

A review on Structural, optical, magnetic and electrical properties of zinc oxide doped with iron

Abstract

This study reviewed the structural, optical, magnetic and electrical properties of zinc oxide (ZnO) nano-particles doped with iron relative to zinc oxide. ZnO is an excellent nanomaterial in semiconductor industries because of its properties such as low cost, high stability and high absorbance. However, its application has been limited because of some undesirable characteristics. Hence, doping with iron has proven essential in overcoming these limitations. The X-ray diffraction studies show that the structure is hexagonal with wurtzite structure, ZnO films prefer to grow in the (002) direction. This is because each apex is parallel to the c-axis in the wurtzite structure. This review reported that optical band gap ZnO nano-particles doped with iron was found to be in the range to (3.26 – 3.35) eV. The electrical properties of these films indicated low resistivity and consequently high electrical conductivity at high dopant iron concentration. Consequently, higher electrical resistivity was observed with lower conductivity at lower iron dopant concentration. Higher iron-doped ZnO ($x=0.10$ and 0.20) samples exhibited obvious ferromagnetic behaviors suggesting that saturation magnetization of $Zn_{1-x}Fe_xO$ nano-particles increases with the increasing of Fe doping concentration compared with pure ZnO. Hence the SEM of iron-doped zinc oxide nano-particles consists of ellipsoidal shape particles, which are well dispersed with smooth surface and uniform size.

Keywords: zinc oxide, nano-particles, iron, Optical, Structural, Magnetic Properties

1.0 Introduction

Nano-particles are micro particles whose particles are usually less than 100nm (Onu et al. 2023). Though they are larger than molecules/atoms but they are lesser in size than objects depicted by Newton's laws. The development of improved nano-composite materials is gaining global attention among scientists because of the numerous applications of the materials in materials development and development. Currently, there is ongoing extensive research on the synthesis of essential and vital third generation semiconductor nano-particles that will have comprehensive relevance on account of their multi-functional characteristics (Faramawy et al. 2022). These nano-composites have copious importance in semiconducting industries, optoelectronic industries and photocatalytic industries (Madkhali et al. 2022). Furthermore, they can be applied in the environmental field for water treatment, gas sensors, solar photocatalysis, etc (Theerthagiri et al. 2019; Hamid et al. 2017; Kumar et al. 2015); in electro-optical fields for solar cells, optical waveguides, light-emitting devices etc (Hwang et al. 2007); in biological fields for biomedical and antibacterial purposes, biosensors etc (Tereshchenko et al. 2016); in electronics for light emitting diode, thin film transistors, etc (Kumar et al. 2015).

2.0 Zinc oxide nano-particles

Zinc oxide (ZnO) is an n-type semi-conductor nano-particle that have many important that have wide spread applications across different industries (Dhiman et al. 2013; Faramawy et al. 2022). This is mainly because of its exceptional chemical and physical characteristics, such as a large direct energy gap (e.g., 3.37 eV at 300 K), excellent conditions of ultraviolet and blue emissions, and a large excitation binding energy (60 meV) at room temperature, which makes it superior to numerous other nanomaterials (Rong et al. 2018). Besides, ZnO is environmental friendly and has hexagonal wurtzite (WZ) which is relatively stable. It has ability to alter electrical conductivity of gaseous samples. Also, ZnO has outstanding properties such as low cost, high stability, with intrinsic ability to absorb UV radiations (Madkhali et al. 2022; Liu et al. 2011). The remarkable morphological features of ZnO make it a striking material that is widely used as a counterpart of a noteworthy scope of applications. However, there are significant drawbacks in the application of ZnO nano-particles. These drawbacks mitigate the effective application of ZnO nano-particles for industrial purposes. The most important one is their toxicity. The toxic effects are usually due to their high solubility which leads to cytotoxicity

and oxidative stress (Carofiglio et al. 2021). Besides, some undesirable optical, electrical and structural properties of pure ZnO nano-particles limit its application. Example, the wide band gap of the ZnO nano-particles depicts low photocatalytic ability in UV rays. Quick recombination of the hole-electron pairs is another major constraint of the ZnO nano-particles (Ebrahimi et al. 2019; Xu et al. 2010). These drawbacks can be overcome with required improvement in the properties of the ZnO through doping of the ZnO nano-particles with transition metals.

