Investigation on anneal-tuned properties of ZnFe$_2$O$_4$ nanoparticles for use in humidity sensors

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Abstract
The effect of different annealing temperatures on structural, optical and magnetic properties of ZnFe$_2$O$_4$ nanoparticles prepared using the coprecipitation technique has been investigated. With the increase in annealing temperature, crystallinity and average crystallite size of nanoparticles increased. The average crystallite size was found to be 5.55 nm, 6.62 nm and 32.9 nm for the samples annealed at 300 °C, 500 °C and 700 °C, respectively. The X-ray diffraction and Fourier-transform infrared spectroscopy revealed the formation of a cubic spinel structure. The optical direct and indirect bandgap energy decreased with an increase in annealing temperature. The saturation magnetization increased from 16.38 emu/g to 25.91 emu/g. The M–H curves depicted the magnetic phase transition from superparamagnetic to ferrimagnetic. The electrical properties were investigated using an impedance analyzer in the frequency range of 300 Hz to 1 MHz. The conduction properties showed enhancement with increased annealing. The humidity sensing properties were investigated in the range of 15–90% RH and revealed a strong dependence of adsorption capacity on the annealing temperature. Electrical conductivity improved with increased humidity. Excellent humidity sensitivity was observed for ferrites annealed at 700 °C attributed to increased crystallinity and reduced lattice strain making them a potential candidate for use in humidity sensors.

Keywords Ferrites · AC conductivity · Magnetism · Optical properties · Humidity · Sensors

1 Introduction
The unique combination of structural, magnetic and dielectric properties of spinel ferrites makes them the most studied ceramic materials in recent years. These properties promote the use of ferrites in various fields like high-frequency applications, electronic devices and biomedical field as catalysts, inductors, sensors, transformer cores, choke coils, filters, for drug delivery and nonreciprocal devices, etc. [1–11]. Great attention is focused nowadays on sensors due to increased environmental concerns. Precise humidity measurement is an important aspect in areas like the agricultural sector, manufacturing industries, food storage applications as well as indoor and outdoor air quality [12–17]. Optimal humidity level holds utmost importance in industries for increasing production efficiency [18, 19]. The increased relative humidity is associated with decreased SARS-CoV-2 transmission, the first pandemic of the twenty-first century [20–23]. Detrimental effects are observed on plant growth with imbalance humidity levels [24–26]. A good humidity sensor possesses several characteristics like high sensitivity, chemical and thermal stability, reversibility and fast response time. Spinel magnetic oxides hold a great advantage for potential use in humidity sensors due to their porous structure, large surface-to-volume ratio, humidity varying resistivity, low cost, ease of synthesis and suitability in a diverse operating environment [27–31]. Humidity, in general, is specified by ‘relative humidity (RH),’ which represents the amount of water vapor in the air at a given temperature. Zinc ferrite, a class of normal spinel ferrite, is reported to be highly stable with good response, high sensitivity, magnificient magnetic properties, tunable optical properties and excellent electrical properties [32–35]. An extensive literature survey reveals a vast majority of sensors are based on ZnFe$_2$O$_4$ nanoparticles [36–40]. Also, ZnFe$_2$O$_4$ shows the highest humidity sensitivity among other ferrite nanoparticles [41]. The literature lacks the study of humidity response of annealed...
ZnFe$_2$O$_4$ nanoparticles. The sensing properties of a spinel ferrite are characterized by their magnetic and electrical properties which depend on structure, crystallinity, size and composition. Heat treatment improves the crystallinity of materials by enhancing chemical ordering which influences the physicochemical properties of prepared nanocrystalline materials [42–44].

In the present study structural, magnetic, optical and electric properties of ZnFe$_2$O$_4$ nanoparticles prepared by coprecipitation technique and annealed at different temperatures were investigated. The effect of varying relative humidity on the electrical response of annealed samples is studied for potential use in humidity sensors.

