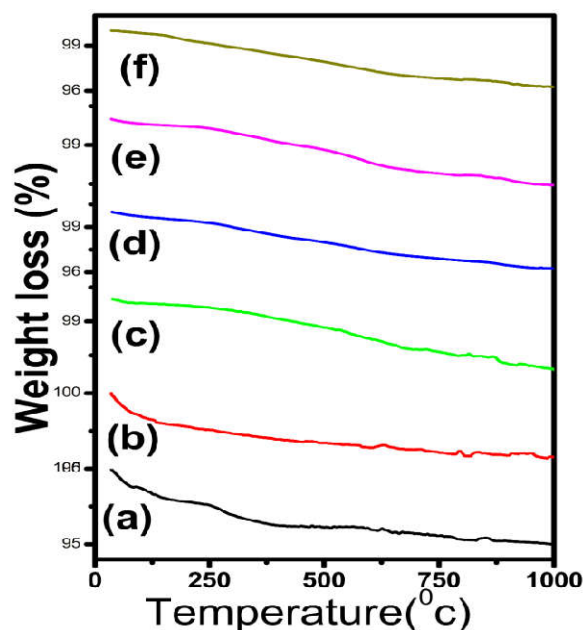


Fig 2 TGA plots of (a) commercial HAp, (b) commercial TiO₂, (c) 0.5%TiO₂ + HAp, (d) 1% TiO₂ + HAp, (e) 1.5% TiO₂ + HAp and (f) 2% TiO₂ + HAp, showing no remarkable weight loss and thus confirming the thermal stability of TiO₂-HAp system.

Fourier transforms infrared spectroscopy (FTIR)

FTIR spectra of commercial TiO₂, commercial HAp and TiO₂ blended HAp, recorded at room temperature in the range of wave number 4000-400 cm⁻¹, and are shown in a (fig.3). The spectra for commercial HAp. displays the peaks at 1041, 1099 and 578 cm⁻¹ which is assigned as the stretching and bending motion of (PO₄³⁻) of HAp, The presence of peak in the region 869-1451 cm⁻¹ was due to absorbed carbon dioxide (CO₃²⁻), the broad band at about 3433 cm⁻¹ corresponds to the absorbed hydrate (O-H) and the peak 3563 cm⁻¹ belongs to the stretching vibration of lattice OH⁻ ions²². The spectra TiO₂ peak at 780 cm⁻¹ due to the vibration of the (Ti-O) bonds in the TiO₂ lattice, the sharp peak at 1072 cm⁻¹ corresponds to bending vibrations of water. Peak observed at 1633 cm⁻¹ corresponds to the C=O vibrations. Peaks observed at 2390-2902 cm⁻¹ corresponds to the (C-H) stretching vibrations. Peak at 3450 cm⁻¹ corresponds to stretching vibration of (O-H) bond²³.

Table 1 Some important functional group assignments of Hydroxyapatite (HAp).

Wave number (cm ⁻¹)	Stretching mode	Functional group
1041-1099 and 578	stretching and bending motion	PO ₄ ³⁻
869-1451	Asymmetric stretching	CO ₃ ²⁻
3433	absorbed hydrate	hydrated O-H
3563	Ion stretching	OH ⁻

Table 2 Some important functional group assignments of Titania (TiO₂).

Wave number (cm ⁻¹)	Stretching mode	Functional group
780	Stretching vibration	Ti-O
1072	bending vibrations of water	H ₂ O
1633	Stretching vibration	C=O
2390-2902	stretching vibrations	C-H
3450	stretching vibration	O-H

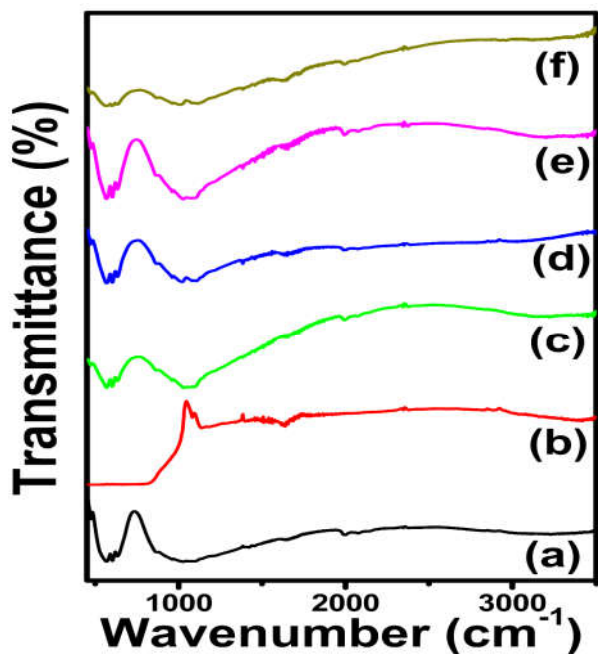


Fig 3 FTIR analysis: (a) commercial HAp, (b) commercial TiO₂, (c) 0.5% TiO₂ + HAp, (d) 1% TiO₂ + HAp, (e) 1.5%TiO₂ + HAp and (f) 2% TiO₂ + HAp, showing the prominent functional groups.

