

Synthesis and Structural Characterization of Tin-Doped Iron Oxide Nano Crystalline Materials

M.Marimuthu^{*1}, Arunkumar², Balakrishnan³, B.Saravanan⁴, N.Elakkiya⁵
and Abirameswari.R⁶

^{1, 2&5} Department of Mechanical engineering Ramakrishna college of engineering Perambalur

^{3,5& 6} Department of science and humanities Ramakrishna College of engineering Perambalur

Corresponding author: mmarimuthu03@gmail.com

Abstract

Tin oxide nanoparticles have been synthesized by a hydrolysis and co-precipitation method from iron (II) acetate, and tin tetrachloride. The mixed oxide was characterized as a tetragonal cassiterite structure by X-ray diffraction (XRD). X-ray photoelectron spectroscopy (XPS) revealed an electronic interaction between tin and iron atoms in the oxide structure. The addition of iron species to the tin oxide led to a decrease in the crystallite size and changes in the oxidation states of iron cations in the surface region. Based on sensitivity measurements in a semiconductor, iron doping resulted in a shift of the maximum sensitivity toward the lower temperature side.

Keywords: NANOMATERIALS, DOPING, IRON, TIN OXIDE, XRD, FTIR, AND XRD

Date of Submission: 24-03-2023

Date of acceptance: 07-04-2023

I. Introduction

Tin-doped iron oxide, also known as Sn-doped Fe₃O₄ or Sn-Fe₃O₄, is a magnetic nanoparticle material that has gained attention in various fields such as biomedicine, catalysis, and environmental remediation due to its unique properties. The material is composed of iron oxide (Fe₃O₄) nanoparticles doped with tin (Sn), which can be synthesized through different methods including co-precipitation, sol-gel, and hydrothermal techniques. The doping of tin in the iron oxide lattice results in changes in the magnetic, electronic, and structural properties of the material. One of the significant advantages of Sn-Fe₃O₄ is its biocompatibility, which makes it an attractive candidate for biomedical applications such as magnetic resonance imaging (MRI), drug delivery, and hyperthermia treatment. The magnetic properties of Sn-Fe₃O₄ allow it to be used as a contrast agent for MRI, while its surface can be functionalized with bimolecular for targeted drug delivery. In catalysis, Sn-Fe₃O₄ has been used as a catalyst for various reactions such as Fischer-Tropsch synthesis, hydrogenation, and degradation of pollutants. The material's high surface area, magnetic properties, and stability make it a promising candidate for environmental remediation applications such as the removal of heavy metals and dyes from waste water. Low levels of transition metal dopants in semi-conducting oxides have a major impact on the catalytic activity or electrical conductivity of the materials, because they possess variable electronic structures. For example, Fe²⁺/Fe³⁺ redox species in titanium oxide can adjust acid-base properties, thus influencing surface reactivity [1]. Some authors report that Fe³⁺ dopant in V-Ti mixed oxide improves catalytic activity [2] and inhibits phase transition [3]. Moreover, V⁵⁺-dopant in ZnFe₂O₄ serve as an electron donor, increasing n-type electrical conductivity [4]. Addition of V³⁺ into SnO₂ [5] and Ti⁴⁺ into Recent papers that have appeared about mixed oxides composed of three metals as gas-sensing elements include the work on V⁵⁺ in ZnFe₂O₄ [4] and Ba²⁺ in SmCoO₃ [7]. In the present study, our attempts electronic states through addition of Fe²⁺ ions into the V-Sn oxide. We synthesized Fe-V-Sn mixed oxide nanostructures and characterized their crystal structure, surface state and gas-sensing response.