2 Experimental

2.1 Materials and preparation

The chemicals used during the preparation were of analytical grade and used without further purification. The process involved the preparation of separate homogenous solutions of Zn(NO$_3$)$_2$.6H$_2$O and Fe(NO$_3$)$_3$.9H$_2$O in distilled water according to the stoichiometric ratio. The solutions were mixed properly using a magnetic stirrer maintained at a temperature of 65 °C. Oleic acid was used as a surfactant to avoid the agglomeration and oxidation of the particles. Ammonia was added dropwise to the solution till pH 10 was attained to obtain precipitates. The precipitates so formed were subjected to increased heat (80 °C) to transform precipitates into hydroxides. The obtained product was washed with distilled water to remove unwanted salt residues. The dried sample was then powdered using a pestle to get ZnFe$_2$O$_4$ magnetic nanoparticles (MNPs). The prepared ferrite powder was annealed at 300 °C, 500 °C and 700 °C to improve crystalline properties. Cylindrical pellets of diameter 10 mm and thickness 3 mm were made from annealed samples under a force of 10 ton/cm$^2$.

2.2 Characterization

The crystallinity, structure and phase purity of the annealed samples were identified using X-ray diffraction (Rigaku Ultima-IV powder X-ray diffractometer) employing Cu-Ka radiation over the range of 20–70 °C and Fourier-transform infrared (FTIR) spectra recorded using a PerkinElmer Frontier FTIR spectrophotometer in the range 4000–450 cm$^{-1}$. The thermal stability of MNPs was studied using thermogravimetric analysis (TGA) in the temperature range of room temperature–800 °C at a heating rate of 10 °C/min. The optical parameters were measured using an ultraviolet–visible spectrophotometer (PerkinElmer Lambda 950 UV–VIS) in the wavelength range of 200–1600 nm. For magnetic measurements, a vibrating-sample magnetometer (LakeShore model 7400 VSM) is used within the range of ± 1 T. Prepared pellets were exposed to different humidity values in the range of 15 to 90% RH in a closed chamber for a constant time. Dielectric properties of pellets before and after humidity exposure were analyzed using an impedance analyzer in the frequency range of 300 Hz–1 MHz.

3 Results and discussion

3.1 X-ray diffraction (XRD) study

Figure 1 shows the XRD pattern of ZnFe$_2$O$_4$ nanoparticles annealed at 300 °C, 500 °C and 700 °C indexed as Z3, Z5 and Z7, respectively. Broad peaks with less intensity representing crystalline structure with dominant amorphous phase are observed for Z3 and Z5, whereas Z7 showed high-intensity peaks depicting enhanced crystalline structure. All the diffraction peaks (220), (311), (222), (400), (422), (511) and (440) observed match well with the standard diffraction data (JCPDS card no. 22–1012) and confirm the formation of cubic spinel structure with Fd3m space group. The peak corresponding to the (311) plane, demonstrating the highest intensity, was employed to calculate average crystallite size (D) using the Debye–Scherrer formula [45].

$$D = k\lambda / \beta \cos \theta$$

In the above equation, k, $\lambda$, $\beta$ and $\theta$ denote the shape factor (0.9), X-ray wavelength (1.5406 Å), full width at half maximum of diffraction peak and Bragg’s diffraction angle, respectively. The lattice constant (a), X-ray density ($\rho_x$), $\rho_x$, $\sigma_x$, $\tau_x$.
specific surface area (S), bulk density (ρ) and porosity (P) are calculated using Eqs. 2–6 [46, 47].

\[ a = d \sqrt{h^2 + k^2 + l^2} \]  

(2)

\[ \rho x = \frac{Z \times M}{N \times a^3} \]  

(3)

\[ S = \frac{6}{\rho} \times D \]  

(4)

\[ \rho = \frac{m}{\pi r^2 h} \]  

(5)

\[ P = 1 - \frac{\rho}{\rho x} \]  

(6)

In the above equations, d is the interplanar spacing and h, k and l are Miller indices of the plane. The results obtained are shown in Table 1.

The average crystallite size of Z3 was found to be 5.55 nm and showed a minor increase for Z5. A significant increase in average crystallite size was observed for Z7. The increase in thermal energy of grains due to annealing promotes atomic diffusion resulting in increasingly larger grain size [48].

The increase in lattice constant with annealing temperature is attributed to the redistribution of cations among tetrahedral sites (A-sites) and octahedral sites (B-sites) [49]. Annealing the prepared samples also reduces the lattice defects and strains, thereby causing lattice expansion. X-ray density, specific surface area and porosity decrease with annealing. Humidity sensing being a surface phenomenon significantly depends on the porosity and grain size. A higher surface area with a suitable pore size leads to enhanced humidity adsorption [50].

