Preparation and Gas Sensing Study of NiO Thin Films

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Abstract

Thin film technology offers significant advantages over thick films, making it highly desirable for a wide range of applications including solar cells, gas sensors, photovoltaic devices, biomedical systems, and supercapacitors. In this study, nickel oxide (NiO) thin films were successfully synthesized using the spin coating technique, employing nickel nitrate as the precursor. The films were deposited on glass substrates with dimensions of 2 × 2 cm. The gas sensing performance of the NiO thin films was evaluated against various oxidizing and reducing gases such as NH_3, LPG, NO_2, CH_4, and ethanol. Among these, the films exhibited the highest sensitivity to LPG, with a maximum response of 85.76% at an optimal operating temperature of 120 °C. The films demonstrated excellent selectivity towards LPG, along with a rapid response time (~09s) and a swift recovery time (~32 s), highlighting their potential for efficient gas sensing applications.

Keywords: Thin Film, Gas Sensors, Glass Substrates, Sensitivity, Selectivity, Response Time

1. Introduction:

Thin Film Technology (TFT) refers to the process of depositing extremely thin layers of material ranging from a few nanometers to several micrometers onto a substrate surface [1]. These thin films can be composed of metals, semiconductors, insulators, or a combination thereof, and are engineered to achieve specific electrical, optical, thermal, or mechanical properties [1, 2]. TFT is widely used in various high-performance applications such as microelectronics, photovoltaics, sensors, and optoelectronic devices. The precise control over film thickness, uniformity, and composition makes TFT a crucial technique in fabricating devices like solar cells, thin-film transistors, gas sensors, and display panels. Common deposition methods include physical vapor deposition (PVD), chemical vapor deposition (CVD), and solution-based techniques like spin coating and dip coating [2-4]. The ability to create high-quality films with tailored properties on diverse substrates enables the development of compact, efficient, and cost-effective devices across multiple industries [4, 5].

The sol-gel and spin coating methods are widely used techniques in thin film fabrication, particularly for producing metal oxide films with controlled morphology and composition [8]. The sol-gel method is a chemical synthesis process that involves the transition of a system from a liquid “sol” (a colloidal suspension of particles) into a solid “gel” phase. This technique typically starts with metal alkoxides or metal salts as precursors, which undergo hydrolysis and condensation reactions to form a homogeneous sol. Upon aging and drying, the sol gradually transforms into a gel, which can then be heat-treated (calcined) to produce a dense and crystalline oxide material [8, 9]. On the other hand, spin coating is a deposition technique where the sol is dropped onto a substrate, which is then rapidly rotated at high speeds [9, 10]. The centrifugal force spreads the sol uniformly across the surface, forming a thin film. The thickness and

uniformity of the film can be precisely controlled by adjusting parameters such as spin speed, viscosity of the sol, and spin duration [11, 12]. Together, the sol-gel and spin coating methods provide a simple, cost-effective, and scalable approach for producing high-quality thin films with excellent control over structural and functional properties, making them ideal for applications in electronics, optics, and sensing technologies [13-15].

Gas sensors are essential devices used to detect the presence and concentration of various gases in the environment, playing a crucial role in ensuring safety, health, and process control across multiple sectors [16]. The need for gas sensors arises from the increasing use of flammable, toxic, and hazardous gases in domestic, industrial, and environmental settings. Leaks or accidental releases of gases like LPG, methane, carbon monoxide, ammonia, and hydrogen sulfide can lead to fire hazards, explosions, or serious health risks [16, 17]. Gas sensors enable early detection of such leaks, allowing timely preventive actions, reducing the risk of accidents, and saving lives. In industries such as chemical manufacturing, mining, agriculture, and healthcare, gas sensors help maintain air quality, ensure regulatory compliance, and optimize operational efficiency [17, 18]. With advancements in nanotechnology and material science, modern gas sensors offer high sensitivity, selectivity, fast response, and compact design, making them indispensable for smart safety systems, environmental monitoring, and emerging technologies like IoT-enabled smart homes and wearable health monitors [19, 20].