3.0 Zinc oxide doped with iron

One of the ways of improving the qualities of zinc oxide is by doping it with other metallic elements. Doping is the process of adding an ion that wasn't initially present in the starting material into a crystal lattice. Doping of ZnO with transition metals has the capability of decreasing the heat conductivity while simultaneously increasing the electrical conductivity of the nanoparticle (Pashkevich et al. 2018). Doping can also be very helpful in modifying the energy band-gap, which will directly affect the photocatalytic capabilities of ZnO. Doping is a potent technique for changing the electrical and optical characteristics of semiconducting materials like ZnO (Carofiglio, et al. 2021). Doping ions cause the creation of novel electronic transitions between energy levels and more defect states in the energy band structure which may result to a decrease in the bandgap of the ZnO and an extension of the matching absorption spectral area from the UV to visible light.

Iron has proved to be one of the most common metals used in doping zinc to improve its properties. Different zinc and iron precursors have been reportedly used in synthesizing zinc oxide particles doped with iron (Table 1). Different conditions and techniques have also been used to synthesize zinc oxide doped with iron.

Table 1: Different precursors and techniques used in synthesis of zinc oxide doped with iron

Zinc precursor	Iron precursor	Synthesis Technique	Synthesis Conditions	Reference
Zn(NO ₃) ₂ ·6H ₂ O	Fe (NO ₃) ₂ ·6H ₂ O	Dissolution / electron beam gun	Substrate temperature = 150°C, Evaporation rate = 2 nm/min Vacuum pressure = 9 mbar Agitated time = 3 hours	Mahmouda et al . 2023
Zn(NO ₃) ₂ ·6H ₂ O (99.99%)	Fe(NO ₃) ₃ ·9H ₂ O (99.99%)	Hydrothermal microwave method	Synthesis time = 20 min pH = 9.5 Pressure = 5.8–6.2 MPa Drying temperature = 60°C Drying time = 18 h	Rosowska et al. (2020)
Zn (CH ₃ COO) ₂ ·2H ₂ O, > 99.5%	Iron nitrate nonahydrate [Fe (NO) ₃ ·9H ₂ O, 98%],	Microwave method / spin coating technique.	Rotation speed = 2500 rpm Rotation time = 30 s Drying temperature = 120 °C Drying time = 4–5 min Annealing temperature = 300 °C.	Ganga et al. 2019
ZnO films (99.95%)	Fe	Sputtering deposition	Pre-sputtering pressure = 1.5 x 10 ⁻⁶ mbar) Sputtering pressure = 50 x 10 ⁻⁴ mbar) Ar gas pressure = 0.5 mTorr Ar gas flow = 20 sccm Sputtering time = 10 min Sputtering voltage = 350 – 390 Film thickness = 150 nm	Faramawy et al. 2022
Zinc acetate	Iron nitrate	Self-modified sol–gel technique	Solution temperature = 60°C Solution time = 2 hours Annealing temperature = 600 Annealing time = 6 hours	Elsheikh et al. (2022)
ZnO (99.99%)	Iron (99.99%)	Magnetron co-sputtering technique	Base pressure = 7×10 ⁻⁵ Pa Flow ratio = 2:1. Total gas-flow rate = 40 sccm, Chamber pressure = 0.5 Pa, Substrate temperature = 400 °C.	Wenjuan and Xueming (2009)
ZnCl ₂ (50 mg/L solution)	FeCl ₂ and FeCl ₃	One-step solution plasma process	Voltage = 2.4 kV Frequency = 60 Hz Cathode = Tungsten Anode = Platinum (Pt, 99.99%) Diameter = 2 mm	Ahmad et al. 2021