II. Experimental methods:

The synthesis of Sn doped iron oxide nano particles by simple chemical precipitation method was carried out as follows. First 0.1 mole iron oxide solutions were prepared by dissolving 90% of iron (III) chloride anhydrous (FeCl₃) in 100 ml deionized water and 0.1 mole tin oxide solutions were prepared by dissolving 10% weight of tin (II) chloride dihydrate (SnO₂) in 100 ml deionized water. The surfactant 0.1% weight of hexadecyl trimethyl ammonium bromide (HTAB) dissolved in 50 ml deionized water and all prepared solutions stirred separate 1 hour. After that tin chloride solution added drop by drop with prepared iron oxide solution by using burette. The same way HTAB solution was added drop by drop. The mixed solution stirred for 4hrs and

temperature was maintained at 70°C. Then pH of solution maintained at 9 by adding ammonium hydroxide solution drop wise. The resulting product washed 15 times with double distilled water. Finally the precipitated resulting sample was tried at 120°C and then tried sample annealed at 400°C for five hours. In the same way, the pure iron oxide nano particles are prepared and different tin weight % varied such as 30 weight %, 50 weight % samples are prepared by simple precipitation method.

III. RESULTS AND DISCUSSION

3.1. X-Ray Diffraction (XRD) Structure Analysis

X-ray diffraction (XRD) patterns have been used to confirm the formation of rhombohedra phase in iron oxide nano particles with different doping levels of tin. The XRD patterns of sample (b) at 10 weight% of tin doped iron oxide indicate rhombohedral structure with miller indices (022) (104) (023) (110) (214) (125) (116) and well matched with JCPDS 89-0599[10]. The samples (c) at 30 weight% and 50 weight% of Tin Doped Iron Oxide have similar miller indices, with peaks (110) and (104). The average crystalline size is predicted for the sample (d) as 56 nm, with lattice constants slightly changed from standard values.

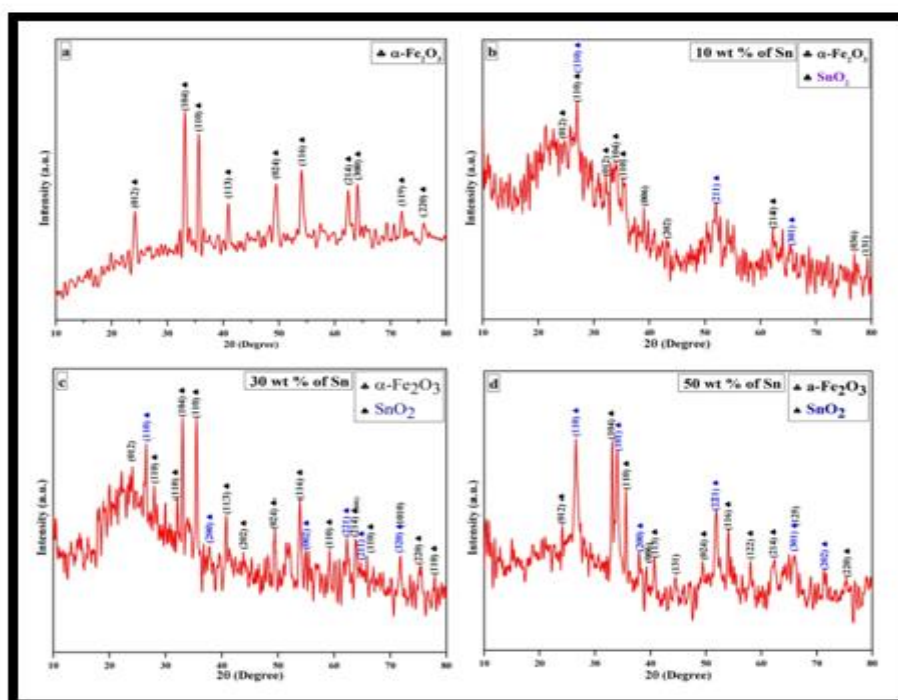


Fig. 1. XRD image of Sn doped iron oxide nanoparticles (a) 0 weight % Sn, (b) 10 weight % Sn, (c) 30 weight % Sn and (d) 50 weight % Sn

