Synthesis and Characterization of PANI/GNS and PANI/GNS/TiO_2 Nanocomposites for Room Temperature LPG Gas Sensing Application

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Abstract

Pure polyaniline (PANI), PANI/GNS (1%), and PANI/GNS (2%)/TiO_2 (20%) were synthesised by the situ chemical oxidation polymerisation method. Titanium dioxide (TiO_2) was synthesised using the sol-gel method and graphene (GNS) functionalized by acidic treatment. The XRD, FTIR, and SEM characterization for structural, functional, and morphological investigations has been carried out. X-ray diffraction of nanocomposite materials shows the highly crystalline nature of synthesized material. FTIR analysis revealed the existence of a functional group in nanocomposite materials. SEM analysis shows highly porous nanocomposite materials are formed, that exhibit a lack of agglomeration with uniform distribution of GNS, and TiO_2 nanoparticles within the PANI matrix. The resistance change response of synthesised material toward LPG was measured at low and high temperatures at 250 parts per million (ppm), 500 ppm, and 1000 ppm of LPG gas, which determined the characteristics like sensitivity, response, and recovery time of the nanocomposite material. Sensitivity shows that nanocomposite material is highly sensitive towards LPG nearly at 37 °C. In ternary nanocomposite, the response time and recovery time are faster as compared to binary nanocomposite material. The response time of the ternary nanocomposite is 16 seconds and the recovery time is 83 seconds. Study shows that PANI/GNS (2%)/TiO_2 (20%) nanocomposite material shows better sensitivity, response and recovery time as compared to PANI/GNS (1%) nanocomposite material towards the higher concentration of LPG gas.

Keywords: PANI, TiO_2, Graphene (GNS), Polymerization, Nanocomposite, LPG, Sensitivity.
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Introduction

Gas sensors are in high demand due to the extensive utilization of various gases for residential and industrial purposes and the rising number of leakage-related incidents. LPG is an explosive gas that poses significant risks to individuals and the environment. Because of its strong flammability, even low concentrations (ppm) represent a considerable risk. The rapid and precise identification of LPG leaks is essential for preventing potential explosions [1]. Liquified petroleum gas (LPG) is a combustible gas mostly made by propane (5-10%), butane (75-80%), and minor quantities of methane, butylene, propylene, and ethylene (1-5%). It poses a significant hazard because of the elevated risk of explosions either from leakage or human mistake as a result, detecting it early on is critical for identifying and effectively controlling it [2]. Nanocomposites are formed by combining two or more nanosized items or nanoparticles. These nanocomposites exhibit distinct physical properties and have various potential applications in multiple fields. The morphology and structure of materials significantly impact the sensing characteristics. Polyaniline (PANI) nanocomposites have been found to possess superior sensing properties due to their high surface-to-volume ratio resulting from the nanostructure. This is evident in sensitivity, response time, and other relevant factors [3]. Conducting polymers are a low-cost sensing material with quick response time, high sensitivity, and room temperature functionality. They also allow for chemical and physical qualities using various substituents [4]. Due to its distinct electrochemical properties, polyaniline (PANI) is the preferred conducting polymer for detecting LPG related to chain nitrogen, as well as its tunable chemical and electrical properties, ease of synthesis, ability to sense ambient temperature and cost-effectiveness [5]. The conductivity of a polymer depends on its capacity to transfer charge carriers along its backbone and hop across chains. Interactions with polyaniline can modify these processes, affecting conductivity [6]. Compared to plain polymers, conducting polymer composites based on graphene exhibit superior properties [7]. Graphene, in contrast to most porous carbon-based materials, is two-dimensional in nature and a single sheet of sp2-hybridized carbon with exceptional conductivity, mechanical strength, and surface area [8]. Graphene (GNS) has attracted a lot of potential applications as gas sensor material this is because of its inherent physical and electrical properties, including its high surface-to-volume ratios, elevated tensile strength, chemical inertness, and outstanding thermal and electrical conductivity [9]. To improve the functionality of electrical sensors that operate at room temperature, it is possible to include sensing materials, including conducting polymers, into the surface of GNS sheets sensors by combining their superior properties [10]. Graphene or CNT interacts with conducting polymers to store energy by electronic and ionic charge separation, as well as charge transfer across the electrode-electrolyte interface [11]. Gas-sensing sensors use inorganic elements, such as metal oxide semiconductors, to detect gas molecules by measuring changes in conductivity. At present, there is an ongoing investigation into semiconducting metal oxides such as TiO_2, WO_3, In_2O, CuO, SnO_2, ZnO, and V_2O_5, focusing on their possible applications in gas sensing [12]. Nanocrystalline TiO2 is a prominent inorganic material for detecting gases such as NO2, H2, NH3, and LPG [13]. Researchers have developed composite PANI/TiO2 materials for use as gas sensors [14]. Titanium dioxide is a significant n-type semiconductor owing to its superior electrical and photocatalytic characteristics [15]. Hybrid carbon-based nanostructures present promising materials for various applications, including flexible batteries, biosensors, solar cells, supercapacitors, chemical sensors, and gas sensing [16]. Researchers have used nanostructured materials, organic/inorganic nanocomposites, conducting polymers and carbon-containing materials to develop gas sensor devices that detect differences in resistance these devices have demonstrated enhanced performance even at ambient temperature [17]. This study effectively produced PANI, PANI/GNS (1%), and PANI/GNS (2%)/TiO_2 (20%) nanocomposites using the chemical oxidation polymerization process for LPG gas sensing. The gas-sensing capabilities of the nanocomposites were investigated at both ambient and high temperatures to evaluate the impact of graphene and TiO_2 on the gas-sensing properties of the conducting polymer polyaniline.

