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Preparation and Characterization of PANI/GO/WO_3 Nanocomposites: Structural Properties and Their Applications in Gas Sensing

This work aims to examine the synthesis, optical properties, and gas-sensing abilities of PANI/GO/WO_3 nanocomposites. An FTIR study was conducted to examine the chemical contents of nanocomposites. The synthesis effort commenced with the application of chemical oxidative polymerization of polyaniline (PANI). The detection of H_2S gas was assessed across an extensive temperature range. The integration of metal oxide (WO_3) and graphene oxide (GO) into the PANI matrix led to a reduction in structural stability. The findings indicated that the integration of these two materials enhanced gas detection. The nanocomposites exhibited exceptional performance due to their enhanced sensitivity, rapid response time, and selectivity for H_2S, even under elevated temperatures. Research has shown that nanocomposites composed of PANI, GO, and WO_3 has the capability for gas sensing applications in agricultural and industrial monitoring. Future experiments should concentrate on refining the material composition and processing parameters to improve the system's sensitivity, selectivity, and long-term operational stability.

Keywords: Polyaniline; Graphene oxide, Tungsten trioxide; H_2S detection
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1. Introduction

Since the turn of the century, nanomaterials have grown in sophistication as a result of the distinctive characteristics they possess. The development of gas sensing, energy storage, electronics, and catalysis has been influenced by a wide variety of causes [1]. PANI possesses an extraordinary conductivity that can be attributed to its chemical stability, its electrical conductivity that can be adjusted, and its uncomplicated production procedure. PANI may operate inefficiently under some circumstances; hence, it is necessary to make improvements to it. The huge surface area, electrical properties, and the ability to interact with both organic and inorganic molecules that graphene oxide (GO) possesses make it an excellent choice for use in conjunction with polyaniline [2]. The sensitivity and selectivity of gas sensing in PANI/GO composites are both improved by the addition of tungsten trioxide (WO_3). Having this ability is absolutely necessary in order to recognize hydrogen sulfide (H_2S), which is a hazardous industrial chemical [3]. In order to protect workers and monitor environmental conditions, H_2S detection sensors need to have a high level of sensitivity since they are also very effective. Synthesis of advanced nanocomposites made from PANI/GO/WO_3 is the primary objective with this project. For the purpose of analyzing their structure, FTIR was applied [4].

Gas sensing was tested at various temperatures to determine how material composition affects sensor sensitivity and response [5]. Nanomaterials for advanced gas sensors are the goal of this study. Gas detection technology for industrial and environmental usage will improve. This will be done by lowering response times and enhancing gas detection.

2. Experimental Work

Pure polyaniline was made in a 0°C ice bath. Add aniline and 0.3 M hydrochloric acid to a 100-mL magnetic stirred volumetric flask to start polymerization. Following a similar technique, two grams of ammonium persulfate were combined with fifty milliliters of distilled water for a period of fifteen minutes. Next, modest volumes of aniline-hydrochloric acid mixture were added to this solution. As the addition process proceeds, the solution turns dark green over five hours while the ice bath temperature remains steady. The reaction mixture is then left to stand overnight. The resulting polymer is filtered through filter paper, thoroughly rinsed with distilled water, baked in an oven at 40°C for an hour, and then ground into a fine powder.

After the early polymerization processes, which include combining aniline and hydrochloric acid with an ammonium persulfate solution, GO is added to the reaction mixture in different weight percentages. This method takes 5 hours of churning in an ice bath. The combination is then set to react overnight. The final product is then filtered, rinsed repeatedly with distilled water, dried for one hour at 40°C in an oven, and crushed into a fine powder.

Ten minutes after pure polyaniline compound production, graphene oxide is added. WO_3 is continually combined in various weight ratios. After five hours of agitation in an ice bath, the mixture rests for 24 hours. After filtering, the precipitate is repeatedly rinsed with distilled water. This is done

multiple times. After that, the material is baked at 40 degrees Celsius for an hour, then ground to a fine powder.

3. Results and Discussion

Fourier-transform infrared spectroscopy (FTIR) evaluates composite material structure and operation. In PANI/GO/WO_3 composites, FTIR can study the interaction between the metal oxides of PANI, GO, and WO_3. These composites' vibrational modes, which match their chemical structures, simplify synthesis. These composites could be employed in sensors, electronics, and energy storage. An FTIR study of PANI revealed many functional group-related absorption bands. The signal at 3285 cm^{-1}, caused by N-H bond vibrations, indicates the presence of aniline units.

