

Spin Coated Reduced Graphene Oxide (rGO) Thin Films for NO₂ Detection

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Abstract:

Spin-coated reduced graphene oxide (rGO) thin films were fabricated on glass substrates and investigated for their gas sensing performance toward various reducing and oxidizing gases. Graphene oxide films were deposited using the spin-coating technique. The gas sensing characteristics of the rGO films were systematically studied for H₂S, NO₂, CO₂, NH₃ and CH₄ gases over a wide operating temperature range of 30–150 °C. The experimental results revealed that the rGO thin films exhibited a pronounced and selective response toward NO₂ compared to other tested gases. The maximum gas response (63.48%) was observed at an optimal operating temperature of 120 °C for an NO₂ concentration of 700 ppm. The rGO thin films demonstrated good selectivity, reproducibility and rapid response and recovery characteristics, indicating efficient adsorption and desorption processes. The observed sensing performance highlights the suitability of spin-coated rGO thin films for reliable and selective NO₂ detection. The obtained results suggest that rGO-based thin film sensors prepared by a simple and cost-effective spin-coating technique hold strong potential for practical gas sensing applications.

Keywords: Reduced graphene oxide; Spin coating; Thin film gas sensor; NO₂ sensing; Selectivity.

1. INTRODUCTION

Air pollution caused by toxic and hazardous gases has become a serious global concern due to rapid industrialization, urbanization, and increased vehicular emissions. Among various air pollutants, nitrogen dioxide (NO₂) is one of the most harmful gases released primarily from automobile exhausts, power plants, and industrial combustion processes [1, 2]. Prolonged exposure to NO₂ can cause severe respiratory problems, lung inflammation, reduced immunity, and aggravation of asthma and cardiovascular diseases. In addition to its adverse effects on human health, NO₂ also plays a significant role in atmospheric reactions leading to the formation of photochemical smog and acid rain. Therefore, continuous monitoring and precise detection of NO₂ at low concentrations are essential for environmental safety, industrial process control, and public health protection [2-4]. Gas sensors based on semiconducting materials have attracted extensive attention due to their simplicity, low cost, and real-time monitoring capability. Conventional metal oxide semiconductor (MOS) gas sensors such as SnO₂, ZnO, WO₃, and TiO₂ have been widely studied for NO₂ detection because of their high sensitivity and stability. However, these sensors typically require high operating temperatures (200–400 °C), which results in high power consumption, poor long-term stability, and limited compatibility with portable and flexible devices [4-6]. Moreover, MOS-based sensors often suffer from poor selectivity and slow response–recovery behavior, which restricts their practical applications. These limitations have motivated researchers to explore alternative sensing materials that can operate at lower temperatures while maintaining high sensitivity and selectivity [6, 7].

In recent years, carbon-based nanomaterials such as carbon nanotubes, graphene, graphene oxide (GO), and reduced graphene oxide (rGO) have emerged as promising candidates for gas sensing applications. Among these materials, graphene has attracted significant interest due to its extraordinary electrical conductivity, high carrier mobility, large specific surface area, and excellent mechanical flexibility [8, 9]. These properties make graphene highly sensitive to surface charge transfer caused by gas molecule adsorption. However, the practical use of pristine graphene in gas sensors is limited by challenges associated with large-scale production, high cost, and the absence of a bandgap, which leads to poor modulation of electrical resistance upon gas exposure [9, 10].

Graphene oxide and reduced graphene oxide offer attractive alternatives to pristine graphene for gas sensing applications. GO contains abundant oxygen-containing functional groups such as hydroxyl, epoxy, and carboxyl groups, which enhance its dispersibility in water and facilitate solution-based processing techniques. Upon reduction, GO is converted into rGO, which partially restores the sp^2 carbon network while retaining a controlled amount of defects and functional groups. These residual functional groups and structural defects act as active adsorption sites for gas molecules, significantly enhancing the gas sensing performance. The rGO exhibits semiconducting behavior with improved electrical conductivity compared to GO, making it suitable for resistive-type gas sensors [9-11].