Materials and Methods

Aniline (99.5%), Ammonium persulfate (98%), sulphuric acid (95-97%), Nitric acid (69%), titanium isopropoxide (TTIP), Methanol (99.8%), absolute Ethanol (99.9%), and Graphene flakes (made available from NPL New Delhi). All the chemicals reach the standards of analytical reagent grade. Distilled water (DW) was used as the medium for producing the material.

Synthesis of TiO_2

The sol-gel method successfully synthesized titanium dioxide (TiO_2). In this method, titanium isopropoxide (TTIP) is used as a source of titanium. In a beaker insert 3.9 ml of TTIP into 60 ml of methanol and then place it on magnetic stirring at 50°C for 1 hour. Then, a muffle furnace dried this white precipitate at 600°C for 1 hour. A motor pistol produced a nanostructured form of TiO_2 [18].

Functionalization of Graphene (F-GNS)

An acidic treatment was used to functionalize graphene (GNS). GNS mix with 6M H_2SO_4 and 6M HNO_3 used in a 3:1 ratio and mix it properly for 15 minutes. Graphene flask was added to the solution and ultrasonically processed for 5 hours at 55°C. GNS was functionalized after using centrifugation, filtration, washing, and drying at 80O C for 16 hr. [19].

Synthesis of Polyaniline (PANI)/functionalized Graphene(F-GNS) (1%) and PANI/GNS (2%)/TiO_2 (20%)

In situ chemical oxidation polymerization method was used for the synthesis of Polyaniline (PANI)/F-GNS (1%) and PANI/F-GNS(2%)/TiO_2(20%) nanocomposites [20]. The weight percentage of GNS to aniline was 1%. Divide the 0.2 M H_2SO_4 solution in 50 ml of deionized water into two equal parts. One part, add 0.2 M aniline and functionalized GNS, then ultrasonicate for 35 minutes. After ultrasonication, stir the mixture for 5–6 hours at 0–5° C to achieve the optimal yield. In another part, add 0.2 M ammonium persulfate (APS) to the stirring monomer solution and add it drop by drop. After mixing the reactants, the solution initially appears greenish and then turns violet. After around 6 to 7 hours, the black precipitate formed. We left the precipitate overnight and diluted it with deionized water until it became colourless. After washing with ethanol, it was oven-dried overnight at 80ºC. The same procedure carried out for synthesize the PANI/GNS/TiO_2 ternary composite, with a weight proportion of GNS (2%) and TiO_2 (20%) used in the 0.2M aniline ratio.