Liquefied Petroleum Gas (LPG) is a widely used fuel composed primarily of propane and butane, commonly employed in domestic, commercial, and industrial sectors for heating, cooking, and energy generation [20]. Despite its efficiency and accessibility, LPG is highly flammable and potentially hazardous when leaked, as it can form explosive mixtures with air and pose severe risks of fire, explosion, and asphyxiation. LPG is heavier than air, which causes it to accumulate at ground level in case of leakage, making early detection critical [21]. The importance of LPG gas detection lies in ensuring safety, preventing accidents, and enabling timely evacuation or intervention. Gas sensors that can rapidly and accurately detect LPG concentrations, especially at low levels, are crucial for use in households, vehicles, storage facilities, and industries. Effective LPG detection systems not only protect lives and property but also support regulatory compliance and improve public safety by enabling automatic shutdown or alarm systems upon gas leak detection [21-23].

The aim of this research paper is to prepare and evaluate nickel oxide thin films for efficient detection of liquefied petroleum gas, addressing the urgent need for reliable gas sensing technologies due to the widespread use and associated hazards of LPG. Leveraging the advantages of thin film technology, NiO thin films were synthesized using the sol-gel method followed by spin coating a low-cost and scalable technique offering excellent control over film morphology and thickness. The study focuses on investigating the gas sensing performance of these films, particularly their sensitivity, selectivity, response, and recovery times when exposed to various oxidizing and reducing gases. Among the tested gases, the films demonstrated maximum sensitivity towards LPG, highlighting their potential for use in household and industrial safety systems.

2. Experimental work

In this process, all AR grade chemicals were used for preparation of NiO thin films. The synthesis of NiO nanoparticles and their subsequent deposition as thin films was carried out using the sol-gel method combined with the spin coating technique. In this process, nickel nitrate hexahydrate [$Ni(NO_3)_2 \cdot 6H_2O$] was used as the precursor for nickel ions. Process of synthesis and preparation of NiO thin films is shown in Fig. 1.