The peaks at 1644 and 1570 cm^{-1} indicate the conjugated structure. These peaks represent PANI backbone vibrational modes in the benzenoid and quinoid rings. The peak at 1485 cm^{-1} suggests that benzene rings exhibit C=C stretching, while aniline units may exhibit C-N stretching around 1301 cm^{-1}. Benzenoid and quinoid complexes show vibrational modes at 1124 and 879 cm^{-1} in their spectral bands. Non-planar deformations at frequencies lower than 700 cm^{-1} are linked to aromatic ring absorption properties. The spectrum shows PANI production and aniline-benzoquinone structure. This increases its electrical conductivity, making it suitable for electrochemical and electronic applications [6-8].

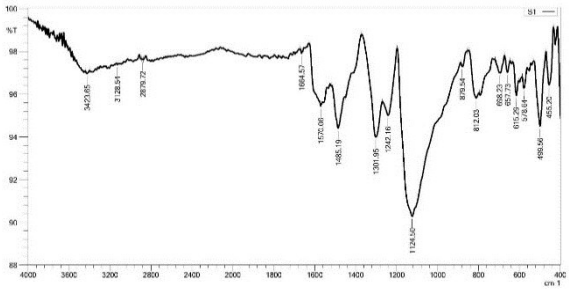

Fig. (1) FTIR spectrum for PANI

The FTIR analysis found structural links between CO and PANI in the PANI/GO 1% composite absorption bands. These links were in the composite. The absorption peak at 1644 cm^{-1} indicates C=C stretching in benzenoid rings, highlighting the material's conjugated backbone. The peak at wavenumber of 1552 cm^{-1} marks the presence of N=Q=N vibrations, where Q represents the quinoid under study. Vibrations indicate structural redox changes in PANI. PANI-GO interaction is indicated by C-N-C vibration absorption at 1481 cm^{-1}. Peaks at 1300 and 1242 cm^{-1} indicate GO presence in the polymer matrix. These peaks match aniline unit C-N bond stretching.

The vibrations observed at 1107 and 1045 cm^{-1}, corresponding to C-O-C and C-O, substantiate the existence of oxygen-containing functional groups within graphene oxide. Furthermore, the peaks observed at 877, 796, and 704 cm^{-1} signify out-of-plane deformations of aromatic rings, thereby bolstering the structural integrity of the composite. This spectral study validates the significant interaction between PANI and GO, improving the composite's electrical and mechanical properties, thereby positioning it as a viable contender for electronic and electrochemical applications [9-11].

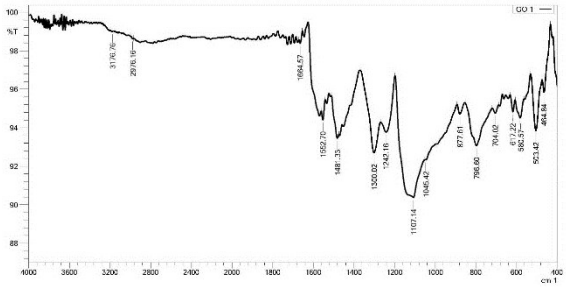

Fig. (2) FTIR spectrum of PANI loaded with 1% GO nanocomposite

The FTIR examination of the PANI/GO 5% composite, depicted in figure 3, displays significant absorption bands that indicate the interactions between polyaniline (PANI) and graphene oxide (GO). The signal at 1658 cm^{-1} corresponds to C=C stretching vibrations in benzenoid rings, affirming the conjugated structure of PANI. The absorbance at 1562 cm^{-1} indicates variations in PANI oxidation states due to N=Q=N vibrations. The band at 1479 cm^{-1} indicates C-N-C stretching, confirming the successful integration of GO and PANI. The peaks seen at wavenumbers of 1301 and 1244 cm^{-1} show that the C-N bonds are stretching within the aniline units, which makes the composite stronger. The absorptions seen at 1128 and 879 cm^{-1} suggest that GO has oxygen functional groups. The levels align with the vibrations associated with the C-O-C and C-O types, respectively. Peaks at 802, 702, and 659 cm^{-1} show the out-of-plane bending of aromatic rings. These improve the composite's structural integrity. Based on these findings, it seems that PANI and GO have effectively interacted, with a higher quantity of GO leading to an improvement in structural changes. Oxygen-related vibrations have been confirmed, indicating the preservation of GO functions. Such behavior could potentially contribute to enhanced electrical conductivity and improved mechanical stability. The composite material demonstrates suitability for applications including sensors, energy storage, and electronic devices, attributed to its structural integrity and functional interactions [12-14].