Reduced graphene oxide has been extensively investigated for the detection of various gases including NO_2 , NH_3 , H_2S , CO, and volatile organic compounds (VOCs). Among these, NO_2 , being a strong oxidizing gas, shows pronounced interaction with rGO due to efficient electron withdrawal from the p-type rGO surface, resulting in a significant change in electrical resistance. Several studies have reported that rGO-based sensors exhibit high sensitivity and fast response to NO_2 even at relatively low operating temperatures [12]. The sensing mechanism is primarily governed by charge transfer between NO_2 molecules and the rGO surface, along with adsorption-desorption dynamics influenced by temperature and surface morphology [11-13].

The fabrication method of rGO thin films plays a crucial role in determining their structural, morphological, and sensing properties. Various deposition techniques such as drop casting, dip coating, spray coating, vacuum filtration, and spin coating have been employed to fabricate rGO-based sensing layers [13, 14]. Among these methods, spin coating is particularly attractive due to its simplicity, low cost, reproducibility, and ability to produce uniform and smooth thin films with controlled thickness. Spin coating is also compatible with large-area substrates and scalable fabrication processes, making it suitable for practical sensor device development [15, 16].

Spin-coated rGO thin films offer several advantages for gas sensing applications, including good adhesion to substrates, uniform surface coverage, and controllable film thickness. These features are essential for achieving stable and repeatable sensing performance. Moreover, spin coating enables easy integration of rGO films onto various substrates such as glass, silicon, and flexible polymer substrates. Glass substrates, in particular, are widely used due to their chemical stability, smooth surface, and insulating nature, which is beneficial for resistive gas sensing measurements [16, 17].

In the present work focuses on the fabrication of reduced graphene oxide thin films using the spin-coating technique and the systematic investigation of their gas sensing properties toward NO_2 . The sensing performance of the rGO thin films is evaluated in comparison with other common gases such as H_2S , CO_2 , NH_3 , and CH_4 over a wide operating temperature range. Emphasis is placed on understanding the effect of operating temperature on gas response, selectivity and response-recovery characteristics. The study aims to demonstrate that spin-coated rGO thin films can serve as efficient, selective, and low-

temperature NO₂ gas sensors, offering a simple and cost-effective approach for environmental monitoring and industrial safety applications.

2. MATERIALS AND METHODS

All AR grade chemicals were used in this work. Graphene oxide (GO) powder (50 mg) was dispersed in 100 mL of deionized water to obtain a 0.5 mg/mL GO sol and ultrasonicated for 45 minutes to ensure uniform dispersion. Tetraethyl orthosilicate (TEOS, 5 ml) was then added dropwise to the GO sol under continuous magnetic stirring, then addition of 1 mL of 0.1 M hydrochloric acid as a catalyst for the sol-gel reaction [18]. The mixture was stirred for 2 hours at room temperature to form a stable and homogeneous sol. Glass substrates were cleaned with acetone and deionized water for 10 minutes each and dried under IR lamp for 20-30 min. The prepared GO sol was deposited onto the substrates using a spin coater at 2000 rpm for 30 seconds. The coated films were pre-dried at 80 °C for 10 minutes under IR lamp to remove residual solvents. This spin-coating and drying process was repeated 2–3 times to achieve the desired film thickness. Finally, the deposited GO thin films were thermally reduced to rGO by annealing at 300 °C for 1 hour in the muffle furnace. The resulting rGO thin films were used for gas sensing study [19-20].

3. RESULTS AND DISCUSSION

The static gas sensing system shown in Fig. 1 consists of a closed gas chamber (25 L) integrated with a resistive heater, temperature control unit, electrical measurement circuit and gas injection arrangement, designed to evaluate the sensing performance of thin film sensors under controlled conditions. The sensing element, in the form of a thin film deposited on an insulating glass substrate, is placed inside the gas chamber on a metallic heater block. The operating temperature of the sensor is controlled in the range of 30–150 °C using a temperature controller connected to a dimmerstat (0–230 V AC) powered by the AC mains. A K-type thermocouple (T_c) positioned close to the sensor surface continuously monitors the temperature and provides feedback to maintain temperature stability within ± 1 °C. For electrical measurements, a constant DC bias voltage ($V_{app} = 30\text{VDC}$) is applied across the sensor, which is connected in series with a reference resistor ($R_{ref} = 10\text{ M}\Omega$) to form a voltage divider circuit. The voltage drop across the reference resistor is measured using a digital voltmeter (DVM) with millivolt resolution and the resistance of the sensing film is calculated using standard electrical relations. Initially, the sensor resistance is measured in ambient air at the desired operating temperature to establish a baseline [21, 22]. A known volume of target gas corresponding to the required concentration (typically 100–1000 ppm) is then injected into the chamber through the gas inlet using a calibrated gas syringe (25 ml), while the total volume of the chamber remains constant, thereby operating in static mode. Using this static gas sensing system, important sensor parameters such as gas response, selectivity, response/recovery time, and operating temperature dependence are systematically evaluated [22, 23].