3.2. Fourier Transform Infrared Spectroscopy (FT-IR) Spectrum:

Fourier transform infrared spectrum was recorded for the sample (a)-(d) and it is shown in figure (5.2). This is an easy way to identify the presence of certain functional groups in the sample. The FT-IR spectra belong to Alpha-iron oxide nanoparticles, which is prepared by using precipitation method and is calcinated at 400 °C. The figure (a-d) indicates a strong vibrational band at 3370 cm⁻¹ for sample (a), 3392 cm⁻¹ for sample (b), 3382 cm⁻¹ for sample (c) and 3380 cm⁻¹ for sample (d) correspond to O-H stretching[8]. The presence of O-H vibration in the spectrum for the respective samples are appearing due to adsorbed water vapour from the surroundings when it is exposed atmosphere, which (O-H) are due to the expected adsorbed water in the sample[2].

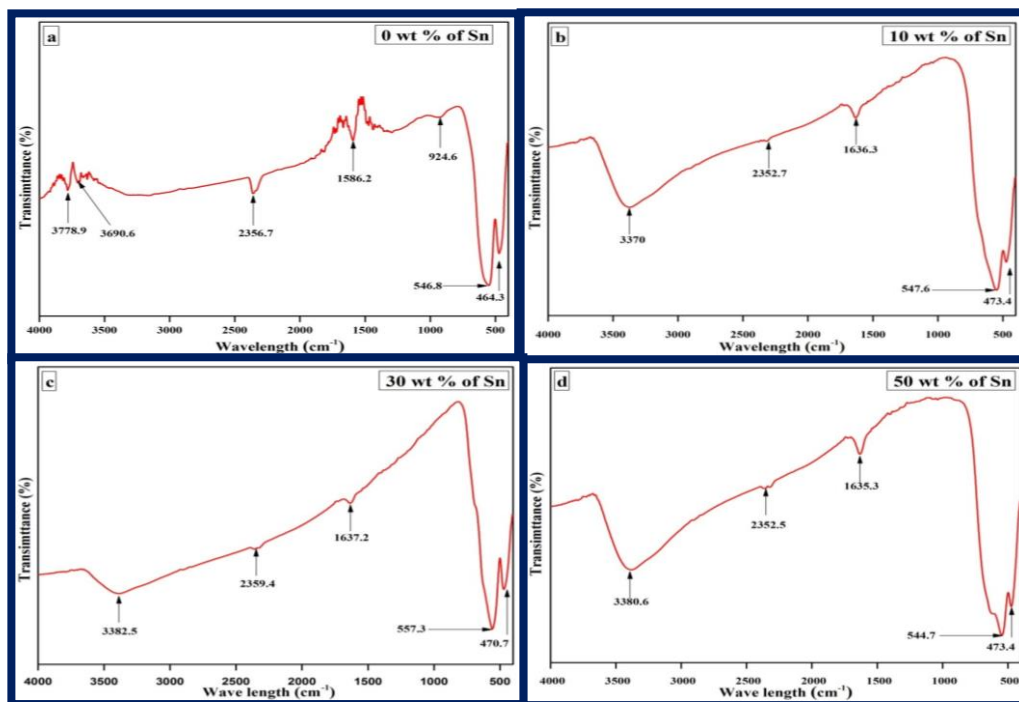


Fig. 2. FT-IR image of Sn doped iron oxide nanoparticles
 (a) 0 weight % Sn, (b) 10 weight % Sn, (c) 30 weight % Sn and (d) 50 weight % Sn

3.3. Morphological studies

Scanning Electron Microscope (SEM):

The SEM images of pure iron oxide nanoparticles and iron oxide doped with tin for different weight% levels show that the spherical shaped particle with less agglomeration is formed after 10 weight% and grows into larger size at 30 weight%. At 50 weight% of tin, most of the smaller spherical particles are disappearing and remaining particles are agglomerated and become a larger grain.