### 3.2 Infrared spectral analysis

The Fourier-transform infrared spectra for Z3, Z5 and Z7 are shown in Fig. 2. It gives an insight into atomic bond vibrations in the infrared region. The perceptible bands in the spectra can be identified based on Waldron’s theory [51]. The intensity variation in the observed peaks signifies the amount of the corresponding functional group. The bands at ~3400 cm\(^{-1}\) and 1600 cm\(^{-1}\) depict the presence of adsorbed water on the NPs surface. These significant absorption bands indicate the hygroscopic nature of the prepared ferrites and their suitability in humidity sensing applications. The intensity of these peaks represents the amount of residual water in the sample. The peaks at ~2900 cm\(^{-1}\) and 1300 cm\(^{-1}\) confirm the presence of organic compounds used during the synthesis process. The decreased intensity of these peaks demonstrates the removal of water and surfactant on annealing. A characteristic peak at ~550 cm\(^{-1}\) relates to the intrinsic vibration of metal–oxygen (M–O) bonds at A-sites. The peak shift toward lower wave number with increased intensity revealing the reduced tetrahedral bond strength with an increased amount of the functional group. The observation suggests the increased occupancy of Zn\(^{2+}\) ions at A-sites with increased annealing temperature, thereby elongating the tetrahedral bond. It eventually leads to lattice expansion. The results are in agreement with XRD data analysis.

### 3.3 Thermal analysis

The thermogravimetric analysis gives useful information about the decomposition behavior of spinel ferrite NPs with temperature. The TGA and its derivative (DTG) curve for

| Sample | D (nm) | a (Å) | ρx(g/cc) | S(m\(^2\)/g) | ρ(g/cm\(^2\)) | P | Direct bandgap (eV) | Indirect bandgap (eV) |
|--------|-------|-------|----------|-------------|--------------|---|-------------------|-------------------|
| Z3     | 5.6   | 8.389 | 5.42     | 199.30      | 1.17         | 0.78 | 2.83             | 0.31              |
| Z5     | 6.6   | 8.395 | 5.41     | 167.44      | 1.66         | 0.69 | 2.71             | 0.22              |
| Z7     | 32.9  | 8.402 | 5.40     | 94.17       | 2.01         | 0.63 | –                | –                 |

Fig. 2 FTIR spectra of Z3, Z5 and Z7. (Inset- Peak corresponding to tetrahedral metal–oxygen bond)
as-prepared ZnFe₂O₄ NPs are shown in Fig. 3. The curve shows a two-step decomposition process. The first weight loss (2.79%) at ~100 °C is due to the removal of adsorbed water during the synthesis. The second major weight loss (56.3%) between 250 and 450 °C accounts for the removal of oleic acid used as a surfactant to bind the NPs [52]. At this stage, the amorphous dominating crystalline state is completely transformed to a regular nanocrystalline structure. For a temperature higher than 450 °C, the sample becomes thermally stable. Thus, it can be inferred that heat treatment above 450 °C removes all organic compounds to form pure phase spinel ferrite NPs.

3.4 Optical studies

Figure 4 shows the absorption spectra of samples annealed at 300 °C and 500 °C to study the effect of annealing on the optical properties of ZnFe₂O₄ nanoparticles. Both FTIR and UV–VIS spectroscopy is useful to investigate the vibrational energy levels and optical energy levels, respectively. The energy in FTIR does not cause electron excitation but it is sufficient to affect the chemical bonds, thereby causing the stretching and bending of molecules. The UV–VIS absorption intensity increases with an increase in annealing temperature. The electrons absorb the light energy and get excited. Due to the instability of higher energy states, they return to the ground state by emitting energy equivalent to the absorbed energy in the form of heat or light. Quantitatively, the intensity of absorption reflects the presence of chromophores in the molecules. The absorption peak at ~300 nm corresponds to the inter-sublattice charge transfer transition (ISCT) from 3d⁵ to 3d⁴4s¹ in Fe³⁺ ions. The absorption band due to inter-valence charge transfer transition (IVCT) among Fe³⁺ ions at B-sites is noted at ~1400 nm [53, 54].

The optical direct and indirect bandgap of the samples can be estimated using Tauc’s relation [55].

\[ a\nu = A[(\nu - E_g)^n] \]

(7)

In the above equation, \( a \), \( \nu \), \( E_g \) and \( A \) are the absorption coefficient, photon energy, bandgap and proportionality constant, respectively. The value of \( n \) defines the type of electronic transition, i.e., \( n = 1/2 \) for direct bandgap transition and \( n = 2 \) for indirect bandgap transition.