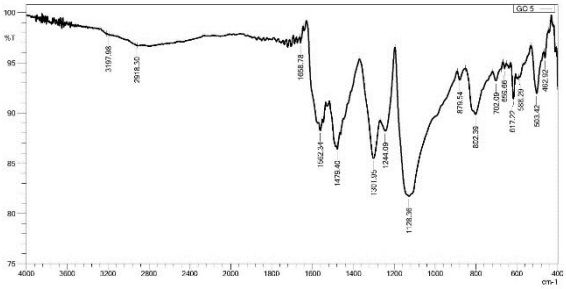

Fig. (3) FTIR spectrum of PANI loaded with 5% GO nanocomposite

When WO_3 is added to the PAN and GO matrix, the FTIR examination of the composite (Fig. 4) shows significant structural alterations. PANI's conjugated structure is confirmed by the absorption peak at 1568 cm^{-1}, which indicates C=C stretching in benzenoid and quinoid rings. N=Q=N vibrations in the 1550 cm^{-1} band suggest oxidation state changes in PANI. Absorption at 1481 and 1448 cm^{-1} points to C-N-C stretching, indicating strong interactions between PANI, GO, and WO_3. Successful integration of GO and WO_3 into the polymer matrix is confirmed by peaks at 1301 cm^{-1} and 1246 cm^{-1}, attributable to C-N stretching in aniline units. Absorption at 1134 and 1043 cm^{-1} indicates oxygen-containing functional groups from GO through C-O-C and C-O vibrations. Peaks at 877, 796, and 705 cm^{-1} indicate aromatic ring out-of-plane bending, preserving composite structure. Tungsten oxide is confirmed by additional peaks at wavenumbers of 619, 576, and 503 cm^{-1}, which correspond to W-O vibrations. Spectral changes suggest substantial interactions between PANI, GO, and WO_3, improving the composite's electrical conductivity, mechanical stability, and charge transfer. Adding WO_3 is expected to enhance electrochemical performance, making it a potential material for sensors, energy storage, and electrical devices [15-17].

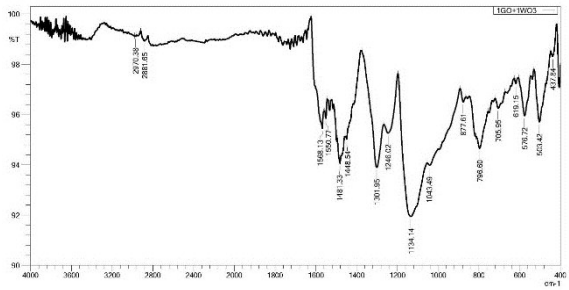

Fig. (4) FTIR spectrum of PANI loaded with1% GO/1% WO_3 nanocomposite

Figure (5) shows the FTIR analysis of the PANI/5% GO/3% WO_3 composite, highlighting structural alterations caused by the integration of GO and WO_3 into the PANI matrix. The absorption signal at 1570 cm^{-1} indicates C=C stretching in benzenoid and quinoid rings, validating the conjugated structure of PANI. Strong interactions between PANI, GO, and WO_3 are shown by peaks at 1531 and 1483 cm^{-1}, which correspond to N=Q=N and C-N-C vibrations.

The peaks at 1303 and 1240 cm^{-1} are attributed to C-N stretching in aniline units, confirming the incorporation of GO and WO_3 into the polymer network. Absorption at 1138 and 1056 cm^{-1} corresponds to C-O-C and C-O vibrations, verifying the retention of oxygen functional groups from GO. The peaks at 995, 877, and 800 cm^{-1} are linked to out-of-plane bending of aromatic rings, maintaining the structural stability of the composite. Additionally, new bands at 704, 648, and 586 cm^{-1} correspond to W-O vibrations, confirming the presence of tungsten oxide.

The observed spectral shifts and new absorption bands indicate strong synergistic interactions between PANI, GO, and WO_3. The presence of WO_3 enhances charge transfer efficiency and mechanical stability, potentially improving electrochemical performance. This composite material exhibits promising properties for applications in energy storage, sensors, and electronic devices, where improved conductivity and structural stability are crucial [18-20].

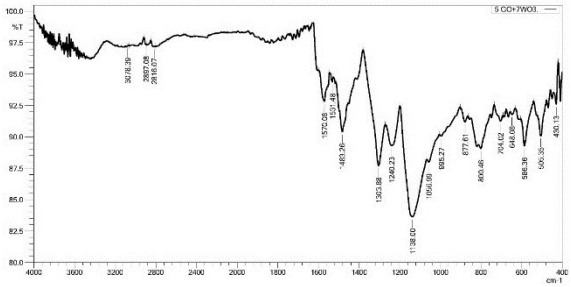

Fig. (5) FTIR for PANI loaded with 5% GO/3% WO_3 nanocomposite

Gas sensing using H_2S is crucial for environmental and industrial applications, requiring highly sensitive and selective materials. Composite structures like PANI/GO/WO_3 offer enhanced performance due to the superior conductivity of PANI, the unique properties of metal oxides, and the high surface area of graphene. These advanced materials enable efficient H_2S detection at low concentrations, making them promising candidates for next-generation gas sensors [21].