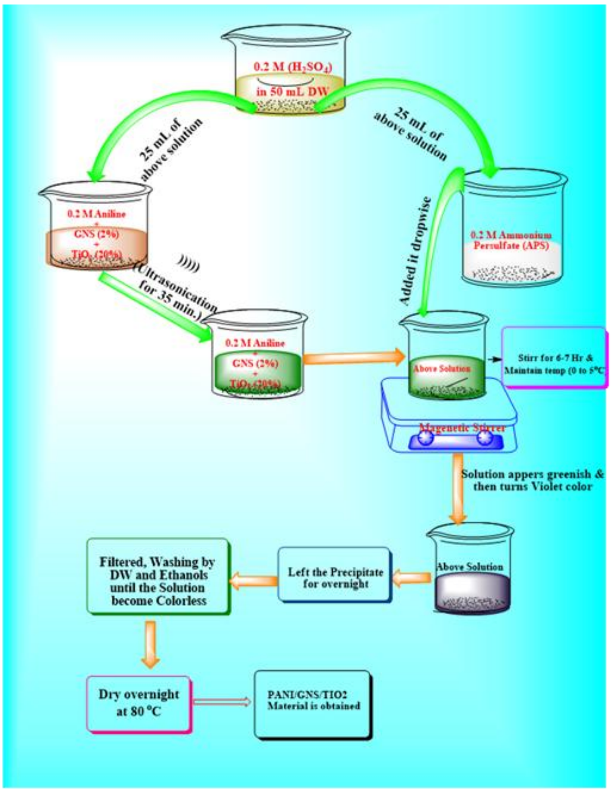


Figure 1: Flowchart of synthesis of PANI/GNS (1%) and PANI/GNS (2%)/TiO_2 (20%) Nanocomposite

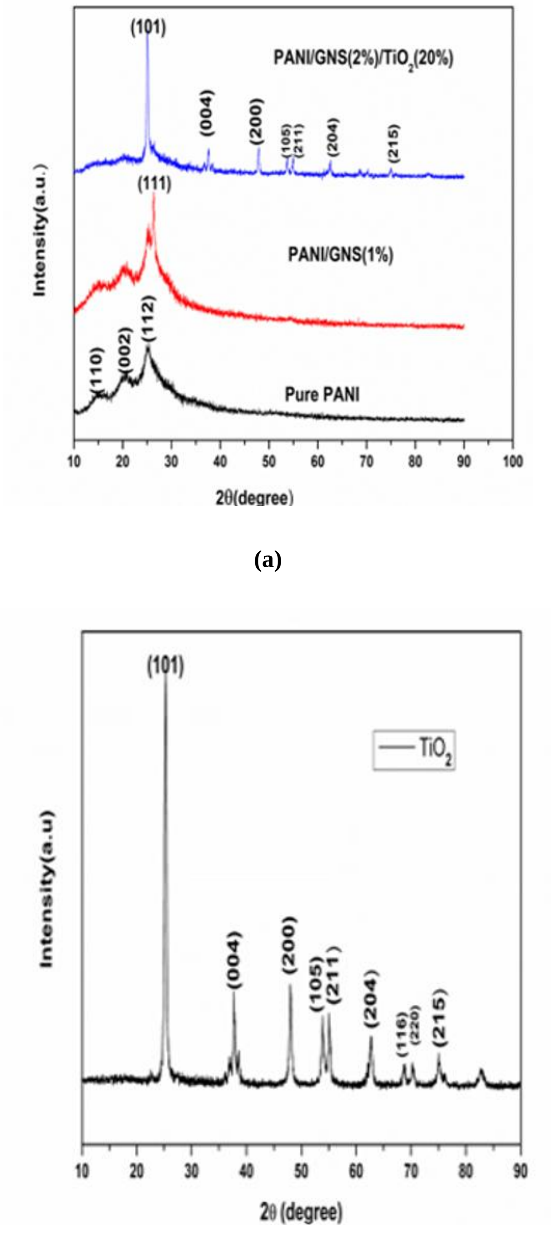
Characterizations

Table 1: Table shows characterization techniques and the instrument model used for characterization of materials.

