

**Special Issue of First International Conference on Advancements in Engineering & Technology (ICAET- 2020)****Hydrothermal Synthesis of Fe₂O₃ for the Humidity Sensing Application**Kavita P. Patil¹, V.R Khadse², K. B Mahajan³, R. B. Waghulde⁴,^{1,2,3}School of Physical Sciences, KCE Society's Moolji Jaitha College, Jalgaon, India.⁴DNCVP Shirish Madhularao Chaudhari College, Jalgaon, India.kavitas_patil@rediffmail.com¹, rbwaghulade@yahoo.co.in⁴**Abstract**

Fe₂O₃ nanorods have been successfully synthesized by a hydrothermal method using stainless steel Teflon autoclave. The solution of FeCl₃ and Urea was transferred to Teflon-lined stainless-steel autoclave and then maintained the temperature 180°C for 24 hrs. As prepared Fe₂O₃ powder were characterized by X-ray Diffraction (XRD) and the result shows the formation of Hematite phase of Fe₂O₃ after calcination at 400°C of 2h. Field Emission Scanning Electron Microscopy (FESEM) was used to study the morphology of synthesized Fe₂O₃ powder and the result indicates that bunch of nanorods were formed with width 44 nm and length of 220 nm. Also, Fourier Transform Infrared spectroscopy (FTIR) and X-ray Photoelectron Spectroscopy (XPS) techniques were used to characterize synthesized Fe₂O₃ nanorods. To study the humidity sensing performance of Fe₂O₃ nanorods, sensor was synthesized as interdigitated electrode coated with sensing material on top of the electrode. The present study investigated good humidity sensing performance of Fe₂O₃ nanorods at room temperature in humidity (RH) range of 11%-97% at DC condition.

Keywords: Fe₂O₃, nanorod, hydrothermal, Humidity,**1. Introduction**

Metal oxides are the promising material for the various applications in the field of electrical and optical device development. Also, to control environmental conditions, metal oxide nanostructures are extensively used as Humidity Sensor [1-3]. As a result, it is a challenging task of design a good humidity sensor. The performance of humidity sensor is improved by taking efforts in betterment of sensor performance parameters such as sensing elements, structure design, principle of mechanism and fabrication technologies [4]. Metal oxides have wide ranges of properties, including large band-gap, transitions from insulators to metallic and superconducting nature. Among these metal oxides, the iron oxide (Fe₂O₃) has significantly used in various application because of its stability, high electrical conductivity, large

band gap, low cost and environmentally friendly [5-8]. The α-Fe₂O₃ is an n-type semiconductor with a thermodynamically stable with a band gap of 2.10 eV. As a result, a variety of nanostructures of iron oxide like nanoparticles, nanorods, nanocubes, nanorings, nanonails, nanodiscs and nanoflowers by using different synthesis route has been reported to enhance material properties. These nanostructures of α-Fe₂O₃ are extremely promising and attractive materials for their wide range of applications, such as catalysis, gas sensors, magnetic recording materials, lithium rechargeable batteries, biosensor, pigments and paints [9-13].

In a present work, nanorods of α-Fe₂O₃ were synthesized by hydrothermal method. The synthesized α-Fe₂O₃ nanorods were characterized

by XRD, FESEM, XPS and FTIR. And further humidity sensing performance of α -Fe₂O₃ nanorods were examined at room temperature.

2. Experimental

2.1 Materials

The purity of all chemicals was of analytical grade and there is no subsequent purification process. Ferric chloride (FeCl₃ 6H₂O) was procured from Sigma-Aldrich, India and Urea was procured from Qualigens Fine Chemicals, India.

2.2 Preparation of Iron Oxide nanorods.

In this work, Hematite α -Fe₂O₃ nanorods were synthesized by hydrothermal method. In the typical experiment, 0.1M solution of FeCl₃ was prepared in 120ml distilled water, and then urea was added into the solution with continuous magnetic stirring for 30 min. The prepared solution was transferred to Teflon-lined stainless-steel autoclave and then maintained the temperature 180⁰C for 24hrs. The resulting precipitated was filtered, washed several times by double distilled water and dried at 60⁰ C for 6hrs.

The final precursor was calcinated at 400⁰C for 2hrs to form final product of Hematite α -Fe₂O₃ nanorods.