Sensor response of PANI to H_2S at different temperatures (30, 100, and 150°C) shown in Fig. (6), the sensor exhibits a gradual decrease in resistance upon exposure to H_2S, followed by fluctuations indicating an incomplete recovery process. The response suggests that PANI, as a p-type semiconductor, undergoes charge carrier modulation due to interactions with H_2S molecules. The observed resistance decrease is likely due to the reduction of adsorbed oxygen species and the direct interaction of H_2S with the PANI surface, leading to increased hole concentration [22]. Nonetheless, the protracted and

erratic recovery phase underscores the constraints associated with room-temperature operation, especially regarding desorption efficiency [23].

After being exposed to H_2S at 100°C, the resistance decreases significantly and becomes constant. Thus, heat improves gas molecule adsorption onto PANI. Effective desorption kinetics affect sensor reliability and durability [24]. This association is strengthened by the greater stability during ambient temperature recovery. This temperature increases the sensors' sensitivity to detect even trace levels of H_2S, a major improvement [25]. Resistance decreases at 150°C, indicating H_2S exposure. Temperature accelerates H_2S-sensor material reaction kinetics, improving charge transfer efficiency. A progressive decline in charge transfer shows this. Desorption is more successful at this temperature, resulting in more recovery and lower resistance [26]. Higher operating temperatures enable faster detection and recovery. Sensors stabilize and react faster at increasing temperatures [27].

PANI has great potential as a gas-sensitive H_2S detector, according to the findings. This is shown by showing how temperature affects reaction magnitude, stability, and recovery efficiency. Monitoring the reaction is achievable at low temperatures, but the sensor works better at high temperatures since adsorption and desorption occur faster. Polyaniline can increase H_2S sensor sensitivity, selectivity, and long-term stability by changing operational settings. This is possible due to the material's conductivity variations and heat activation [28].

Figure (7) illustrates the response of the 1% GO-based sensor to H_2S gas across different temperatures, indicating that the sensing mechanism varies with temperature. At 30°C, the resistance demonstrates a modest initial decline, succeeded by variations. At ambient temperature, H_2S molecules engage in the processes of adsorption and desorption, resulting in a reaction characterized by instability [29]. The minor variation in resistance indicates that H_2S and the sensing material exhibit a weak interaction at lower temperatures. This is likely due to the diminished efficiency of charge transfer [30].

At 100°C, the response is more pronounced, showing an initial drop in resistance followed by a gradual increase. This behavior suggests that H_2S adsorption initially dominates, reducing the resistance due to electron donation from H_2S to the sensing material [31]. However, as the exposure continues, the resistance starts increasing, indicating a complex interaction where desorption or sensor surface saturation might play a role [32]. The improved response compared to 30°C indicates that higher temperatures enhance the reactivity of H_2S with the sensor surface [33].

At 150°C, the resistance exhibits a sharp drop upon H_2S exposure, followed by a relatively stable response. This suggests that at elevated temperatures, the sensor experiences strong charge transfer interactions, leading to significant conductivity changes [34]. The sustained lower resistance indicates enhanced adsorption of H_2S, while the stability of the response suggests that desorption is minimized at this temperature, making the sensor more reliable [35].

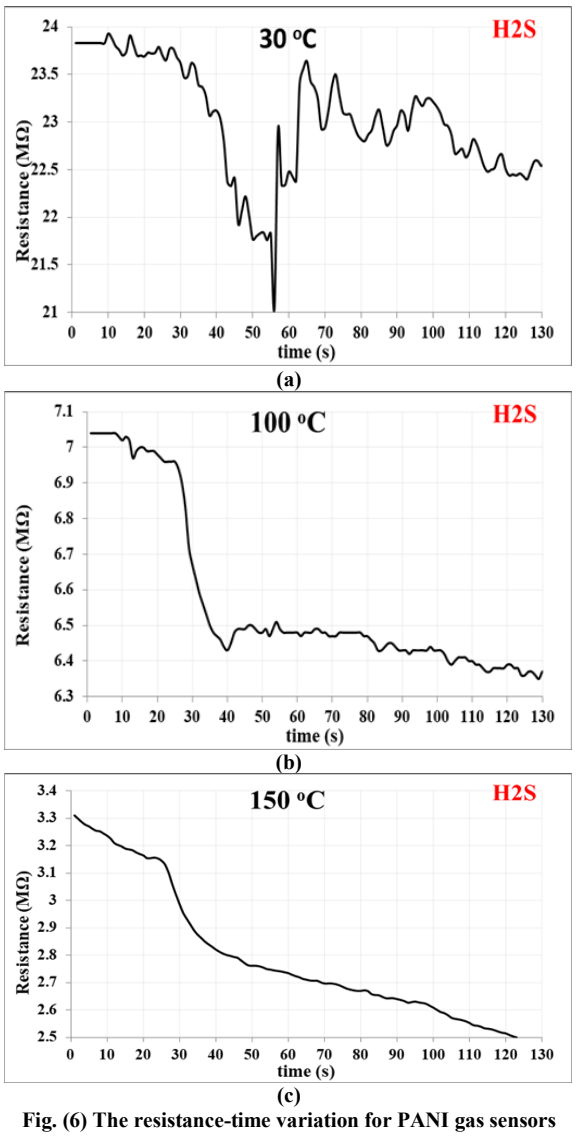

Fig. (6) The resistance-time variation for PANI gas sensors

Overall, the 1% GO sensor demonstrates an increasing sensitivity with temperature. The resistance drop becomes more pronounced at higher temperatures, indicating enhanced gas-sensing performance [36]. However, the fluctuating response at lower temperatures suggests that optimization is needed for stable and reproducible sensing behavior [37].

