

Room Temperature Acetone and Ethanol Sensing Using Al-Substituted NiFe_2O_4 Nanoparticles: Synthesis, Characterization, and Performance

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Abstract: This study reports the synthesis and characterisation of aluminium-doped nickel ferrite ($\text{NiFe}_{2-x}\text{Al}_x\text{O}_4$, where $x = 0, 0.02$, and 0.04) nanomaterials prepared by citric acid-modified sol-gel method. The materials were calcined at 600°C for 2 hours and sintered at 600°C for 3 hours. X-ray diffraction analysis confirmed the formation of a single-phase cubic spinel structure with lattice parameters ranging from 8.27 to 8.22 Å. The crystallite size decreased from 47 nm to 42 nm with increasing aluminium content, as calculated by the Williamson-Hall method. FTIR spectroscopy revealed the characteristic metal-oxygen vibrations of the ferrite structure. The FESEM images show nanoscale grains with good porosity. The gas-sensing performance was tested against acetone and ethanol vapours at room temperature. The $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$ sample showed the highest response, with R_g/R_a values of 30.15 and 44.71 for acetone and ethanol, respectively, at 200 ppm. The room-temperature sensing capability makes these materials promising for practical applications in gas-detection devices.

Key words: Nickel ferrite, Aluminum doping, Sol-gel synthesis, Gas sensor, Room temperature sensing, Acetone detection, Ethanol detection

1 Introduction

Environmental pollution, industrial safety and public health have raised concerns for detecting harmful gases and volatile organic compounds (VOCs). The materials used in gas sensors enable the continuous monitoring of harmful gases across various applications [1]. Gas sensors track air quality by detecting toxic gases such as NO_2 , CO , and VOCs that contribute to smog formation and respiratory diseases. In industrial environments, sensors enable the early detection of harmful gases, thereby protecting workers from hazardous chemicals [2]. Gas sensors are also being developed for non-invasive disease diagnosis through breath analysis, as specific VOCs serve as biomark-

ers, such as acetone in diabetes and hydrocarbons in lung cancer [3]. High sensitivity and selectivity are essential for detecting these gases at low concentrations.

Metal oxide semiconductor (MOS) sensors have attracted attention due to their low cost, high sensitivity, simple fabrication, and compatibility with mass production [4]. MOS gas sensors work through surface-controlled charge transfer reactions. When exposed to air, oxygen molecules adsorb onto the sensor surface, causing electron capture in n-type semiconductors and hole capture in p-type semiconductors. This forms charged oxygen species such as O_2^- , O^- , and O^{2-} , depending on operating temperature [5]. This adsorption creates a depletion or accumulation layer near the

surface, modulating electrical conductivity. When gas is detected, the adsorbed charge carrier releases back into the material, causing changes in electrical resistivity [6]. Nanoscale materials enhance sensor performance by providing greater surface area. Nanostructures like nanoparticles, nanorods, and thin films can be produced using hydrothermal synthesis, sol-gel processing, and screen printing [4].

Spinel ferrites have emerged as promising candidates for gas sensors due to their unique structural, electronic, and catalytic properties. With the general formula MFe_2O_4 (where M represents a divalent metal cation), they belong to the space group Fd-3m, with metal cations occupying tetrahedral (A) and octahedral (B) sites [7]. This arrangement provides thermal and chemical stability, making them suitable for use in all environments and at high temperatures [8]. Spinel ferrites' inherent catalytic ability promotes oxidation reactions due to multiple metal cations with variable oxidation states (Fe^{3+}/Fe^{2+}), facilitating redox reactions and catalytic oxidation of VOCs and reducing gases [2]. This catalytic behaviour enables faster response and recovery times than non-catalytic oxides. The electrical properties can be tuned by varying metal cation composition, controlling carrier concentration, conductivity type, and bandgap energy [5]. Their morphology can be modified at the nanoscale to achieve porous, hollow, and hierarchical structures, providing high surface areas for gas adsorption and improving the sensing performance [2]. Recent studies have demonstrated that rare-earth doped ferrites can further enhance gas sensing capabilities, with Nd^{3+} doping in Cd-Zn ferrites increasing LPG sensitivity by 4-20 times and reducing response times [9]. These features, with tunable electronic properties, make spinel ferrites ideal for high-performance gas sensors.

Among various spinel ferrites, nickel ferrite ($NiFe_2O_4$) has attracted scientists and engineers as a gas-sensor material due to its electronic structure and semiconduct-

ing properties. $NiFe_2O_4$ (NFO) exists in an inverse spinel structure where Ni^{2+} ions occupy octahedral sites, while Fe^{3+} ions distribute between tetrahedral and octahedral sites [7]. This cation distribution creates an electronic environment influencing the material's properties. While most metal oxide sensors show n-type semiconducting behaviour, NFO exhibits p-type conductivity, with holes as the majority carriers [10]. The p-type nature comes from Ni^{2+}/Ni^{3+} redox couples and hole transport between nickel ions in octahedral sites. This mechanism produces a chemiresistive response where reducing gases increase the resistance by desorbing surface oxygen and replenishing holes [10]. Recent work has confirmed the p-type chemiresistive response of $NiFe_2O_4$ nanoparticles, showing detection capabilities down to ~ 10 ppm for ethanol with fast response times below 10 s [10].

NFO provides key benefits for gas sensing. It has a catalytic activity that enables a fast response and recovery [11]. Its bandgap of 1.7 eV in ultrathin nanostructures ensures chemical reactivity and thermal stability at operating temperatures (150-300°C) [11]. NFO can form various nanostructures like particles, rods, sheets, and porous architectures, allowing control of surface area and active sites [4].

Previous research shows NFO's gas sensing capabilities for various VOCs and toxic gases. For acetone, NFO nanoparticles showed a response of 120% to 100 ppm at 190°C [4]. Pt-coated mesoporous NFO nanorods achieved a response of 48.2 to 100 ppm at 160°C with 1 ppm detection limit [12]. Recent advances have shown that amorphous Pt decoration on NFO nanorods can increase the acetone response by 37 times while lowering the optimal operating temperature [13]. For toluene, porous ultrathin NFO nanosheets demonstrated a sensitivity with a 50 ppb detection limit at 220°C, with response and recovery times of 11.6 and 41.9s, respectively [11]. MOF-derived porous NFO nanooctahedrons detected toluene at 1 ppm at 260°C [2]. Ethyl acetate detection using porous NFO gives

a response of 64.27 to 200 ppm at 120°C with 0.26 ppm detection limit [5]. For formaldehyde, NiO/NFO composite nanotetrahedrons showed a response of 33.3 to 200 ppm at 240°C with a 200 ppb detection limit [14]. Heterojunction approaches have proven effective, with NiO/NFO heterostructures reducing the acetone operating temperature to 170°C and increasing the response by 4.7 times compared to pure NFO [15]. NFO nanospheres demonstrated a specific response to xylene (68.5% at 70 ppm) with reproducibility over 70 days [1], and recent hydrothermal ultrasonication synthesis methods have produced NFO nanospheres with strong selectivity for xylene across 25-150 the temperature range [1]. These studies show that NFO-based sensors can detect VOCs from the ppb to ppm levels, with performance affected by morphology, surface area, and operating conditions.

NFO has potential for gas sensing, but its performance can be enhanced through compositional modifications. Doping or substituting iron or Nickel with other elements can tailor the structural and catalytic properties of spinel ferrites [5]. The incorporation of cations modifies the properties of NFO through several mechanisms.