2.3 Humidity sensing measurement.

2.3.1 Preparation of sensor electrode.

The sensor consist of an interdigitated electrode coated with sensing material on top of the electrode as shown in Fig.1.(a) The electrode consists of five pairs of Cu tracks .The width and gap between two successive tracks is about 1 mm.

2.3.2 Humidity sensing measurement.

In the present study, humidity sensing performance of α -Fe₂O₃ nanorods were examined at room temperature (27⁰C) using indigenously developed humidity sensing set-up as shown in Fig.1.(b). In the typical experimental method, humidification inside the chamber was developed by saturated salt solution (K₂SO₄) by adding distilled water drop by drop in salt K₂SO₄ at room temperature. The variations in electrical current (nA) with different RH level were manually recorded by digital picoammeter (SES, Model: DPM-111).

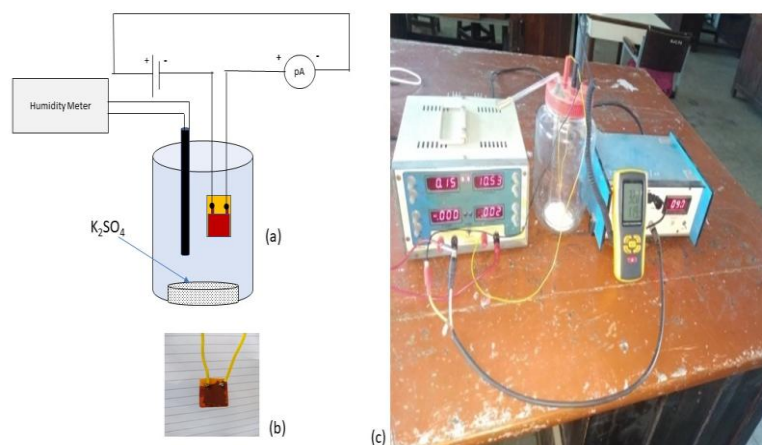


Fig.1: Synthesized α -Fe₂O₃ nanorods as humidity sensor (a) Experimental Circuit Diagram, (b) Sensor Electrode and (c) Humidity sensing set-up.

3. Result and Discussion

3.1 Characterization of α -Fe₂O₃ nanorods

X-ray diffraction (XRD) analysis were performed with a Bruker D8 Advance X-ray diffractometer with CuK-alpha radiation to identification of phase and crystallite size. The XRD patterns of synthesized nanorod powder shown in Fig.2 (a) identifies the hematite phase of iron oxide (α -Fe₂O₃) and well matches with the standard data

(JCDPS file no. 86-0550) of rhombohedral geometry with cell constants $a = 5.035 \text{ \AA}$ and $c = 13.74 \text{ \AA}$. In addition, no other impurity peak and phase peak were detected, indicating high purity of the product. The intense peaks of α -Fe₂O₃ are 24.390⁰, 33.110⁰, 35.640⁰, 40.840⁰, 49.240⁰, 54.090⁰, 56.270⁰, 62.830⁰, 64.160⁰ with corresponding plane (012), (104), (110),(113), (024), (116), (122), (214) and (300).