For the 5% GO-based sensor, the response to H_2S gas at different temperatures shown in Fig. (10) exhibits distinct trends, indicating a strong temperature dependence of the sensing mechanism. At 30°C, the resistance initially decreases slightly, followed by significant fluctuations throughout the exposure period.

This behavior suggests that at room temperature, the adsorption and desorption of H_2S molecules are competing processes, leading to an unstable sensing response [38]. The weak and inconsistent change in resistance implies limited interaction between H_2S and the sensing material at low temperatures, likely due to insufficient energy for effective charge transfer or chemical reaction [39].

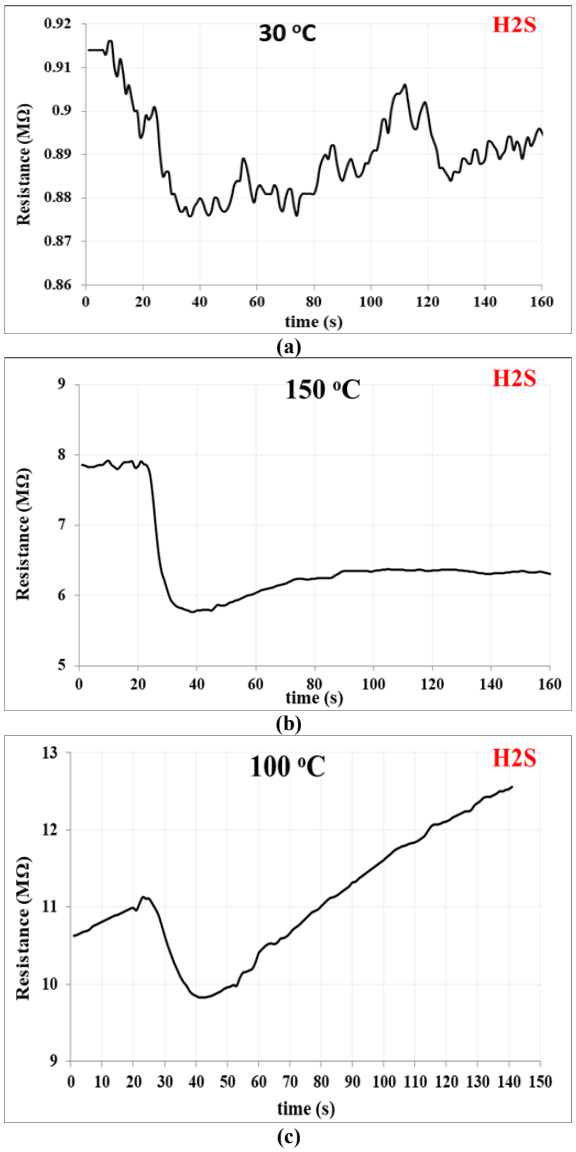

Fig. (7) The resistance-time variation for PANI/GO1% nanocomposites gas sensors

At 100°C, the sensor demonstrates a pronounced drop in resistance upon H_2S exposure, indicating enhanced interaction between the gas molecules and the sensing material [40]. The response demonstrates several fluctuations, indicating a dynamic adsorption-desorption equilibrium [41]. The observed temporary recovery and subsequent resistance oscillations suggest that the sensor exhibits responsiveness; however, it fails to establish a fully stable baseline. This instability may be linked to fluctuations in charge carrier concentration and surface interactions at the given temperature [42].

The resistance demonstrates a more gradual, stable, and defined reduction at 150°C in comparison to lower temperatures. The evidence suggests that the sensor exhibits enhanced charge transfer at elevated temperatures, leading to a more stable and extended response [43]. The continuous decrease in resistance indicates a significant interaction between H_2S molecules and the GO-modified surface, which improves electron donation and conductivity [44]. The lack of complete resistance recovery indicates that desorption at this temperature remains incomplete, possibly necessitating additional heating or an external stimulus for full regeneration [45].

Overall, the results highlight that increasing the GO concentration to 5% alters the sensor's response dynamics, with higher temperatures enhancing sensitivity and stability [46]. However, the fluctuating response at lower temperatures suggests that optimization is required to achieve consistent and reproducible sensing performance across different operating conditions [47].