Sr. No.	Characterization Techniques	Instrument Model
1	X-Ray diffraction (XRD)	Rigaku Mini-Flex 600, Rigaku Japan
2	Fourier Transform Infrared radiation (FTIR)	Model: Spectrum 400FT-IR/FIR, Spectrometer. Perkin Elmer, USA
3	Scanning electron microscopy (SEM)	SEM Model - Carl Zeiss EVO-18
4	The average particle size	Image-J application
5	Gas sensing	Tabletop static Gas sensing unit equipped with two probes and temperature controller

1. XRD analysis

Figure 2 (a) shows XRD patterns for Pure Polyaniline (PANI) characteristic peaks are observed at 2θ= 15.24°, 20.78°, and 25.26°, corresponding to the (110), (002), and (112) crystal planes. This indicates that the material is semi-crystalline in nature [21]. In Figure 2 (b) Distinct diffraction peaks for pure TiO_2 are noted at approximately 2θ values of 25.28°, 37.81°, 47.99°, 53.95°, 55°, 62.9°, and 75°. These peaks align with the crystallographic orientations of (101), (004), (200), (105), (211), (204), (116), (220), and (215) a tetragonal structure of the material has obtained. The individual peaks observed in the TiO_2 sample may be accurately matched to the anatase phase (JCPDS-ICDD card: 21-1272) [15]. When graphene was added to the PANI matrix, a strong and clear extra diffraction peak of GNS was



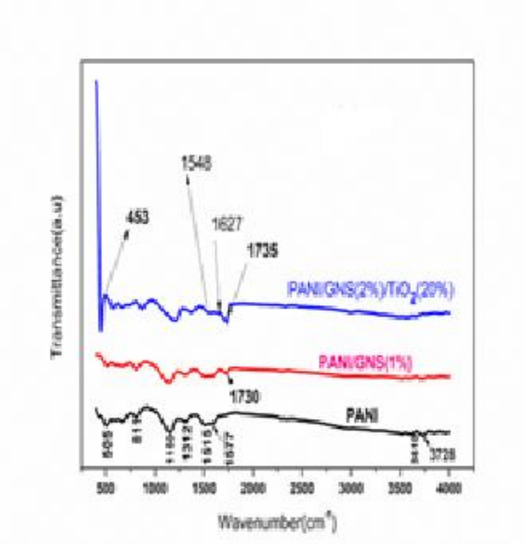
(a)

(b)

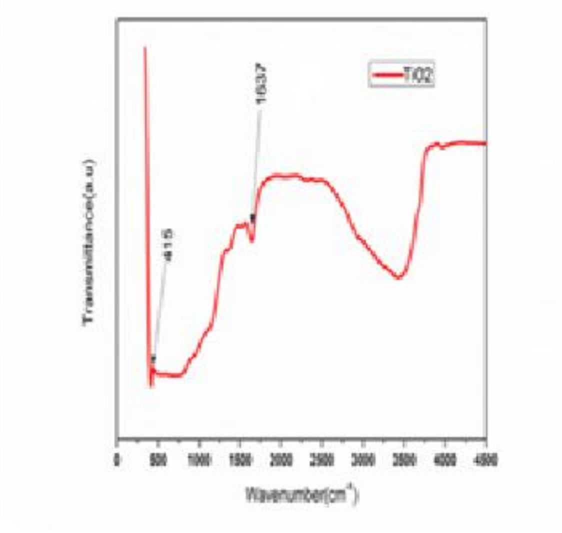
Figure 2: **(a)** Polyaniline (PANI), PANI/GNS (1%), and PANI/GNS (2%)/TiO_2(20%) **(b)** Pure TiO_2

seen at 26.46°, which is the same angle as the (111) crystal plane. When TiO_2 (20%) was added to the PANI/GNS (2%) composite which observed sharp and intense diffraction peaks are observed in the PANI/GNS(2%)/TiO_2(20%) nanocomposite matrix, which overlaps with the peak of the TiO2, GNS and PANI matrix. The results display sharp and intense defined peaks, indicating the highly crystallographic nature of the synthesized material.