Substituting cations alters the crystal-field environment and superexchange interactions among metal ions. Different cations have distinct ionic radii, electronegativities, and oxidation states, influencing electronic density near the Fermi level and band structure [3]. This modifies carrier concentration and mobility, affecting electrical conductivity and sensor performance. Substitution impacts charge-carrier concentration by introducing charge compensation mechanisms or changing metal ion ratios in various oxidation states. Adding trivalent cations like Al^{3+} can influence the $\text{Ni}^{2+}/\text{Ni}^{3+}$ balance and alter hole concentration in p-type NFO [10]. Additionally, cation substitution affects the grain size, crystallite size, and surface morphology. Substituent cations may inhibit grain growth, resulting in smaller crystallites

with higher grain boundary density [3]. This increases the surface area and enhances gas the adsorption sites. Substitution also modifies the catalytic activity by changing the surface chemistry and oxygen vacancy availability. Oxygen vacancies, acting as gas adsorption sites, can be regulated by selecting substituents with different valences and ionic sizes [5]. Studies on mixed ferrite systems have shown that cation substitution significantly alters activation energies and sensing kinetics, with $\text{Zn}_{0.5}\text{Ni}_{0.5}\text{Fe}_2\text{O}_4$ exhibiting different activation energies for ethanol (0.26 eV) versus acetone (0.34 eV), affecting selectivity [16]. Replacing aluminium (Al^{3+}) is important for modifying NFO. With an ionic radius of 0.535 Å, Al^{3+} is smaller than Fe^{3+} (0.645 Å), causing lattice contraction [3]. Al^{3+} incorporation into $\text{NiFe}_{2-x}\text{Al}_x\text{O}_4$ maintains inverse spinel symmetry while changing lattice parameters. Studies show increasing Al content decreases crystallite size, potentially improving gas sensing surface area [3]. Al substitution increases resistivity and modifies dielectric behaviour, affecting carrier mobility [3]. These modifications suggest Al substitution could impact gas sensing performance, though its influence on VOC detection needs investigation.

Research on substituting cations for nickel in NFO has shown that composition changes can improve sensing performance. Replacing NFO films with Sn enhances gas sensing at room temperature [6], while adding Ru increases hydrogen sensitivity [5]. These substitutions demonstrate that selecting appropriate cations can improve sensitivity, selectivity, temperature, and response dynamics. Computational studies combined with experimental work on Ni-Zn ferrites have suggested that certain compositions may achieve high-sensitivity VOC sensing at room temperature, with surface hydroxyl groups playing key roles in adsorption mechanisms [17].

Recent progress in spinel ferrite gas sensors has focused on morphological modification, surface functionality, and composites to improve the sensing performance. Syn-

thesis techniques like MOF-derived methods, hydrothermal processes, and template-assisted strategies have produced high-surface area nanostructures [2][5]. Surface modification with noble metals like Pt and Pd enhances sensitivity and lowers operating temperatures [12]. Recent work on Pt-functionalized hierarchically structured flower-like NFO sheets has shown high-performance acetone sensing capabilities [18]. Composites combining NFO with metal oxides like NiO show improved selectivity and reduced detection limits [14]. Comparative reviews of nickel and cobalt spinel ferrite nanoparticles have highlighted their promise for pollutant removal and gas sensing, with some ferrite compositions demonstrating room-temperature sensing capabilities [19]. Alternative synthesis routes such as coprecipitation have produced ZnFe_2O_4 and $\text{ZnO}/\text{ZnFe}_2\text{O}_4$ structures with very high responses to acetone and ethanol [20]

Despite extensive research on pure NFO and morphological modifications, systematic studies on Al substitution's effect on NFO's VOC sensing properties remain limited. Research on Al-substituted NFO has mainly focused on structural, magnetic, dielectric, and thermal properties [3], while gas sensing performance remains unexplored. This gap is significant, as Al substitution's structural and electronic modifications could influence key sensing parameters including sensitivity, detection limits, selectivity, operating temperature, and stability. The relationship between Al substitution level, defect chemistry, and VOC sensing behaviour has not yet been established.

Understanding how Al substitution affects crystallite size, surface area, carrier concentration, and catalytic activity is crucial for improving ferrite based gas sensors.

This study investigates gas-sensing properties of Al-substituted NFO ($\text{NiFe}_{2-x}\text{Al}_x\text{O}_4$) nanoparticles toward volatile organic compounds. The objectives are: (1) to synthesize $\text{NiFe}_{2-x}\text{Al}_x\text{O}_4$ nanoparticles with varying Al substitution using a sol-gel method;

(2) to characterize structural, morphological, and compositional properties using X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), and Fourier Transform Infra-red (FTIR) spectroscopy; (3) to make pellet gas sensors from Al-substituted NFO nanoparticles; (4) to evaluate gas sensing performance toward VOCs and (5) to correlate sensing behaviour with structural modifications induced by Al substitution. This investigation provides insights into cation substitution's role in modulating gas-sensing properties of spinel ferrites and contribute to developing improved VOC sensors for environmental monitoring, industrial safety, and healthcare applications. Here study has been particular focused on acetone and ethanol gas sensing which is crucial because acetone is a validated biomarker for diabetes mellitus, detectable in exhaled breath at elevated concentrations in diabetic patients, enabling non-invasive glucose monitoring, and ethanol is critical for breath alcohol testing, workplace safety monitoring, and detection of volatile organic compounds (VOCs) in industrial environments [21]. Both gases are important classes of VOCs relevant to public health and environmental safety.

2 Experimental Details

2.1 Synthesis of $\text{NiFe}_{2-x}\text{Al}_x\text{O}_4$ Nanomaterials

The Al-doped NFO nanomaterials were synthesized using the citric acid modified sol-gel method for which different precursor such as nickel nitrate hexahydrate [$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\geq 98\%$], iron nitrate nonahydrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\geq 98\%$], aluminum nitrate nonahydrate [$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\geq 98\%$], and citric acid monohydrate [$\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$, $\geq 99\%$] were taken. All chemicals were procured from SRL and used without further purification. Three compositions were prepared: NFO (Al_1), $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$ (Al_2), and $\text{NiFe}_{1.96}\text{Al}_{0.04}\text{O}_4$ (Al_3). For the synthesis of

each composition, the appropriate amounts of metal nitrates were taken based on the stoichiometric ratio. The metal nitrates were dissolved in distilled water to obtain a homogeneous solution under continuous magnetic stirring at room temperature. Citric acid was added to the solution in a 1:1 molar ratio with respect to the total metal ions. The solution was stirred continuously at 80°C until it formed a viscous gel. The temperature was then raised to 120°C to induce self-combustion in the gel, producing a fluffy powder. The obtained powder was ground into a fine powder using a mortar and pestle. The powder was then calcined in a muffle furnace at 600°C for 2 hours with a heating rate of 5°C per minute. After calcination, the powder was again ground to ensure uniformity. For gas-sensing measurements, the calcined powder was pressed into circular pellets using a hydraulic press at 3 tons. The pellets had a diameter of approximately 10 mm and a thickness of about 1.5 mm, which were further sintered at 600°C for 3 hours to improve their mechanical strength and electrical contacts.

3 Characterization Techniques

The phase purity and structural parameters of the synthesised materials were studied using XRD. XRD patterns were recorded at room temperature using a diffractometer with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) over the range 20°-80°. FTIR analysis was performed to identify the vibrational bonds to support the XRD phase-purity information. The spectra were recorded in the wavenumber range of 400–1200 cm $^{-1}$ at room temperature using the KBr pellet technique. The surface morphology and microstructures of the samples were observed using FESEM. The samples were coated with a thin layer of gold to make them electrically conductive before imaging. The gas-sensing properties of the sintered pellets were measured at room tempera-

ture, v utilising a Keithley 2420 sourceme-ter. Silver paste was applied to both sides of the pellets to create electrical contact. Copper wires were attached to the silver electrode. The sensor was placed inside a sealed glass chamber with gas inlet and out-let ports. The chamber's internal pressure was attained using a rotary pump. The target gases (acetone and ethanol) were introduced into the chamber by injecting a known volume of liquid through the septum. The gas concentration in ppm, related to the volume of liquid, was calculated using the equation [22]:

$$C = \frac{22.4\rho TV_S}{273MV} \times 1000 \quad (1)$$

In this context, C represents the concentra-tion of the gas in parts per million (ppm), T denotes the testing or operating temper-ature measured in kelvin, ρ indicates the density of the liquid sample (target gas) in grammes per millilitre (g/ml), V_S refers to the volume of the liquid sample (target gas) in microlitres, M signifies the molec-ular weight of target gas in grammes per mole (g/mol), and V stands for the vol-ume of the chamber in litres. The liquid evaporated inside the chamber and mixed with air to create the desired gas concen-trations. Concentrations of 1 ppm, 10 ppm, 20 ppm, 50 ppm, 100 ppm, and 200 ppm were tested. The electrical resistance of the sensor was measured using a digital multi-meter. The resistance in air (R_a) was first recorded. Subsequently, the target gas was introduced, and the gas resistance (R_g) was measured continuously until it stabilised. Thereafter, the chamber was opened to al-low fresh air to enter, and the recovery of resistance was monitored.