The response versus time of (1% GO + 1% WO_3) shown in Fig. (9). At 30°C, the sensor exhibits a significant drop in resistance upon exposure to H_2S, followed by a gradual recovery phase. The initial sharp decrease suggests a strong interaction between H_2S molecules and the sensing material, leading to increased charge carrier concentration and enhanced conductivity [48]. However, the incomplete recovery indicates slowly desorption of gas molecules at this low temperature, which may limit the sensor's ability to return to its baseline resistance quickly [49].

At 100°C, the sensor response becomes more dynamic, with a pronounced increase in resistance upon gas exposure, followed by a gradual decline. This behavior suggests that the adsorption mechanism at this temperature differs from that at 30°C, likely due to enhanced chemisorption effects facilitated by increased thermal energy [50]. The observed increase in resistance on WO_3 may result from either electron depletion or interaction with surface oxygen species induced by H_2S molecules [51]. This will result in a decreased number of free carriers. The adsorption-desorption process during the incremental recovery phase exhibits a more balanced response at higher temperatures compared to previous observations [52].

At 150°C, resistance significantly increases upon exposure to H_2S, followed by a gradual decline over time. An early surface reactivity with oxidized species likely accounts for the transient decrease in charge carriers resulting from the significant rise [53]. As resistance decreases, the equilibrium between adsorption and desorption appears to stabilize at elevated temperatures [54]. The improved recovery rate at this temperature indicates enhanced gas desorption

kinetics, which is crucial for the stability and reusability of sensors [55]. The results demonstrate that the amalgamation of 1% GO and 1% WO_3 exhibits a notable temperature-dependent reaction to H_2S, characterized by different adsorption and charge transfer pathways across various working temperatures [56]. Reduced temperatures facilitate gas adsorption while impeding desorption; in contrast, elevated temperatures accelerate dynamic responses and recovery rates. Optimization of the sensor's sensitivity and stability can be achieved through the adjustment of material composition and operating conditions, aiming for an equilibrium between response magnitude and recovery efficiency [57].