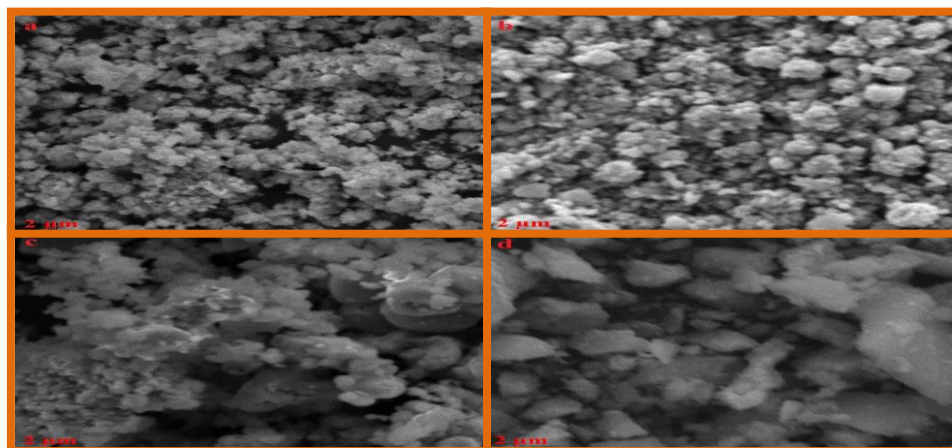


Fig. 3. SEM image of Sn doped iron oxide nanoparticles
 (a) 0 weight % Sn, (b) 10 weight % Sn, (c) 30 weight % Sn and (d) 50 weight % Sn

3.4. Energy-dispersive spectrum (EDAX) Studies

The EDAX spectrum of pure iron oxide nanoparticles shows the elemental presence of Fe, Tin, O, Cl and C. The doping level of Tin and Fe is identified, with stoichiometry stoichiometry of 89% of Fe and 9% of Tin. The atomic% of Tin is gradually increased according to doping level, and the peaks of Cl, O and C are due to the impact of atmosphere and raw materials. Iron oxide nanoparticles conductivity ranges from 1003 to 10-08 Siemens per seconds, with a symmetric, linear behavior and formation of good ohmic contacts[9].

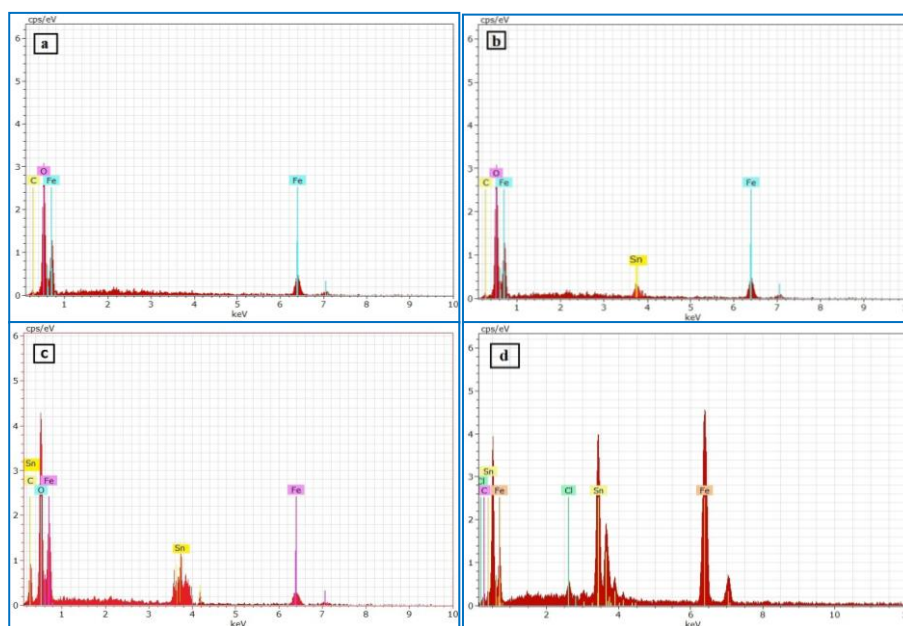


Fig.4. EDAX image of Sn doped iron oxide nanoparticles
(a) 0 weight % Sn, (b) 10 weight % Sn, (c) 30 weight % Sn and (d) 50 weight % Sn