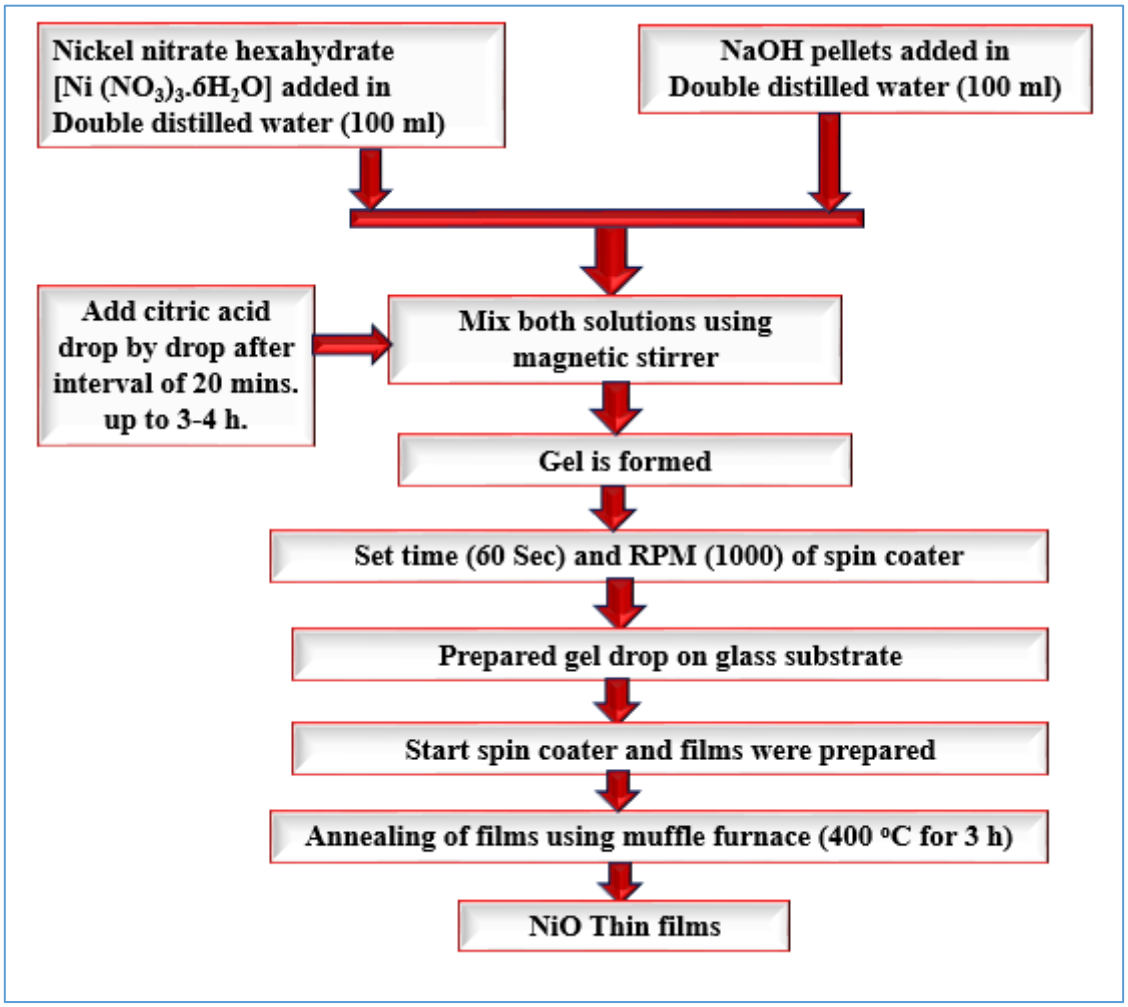

Figure 1. Steps for synthesis and thin film preparation of NiO

3. Result and discussion

The gas sensing study of NiO thick films were carried out using half bridge method and static gas sensing system as reveal in Fig. 2 [24, 25]. The schematic diagram illustrates a gas sensing experimental setup designed to evaluate the performance of a gas sensor. At the core of the setup is the sensor mounted on a heater and placed over an insulating base. This entire assembly rests on a base plate, which is connected to a 230 V AC power supply to heat the sensor to the required operating temperature. A thermocouple is attached near the sensor to monitor the temperature, and its reading is displayed via a temperature indicator.A glass dome (25 lit.) is placed over the sensor to create a controlled environment for gas exposure. Gases are introduced into this chamber through a gas injection unit, typically using a syringe for precise volume (ppm) control. The change in sensor resistance upon exposure to the target gas is measured using a digital multimeter (DMM), as shown in the upper right corner of the diagram. The multimeter records the change in voltage across the sensor, which correlates with its gas sensitivity. This setup allows for controlled testing of the sensor's response, recovery time, sensitivity, and selectivity to various gases under defined temperature conditions [25, 26].