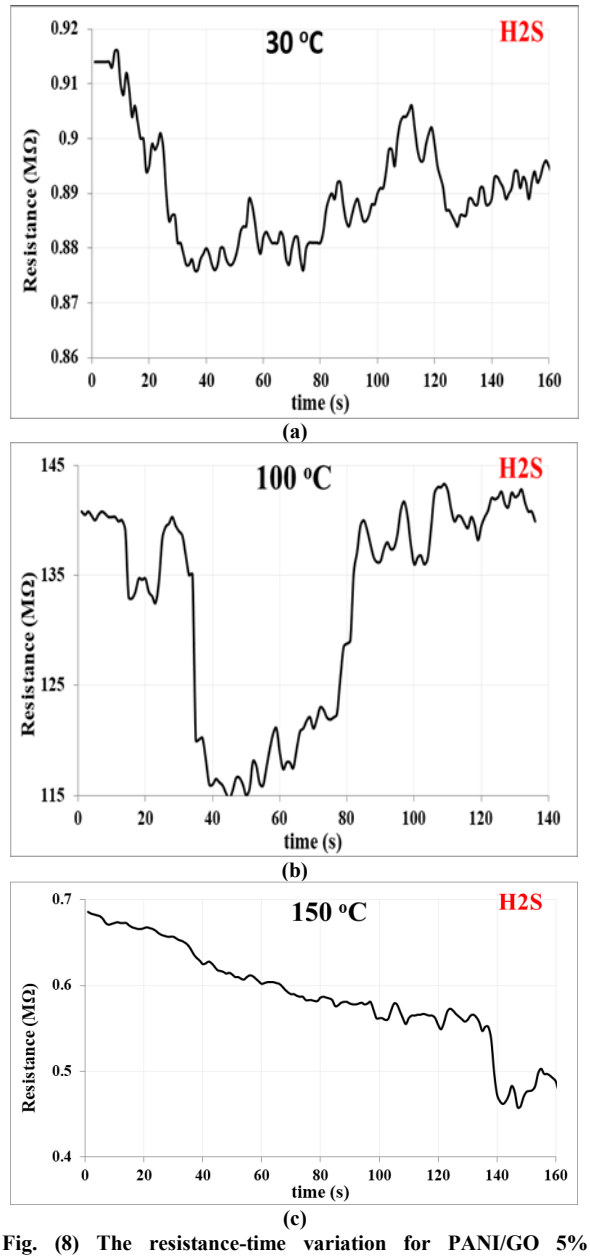

Fig. (8) The resistance-time variation for PANI/GO 5% nanocomposites gas sensors

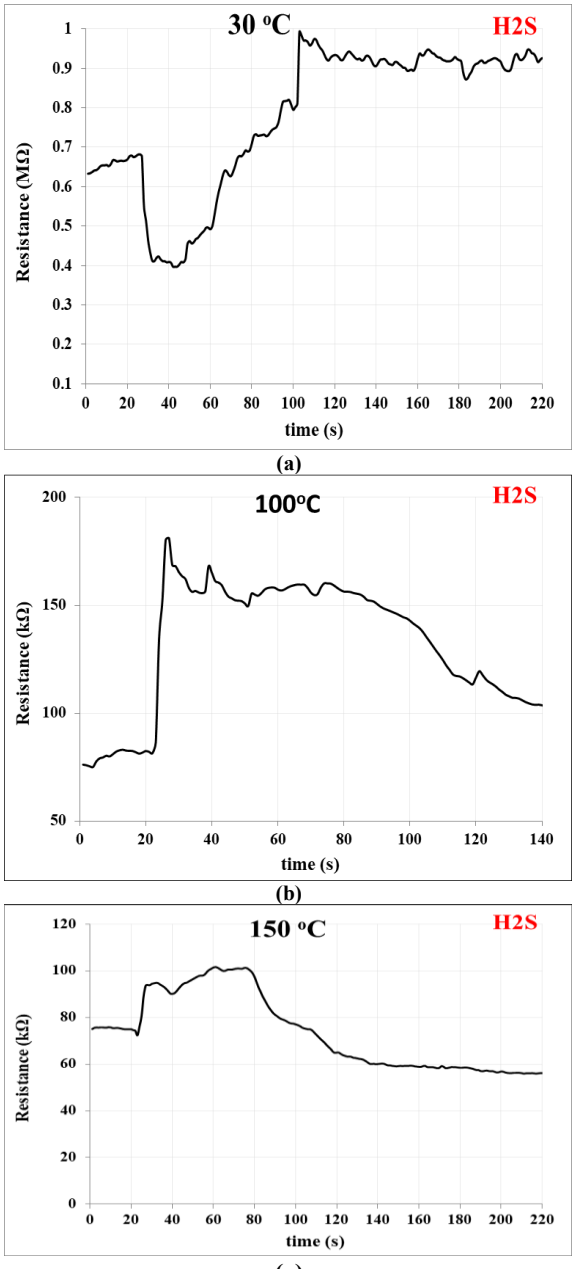

Fig. (9) The resistance-time variation for PANI/1%GO/3%/WO_3 nanocomposites gas sensors

The response versus time for (5% GO + 3% WO_3) shown in Fig. (10). At 30°C, the sensor demonstrates an initial sharp rise in resistance, followed by a rapid decline upon exposure to H_2S. This behavior suggests a strong interaction between the gas molecules and surface-adsorbed oxygen species [58]. The resistance drop indicates charge carrier transfer, likely due to the reduction of pre-adsorbed oxygen by H_2S, leading to enhanced conductivity [59]. However, the incomplete recovery phase at this low temperature highlights a slow desorption process, which may limit sensor stability and reusability under ambient conditions [60].

Resistance rises significantly at 100°C, stabilizes at a plateau, and subsequently declines [61]. Studies suggest that H_2S molecules interact with WO_3 and GO nanostructures, reducing charge carriers and increasing resistance [62]. The slow drop indicates desorption, allowing sensor partial recovery [63]. The measured temperature balances adsorption and desorption, making it more stable for gas detection [64].

Resistance initially rises at 150°C, then gradually decreases [65]. High resistance results from H_2S molecule interactions with sensor active sites, causing electron depletion [66]. Lower resistance means faster desorption kinetics at higher temperatures, accelerating recovery [67].