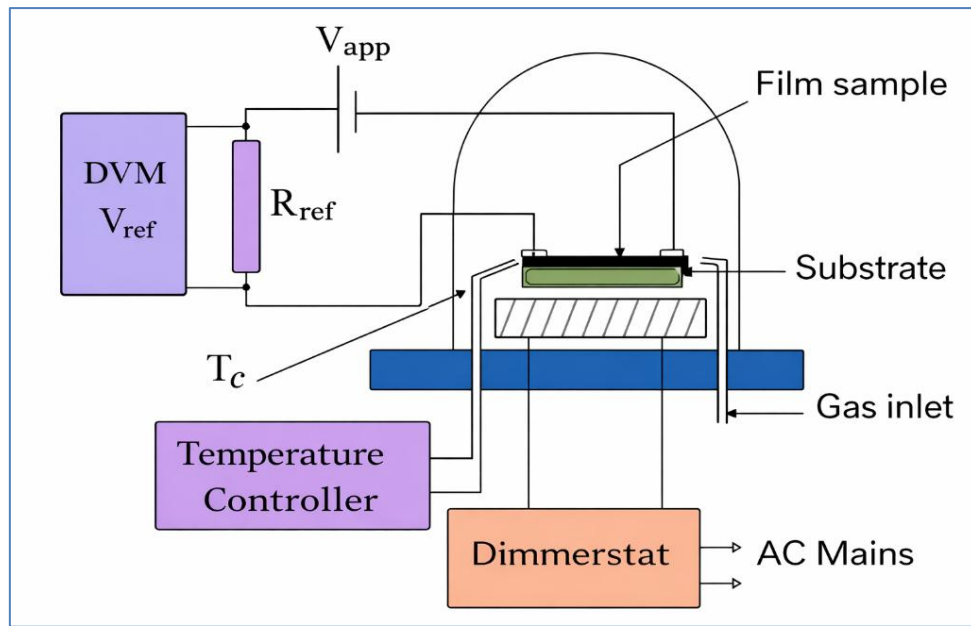


Fig.1. Schematic diagram of static gas sensing system

Gas response, selectivity, response time, recovery time, and reproducibility are the key performance parameters used to evaluate the sensing behavior of gas sensors. The gas response of a sensor is defined as the relative change in its electrical resistance (or conductance) when exposed to a target gas compared to its baseline value in air at a fixed operating temperature. It is commonly expressed as given in eq. 1 [23, 24].

$$\text{Gas response (\%)} = (R_a - R_g) / R_a \times 100 \quad (\text{Eq. 1})$$

Where,

R_a - sensor resistances in air and R_g - sensor resistances the presence of the target gas.