SEM analysis

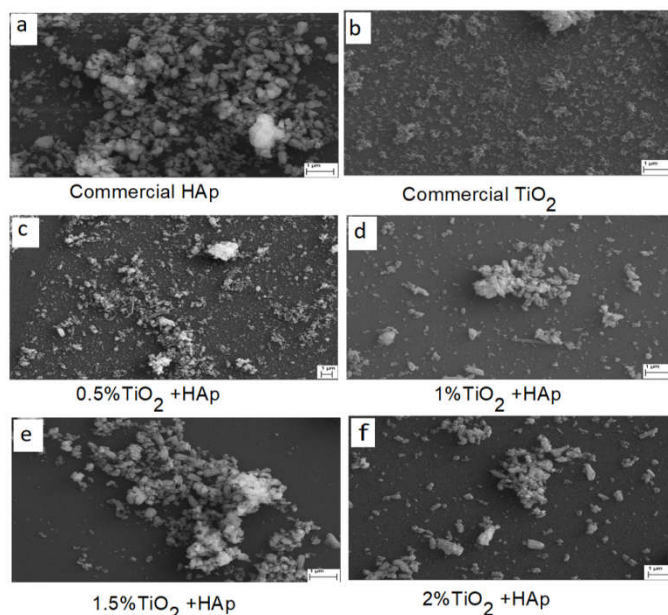


Fig 4 SEM analysis: (a) commercial HAp, (b) commercial TiO₂, (c) 0.5% TiO₂ + HAp, (d) 1% TiO₂ + HAp, (e) 1.5%TiO₂ + HAp, and (f) 2% TiO₂ + HAp.

The scanning electron microscope (SEM) was utilized for surface morphological study of HAp, TiO₂ nanoparticles, and their composites. The powder material was dissolved in Ethanol and spread on the sample holder to see clear view nanoparticles and fine crystallites of HAp and TiO₂. Fig.4. shows the SEM images of the HAp formed were agglomerated. The surface of TiO₂ with a large number of small grain sized particles. The composites are found to be highly porous.

Gas sensing performance

Operating temperature

The sensitivity variation of ammonia gas vapors with its concentration of 10 ppm for pure HAp thick film, TiO₂ film and TiO₂-HAp blended films is shown in Fig.5. It is seen that almost all substrate materials show highest gas sensitivity at room temperature which is one of the unique properties of these substrate to detect ammonia vapors. The increase in sensitivity is due to doping of TiO₂ in HAp material. It is observed that the sensor substrate shows increasing gas response at lower temperature as seen from Fig.5. Thus, TiO₂ doping in HAp changes maximum sensitivity as well as the temperature at which it occurs. The maximum sensitivity of ammonia gas for commercial & composites materials (in order of descending parameter) is found to be commercial TiO₂>2%TiO₂-HAp>1.5%TiO₂-HAp>1%TiO₂-HAp>0.5%TiO₂-HAp. Thus the doping of TiO₂ in HAp gives enhancement is sensitivity factor for ammonia vapors, this increment in sensitivity is attributed to the fact that ammonia is reducing gas^{24, 25}.

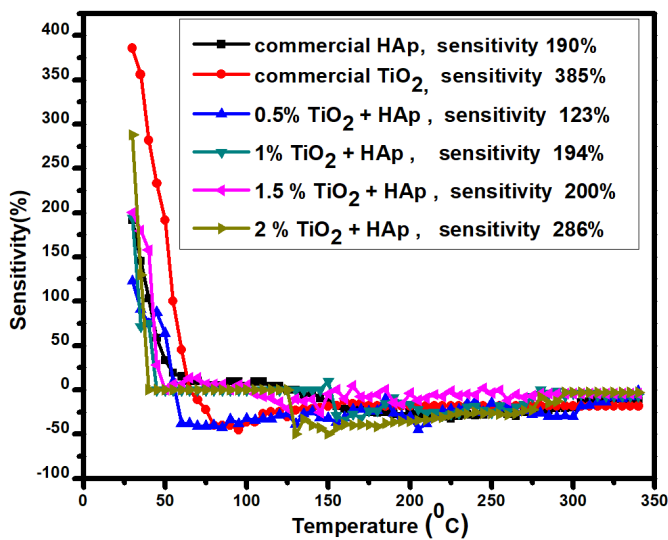


Fig 5 Variation of gas response with temperature for ammonia (NH₃) gas load of 10ppm.