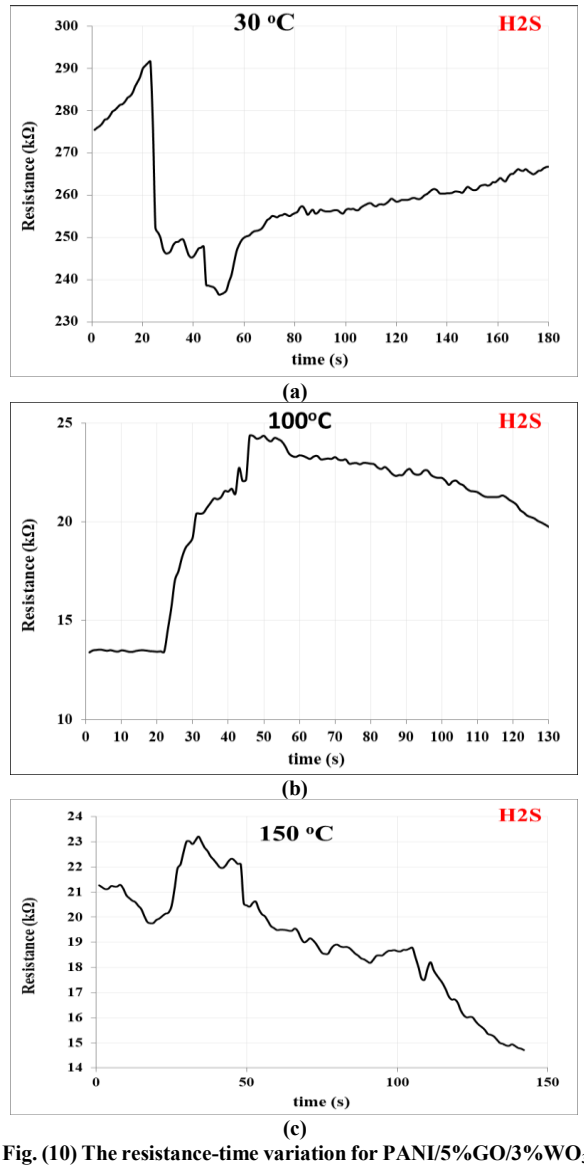

Fig. (10) The resistance-time variation for PANI/5%GO/3%WO_3 nanocomposites gas sensors

The enhanced response and recovery trends at this temperature underscore the importance of thermal activation in improving sensor efficacy [68]. The findings indicate that the 5% GO and 3% WO_3 composite demonstrates a temperature-dependent response to H_2S, characterized by distinct adsorption and desorption mechanisms influencing its behavior [69]. At reduced temperatures, strong adsorption prevails; however, slow desorption constrains recovery [70]. Higher temperatures result in an increased desorption rate, which facilitates a quicker sensor reset and enhances repeatability [71]. The sharp response and recovery dynamics indicate that this nanocomposite holds great potential for high-sensitivity gas detection applications [72]. Further optimization of the material composition and operating conditions could enhance the sensor's efficiency, ensuring an optimal balance between sensitivity, response speed, and stability [73-78].

It can be noticed from table (1) and Fig. (11) that the all type of nanomaterials enhanced the sensitivity, but in low ratio, the increase of loading ratio retarded the sensitivity values .On the other hand the sensitivity improved by rising the working temperature of pristine PANI while the sensitivity get to retarded at high elevated working temperatures for other nanocomposites gas sensors.

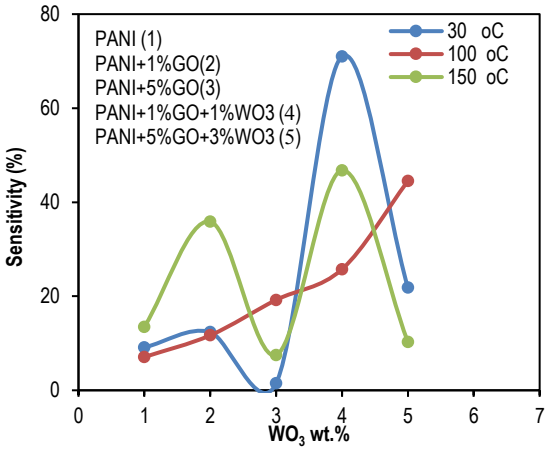

Fig. (11) Relation between sensitivity and loading type at different working temperatures of PANI loaded with different nanomaterials gas sensors toward H_2S gas

4. Conclusion

This study focused on the synthesis and characterization of PANI/GO/WO_3 nanocomposites, investigating their structural and using FTIR spectroscopy and evaluating their gas-sensing performance, particularly for H_2S detection. The results demonstrated that incorporating graphene oxide and metal oxides into the polyaniline matrix significantly enhanced the composites' structural stability, optical characteristics, and gas-sensing capabilities. The synthesized materials exhibited improved sensitivity and response stability at ambient and moderate temperatures, confirming their potential for real-world applications. The findings suggest that optimizing the composition of these nanocomposites can further

enhance their performance, making them promising candidates for highly sensitive and selective gas sensors. The improved conductivity, rapid response, and stability observed in these materials highlight their potential for industrial and environmental applications where efficient toxic gas detection is crucial. Future research should explore additional modifications in material composition and processing conditions to fine-tune their selectivity, response time, and long-term operational stability.

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Table (1) Values of sensitivity%, response and recover times of PANI loaded with different nanomaterials gas sensors toward H_2S gas

Nanocomposites sample	Temperature (°C)	Sensitivity (%)	Response Time (s)	Recovery Time (s)
PANI	30	9.13	24.3	89.1
	100	7.08	21.6	93.6
	150	13.52	21.6	65.7
PANI+1%GO	30	12.39	21.6	95.4
	100	11.70	13.5	70.2
	150	35.88	17.1	54.9
PANI+5%GO	30	1.48	19.8	91.8
	100	19.22	21.6	63.9
	150	7.50	21.6	63
PANI+1%GO+1%WO3	30	71.03	20	95
	100	25.74	26	88
	150	46.77	30	78
PANI+5%GO+3%WO3	30	21.84	23	94
	100	44.50	25	65
	150	10.30	21	50