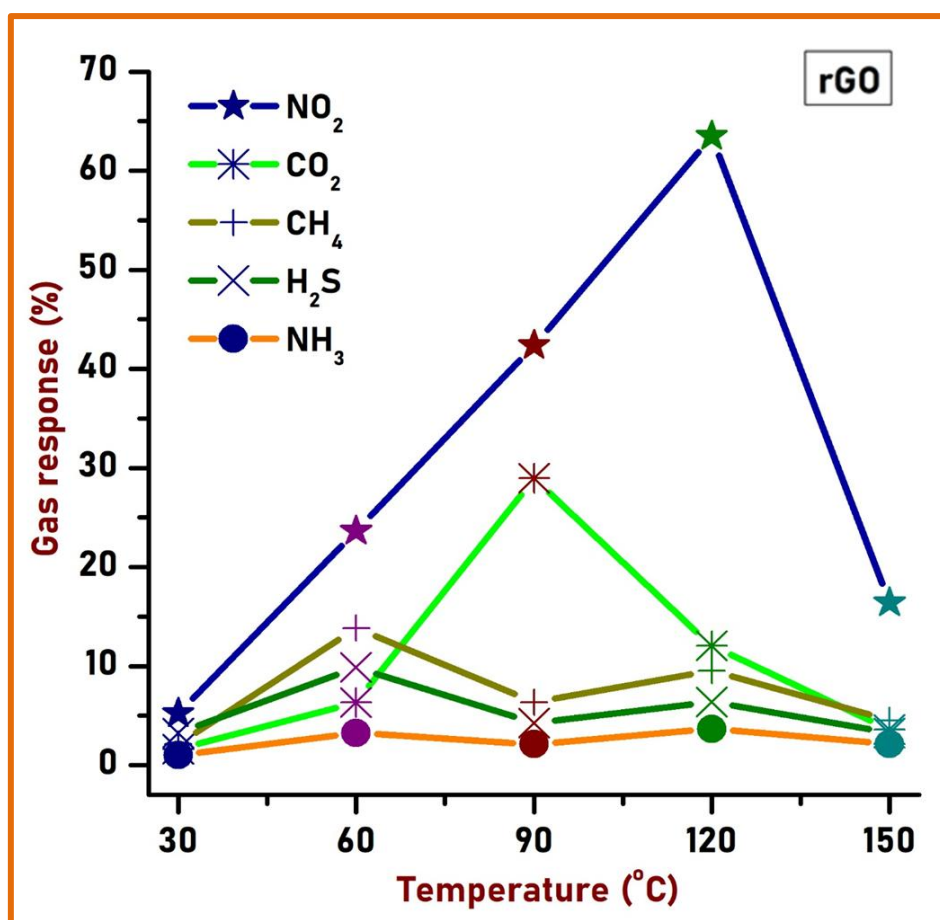


Fig. 2. Gas response versus operating temperature plot of rGO thin films

Figure 2 illustrates the variation of gas response of rGO thin films as a function of operating temperature for different target gases, such as NO₂, CO₂, CH₄, H₂S, and NH₃. It is evident from the plot that the gas response strongly depends on the operating temperature and the nature of the gas. At lower temperatures (30 °C), the rGO thin films exhibit relatively low responses for all the tested gases, which can be attributed to insufficient thermal energy for effective adsorption and surface reaction of gas molecules on the rGO surface. As the operating temperature increases from 30 to 90 °C, a gradual enhancement in gas response is observed for most gases due to increased adsorption kinetics and improved charge transfer between gas molecules and the rGO sensing layer. Among all the tested gases, NO₂ shows a significantly higher response compared to CO₂, CH₄, H₂S, and NH₃ across the entire temperature range, indicating the strong oxidizing nature of NO₂ and its pronounced interaction with the p-type rGO surface [24, 25]. The NO₂ response increases sharply with temperature and reaches a maximum value of 63.48% at an optimal operating temperature of 120 °C. This enhanced response is attributed to efficient adsorption of NO₂ molecules and strong electron withdrawal from the rGO surface, leading to a substantial increase in hole concentration and a pronounced change in resistance. Beyond 120 °C, the response to NO₂ decreases at 150 °C, which is likely due to the dominance of desorption processes at higher temperatures, reducing the number of adsorbed NO₂ molecules on the sensing surface. It is also found that, the responses to CO₂, CH₄, H₂S, and NH₃ are comparatively lower and show different temperature-dependent behaviors. CO₂ exhibits a moderate response with a maximum value at around 90 °C, while CH₄ and H₂S show relatively small responses with slight variations over the temperature range. NH₃ demonstrates the lowest response among the tested gases, indicating weak interaction with the rGO surface under the given experimental conditions. The clear difference in response magnitude between NO₂ and other gases at 120 °C highlights the excellent selectivity of rGO thin films toward

NO₂. The gas response versus temperature characteristics confirm that the sensing performance of rGO thin films is governed by a balance between adsorption and desorption processes, and the optimal operating temperature of 120 °C provides the best sensitivity and selectivity for NO₂ [25-27].