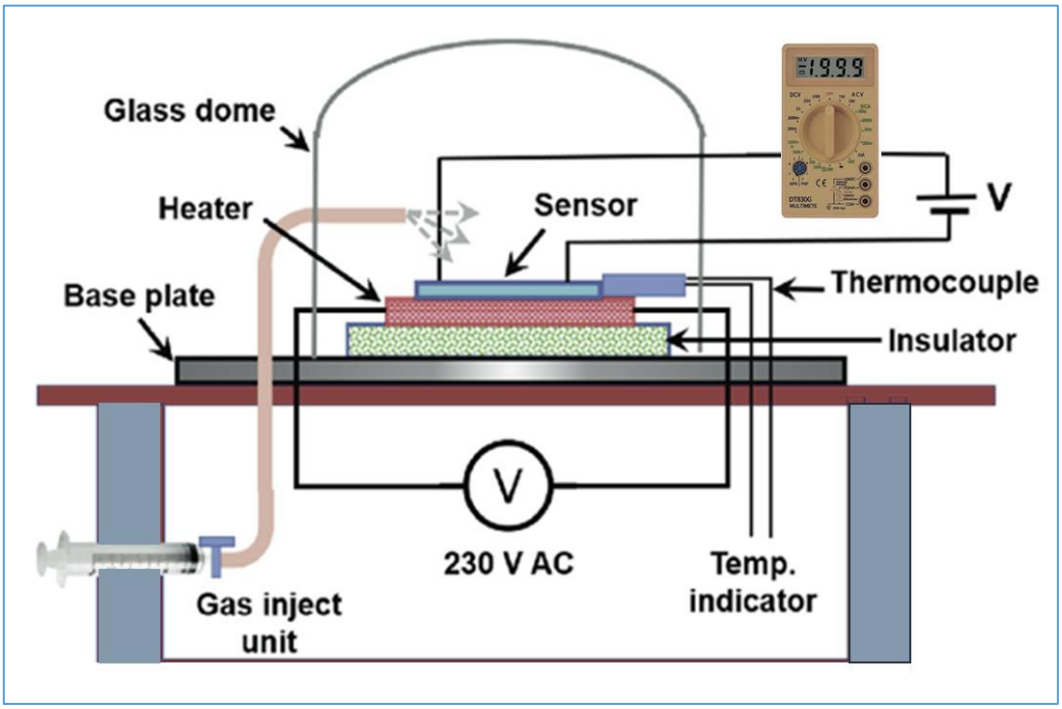

Figure 2. Schematic diagram of gas sensing set-up

The sensitivity of a gas sensor refers to its ability to detect even small changes in the concentration of a target gas and respond accordingly [26]. It is a critical parameter that defines how effectively the sensor can measure low levels of gas presence. The sensitivity of the NiO films is estimated using Eq. 1 [26, 27].

$$Ra-Rg/Ra \times 100 \qquad \text{(Eq. 1)}$$

Where,

Ra- resistance of the film in air, and Rg- resistance of the film in presence of gas.

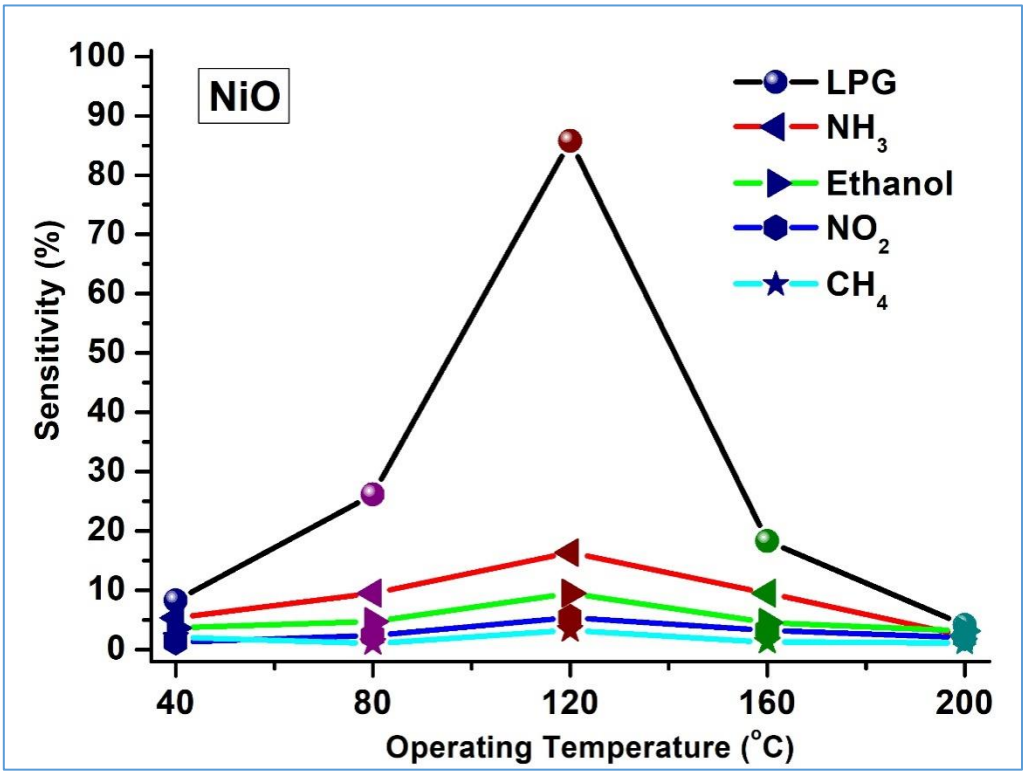

Figure 3. Sensitivity versus operating temperature plot of NiO thin films

Fig.3 illustrates the sensitivity of NiO thin films as a function of operating temperature for various gases including LPG, NH_3, ethanol, NO_2, and CH_4. The plot reveals a distinct temperature-dependent sensitivity behavior for each gas. Among the gases tested, LPG exhibits the highest sensitivity, with a sharp peak of approximately 85.76 % at 120 °C. This indicates that NiO thin films are particularly responsive to LPG at this specific temperature, suggesting optimal adsorption and reaction kinetics. For the other gases NH_3, ethanol, NO_2, and CH_4 the sensitivity remains relatively low across the entire temperature range (40–200 °C), with minor increases around 120 °C but no significant peaks [26, 27]. NH_3 shows the second-highest response after LPG, though its sensitivity is modest in comparison. This overall trend implies that NiO thin films are selectively more sensitive to LPG, especially at moderate temperatures, while they are less responsive to other tested gases.The decline in sensitivity at temperatures beyond 120 °C, especially for LPG, suggests that desorption dominates at higher temperatures, reducing the film's ability to interact with gas molecules effectively. Therefore, 120 °C can be considered the optimal operating temperature for detecting LPG using NiO thin films, while other gases may require different materials or modified NiO structures for enhanced sensitivity [27, 28].