The extrapolation of the linear part of plots of \((a\nu)^2\) versus \(\nu\) and \((a\nu)^{1/2}\) versus \(\nu\) (shown in Fig. 5) to intersect x-axis gives the value of bandgap energy which is tabulated in Table 2. The decrease in optical bandgap with an increase in annealing is attributed to the electron confinement at the nanoscale. The phenomenon can be explained using the model of a potential well with infinite walls. The confinement of electrons in the conduction band and holes in the valence band can be altered by varying the lattice parameter. The charge carriers are weakly confined in crystals with larger lattice parameter and require less energy for electron excitation [56]. The results validate Brass’s effective mass model [57].

3.5 Magnetic properties

Figure 6 shows the hysteresis curve of the samples recorded at room temperature. The VSM data are used to extract the magnetic parameters such as saturation magnetization \( M_s \), retentivity \( M_r \) and coercivity \( H_c \). These values are further used to calculate the squareness ratio \( S \), anisotropy constant \( K \) and magnetic moment \( \eta_B \) [58]. The obtained values are presented in Table 2.
Magnetic parameters strongly depend on average crystal-
lite size. The observed magnetic behavior reflects modifica-
tion in the structure of NPs. The hysteresis-absent curves for
Z3 and Z5 show superparamagnetic nature with negligible
retentivity and small values of coercivity. The M-H curve for
Z7 depicts a ferrimagnetic behavior. The saturation magneti-
zation increases with an increase in annealing temperature
attributable to the increased average crystallite size. Accord-
ning to the dead layer theory, as smaller crystallites possess a
larger surface-to-volume ratio, more atoms are captured by
the surface relative to the core. This induces surface disor-
der which reduces the saturation magnetization. The reduc-
tion in surface disorder enhances magnetization [59]. The
increase in coercivity with annealing demonstrates the single
domain nature of MNPs [60]. The behavior of superpara-
magnetism for ZnFe$_2$O$_4$ nanoparticles with average crystal-
lite size ~ 5 nm is reported previously [61, 62]. The values
of squareness ratio lie below 0.5 depicting magnetostatic
particle interactions. The magnetic moment increases with
annealing due to the movement of Zn$^{2+}$ ions from B-sites to
A-sites. It is inferred that annealing above 500 °C results in
the transformation of superparamagnetic NPs with dominat-
ing amorphous phase to ferrimagnetic NPs with enhanced
crystalline structure simultaneously maintaining the single-
domain particles.

**Table 2** Magnetic parameters of Z3, Z5 and Z7

| Sample | Saturation magnetization (emu/g) | Magnetic coercivity (Oe) | Retentivity (emu/g) | Anisotropy constant (erg/Oe) | Squareness ratio | $\eta_B (\mu_B)$ |
|--------|---------------------------------|--------------------------|---------------------|-----------------------------|-----------------|-----------------|
| Z3     | 16.38                           | 34.50                    | 0.29                | 576.64                      | 0.017           | 0.70            |
| Z5     | 18.60                           | 36.82                    | 0.30                | 698.94                      | 0.016           | 0.80            |
| Z7     | 25.91                           | 94.74                    | 1.31                | 2504.81                     | 0.050           | 1.11            |

**Fig. 5** Tauc plot for direct and optical bandgap of Z3 and Z5

**Fig. 6** M-H curves for Z3, Z5 and Z7
3.6 Electrical properties

The variation in AC electrical conductivity of prepared ferrites with frequency at room temperature is shown in Fig. 7. The effect of annealing on the electrical properties of prepared ferrites is depicted in the figure. The conductivity initially increases slowly at lower frequencies followed by a steep increase at higher frequencies. The relation between AC conductivity and frequency is given by Eq. 8 [63].

\[ \sigma_{\text{AC}}(\omega, T) = B(T)\omega^n(T) \]  

(8)

In the above equation, B and n are temperature-dependent constants. The increase in the conductivity values can be explained using Maxwell–Wagner polarization theory and Koop’s two-layer model [64]. The nonconducting grain boundaries are dominant at low frequencies. These insulating grain boundaries trap the electrons, resulting in low AC conductivity values. The value of n belonging to the range 0–1 depicts short-range transitions of frequency-dependent AC conductivity [65].

The AC conductivity values increase with an increase in annealing temperature. The observation can be attributed to the increased crystallite size, cation redistribution and reduced defects [66].