Selectivity refers to the ability of the sensor to preferentially respond to a specific target gas in the presence of other interfering gases; a highly selective sensor exhibits a significantly higher response to the target gas than to other gases under identical operating conditions. Figure 3 shows the selectivity histogram of rGO thin films toward different target gases measured at the optimum operating temperature of 120 °C. Selectivity is defined as the relative response of the sensor to a specific gas compared to other interfering gases under identical experimental conditions [27, 28]. As evident from the histogram, the rGO thin films exhibit excellent selectivity toward NO₂, with the selectivity value normalized to 100%. In contrast, significantly lower selectivity values are observed for CO₂ (18.98%), CH₄ (15.01%), H₂S (10.06%), and NH₃ (5.75%), indicating weak interaction of these gases with the rGO surface.

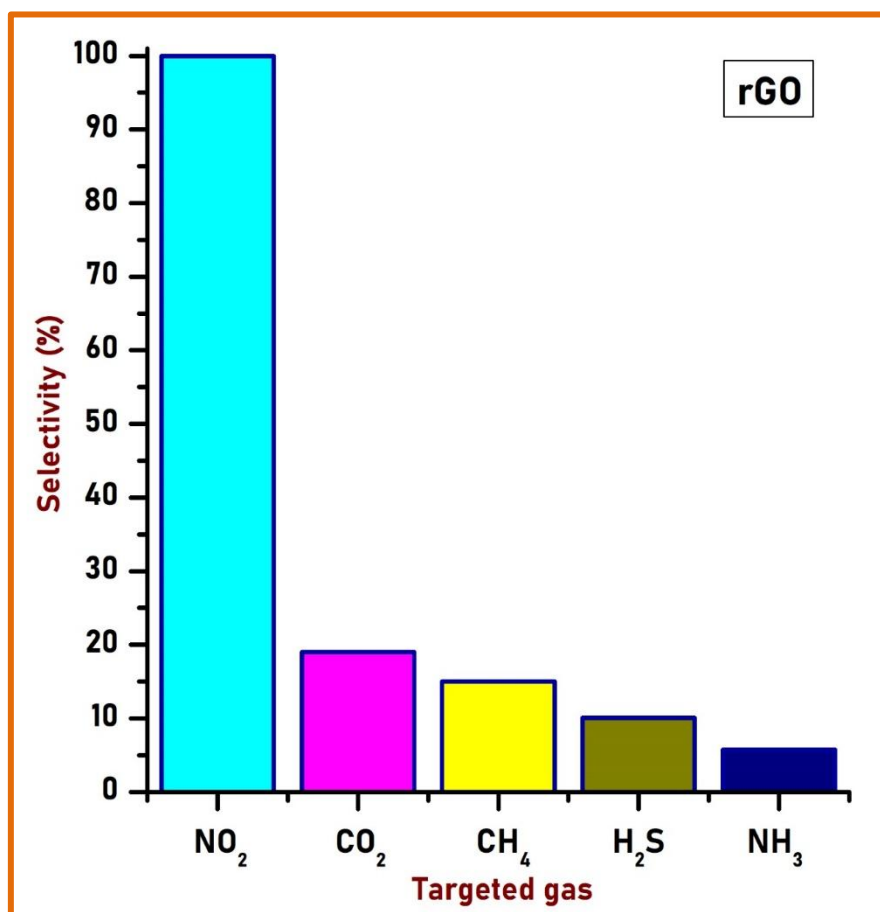


Fig. 3. Selectivity histogram of rGO thin films

Response time is defined as the time required for the sensor to reach 90% of its maximum resistance change after the introduction of the target gas, indicating the speed of adsorption and surface reaction processes. Conversely, recovery time is the time taken by the sensor to return to 90% of its original baseline resistance after the removal of the target gas and exposure to air, reflecting the efficiency of gas desorption from the sensor surface. Figure 4 shows the dynamic response–recovery characteristics of the rGO thin film sensor toward NO₂ gas measured at the optimal operating temperature of 120 °C [23, 25]. The plot represents the variation of gas response (%) as a function of time during successive ON–OFF cycles of NO₂ exposure. When NO₂ gas is introduced into the sensing chamber (ON state), the sensor response increases rapidly, indicating quick adsorption of NO₂ molecules on the rGO surface.