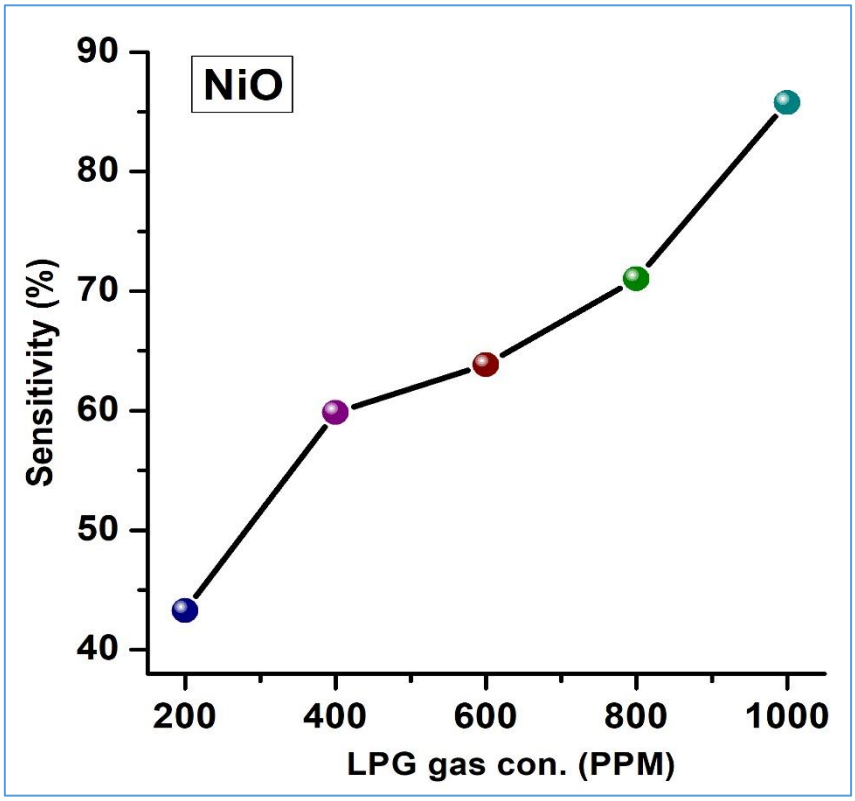

Figure 4. Sensitivity versus LPG gas concentration plot of NiO thin films

Fig. 4 depicts the sensitivity of NiO thin films as a function of LPG gas concentration, ranging from 200 to 1000 ppm. The plot shows a clear trend of increasing sensitivity with rising LPG concentration, indicating that NiO thin films exhibit a strong and proportional response to LPG. At 200 ppm, the sensitivity starts at approximately 43.26%, and as the concentration increases, the sensitivity enhances progressively, reaching about 85.16 % at 1000 ppm.This increasing trend suggests that more LPG molecules are interacting with the surface of the NiO thin film as concentration rises, leading to greater changes in electrical resistance and, consequently, higher sensitivity. The nearly linear or slightly nonlinear upward curve also implies a consistent and reliable sensor performance across a wide concentration range, making NiO thin films highly suitable for detecting varying levels of LPG in practical applications [25, 27].The observed behavior highlights the potential of NiO-based sensors in detecting LPG leaks or monitoring LPG levels in industrial and domestic applications [28, 29]. This trend, when coupled with the optimal operating

temperature observed in Fig. 3, reinforces the efficiency and selectivity of NiO thin films as promising gas sensing materials for LPG.