4 Results and Discussion

4.1 X-ray Diffraction Analysis

The XRD patterns of the three synthesised samples, i.e. Al1, Al2, and Al3 are shown in Figure 1. All the samples show clear diffraction peaks that match well with the

standard pattern of NFO, which belongs to the space group Fd-3m, a signature of the cubic spinel structure. The peaks were indexed as the Fd-3m space group sym-

metry. No additional peaks from impurity phases were detected, confirming that a pure single-phase NFO was formed in all the samples.

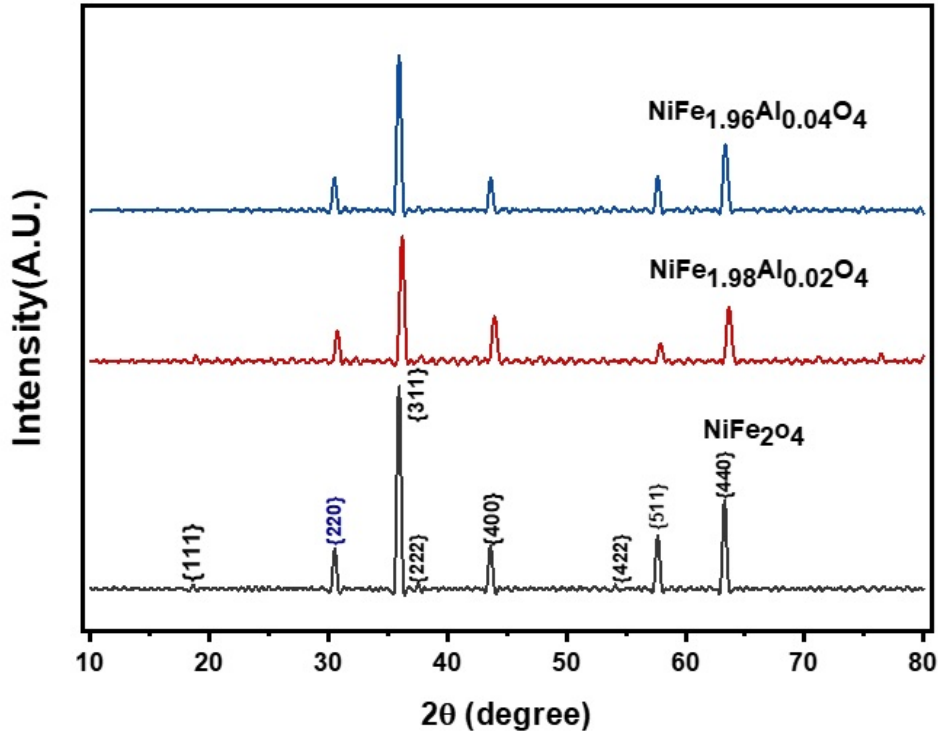


Figure 1: XRD Pattern of $\text{NiFe}_{2-x}\text{Al}_x\text{O}_4$ ($x=0.0, 0.02$ and 0.04)

A careful observation of the XRD patterns revealed that the peaks shifted slightly to higher angles with increasing Al content. This shift indicates that the crystal lattice is becoming smaller. This happens because Al^{3+} has a smaller ionic radius ($0.535 \text{ \AA}$) compared to Fe^{3+} , which has an ionic radius of $0.645 \text{ \AA}$ [23]. When smaller Al ions replace larger Fe ions in the crystal structure, the lattice shrinks slightly. This is called lattice contraction. The lattice parameter (a) was calculated from the XRD data using the formula for cubic crystal systems [24]:

$$d = a \times \sqrt{h^2 + k^2 + l^2} \quad (2)$$

where d is the interplanar spacing, and $(h \ k \ l)$ are the Miller indices of the crystal plane. There are several intense diffraction peaks corresponding to the (111), (311),

(220), (222), (400), (422), (511) and (440) planes, which are characteristic of the cubic spinel structure. The (311) plane, being the strongest reflection in the XRD pattern, was given primary consideration to calculate lattice parameter using Eq. (2). The values of the lattice parameters are listed in Table 1. The lattice parameter decreased from $8.27 \text{ \AA}$ for pure NFO to $8.22 \text{ \AA}$ for the sample with the highest Al doping ($x = 0.04$). This confirms the successful incorporation of Al ions into the NFO structure. The crystallite size of the nanoparticles was calculated using the Scherrer Formula [24]:

$$D_s = \frac{k\lambda}{\beta \cos \theta} \quad (3)$$

where D_s is the crystallite size, K is the shape factor (usually taken as 0.9), λ is the X-ray wavelength ($1.5406 \text{ \AA}$ for Cu- K_α), β is the full width at half maximum

(FWHM) of the diffraction peak in radians, and θ is the Bragg angle. The FWHM was determined by fitting the highest intensity peak (311) with a Lorentzian function as shown in Figure 2 for the Al2 sample. The

crystallite size obtained from eq. (2) are listed in Table 1 below. We can see that the crystallite size decreases with increasing aluminum doping.

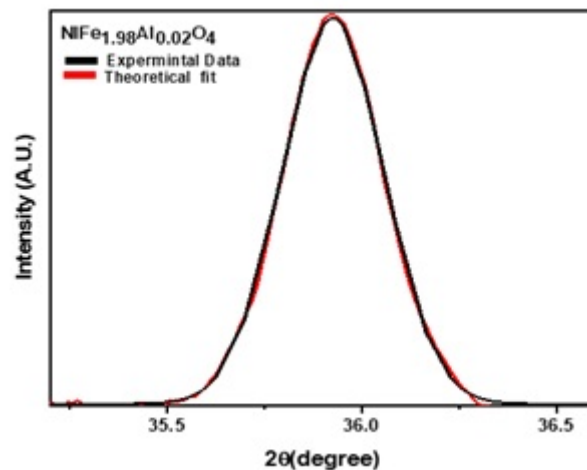


Figure 2: Lorentzian Fit of highest intensity peak from (311) plane of $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$.

In Eq. (3), β is taken from the highest intensity peak, which corresponds to the reflection from the (311) plane, to incorporate the reflection from all planes. Eq. (3) has been modified by taking the log on both sides, and the resulting formula is the modified Scherrer equation, which is defined below [24]:

$$\ln(\beta) = \ln \frac{K\lambda}{D_{MS}} + \ln \frac{1}{\cos\theta} \quad (4)$$

where D_{MS} is the crystallite size obtained from the modified Scherrer equation, and

Modified Scherrer has contributions from all reflecting planes, but both the Scherrer equation and the modified Scherrer equation ignore the contribution from strain. So to consider contribution from strain, Williamson Hall (WH) formula has been used to calculate particle size and strain, which is defined as [24]:

$$\beta \cos\theta = \frac{k\lambda}{D_{WH}} + 4\epsilon \sin\theta \quad (5)$$

where D_{WH} is crystallite size obtained from

other symbols have their usual meaning. In Eq. (4), the FWHM (β) was determined for each peak individually. The modified Scherrer method gives crystallite sizes of 38 nm, 35 nm, and 31 nm for Al1, Al2, and Al3 samples, respectively (Table 1). These values are slightly larger than those obtained from the simple Scherrer formula because the β is averaged by taking contributions from all reflecting planes. Figure 3 shows the plot used for the modified Scherrer calculation for the Al2 sample.