Zn(CH ₃ CO ₂) ₂	FeCl ₃	Co-precipitation method	Solution volume = 50ml Drying temperature = 120 °C Drying time = 2 h	Mondal et al. 2019
Zn(C ₅ H ₇ O ₂) ₂ · xH ₂ O	Fe(C ₅ H ₇ O ₂) ₃	Sonochemically ultrasound technique	Ultrasound frequency = 30 ± 3 KHz Ultrasound time = 40s Vacuum pressure = 700–750 mm Hg Vacuum temperature = 65 – 100 °C	Anirban et al. 2016
Zinc acetate dihydrate(Zn(CH ₃ COO) ₂ .2H ₂ O)(99%)	Iron (III) nitrate	Solution chemical method	Solution pH = 9.8 Reflux temperature = 90°C Reflux time = 1 hour Calcination temperature = 200-300 °C	Wiengnon and Anuragodom, 2019.
Zn(NO ₃) ₂ (45mL of 1M)	FeCl ₃ (1% of .1M)	Chemical dissolution method	pH = 9.0 Stirring speed = 250rpm Stirring temperature = 38 ± 1 C Stirring time = 30 mins Drying time = 5h Drying temperature = 65 °C	Muthukumar et al. (2018)
Zinc nitrate hexahydrate (Zn(NO ₃) ₂ .6H ₂ O)	Iron(III) nitrate nonahydrate (Fe(NO ₃) ₃ .9 H ₂ O)	Solution combustion method	Furnace temperature = 500°C Time of combustion initiation= 5 mins	Mahour et al. (2019)
Zinc acetate dihydrate(Zn(CH ₃ COO) ₂ .2H ₂ O)	ferric nitrate (Fe(NO ₃) ₃ .9 H ₂ O, ≥99.0%, AR)	Hydrothermal method	Stirring time = 30 mins Hydrothermal temperature = 180°C Hydrothermal time = 24 hours Washing conditions = 60°C for 24h	Wu et al. 2014
Zinc acetate dihydrate(Zn(CH ₃ COO) ₂ .2H ₂ O)	Fe	Low temperature sol-gel method	Magnetic stirring time = 120 mins Magnetic stirring temperature = 80 °C	Ashraf et al. (2015)
Zn (99%), oxygen and argon	Fe	DC magnetron sputtering	Deposition time = 60 mins Sputtering time = 100W Cleaning time = 15-20 mins	Gao et al. 2013
Zinc chloride anhydrate	ferric chloride anhydrate	Co-precipitation method	Mixing temperature =65 ± 5 °C Mixing time = 30 mins Centrifugation speed = 3000 rpm	Himawan et al. 2019

			Calcinations time = 3 hours Drying condition = 80°C for 3hours	
ZnO (99.99%)	Fe (99.5%), FeO (99.9%), Fe ₂ O ₃ (99.99%)	Mechanical milling	Steel ball diameter = 12mm Ball-to-sample mass ratio = 11.5:1 Oscillation frequency = 32 Hz	Meyer and Damonte (2015)
Zinc Nitrate (98%)	Iron Nitrate (99%)	Sol-gel process	Stirring time = 1hour Solution pH = 7 Furnace temperature = 564°C Furnace time = 6 hours Cooling rate = 274K/min	Dhiman et al. (2013)
Zinc oxide ZnO	FeO, Fe ₂ O ₃ , Fe ₃ O ₄	Open air powder sintering technique	Compact pressure = 200MPa Sintering temperature = 1200 °C Sintering time = 2 hours Cooling rate = 200–300 K/h	Pashkevich et al. (2018)
Zinc acetate dihydrate (Zn(CH ₃ COO) ₂ ·2 H ₂ O, ≥99.0%,	Ferric nitrate nonahydrate (Fe(NO ₃) ₃ ·9 H ₂ O	Wet chemical process	Stabilizing agent = Oleic acid Stirring temperature= 70°C Stirring time = 10 mins	Carofiglio et al. (2021)
Zinc acetate dehydrate Zn(CH ₃ COOH) ₂ · H ₂ O	FeSO ₄ ·7H ₂ O	Spin coating technique	Conc of zinc acetate 0.35M Solution temperature = 50°C Solution stirring time = 1 hour Coating time = 24 hours	Kafle et al. (2016)
Zinc acetate	Ferric chloride	Chemical spray pyrolysis	Substrate temperature = 400°C Solution flow rate = 5ml/min Gas flow rate= 2.5 kg/cm ²	Srinivasulu et al. (2017)