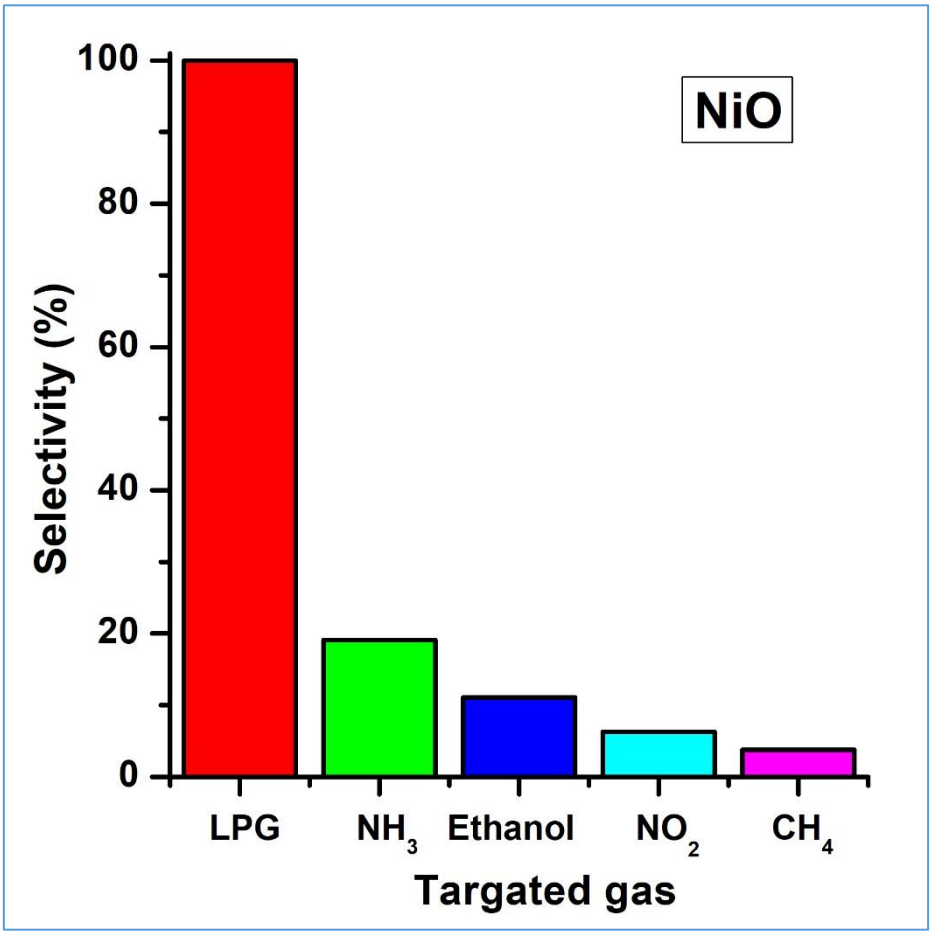

Figure 5. Selectivity histogram of NiO thin films

Fig. 5 presents the selectivity histogram of NiO thin films towards various target gases, namely LPG, NH_3, ethanol, NO_2, and CH_4. The bar graph clearly demonstrates that NiO thin films exhibit exceptional selectivity for LPG, with a selectivity value nearing 100%. This indicates a highly preferential response of the NiO sensing material towards LPG over the other gases tested.In contrast, the selectivity towards NH_3, ethanol, NO_2, and CH_4 is significantly lower, all falling below 20%, with CH_4 showing the least selectivity [30]. This sharp disparity confirms that NiO thin films are not only highly sensitive to LPG, as seen in earlier figures, but also strongly selective, making them excellent candidates for LPG-specific gas sensing applications.Such high selectivity is critical in real-world sensor applications, as it minimizes cross-sensitivity and false responses to other interfering gases commonly present in the environment. The strong selectivity towards LPG may be attributed to favorable interactions between LPG molecules and the surface chemistry of NiO, such as specific adsorption sites or catalytic activity that enhances reaction efficiency [30, 31].