WH formula, ϵ is strain and other symbols have their usual meaning. Similar to the Modified Scherrer Formula, β is taken for each peak separately, that is, for each peak corresponding to the recorded Bragg's angle. If we plot $\beta \cos\theta$ versus $4\sin\theta$ for different diffraction peaks, we get a straight line. The slope of the line gives the strain, and the y-intercept gives the crystallite size of the corresponding phase. Figure 4 shows the WH plot for the Al3 sample. Similar plots were made for all samples.

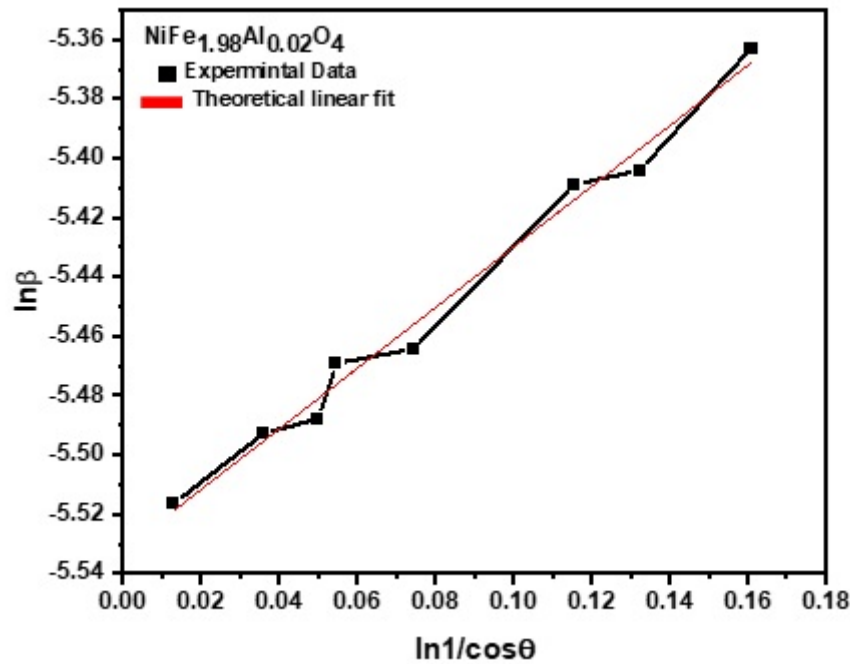


Figure 3: Plot of modified Scherrer equation for $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$.

The crystallite sizes obtained from the WH method are 47 nm, 44 nm, and 42 nm for Al1, Al2, and Al3 samples, respectively (Table 1). These values are larger than those from the Scherrer methods because the WH method removes the effect of strain broadening. The lattice strain values calculated from the WH plots are listed in Table 1. The strain increased with Al doping. This is expected because when Al ions replace iron ions, they create a mismatch in the

crystal structure due to the size difference. This mismatch creates stress in the lattice, which appears as strain [25]. The decrease in crystallite size with Al doping can be attributed to Al's effect on crystal growth. During calcination, the nanocrystals grow by combining smaller particles. Al ions can hinder this growth process by creating defects and strain in the lattice. This prevents the crystals from growing too large, resulting in smaller crystallite sizes [26].

Table 1: D_S : Particle size calculated by Scherrer Formula; D_{MS} : Particle Size calculated by modified Scherrer Formula; D_{WH} : Particle Size Calculated by Williamson Hall Plot; a : lattice parameter, ϵ : lattice strain calculated from Williamson Hall plot

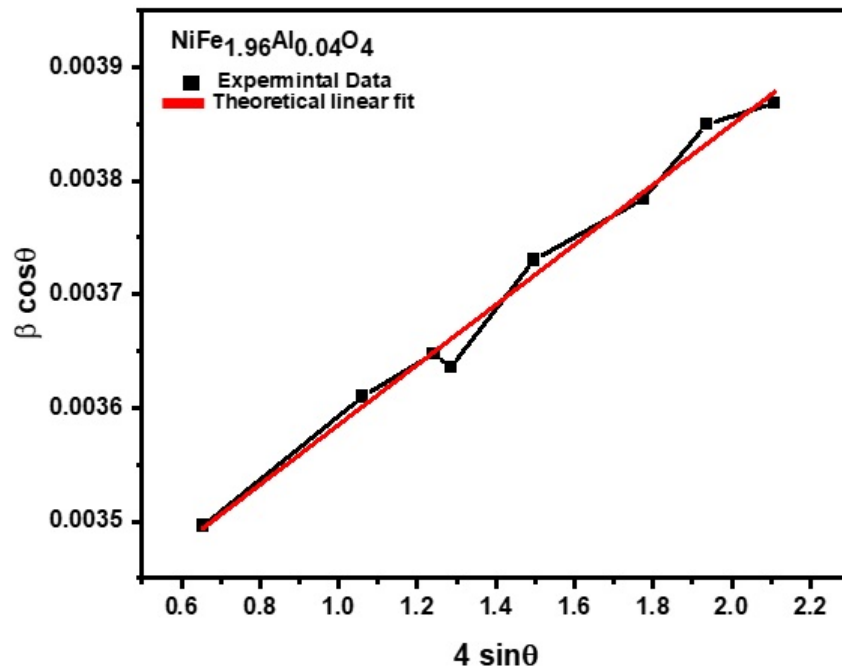
Sample Name	a (Å)	D_S (nm)	D_{MS} (nm)	D_{WH} (nm)	ϵ
NiFe_2O_4	8.27	33	38	47	1.98×10^{-4}
$\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$	8.25	30	35	44	2.10×10^{-4}
$\text{NiFe}_{1.96}\text{Al}_{0.04}\text{O}_4$	8.22	28	31	42	2.80×10^{-4}

Further, the X-ray density (d_x) was calculated using the following formula [24]:

$$d_x = \frac{8M}{Na^3} \quad (6)$$

where M is the molecular weight of the sample, N is Avogadro's number (6.022×10^{23}),

and a is the lattice parameter of the sample. The bulk density (d_B) was experimentally determined from the mass and dimensions of the sintered pellets. The porosity (P) was then calculated using [24]:

Figure 4: Williamson Hall Plot for $\text{NiFe}_{1.96}\text{Al}_{0.04}\text{O}_4$.

$$P = 1 - \frac{d_B}{d_x} \quad (7)$$

Table 2 shows the calculated values of d_x , measured d_B and calculated P , bulk density, and porosity for all samples. Here, d_x and d_B appears to be contradicting these each other as they show opposite trend. But, this is not a contradiction but reflects two fundamentally different density measures. d_x is the theoretical crystallographic density calculated from unit cell parameters and represents the density of the perfect crystal lattice without defects or pores. It increases with Al doping ($5.504 \rightarrow 5.564 \text{ g/cm}^3$) because Al^{3+} substitution causes lattice contraction ($8.27 \rightarrow 8.22 \text{ \AA}$), making the unit cell more compact despite Al being lighter than Fe. On the other hand d_B is the macroscopic density measured ex-

perimentally, accounting for all structural features including pores, voids, and grain boundaries. It decreases ($4.044 \rightarrow 3.713 \text{ g/cm}^3$) because Al doping increases porosity ($26.54\% \rightarrow 33.27\%$), creating more void space in the material. This means that the Al-doped samples were less dense than pure NFO. The decrease in d_B is related to the increased porosity of the samples. Higher porosity is highly beneficial for gas-sensing applications. Porous materials allow gas molecules to penetrate deep into the sensor material, increasing the contact area between gas and sensor. This leads to higher sensitivity and faster response [27]. The increased porosity in Al-doped samples can be attributed to greater strain and defects in the crystal structure, which create more voids and channels within the material.