3.1 Properties of iron doped zinc oxide

The purest form of ZnO exhibits diamagnetic properties but the magnetic properties can be enhanced with doping (Carofiglio et al. 2020). Doping with transition metals such as iron can stimulate the alteration from diamagnetism to paramagnetism and ferromagnetism (Ravichandran et al. 2014; Santo et al. 2012; Xu et al. 2009). With the enhanced magnetic properties, the doped ZnO can be applied in bio-imaging processes such as magnetic resonance imaging (MRI) (Carofiglio, et al. 2021). The characteristics of these materials depend not only on the impurity concentration that has been incorporated, but also on whether the impurities are

uniformly distributed in the matrix lattice (wurtzite) or form submicron-sized magnetic clusters that have a particular phase and magnetic structure. The properties of iron-doped ZnO are categorized into structural properties, optical properties, magnetic properties and electrical properties.

4.0 Structural properties

The structural properties of the iron-doped ZnO are usually investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM) and Scanning electron microscopy-energy dispersive X-ray (SEM-EDX). Furthermore some properties such as crystalline size, dislocation density, unit cell volume are used to study the structural properties of the iron-doped ZnO. Other properties such as include x-ray density, lattice parameters and lattice strain can also reveal additional properties of the nano material.

4.1 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a surface imaging method in which the incident electron beam scans across the sample surface and interacts with the sample to generate backscattered and secondary electrons that are used to create an image of the sample. It is a test process that scans a sample with an electron beam to produce a magnified image for analysis. The method is also known as SEM analysis and SEM microscopy, and is used very effectively in microanalysis of morphology and interfacial quality of the nanoparticles (Ozobialu et al. 2022). Scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDX) provides a quick non-destructive determination of the elemental composition of the nano particle.

Wu et al. (2014) observed the morphology and microstructure of the iron doped ZnO nanoparticles through high-resolution transmission electron microscopy (HRTEM). The images of the iron-doped zinc oxide nano-particles ($Zn_{0.95}Fe_{0.05}O$) reveal the presence of randomly distributed ellipsoidal shaped nano-particles. The iron-doped zinc oxide nano-particles depicted ellipsoidal shape particles and well dispersed with smooth surface and uniform size. The crystalline quality improved with the iron doping.

In the morphology analysis of the ZnO and Fe doped ZnO thin films, Faramawy et al. (2022) observed that the pure zinc oxide film surface seems to be agglomerated and spherical in shape. The surface of the films consists of tightly packed grains forming a smooth surface without any voids and cracks whereas the Fe doped zinc oxide films look like black and white islands of

spherical shape. Still, by increasing the DC deposited power for Fe with ZnO deposition, the black spots decreased.

Wu et al. (2014) reported that the synthesized iron-doped zinc oxide nano-particles confirmed the presence of mainly iron, zinc, and oxygen with traces of copper and carbon. In the EDX spectrum Fe doped ZnO thin films, Faramawy et al. (2022) reported that the observed peaks are only attributed to zinc, oxygen and iron as principal elemental components with no other impurities being obtained.

4.2 Photoluminescence analysis

Photoluminescence (PL) can be used to determine the structure of the nano-particles. Both the iron-doped zinc oxide nano-particles and the pure zinc oxides were compared by Wu et al. (2014). The pure ZnO sample contains six main emission peaks but the emission peak position of doped sample exhibits a slight blue shift with increase of Fe³⁺ concentration. This is attributed to defects or the oxygen vacancies in ZnO induced by iron doping. Furthermore, the visible light (VL) emission band is also suppressed with the increase of the iron concentration.

4.3 X-ray diffraction (XRD)

XRD is a scattering of X-rays by the atoms of a crystal that produces an interference effect so that the diffraction pattern gives information on the structure of the crystal or the identity of a crystalline substance. Ashraf et al. (2015) reported that there is no change in the wurtzite structure of ZnO after iron doping hence complete doping of iron was obtained. The full width at half maximum increased with increase in iron doping concentration.