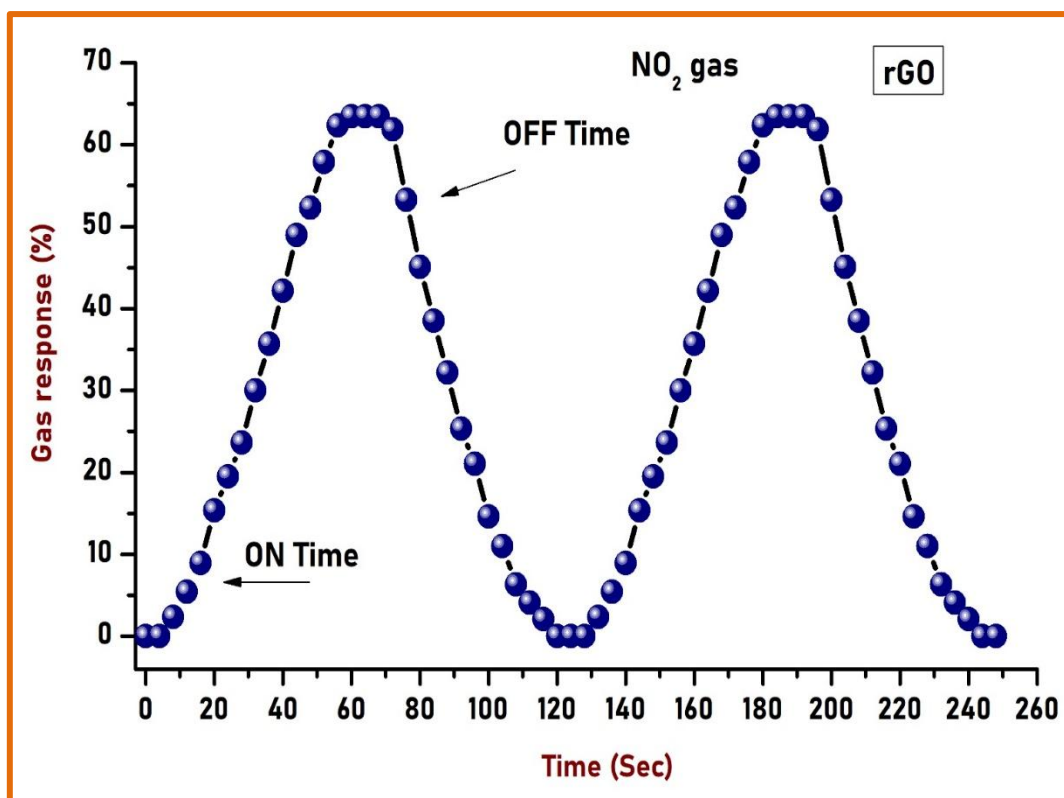


Fig. 4. Response and recovery time plot of rGO thin films to NO₂ gas

The response reaches 90% of its maximum value within a short time, and the response time is estimated to be 19 s, demonstrating the fast sensing behavior of the rGO thin films. Upon switching off the NO₂ gas supply and exposing the sensor to air (OFF state), the response gradually decreases as NO₂ molecules desorb from the rGO surface, and the sensor resistance returns toward its baseline value. The recovery time is found to be 82 s, which reflects efficient desorption and good reversibility of the sensing process. The reproducible response pattern observed during repeated ON–OFF cycles confirms the stability and repeatability of the rGO thin film sensor [23, 27]. The fast response and reasonable recovery times can be attributed to the high surface area, abundant defect sites, and efficient charge transfer characteristics of rGO, which facilitate rapid adsorption and desorption of NO₂ molecules [25, 28].

Figure 5 illustrates the variation of gas response of rGO thin films as a function of NO₂ gas concentration measured at the optimal operating temperature of 120 °C. The NO₂ concentration was varied from 100 to 1000 ppm to evaluate the sensitivity and detection capability of the sensor. It is observed that the gas response increases progressively with increasing NO₂ concentration, indicating a strong dependence of sensor response on gas concentration. At lower concentrations (100 ppm), the rGO thin films exhibit a measurable response of about 16.35%, demonstrating the capability of the sensor to detect NO₂ even at relatively low concentrations [28, 29]. As the concentration increases to 300 and 500 ppm, the response rises significantly to 29.77% and 51.69%, respectively, due to the increased number of NO₂ molecules interacting with the rGO surface and enhanced charge transfer.