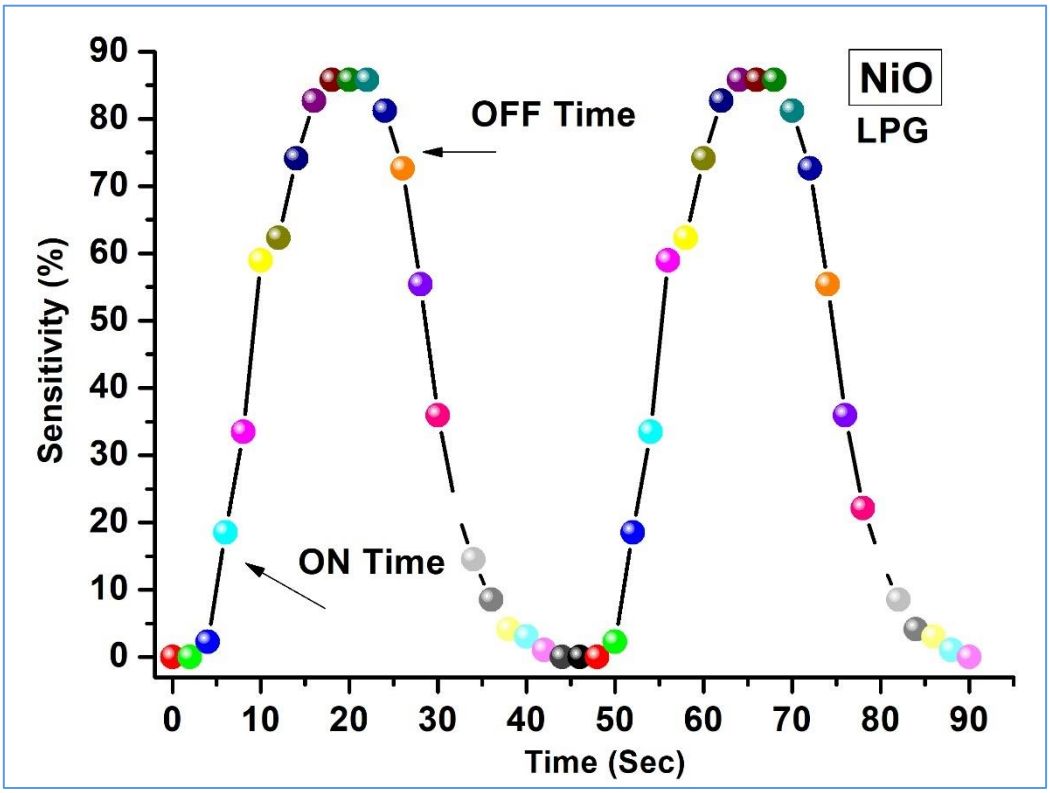

Figure 6. Response and recovery plot of NiO thin films

Fig. 6 illustrates the dynamic response and recovery behavior of NiO thin films upon exposure to and removal of LPG gas. The sensitivity is plotted against time, clearly showing two response-recovery cycles. Upon introduction of LPG (ON time), the sensitivity rapidly increases, reaching a peak of around 85%, indicating a swift and strong response of the NiO sensor to the target gas. This rapid rise suggests efficient adsorption of LPG molecules on the sensor surface [26, 29].When the gas is removed (OFF time), the sensitivity decreases quickly, returning to near baseline values, which signifies effective desorption of LPG molecules and recovery of the sensing surface. The symmetric and cyclic nature of the graph indicates stable sensor performance across multiple cycles, with consistent ON and OFF responses. Such a pattern is desirable in real-time gas sensing applications, as it confirms that the sensor can reliably detect and clear the gas within short time intervals [30]. Theobtained results confirms that NiO thin films not only offer high sensitivity and selectivity to LPG but also exhibit rapid response and recovery times, making them highly efficient for practical gas sensing applications [30, 31].

Conclusions

The NiO thin films synthesized via the spin coating method exhibited excellent sensitivity, selectivity, and rapid response-recovery characteristics, particularly toward liquefied petroleum gas. Among the tested gases NH_3, NO_2, CH_4, and ethanol. LPG demonstrated the highest interaction with the sensing material, with a maximum sensitivity of 85.76% observed at an optimal operating temperature of 120 °C. The temperature-dependent sensitivity profile revealed that gas response increased with temperature, peaking at 120 °C before declining at higher temperatures, indicating this as the ideal operating point for efficient gas detection. A clear linear relationship was observed between LPG concentration and sensitivity, confirming the films' potential for quantitative gas detection over a wide range of concentrations. Selectivity analysis further highlighted the dominance of LPG response over other gases, with LPG selectivity approaching 100%, while the response to other analytes remained significantly lower. The response-recovery plot confirmed the fast and repeatable switching behavior of the sensor, with an impressive response time of approximately 9 seconds and a recovery time of around 32 seconds. Collectively, these

results validate the suitability of NiO thin films as high-performance gas sensors for LPG detection, demonstrating promising potential for real-time environmental monitoring and safety applications.

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