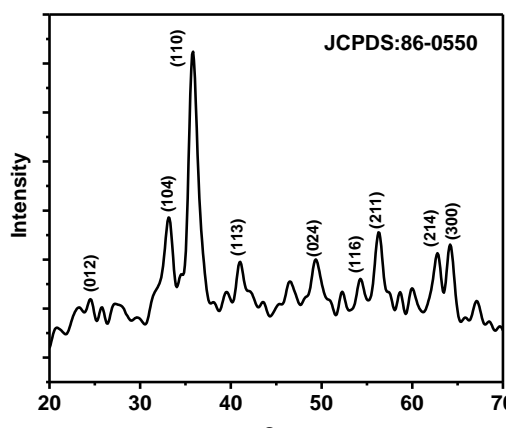


Fig.2 XRD pattern of synthesized $\alpha\text{-Fe}_2\text{O}_3$

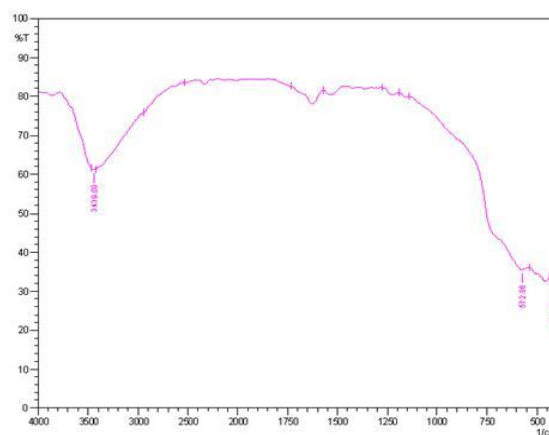


Fig. 3 FTIR spectra of synthesized Fe_2O_3

FTIR of the synthesized $\alpha\text{-Fe}_2\text{O}_3$ nanorods was in the range of 400-4000 cm^{-1} wave number shown in Fig.3 which identifies the chemical bonds as well as functional groups in the compound. The large broad band at 3439 cm^{-1} is due to the O-H stretching vibration in O-H groups. The strong band below 700 cm^{-1} is assigned Fe-O stretching mode. The band corresponding to Fe-O stretching of Fe_2O_3 is seen at 418 and 572 cm^{-1} . The morphology of synthesized iron oxide ($\alpha\text{-Fe}_2\text{O}_3$) powder was carried out by field emission scanning electron (FE-SEM) microscopy with a Hitachi (S-4800, Hitachi, Japan) microscope. As clearly observed that, rods are aligned in the forms of bunch as shown in Fig.4 (a). The image in Fig 4 (b) shows that the outer diameter and length of rod are about $\sim 43\text{nm}$ and $\sim 220\text{nm}$ respectively. The

XPS survey spectrum of the as-prepared product $\alpha\text{-Fe}_2\text{O}_3$ [Fig.5 (a)] clearly shows that nanorods are composed of Fe and O. In the high-resolution Fe 2p spectrum [Fig.5 (b)], two distinct peaks at binding energies of 711.73eV for $\text{Fe}2p_{3/2}$ and 725.22eV for $\text{Fe}2p_{1/2}$ with satellite peak at 719.56eV are observed. This is characteristic of Fe^{3+} in Fe_2O_3 . A high-resolution O1s XPS spectrum [Fig.5 (c)] of $\alpha\text{-Fe}_2\text{O}_3$ can be divided into peaks located at 530.17 and 532.09eV. The observed at 530.17eV is attributed to oxygen atoms that were bound to only iron atoms (Fe-O) whereas the other peak assigned to OH. Furthermore, the average atomic ratio of Fe and O is about 1:1.9, on the basis of the quantification of Fe and O peaks.

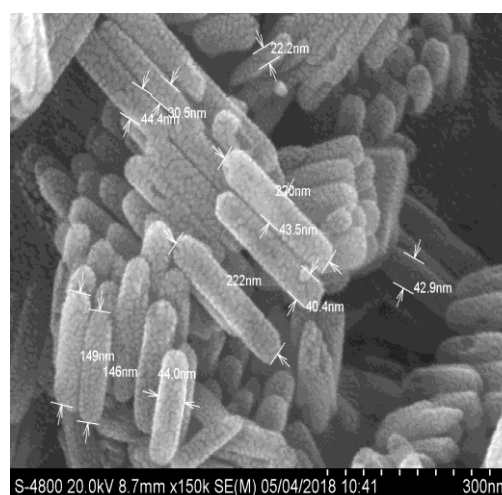
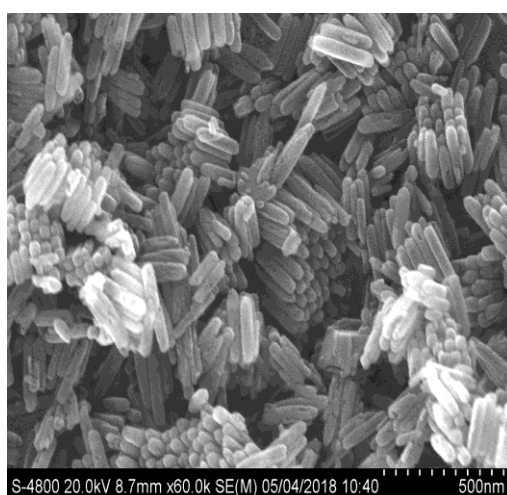