3.7 Humidity sensing study

The variation in AC conductivity with relative humidity at a constant high frequency is shown in Fig. 8. The AC conductivity increases with an increase in relative humidity. The conduction mechanism significantly depends on water adsorption and proton conduction on the surface of ferrite. The adsorption of water vapors results in the dissociation of hydrogen ions which form hydroxyl groups by bonding with lattice Fe ions. This leads to free electron liberation which facilitates the conduction mechanism [67, 68]. The phenomenon is represented in (Eqs. 9 and 10).

\[ H^+ + \text{OO} \leftrightarrow [\text{OH}]^- \]  

(9)

\[ [\text{OH}]^- + \text{Fe} \leftrightarrow [\text{OH}^- - \text{Fe}] + e^- \]  

(10)

In Eq. 11, \( \text{OO} \) is the oxygen present at the lattice site. The plot can be explained by a two-stage process attributed to a low humidity region and a high humidity region. In the first stage, two surface hydroxyls per molecule are formed by surface chemisorptions of water molecules. The conductivity takes place due to the hopping mechanism of electrons in the chemisorbed layer. In the second stage corresponding to the high humidity region, capillary condensation of vapors takes place in the pores. As a consequence both the Grotthuss

| Humidity range (% RH) | Humidity sensitivity (%) |
|-----------------------|--------------------------|
| Z3                    | Z5                       | Z7                       |
| 15–30                 | 35                       | 40.3                     | 42.3                     |
| 30–45                 | 7.1                      | 8.79                     | 12.89                    |
| 45–60                 | 2.07                     | 5.9                      | 11.7                     |
| 60–75                 | 0.6                      | 2.6                      | 4.4                      |
| 75–90                 | 0.53                     | 0.4                      | 1.3                      |

Table 3 Humidity sensitivity for different humidity range for Z3, Z5 and Z7
Investigation on anneal-tuned properties of ZnFe$_2$O$_4$ nanoparticles for use in humidity…

The humidity sensitivity coefficient is calculated using Eq. 11.

\[
\text{Sensitivity coefficient} = 100 \times \frac{\Delta \sigma}{\sigma} \quad (11)
\]

In the above equation, \(\Delta \sigma\) is the increase in conductivity at x% RH and \(\sigma\) is the conductivity at lower RH. The calculated sensitivity coefficients are tabulated in Table 3.

The table reveals that sensitivity decreases as we move from the low RH region to the high RH region. Also, the sensitivity is highest for Z7 despite possessing the largest crystallite size, lowest surface area and porosity. Similar unanticipated observations have been reported in the previous study where annealing has a significant impact on the sensitivity of materials [50, 71–75]. The inverse relation between the lattice strain and sensitivity has been proposed in the literature [72]. The highest sensitivity of Z7 can be attributed to its ordered structure, enhanced crystallinity, greater stability and reduced lattice strain.

4 Conclusion

In this article, we have synthesized ZnFe$_2$O$_4$ nanoparticles using the chemical coprecipitation technique. The prepared NPs were annealed at 300 °C, 500 °C and 700 °C to improve the structural, magnetic, optical and electrical properties. X-ray diffraction revealed the formation of the amorphous-phase dominant crystalline NPs annealed at 300 °C and 500 °C although further annealing helped to enhance crystallinity to a maximum extent. The average crystallite size of the samples belongs to the range of 5–33 nm. FTIR confirmed the spinel ferrite structure of the samples. The thermal analysis demonstrated a two-step weight loss approach to attain a stable form with the complete removal of organic compounds adsorbed during the synthesis process. Absorption spectra in the UV–VIS region depict the enhancement of optical properties with annealing. The magnetic measurements exhibited superparamagnetic to ferrimagnetic phase transition on annealing. The AC conductivity of prepared ferrites in the frequency range of 300 Hz–1 MHz increased with annealing temperature. The humidity sensing properties were examined in a broad relative humidity range of 15–90%. AC conductivity showed a substantial increase with relative humidity. A significant impact of annealing on humidity sensitivity properties of ZnFe$_2$O$_4$ nanoparticles is observed indicating their suitability for efficient humidity sensors operating at room temperature.

Author Contributions
Anu Rana contributed to supervision, software, validation, writing-reviewing and editing; Nitika contributed to data curation, writing-original draft preparation, investigation and formal analysis; Vinod Kumar contributed to conceptualization, methodology, visualization and resources.

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Availability of data and material
The authors declare that all the data supporting the findings of this study are available within the article.

Compliance with Ethical Standards

Conflicts of interest
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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