2. FTIR analysis



(a)

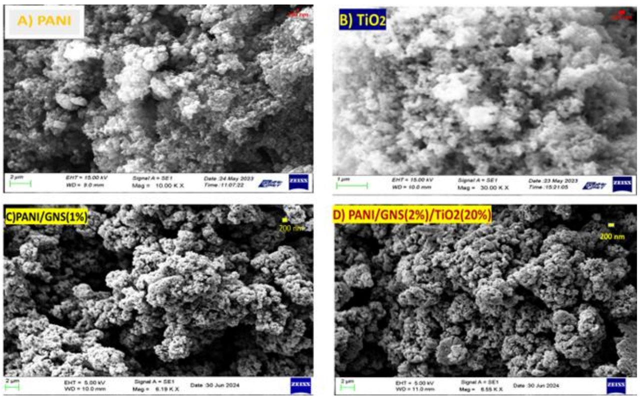


(b)

Figure 3: (a) Polyaniline (PANI), PANI/GNS (1%), PANI/GNS (2%)/TiO_2 (20%) **(b)** Titanium dioxide (TiO_2)

The FTIR spectroscopic analysis of pure polyaniline (PANI), titanium dioxide (TiO_2), PANI/GNS (1%), and PANI/GNS (2%)/TiO_2 (20%) nanocomposites. FTIR spectroscopy investigation of PANI displays multiple distinct peaks. The observed peak at 505 cm^{-1} and 1150 cm^{-1} shows the stretching vibrations of the C-H bond. The peak recorded at 811 cm^{-1} is due to the stretching of N-H bending. The peak at roughly 1312 cm^{-1} corresponds to the stretching vibrations of the C-N bond. The peaks at around 1515 cm^{-1} and 1577 cm^{-1} have been assigned to the stretching vibrations of the C=N bond in the benzenoid and quinoid rings, respectively [22]. The peaks identified in the 3605-3728 cm^{-1} range correspond to the stretching vibrations of the N-H bond in the aromatic amine. In Figure 3(b), the TiO_2 band is detected at a frequency of approximately 415 cm^{-1} during strong vibration. Furthermore, the frequency of 1637 cm^{-1} corresponds with the stretching frequencies of antisymmetric Ti-O-Ti and O-H bonds [15]. In the PANI/GNS (1%) nanocomposite, functionalized graphene displays a peak at 1730 cm^{-1}, and PANI/GNS (2%)/TiO_2 (20%) displays a peak at 1735 cm^{-1} which showing a characteristic band of the stretching mode of C=O carboxylic acid groups [11]. When GNS combines with PANI. The PANI/GNS (2%)/TiO_2 (20%) nanocomposite exhibits a slight shift in PANI frequencies and the presence of the TiO_2 band. The FTIR analysis clarifies the presence of titanium dioxide (TiO_2) and functionalized graphene (GNS) embedded within the polyaniline (PANI) matrix.