Fig. 4: FESEM images of synthesized $\alpha\text{-Fe}_2\text{O}_3$ nanorod

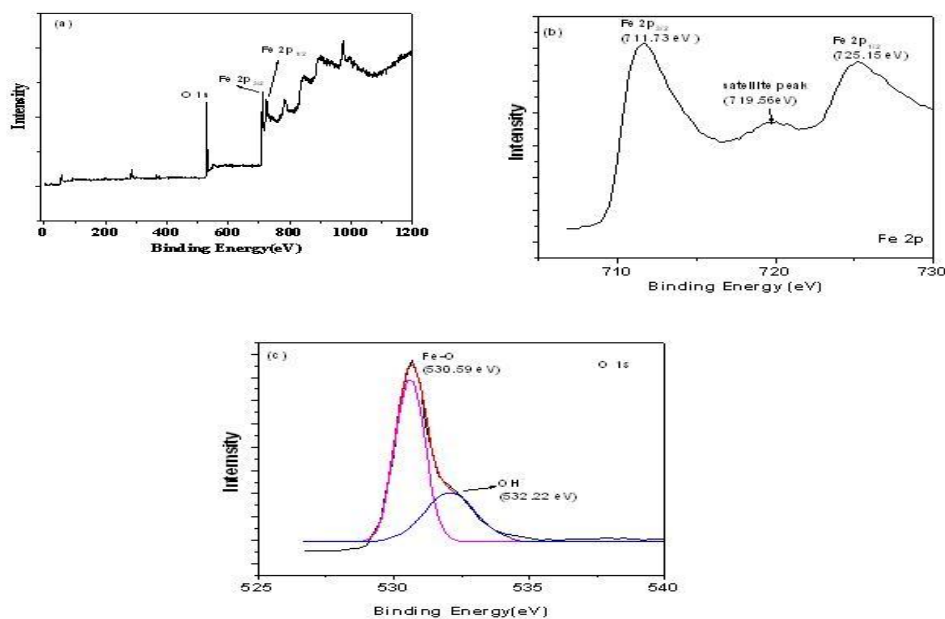


Fig. 5: (a) XPS survey spectrum and XPS spectra (b) Fe 2p and (c) O 1s of synthesized α -Fe₂O₃

3.2 Humidity Sensing Study of α -Fe₂O₃ nanorods

To study the humidity-sensing performance based on α -Fe₂O₃ nanorods, the current (nA) in the sensors was measured as a function of Relative Humidity (10 % RH to 99% RH) at D.C condition and the results are depicted in Fig.6. The current in

the sensor was affected by change in Relative Humidity The current in the sensor changes by two orders of magnitude from 29 nA to 2320 nA RH increases from 11% RH to 99% RH. The change in the current value with RH is attributed to the adsorption of water molecules onto the surface of α -Fe₂O₃ nanorods.

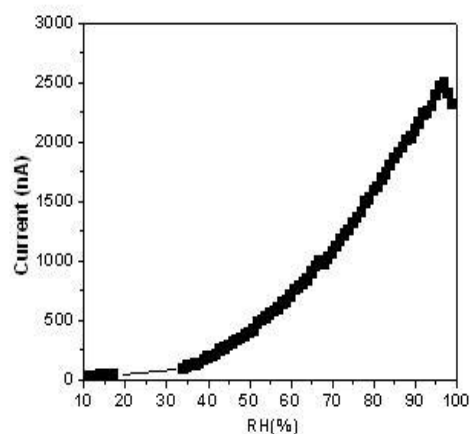


Fig. 6: Variation of current against %RH for synthesized α -Fe₂O₃ nanorods sensing element

Conclusion

α -Fe₂O₃ nanorods have been successfully synthesized by a hydrothermal method by calcinating precursor at 400°C for 2h and their humidity sensing properties are investigated. The synthesized material was characterized by XRD, FESEM, XPS and FTIR to know the phase and

morphology of material. Humidity sensors based on α -Fe₂O₃ nanorods exhibit high and linear response within the whole relative humidity (RH) range of 11%-99% RH at an operating at DC conditions. The corresponding current (nA) changes by approximately two orders of magnitude within the entire humidity range from 11% to 99% RH.

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