Determination of response and recovery time

Fig.6 shows the variation in resistance of nano-HAp sensor material, TiO₂ and composite at room temperature as a function of time, in presence ammonia gas with 10ppm concentration. After injection of test gas (10 ppm ammonia), the resistance of sensor material is decreased slowly with respect to increase in time due to interaction of gas molecules with the sensor material. The sensor matrix shows variation in resistance after the delivery of the gas to be sensed up to 12 min, upon exposing the sensor material to atmospheric air, the resistance reverts back to its original value.

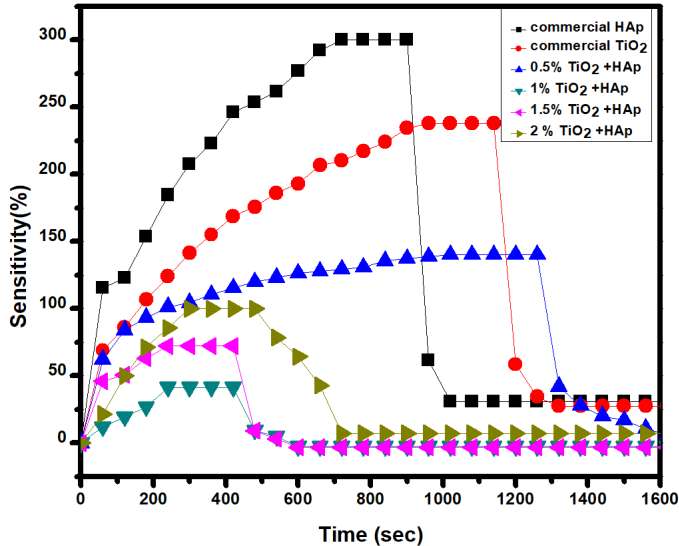


Fig 6. Response and Recovery time for commercial HAp, commercial TiO₂ and their composites for 10 ppm ammonia.

Thus the time required for sensor material to revert to 10% of its original value (resistance/voltage) is termed as recovery time which is achieved after 30 sec. In case of commercial TiO₂ thick film, the variation in resistance is observed up to 18 min and gets back to initial resistance value within 62 sec, while for composite TiO₂ doped HAp, the variation in resistance is observed up to (18, 2, 4 and 5 min) respectively and it get back to initial resistance value within (7 min.80 sec, 90 sec and 4 min).

Determination Gas uptake capacity

Fig.7 shows for determination of gas uptake capacity of sensor material, the thick films of sensing material are exposed to various concentration level of ammonia gas at their respective operating temperatures. The gas response increases with increase in gas concentration, later attains a constant value, described as saturation value or maximum gas uptake. The gas response for commercial HAp and TiO₂ is found to be 600 ppm & 800 ppm respectively. In case of composites such as 0.5 wt%, 1 wt%, 1.5 wt% and 2 wt% TiO₂ doped HAp the gas uptake is in the range of 400- 900 ppm. However, 1% TiO₂ blended HAp shows much higher uptake capacity.

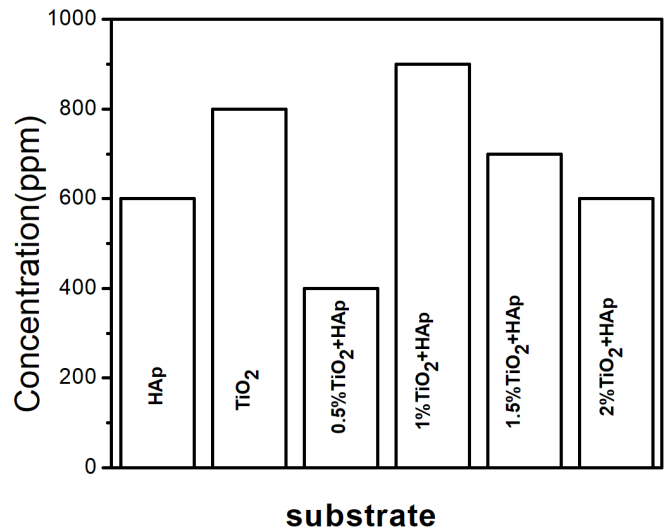
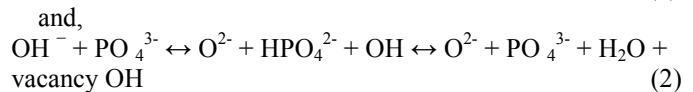


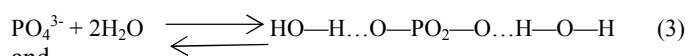
Fig 7 Ammonia gas uptake capacity for commercial HAp, commercial TiO₂, and its composites.