Table 2: d_x : X-ray density; d_B : bulk density; P : Porosity

Sample	$d_x \text{ (gm/cm}^{-3}\text{)}$	$d_B \text{ (gm/cm}^{-3}\text{)}$	$P \text{ (\%)}$
Al1	5.504	4.044	26.54
Al2	5.531	3.894	29.61
Al3	5.564	3.713	33.27

4.2 FTIR Analysis

Figure 5 shows the FTIR spectra of Al2 and Al3. In ferrites, the most important features in FTIR spectra are the metal-oxygen vibration bands. Two strong absorption bands were observed in the FTIR spectra of the spinel ferrites. The first band appears around 550 cm^{-1} and is assigned to the stretching vibrations of metal-oxygen

bonds at tetrahedral sites (Fe-O or Al-O bonds, in which metal ions are surrounded by four oxygen ions). The second band appears around 400 cm^{-1} and is assigned to the stretching vibrations of metal-oxygen bonds at octahedral sites (metal ions surrounded by six oxygen ions). The presence of these bands confirms the formation of spinel ferrite structure [3], [28].

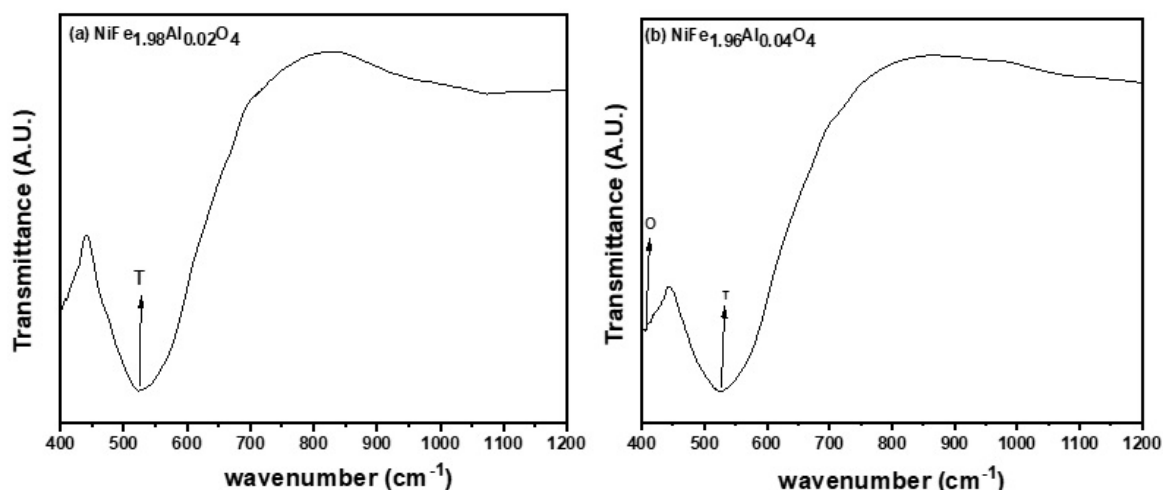


Figure 5: FTIR spectra of (a) $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$ and (b) $\text{NiFe}_{1.96}\text{Al}_{0.04}\text{O}_4$.

Figure 6 shows the FESEM images of Al2 and Al3 samples. From the images, we can observe that the particles have irregular shapes and are distributed across the surface. The particles were in the nanometre size range, which agrees with the crystallite size calculated from XRD data. However, the particles showed little agglomeration due to high surface energy. Despite agglomeration, we can still identify individual grains in the images. The grain boundaries are visible in many places. The average grain size appears to be in the range of 40-60 nm, which is consistent with the Williamson-Hall crystallite size calculations.

4.3 Gas Sensing Analysis

Before conducting the actual gas-sensing measurements, we first tested the semicon-

ducting behaviour of the material synthesised using the van der Pauw technique (data not shown here), which showed that all samples exhibited p-type semiconduct-

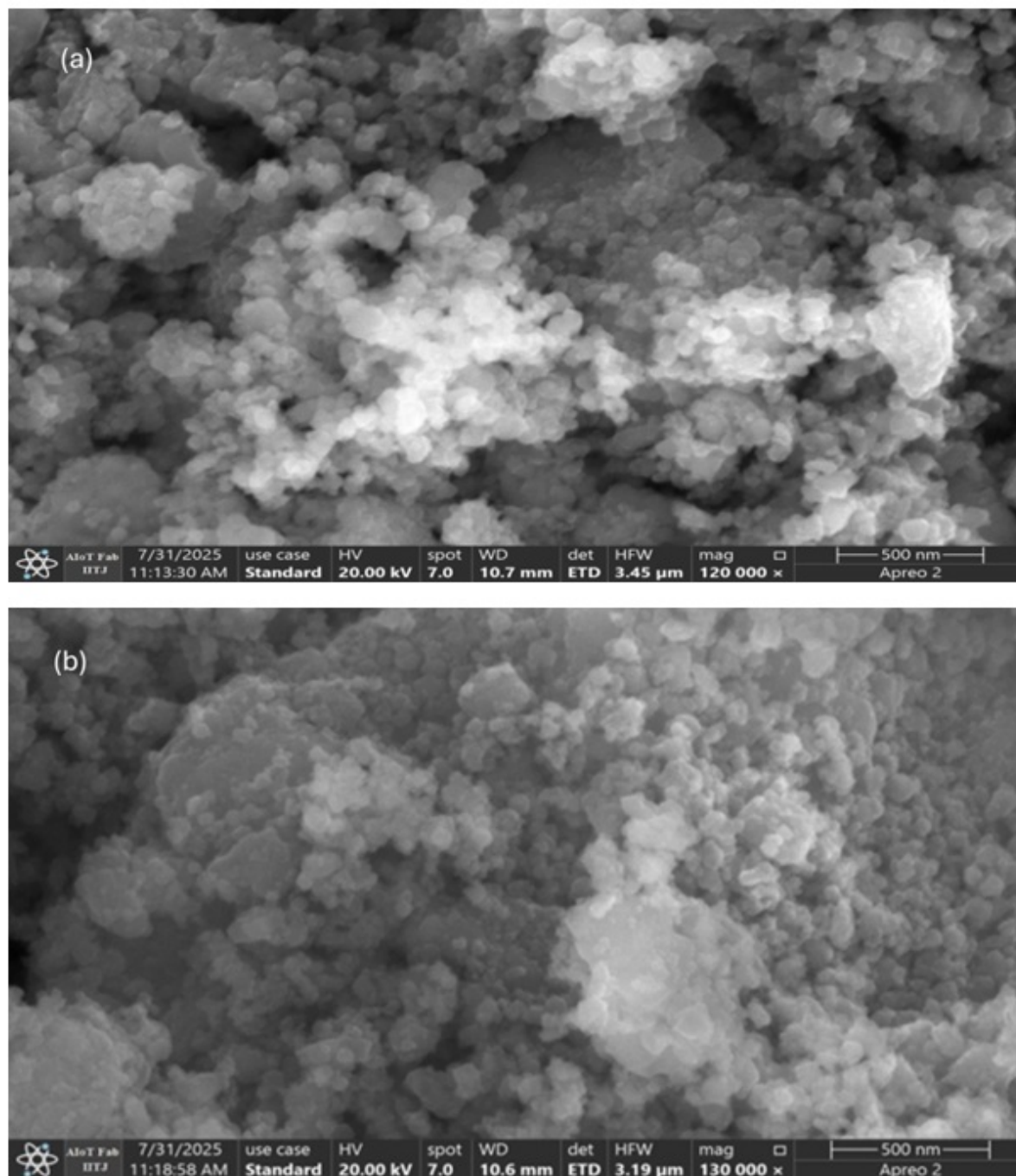


Figure 6: FESEM micrograph of (a) $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$ and (b) $\text{NiFe}_{1.96}\text{Al}_{0.04}\text{O}_4$.

ing behaviour. The primary charge carriers for these materials are holes (h^+), which are generated from oxygen adsorption on the surface of the material as oxygen reacts with electrons from the material surface and gets converted to an oxygen ion (O^-). When the surfaces of these materials are exposed to reducing gases such as acetone and ethanol, electrons are released back to the material surface, reducing hole concentration and increasing resistance in the presence of the target gas (R_g) compared to resistance in air (R_a) [30], [31].