Usually, iron ions were incorporated in the lattice of the host crystals. For Fe-doped ZnO dilute magnetic semiconductors, the peak position of the doped ZnO samples shifts to lower angles compared with pure ZnO, and also there is a decrease in the intensity of peaks with the Fe doping concentration. This shifting as well as decrease in intensity of the characteristic peak clearly indicates the successful incorporation of Fe in the ZnO matrix. It is indicated that Fe ions occupy the Zn ions sites in the hexagonal wurtzite structure and no impurity phase (Wu et al. 2014). The sputtered ZnO and ZnOFe thin films with a c-axis have a preferential orientation due

to the lowest surface free energy. In the equilibrium state, the films grow with the plane of the lowest surface free energy parallel to the surface if there is no effect from the substrate (Faramawy et al. 2022).

4.4 Crystalline size

The crystallite size (D) for iron doped ZnO nano-particles is usually calculated using the Debye–Scherer formula given in equation (1) (Onu et al. 2023)

$$D = \frac{0.9\lambda}{\beta \cos \Theta} \quad (1)$$

Where, β is full width at half-maximum, λ is the X-ray wavelength and θ is Bragg's diffraction angle.

According to Ashraf et al. (2015), there is a decrease in the crystallite size (29-19nm) of the zinc oxide nano-particles with increase in the quantity of iron doping observed. Dislocation density has been found to increase with increase in the iron content of the doped ZnO nano-particles. Dislocation density was calculated by using formula $\frac{1}{D^2}$. There was linear increase in dislocation density with increase in the iron content of the doped ZnO.

Furthermore, there is usually a decrease in lattice parameter and therefore unit cell volume as the concentration of the iron increase in the doped zinc oxide nano-particle. This is mainly because of the difference in ionic radii of zinc and iron ions.

5.0 Magnetic properties

5.1 Magnetization

Room temperature ferromagnetism was usually observed for iron doped zinc oxide. Saturation magnetization was increased from 0.065 to 0.088 emu/g with increase in doping concentration (Ashraf et al. 2015). There was increase in saturation magnetization with increase in dopant concentration of iron. However, even undoped nano-particles can show ferromagnetic behavior. In addition to the Fe doping ions, the presence of lattice defects such as strain and texture may also contribute significantly to the room temperature magnetic properties (Faramawy et al. 2022; Pan et al. 2007). Ferromagnetism in transition metal doped zinc oxide may be because of secondary magnetic phases or due to the oxidation of Fe in mixed valence states (Wang et al. 2010; Kumar et al. 2009).

5.2 Magnetic hysteresis

The room temperature ferromagnetism of the $Zn_{1-x}Fe_xO$ nano-particles could arise from two possible sources. One is extrinsic magnetism and the other is intrinsic magnetism. Extrinsic source includes the formation of clusters of transition elements or secondary phase. Wu et al. (2014) reported on the magnetic hysteresis (M–H) loops of the $Zn_{1-x}Fe_xO$ nano-particles with different doping consistency ($x = 0, 0.01, 0.05, 0.10, \text{ and } 0.20$) measured at room temperature. The saturation magnetization of $Zn_{1-x}Fe_xO$ nano-particles increases with the increasing of Fe doping concentration of ZnO nano-particles.

In view of the Fe^{3+} ions substituted into ZnO lattice, the origin of magnetism in the samples is due to the exchange interaction between local spin polarized electrons (such as the electrons of Fe^{3+} ions) and the conductive electrons. Such interaction can lead to the spin polarization of conductive electrons. Consequently, the spin-polarized conductive electrons undergo an exchange interaction with local spin-polarized electrons of Fe^{3+} ions. Thus, after a successive long-range exchange interaction, almost all Fe^{3+} ions exhibit the same spin direction, resulting in the ferromagnetism of the material (Elilarassi and Chandrasekaran, 2012).

6.0 Optical properties

6.1 Transmittance Spectra

This is usually the ratio of the transmitted power to the incident power in a tiny wavelength interval around the center wavelength and in a certain (sufficiently small) solid angle of aperture. According to Faramawy et al. (2022), the pure ZnO and the iron-doped ZnO nano-particles films' optical transmittance spectra displayed high transmission in the visible spectrum and a significant decline in the UV spectrum. However, doping with iron increased the transmittance. The interaction of the incident long wavelength radiation with the free electrons in the films is what causes the reduction in transmittance (Yusuf et al. 2019). Besides the films also exhibit strong transmission in the visible area of the spectrum, which ends at shorter wavelengths, but there is a clear absorption in the UV region that became more pronounced with increasing Fe concentration.