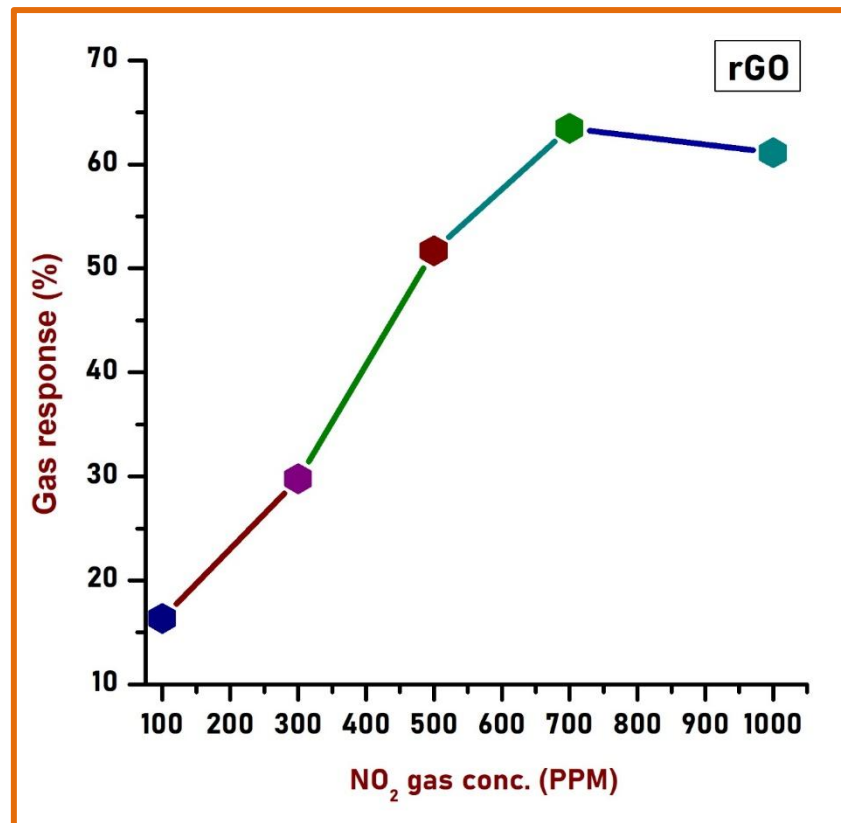


Fig. 5. Gas response versus NO₂ ppm variation plot of rGO thin films

The maximum gas response of 63.48% is observed at an NO₂ concentration of 700 ppm, which corresponds to the optimum sensing condition for the rGO thin films. Beyond this concentration, a slight decrease in response is observed at 1000 ppm (61.11%), which can be attributed to saturation of active adsorption sites on the rGO surface, limiting further charge transfer [30, 31]. The nearly linear increase in response at lower concentrations followed by saturation at higher concentrations suggests that the sensing mechanism is governed by surface adsorption–desorption dynamics. The detectable response at 100 ppm indicates that the rGO thin film sensor possesses a low detection limit for NO₂, making it suitable for practical monitoring of NO₂ over a wide concentration range.

Reproducibility represents the ability of the sensor to produce consistent and repeatable responses upon repeated exposure to the same gas concentration under identical operating conditions, and it is typically assessed by performing multiple sensing cycles and comparing the variation in response magnitude [22, 29].