IV. Conclusions

Iron oxide nanoparticles and tin-doped iron oxide nanoparticles were synthesized using a chemical precipitation method. The resulting samples were characterized using various techniques. X-ray diffraction analysis revealed the presence of a rhombohedral phase, while FT-IR analysis confirmed the formation of both tin and iron oxide phases. Scanning electron microscopy analysis confirmed the morphology of the nanoparticles, and energy-dispersive X-ray spectroscopy confirmed the stoichiometric composition of the samples.

Reference

- [1]. Saravanakannan V, Ganesh T, Babeela C, Girisun TS. Analysis of 2PA and optical limiting behaviour for laser applications: Emphatic on surface nature, impedance and non-linear optical properties of tin doped vanadium nanomaterial. *Optical Materials*. 2023 Jan 1;135:113309.
- [2]. Sapkota P, Ageuy-Zinsou KF. Iron and tin phosphide as polymer electrolyte membrane fuel cell cathode catalysts. *International Journal of Hydrogen Energy*. 2023 Jan 1;48(1):257-67.
- [3]. Lin J, Han X, Liu S, Lv Y, Li X, Zhao Y, Li Y, Wang L, Zhu S. Nitrogen-doped cobalt-iron oxide cocatalyst boosting photoelectrochemical water splitting of BiVO₄ photoanodes. *Applied Catalysis B: Environmental*. 2023 Jan 1;320:121947.
- [4]. Benedet M, Rizzi GA, Gasparotto A, Gauquelin N, Orekhov A, Verbeeck J, Maccato C, Barreca D. Functionalization of graphitic carbon nitride systems by cobalt and cobalt-iron oxides boosts solar water oxidation performances. *Applied Surface Science*. 2023 May 1;618:156652.
- [5]. Kumar P, Thakur N, Kumar K, Jeet K. Photodegradation of methyl orange dye by using *Azadirachta indica* and chemically mediated synthesized cobalt doped α -Fe₂O₃ NPs through co-precipitation method. *Materials Today: Proceedings*. 2023 Feb 4.
- [6]. Kim JH, Lee NK, Kim HW, Pan Z, Sohn WY. Sequential modifications of the surface of hematite (α -Fe₂O₃) photoanodes with amorphous nickel iron oxide (NiFeOx) and cobalt phosphate (Co-Pi). *Journal of Photochemistry and Photobiology A: Chemistry*. 2023 Mar 1;437:114478.
- [7]. Dar MA, Mala NA, Bhat MY, Ahamed SR, Reshi BA, Ashok M, Rather AA. Preserved crystal phase and morphology: Improving the magnetic and electrochemical performance of sulfur doped tin oxide nanoparticles synthesized via the hydrothermal method. *Applied Surface Science Advances*. 2023 Feb 1;13:100360.
- [8]. Hassan SS, Fathy MA, Moussa I, Obaida M, Kamel AH. A novel miniaturized pH potentiometric electrode based on a nano bismuth oxide film deposited on a fluorine doped nano tin oxide glass substrate. *Sensors and Actuators B: Chemical*. 2023 Jan 20:133397.
- [9]. Saijo Y, Murata M, Kajihara T, Hijjiya H, Suzuki Y, Akiyama R, Shimizu M, Shimotsuma Y, Miura K. Speciation of tin ions in oxide glass containing iron oxide through solvent extraction and inductively coupled plasma atomic emission spectrometry after the decomposition utilizing ascorbic acid. *Analytical Sciences*. 2022 Jun;38(6):881-8.
- [10]. Deshmane VV, Shinde S, Halwar DK, Jain G, Patil AV. Tin modified Fe₂O₃ thick films for monitoring environmental and industrial pollutant gases. *Chemistry Africa*. 2022 Aug;5(4):1069-82.