The gas-sensing properties of the synthesised materials were tested against acetone and ethanol vapours at room temperature, a major advantage that eliminates the need for heaters, reduces power consumption, and makes the sensors suitable for portable devices. Table 3 presents the detailed gas-sensing metrics for acetone at different concentrations ranging from 1 ppm to 200 ppm. Figure 7 shows the resistance versus time graphs for all three samples when exposed

to acetone vapour. The response time is the time taken for the sensor to reach maximum value of resistance after gas exposure. For the Al1 sample at 200 ppm, the response time was 130s. For Al2, it was 167s, and for Al3, it was 171s. We can see that the response time increased with Al doping. This is because the increased strain and defects in Al-doped samples may slow down the diffusion of gas molecules into the material. However, at lower concentrations, the trend was different. For example, at 1 ppm, the response times were 49, 44, and 74 s for Al1, Al2, and Al3, respectively. At very low concentrations, the Al2 sample responds faster than pure NFO. Apart from this, the recovery times are generally much faster than response times, ranging from 10 to 31s. Fast recovery is important for practical applications because it allows the sensor to be ready for the next measurement quickly. The Al1 sample showed the fastest recovery in most cases, with recovery times around 10-15s.

Table 3: Gas sensing metrics for Acetone gas

Sample	Metric	Concentration of Gas					
		200 ppm	100 ppm	50 ppm	20 ppm	10 ppm	1 ppm
Al1	Response time (s)	130	103	91	72	61	49
	Recovery time (s)	15	13	14	12	15	10
	R_g/R_a	28.16	17.93	15.23	11.36	8.52	5.62
	$(R_g - R_a)/R_g$ (%)	96.38	94.42	93.43	91.20	88.26	82.22
Al2	Response time (s)	167	151	134	84	65	44
	Recovery time (s)	28	31	14	17	29	18
	R_g/R_a	30.15	28.45	26.56	21.46	18.59	14.61
	$(R_g - R_a)/R_g$ (%)	96.68	96.54	96.23	95.34	94.62	93.15
Al3	Response time (s)	171	152	138	114	107	74
	Recovery time (s)	22	18	14	14	17	11
	R_g/R_a	22.35	17.70	15.70	13.44	12.42	10.39
	$(R_g - R_a)/R_g$ (%)	95.52	94.35	93.63	92.56	91.95	90.37

Figure 8 shows the plot of R_g/R_a (response) versus gas concentration for all samples for both acetone and ethanol. We can see that the response increased with increasing gas concentration. This relationship was not perfectly linear, but it showed a clear trend. At low concentrations (1-20 ppm),

the response increased rapidly. At higher concentrations (50-200 ppm), the rate of increase becomes slower. This behaviour is typical of gas sensors and is related to the saturation of adsorption sites on the sensor surface [32].

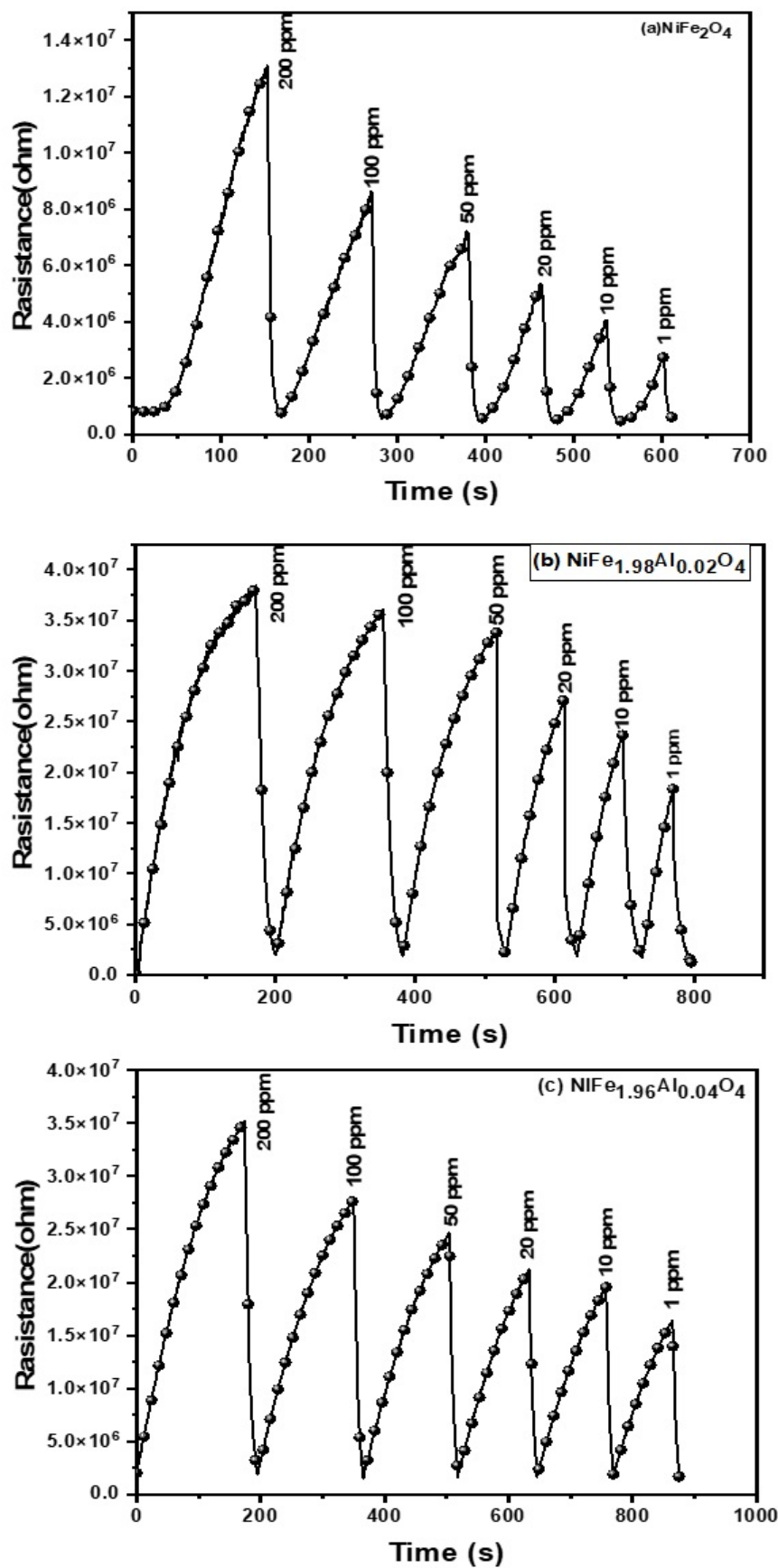


Figure 7: Resistance versus time graph (a) NiFe_2O_4 , (b) $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$ and (c) $\text{NiFe}_{1.96}\text{Al}_{0.04}\text{O}_4$ for acetone vapour.