6.2 Optical band energy gap and Urbach energy

The optical band gap energy (E_g) is defined as the sum of the bulk band gap energy, the electron and hole confinement energies. It is the minimum energy required to make an electron pairing in the semi-conductor. It is given by equation 2.

$$\alpha h\nu = A(h\nu - E_g)^n \quad (2)$$

Where α is the absorption coefficient, $h\nu$ is the photon energy, A is a constant, E_g is the bandgap, and n is for modes of transition (1/2) for direct transition and 2 is for indirect transition)

The optical band gap is the threshold for photons to be absorbed. According to Faramawy et al. (2022), the values of the band gap increased from the pure ZnO nano-particles to the doped ZnO nano-particles.

The optical band gap energy is mainly dependent on the valency of Fe ions in an iron doped ZnO nano-particles (Salem et al. 2016). The optical characteristics of ZnO thin films are significantly influenced by the valence state of Fe. By incorporating Fe onto ZnO films, the valence band's maximum is raised and the conduction band's minimum is lowered, which reduces the band gap. This trend is supported by Wang et al. (2009) and Chen et al.(2007) who reported that increase in the Fe doping concentration in ZnO nano-particles can reduce the band gap. However, when Fe^{3+} ions are substituted for Zn^{2+} ions, this leads to increase in the concentration of extra free carriers. Consequently, the band gap increases and the Fermi level shifts closer to the conduction band.

The absorption spectrum of a pure semiconductor is known to be dramatically altered by doping, leading to degenerate energy levels that cause the Fermi level to rise over the conduction band edge. This change is referred to as a Moss-Burstein shift and is caused by doping-induced band-filling. It leads to significant increase in the band gap energy from the undoped to doped nanoparticle films (Dhiman et al. 2013; Shanmuganathan and Banu, 2014).

On the other hand, the Urbach energy evaluates the degree of structural disorderliness of the nano-films. The Urbach energy increases as the Fe concentration increased. Fe doping is followed by an increase of the strain which can also create structural disorder. The dopant may contribute significantly to the width of localized states within the ZO's optical band. Hence, the Urbach energy is responsible for the valence and conduction bands' tails (Shaban et al. 2017).

7.0 Electrical properties

Electrical conductivity (EC), σ , is a measure of a material's ability to carry an electrical current. It is also defined as how much voltage is required to get an amount of electric current to flow. This is largely determined by the number of electrons in the outermost shell. These electrons determine the ease with which mobile electrons are generated. Zinc oxide nano-particles have low electrical conductivity (Faramawy et al. 2022). Doping zinc oxide with transition metals (such as iron) is one of the major ways of improving the electron transport rates and subsequently increase the electrical conductivity (and reduce the electrical resistivity) in the nano-particles (Ueda et al. 2001).

Cherif et al (2016) reported similar trend of higher electrical resistivity (lower conductivity) at lower dopant concentration. This trend of lower conductivity from pure zinc oxide to Fe doped zinc oxide may be due to carrier traps formation at the film surface causing impediment to charge carriers and due to Fe incorporation, with the formation of interstitial metal atoms causing a decrease in the oxygen vacancies (Cheng et al. 2016). Besides, increasing the concentration of Fe led to an increase in the number of surface defects in the zinc oxide matrix. This will result to the electrical resistivity of ZnO being reduced as the concentration of Fe in ZnO is increased slightly. Hence the conductivity is increased.

8.0 Conclusion

Doping with transition metals such as iron will significantly improve the structural, electrical, magnetic and optical properties of zinc oxide nano-particles. As reported, the refractive index (n) decreased with increasing the Fe concentration of the doped zinc oxide nano-particles which correlated to the optical band gap behavior. Also, an increase of the saturation magnetization values was noted with increase in Fe doped concentration. Furthermore, the resistivity of the zinc oxide nano-particles reduced as the concentration of Fe in ZnO is increased slightly resulting in increase in the conductivity of the doped zinc oxide nano-particles.

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