3. SEM Analysis



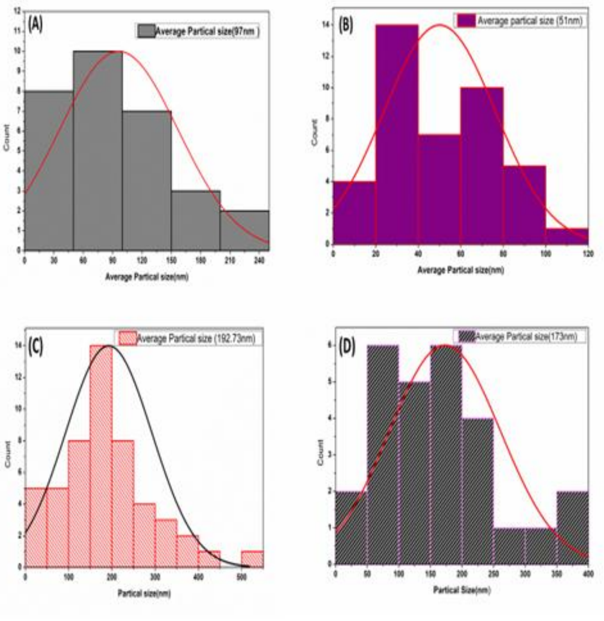


Figure 4: (A) Polyaniline (PANI) **(B)** Titanium Dioxide (TiO_2) **(C)** PANI/GNS (1%) and **(D)** PANI/GNS (2%)/TiO_2 (20%).

In Figure 4 (A) The grain structure of pure PANI is spherical and interconnected networks. However, it is not uniformly distributed throughout, as depicted in the image. The average particle size of this structure was measured to be 97 nm. In figure 4 (B) TiO_2 nanoparticles have a nearly spherical morphology, comprising randomly distributed tiny grains with an average particle size measured to be 51 nm. Figure 4 (C) illustrates the incorporation of polyaniline (PANI) into functionalized graphene (GNS (1%)) which highlights its potential for enhanced absorptivity [19]. PANI/GNS (1%) nanocomposite shows an average particle size of around 192.7nm. In PANI/GNS (2%)/TiO_2(20%) nanocomposite, TiO_2 particles were believed to be embedded within the GNS sophisticated core-shell structure Constructed by PANI chains. Highly Porous nanostructured materials are obtained which is very useful for absorption of LPG gas. The formation of such a structure layer of PANI embedded in Graphene (GNS) and TiO_2 nanoparticles results in nanocomposite with increased surface area. This increased surface area is advantageous for attracting molecules. which shows an average particle size of around 173 nm. Nanocomposites with GNS and TIO_2 nanoparticles distributed uniformly throughout the PANI matrix show no signs of agglomeration.

Gas sensing experimental setup

The table top static gas sensing apparatus used to measure the resistance of LPG It has equipped with two probe systems, a stainless steel 304 closed chamber and a digital temperature controller with a range of (250 °C). The nanocomposites pallet (1.94 mm thickness and 12 mm diameter) is placed on the surface of the alumina substrate and two probes are placed on the surface of the pallets then insert a specified volume (corresponding to 250 ppm, 500 ppm, and 1000 ppm) of LPG gas into the experimental chamber by using a syringe. Extracted the electrical leads from the chamber to enable the monitoring of electrical parameters. The resistance of the pallet is measured with a specific temperature. a multimeter used to measure the resistance variation. The resistance variation of the sensing material at various temperatures measures it to both clean air and LPG gas. Measure the resistance of the sample, which should fall within a range from 2 KΩ to 20 MΩ, using an adjustable scale that depends upon temperature and gas concentration.