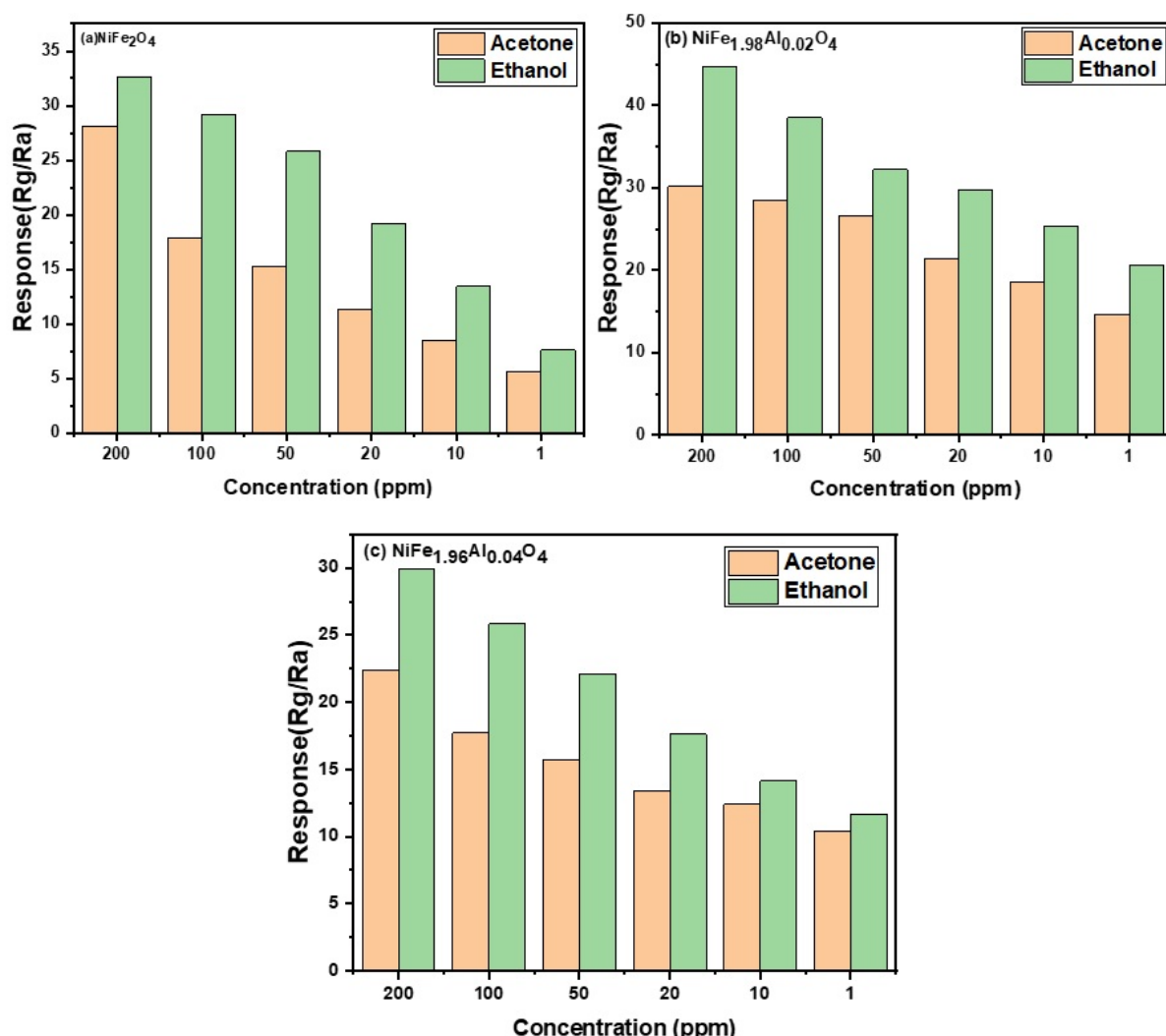


Figure 8: R_g/R_a vs. concentration graph of (a) NiFe_2O_4 , (b) $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$ and (c) $\text{NiFe}_{1.96}\text{Al}_{0.04}\text{O}_4$ for acetone and Ethanol.

Table 4 presents the gas-sensing metrics for ethanol at different concentrations. Figure 9 shows the resistance-versus-time graphs for ethanol sensing. The response to ethanol was generally higher than that to acetone across all samples. At 200 ppm, the R_g/R_a values were 32.64 for Al1, 44.71

for Al2, and 29.94 for Al3. Again, the Al2 sample shows the best performance with the highest response value of 44.71. The percentage responses for ethanol are also very high, exceeding 95% for most samples and concentrations. This indicates excellent sensitivity to ethanol vapor.

The response times for ethanol were comparable to those for acetone, ranging from 45 to 170s depending on the sample and concentration. The Al1 sample generally showed faster response times compared to Al-doped samples. The recovery times for ethanol were similar to those for acetone, mostly in the range of 11-33s. The sensors recovered quickly after ethanol was

removed, which is important for repeated measurements. Apart from this, we can clearly see from Figure 8 that ethanol produces a higher response than acetone at all concentrations. The higher response to ethanol suggests that the sensor has some selectivity towards ethanol over acetone.

In practical applications, repeatability is an important characteristic of gas sensors.