Gas sensing mechanism with Hap

The HAp with chemical compositions as [Ca₁₀(PO₄)₆(OH)₂] behaves as an ionic conductor and is highly porous. In the first place, porosity provides physisorption of gas molecules. At higher temperatures, presence of H⁺ and OH⁻ ions of hydroxyl group provides conductivity^{26,27}, while at low temperatures, the conductivity, if any, is attributed due to proton transfer between OH⁻ ions or jumping of protons from OH⁻ to PO₄³⁻ ion^{28, 29}.



Hydroxyapatite possesses P-OH groups and/or several ionic species like Ca²⁺, PO₄³⁻, and OH⁻. The protons (H⁺), oxide ions (O²⁻) and the lattice hydroxyl ions (OH⁻) are responsible for bonding with molecule, providing noticeable change in conduction³⁰. HAp favour hydrogen bonding by means of interaction of PO₄³⁻ with H₂O [Eq.2 and Eq.3]. Moreover, NH₃ has tendency to bond with H₂O via hydrogen bond surface [Eq. 4].



HAp behaves as an n-type semiconductor wherein electric resistance decreases upon exposure to reducing species such as NH₃²⁸.

Gas sensing mechanism with TiO₂

TiO₂ detection of gas vapors on TiO₂ depends on the charger in depletion layer at the TiO₂ grain boundaries influenced by the adsorbed reducing or oxidizing gases. The variation in depletion region leads to shift in the energy barrier height for free charge carries, ultimately leading to conductivity charges of the sensing materials comprising of TiO₂ blended HAp material. Ammonia, being reducing gas gives negative charge which is introduced in the materials decreasing the positive (hole) charge carrier concentration in HAp. This results in increasing the sensitivity of TiO₂ blended HAp as in comprising with commercial HAp. Ammonia acts as a reducing agent for all the metal oxide semiconductors, A metal oxide can absorb oxygen from the atmosphere both as the O²⁻ and O⁻ species, can be described by the following formulae:

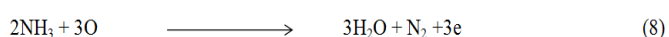
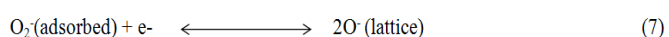
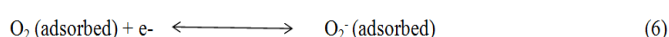
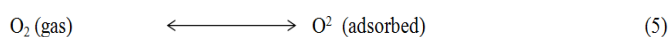


Table 3 Classification of materials with adsorbed gases.

Reducing Gases	Oxidizing Gases	Classification
Resistance decrease	Resistance increase	n-type
Resistance increase	Resistance decrease	p-type

During the adsorption of oxygen species on the surface of sensing element, capturing of electrons from conduction band and the associated decrease in the charge carrier concentration (e⁻) leads to an increase in the resistance of the n-type sensing element until it attains equilibrium. Thus, the surface resistance increases and attains equilibrium during the chemisorptions process. TiO₂ as n-type the resistive decrease when exposed to reducing gas as NH₃, the electron transfer into the conduction band due to increase conductivity³¹.

CONCLUSIONS

Titanium dioxide doped HAp gas sensor is an experimental approach towards the development of an effective gas sensor which works at lower operating temperature with better sensitivity even at low ppm level. HAp is synthesized by wet chemical precipitation method. TiO₂-HAp blending is done by mechanical mixing of 0.5 wt%, 1 wt%, 1.5 wt% and 2 wt% TiO₂ in HAp powder and the thick films are prepared by screen printing method, which are characterized by X-ray Diffraction (XRD) showing the crystalline phase structure. The thermal behavior of the composite materials is examined by Thermo-Gravimetry analysis (TGA) showing high thermal stability of composite material. Fourier Transform Infrared Spectroscopy (FTIR) showed all absorption characteristics of commercial HAp, commercial TiO₂ and composite material. The sensing performance of 1 wt% TiO₂ blended HAp thick films have displayed high sensitivity, faster response/ recovery time for fixed concentration of ammonia (10 ppm) in comparison with other composites. It also possesses higher uptake capacity of 900 ppm of ammonia vapors in ambient conditions. Thus it can be utilized for commercial applications as an efficient sensor substrate for ammonia in sensor devices.

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Conflict of Interest

The authors declare no conflict of interest.

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