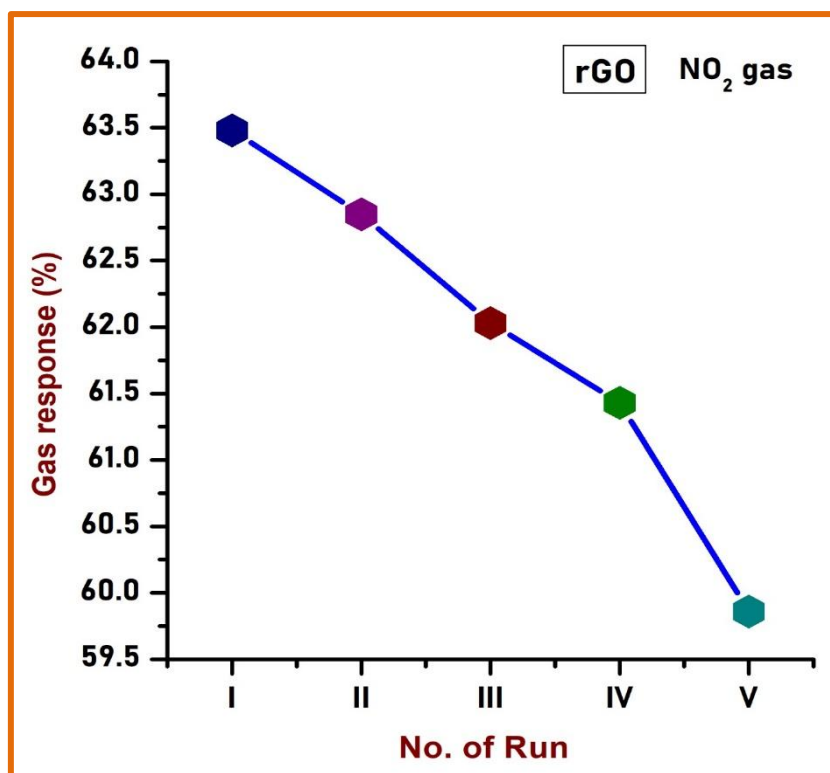


Fig. 6. Reproducibility plot of rGO thin films to NO₂ gas

Figure 6 shows the reproducibility characteristics of the rGO thin film sensor toward NO₂ gas, evaluated through repeated sensing cycles at the optimum operating temperature of 120 °C and a fixed NO₂ concentration of 700 ppm. The plot represents the gas response (%) recorded over five successive runs under identical experimental conditions. It is observed that the rGO thin films exhibit a high and consistent response in each cycle, with response values of 63.48%, 62.85%, 62.03%, 61.43%, and 59.86% for runs I to V, respectively. Although a slight decrease in response is observed with increasing number of runs, the overall variation remains small, indicating stable sensing behavior [29, 30]. The minor reduction in response over successive runs can be attributed to partial saturation of active adsorption sites or slight changes in surface states of the rGO film during repeated exposure to NO₂ gas. Nevertheless, the sensor maintains a high response level with good repeatability, demonstrating reliable adsorption–desorption reversibility and structural stability of the sensing layer [31–33]. The reproducibility results confirm that the spin-coated rGO thin films possess good operational stability and repeatable sensing performance, which are essential requirements for practical and long-term NO₂ gas sensing applications.

CONCLUSIONS

In the present work, reduced graphene oxide thin films were successfully fabricated on glass substrates using a simple and cost-effective sol–gel assisted spin-coating technique. The preparation method resulted in uniform, well-adhered rGO thin films suitable for gas sensing applications. The gas sensing performance of the rGO films was systematically investigated toward various reducing and oxidizing gases, including H₂S, NO₂, CO₂, NH₃, and CH₄, over an operating temperature range of 30–150 °C. Among the tested gases, the rGO thin films exhibited a pronounced and selective response toward NO₂, with a maximum gas response of 63.48% observed at an optimal operating temperature of 120 °C for a gas concentration of 700 ppm. The sensor exhibits a high gas response with fast response and recovery characteristics, achieving a response time of 19 s and a recovery time of 82 s, indicating rapid adsorption and desorption of NO₂ molecules on the rGO surface. The gas response increases systematically with

increasing NO₂ concentration from 100 to 700 ppm, confirming good sensitivity and a low detection capability, while a slight saturation trend at higher concentrations suggests effective utilization of available active sites. The films demonstrated good selectivity, reproducibility, and rapid response and recovery behavior, indicating efficient adsorption–desorption kinetics and stable sensing performance. These results confirm that spin-coated rGO thin films are promising candidates for selective and reliable NO₂ gas sensing. Furthermore, the simplicity, low cost, and scalability of the fabrication process make the developed rGO-based sensor highly suitable for practical environmental monitoring and industrial safety applications.

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