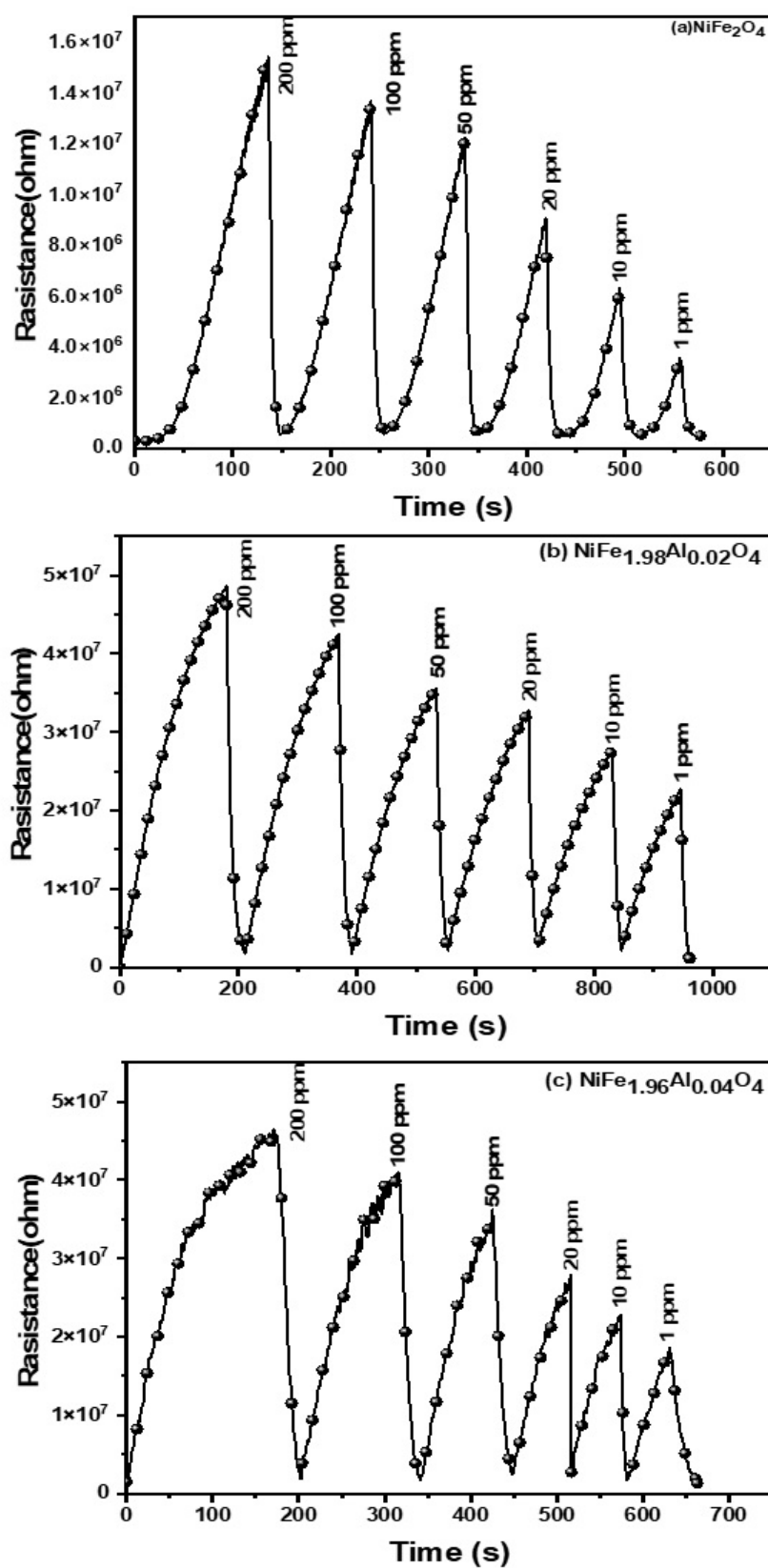


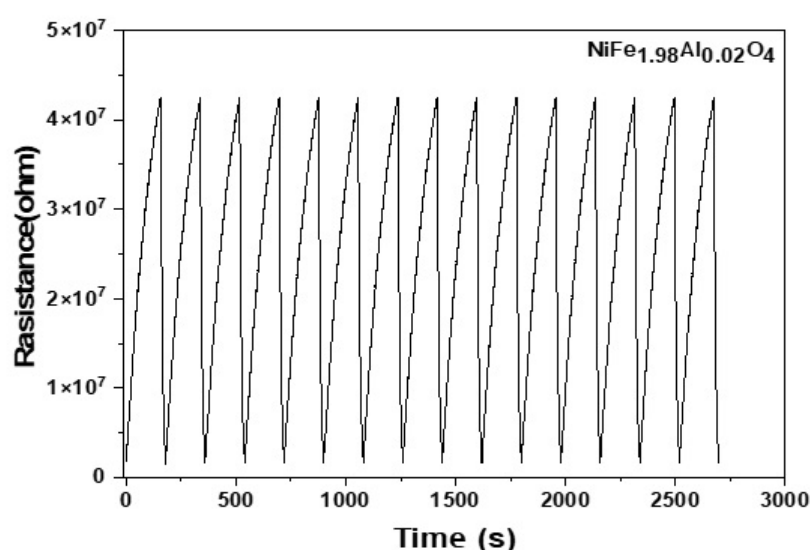
Figure 9: Resistance versus time graph (a) NiFe_2O_4 , (b) $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$ and (c) $\text{NiFe}_{1.96}\text{Al}_{0.04}\text{O}_4$ for Ethanol vapour.

Table 4: Gas sensing metrics for Ethanol gas

Sample	Metric	Gas Concentration					
		200 ppm	100 ppm	50 ppm	20 ppm	10 ppm	1 ppm
Al1	Response time (s)	131	93	82	70	60	45
	Recovery time (s)	12	14	13	16	15	17
	R_g/R_a	32.64	29.15	25.82	19.23	13.42	7.60
	$\frac{R_g - R_a}{R_g}$ (%)	96.64	96.34	96.12	94.80	92.54	86.64
Al2	Response time (s)	169	157	139	136	127	94
	Recovery time (s)	31	26	21	16	15	13
	R_g/R_a	44.71	38.51	32.18	29.78	25.34	20.59
	$\frac{R_g - R_a}{R_g}$ (%)	97.73	97.41	96.78	96.16	95.54	95.14
Al3	Response time (s)	170	113	85	67	51	49
	Recovery time (s)	33	26	23	19	11	25
	R_g/R_a	29.94	25.85	22.12	17.61	14.15	11.66
	$\frac{R_g - R_a}{R_g}$ (%)	96.71	96.13	95.48	94.32	92.94	91.42

A good sensor should give consistent results when tested multiple times under the same conditions. Figure 10 shows the repeatability pattern for ethanol sensing at 100 ppm using the Al2 sample. The graph shows 15 consecutive cycles of gas exposure and recovery. In each cycle, the resistance increases when ethanol is introduced and

decreases when ethanol is removed. The response pattern was very similar in all fifteen cycles, indicating good repeatability. The peak resistance values and the response/recovery times remained consistent across the cycles. This demonstrates that the sensor is stable and reliable for doing multiple measurements.

Figure 10: Repeatability pattern of sensing of Ethanol at 100 ppm for $\text{NiFe}_{1.98}\text{Al}_{0.02}\text{O}_4$.

Comparing the three samples, we concluded that the Al2 sample exhibits the best overall gas-sensing performance. It had the

highest response values for both acetone and ethanol. Although its response and recovery times are slightly longer than those

of pure NFO, its significantly higher sensitivity makes it the most promising composition. The Al3 sample showed a lower response compared to Al2. The higher sensitivity of Al2 can be understood from the fact that the nanostructure and porosity significantly affect sensing. Smaller particles provide more surface area for gas adsorption, while a porous structure enables deeper gas penetration. Gas molecules preferentially adsorbed at grain-boundary defects, influencing material resistance [33]. Aluminum doping influences gas sensing through several mechanisms: (1) It alters the electronic structure, thereby affecting electron mobility. (2) The disparity in size between aluminum and iron ions induces defects that facilitate gas adsorption on the surface. (3) Doping enhances the material's porosity, promoting gas diffusion and increasing the surface area. (4) Aluminum ions on the surface provide additional active sites for reactions. Al2 optimally balances these effects to achieve superior sensing performance.

5 Conclusion

In this study, we successfully synthesised Al-doped NFO nanomaterials ($\text{NiFe}_{2-x}\text{Al}_x\text{O}_4$ with $x = 0, 0.02$, and 0.04) using the citric acid modified sol-gel method. The materials were characterized thoroughly using XRD, FTIR, and FESEM techniques, and their gas sensing properties were evaluated at room temperature. XRD analysis confirmed the formation of single-phase cubic spinel structure in all samples. The lattice parameter decreased from $8.27 \text{ \AA}$ to $8.22 \text{ \AA}$ with increasing Al content, confirming the successful incorporation of Al into the NFO structure. The crystallite size, calculated by WH method, decreased from 47 nm to 42 nm with increasing Al doping. The lattice strain increased with Al content, reaching 2.8×10^{-4} for the highest doped sample. FTIR spectroscopy revealed characteristic metal-oxygen vibration bands around 550 cm^{-1} (tetrahedral sites) and 400 cm^{-1}

(octahedral sites), confirming the spinel ferrite structure. FESEM images showed nanoscale grains with irregular shapes and significant porosity. The porosity increased from 26.54% to 33.27% with Al doping, which is beneficial for gas-sensing applications. The gas-sensing tests demonstrated excellent performance at room temperature. The Al2 sample showed the best results with maximum response values of $R_g/R_a = 30.15$ for acetone and 44.71 for ethanol at 200 ppm concentration. The sensors showed high sensitivity with percentage responses exceeding 90% in most cases. The response times ranged from 44 to 171 s , and recovery times were fast, mostly between 10 and 33 s . The sensors also demonstrated good repeatability and stability over multiple measurement cycles. The enhanced gas-sensing performance of the optimally doped sample can be attributed to the combined effects of increased porosity, appropriate crystallite size, optimal lattice strain, and modified electronic structure due to Al doping. Room-temperature operation is a significant advantage that makes these sensors practical for real-world application. This work demonstrates that Al-doped NFO nanomaterials are promising candidates for low-power, room-temperature gas sensors to detect volatile organic compounds such as acetone and ethanol. These sensors could find applications in breath analysis for medical diagnosis, alcohol detection in breath analysers, industrial safety monitoring, and environmental pollution control. Future work could focus on improving sensor selectivity, reducing response time, and testing their performance with other important gases.

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