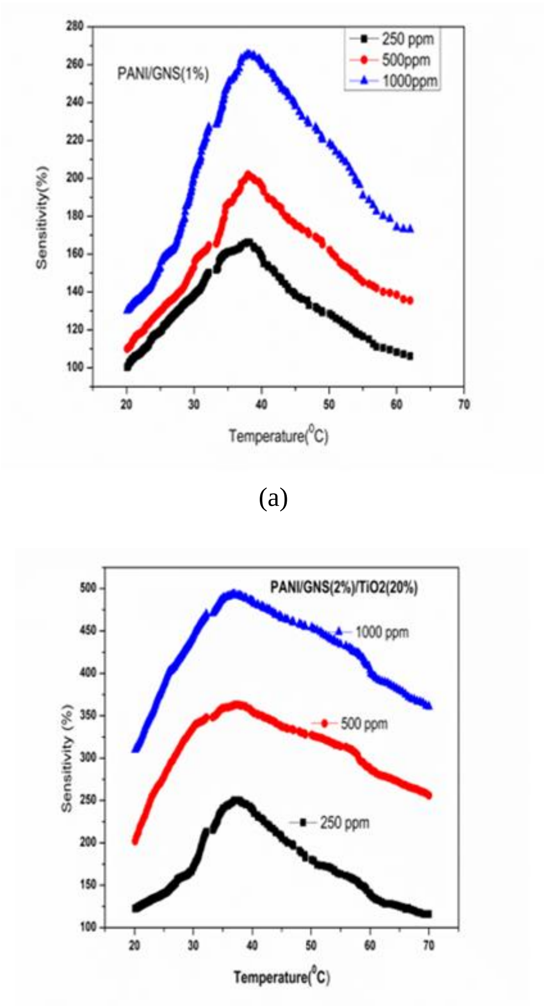
1. Mechanism of LPG sensing

The gas-sensing process of nanocomposite materials is based on changes in barrier height or resistance generated by physisorption and chemisorption on their surface at room temperature or high temperature [12]. Oxygen molecules physically adsorb on the active surface of nanocomposite materials when exposed to air, capturing electrons from the conduction band and trapping them at the surface as various oxygen ions species such O-2(ads), O-(ads) O2-(ads) [16]. The sample later interacts with reducing gases like LPG, which in turn removes the oxidizing ions through a process known as chemisorption. LPG (butane and propane) reacting with superoxide anions which may create by-products such as carbon dioxide (CO_2) and water (H_2O) which react with normal operating temperatures signifies various compositions. The conduction band transfers the released electron after adsorption. The reversing change occurs during their capture. As a result, the resistance of the semiconductor increases in the carrier conduction current which decreases the electron depletion layer of the molecules that make up LPG which tends to transfer an electron to the surface which leads to further increases [23]. At normal temperatures, graphene can change its conductivity in the presence of many chemical compounds, regardless of how differently these compounds behave chemically, making them an extremely sensitive material. Their increased responsiveness to atmospheric conditions is a result of GNS atomic arrangement on their surface and their high area/volume ratio, both of which promote adsorption processes. There are two possible outcomes when the chemical agent interacts with the surface of the GNS, changing their electrical conductivity either the analyte acts as a scattering potential or the GNS facilitates charge transfer from the analyte to the nanomaterials. When the compound that is adsorbed is an electron attractor (hole donation), the conductivity is increased due to charge transfer. Analytes that donate electrons reduce the electrical conductivity of nanomaterials by reducing their hole density [24]. The PANI/GNS blend and PANI/GNS/TiO_2 nanocomposite material interact with pre-adsorbed oxygen species on the active sensing surface upon exposure to LPG at the specified concentration in the gas chamber. The "gas resistance of the sensor (R_{gas})” denotes the interaction between chemisorbed oxygen species on the active surface and injected LPG molecules. LPG molecules, including methane (CH_4), propane (C_3H_8), and butane (C_4H_{10}), engage in electron transfer with adsorbed oxygen species. The adsorption of LPG on the nanocomposite material leads to an increase in resistance, while the removal of the doping anion from the main chain of the PANI also contributes to this resistance increase [25]. Nanostructured sensing materials exhibit a superior surface-to-volume ratio, providing an increased number of sites for the adsorption of analyte molecules, hence enhancing sensitivity relative to thin-film materials. Liquid petroleum gas (LPG) acts as an electron donor, leading to an increase in the resistance of the nanocomposite when exposed to LPG. Graphene's high specific surface area can enhance gas responsiveness at room temperature when combined with metal oxides, leading to improved sensitivity and selectivity [26]. The Sensitivity (%) was calculated by using this formula:

$$\text{Sensitivity}(\%) = \left[\frac{R_{Gas} - R_{Air}}{R_{Air}}\right] \times 100$$

Here, R_{Gas} stands for the material's resistance values while exposed to LPG, and R_{Air} for the sensing material's resistance values when exposed to pure air [27].

2. Sensitivity

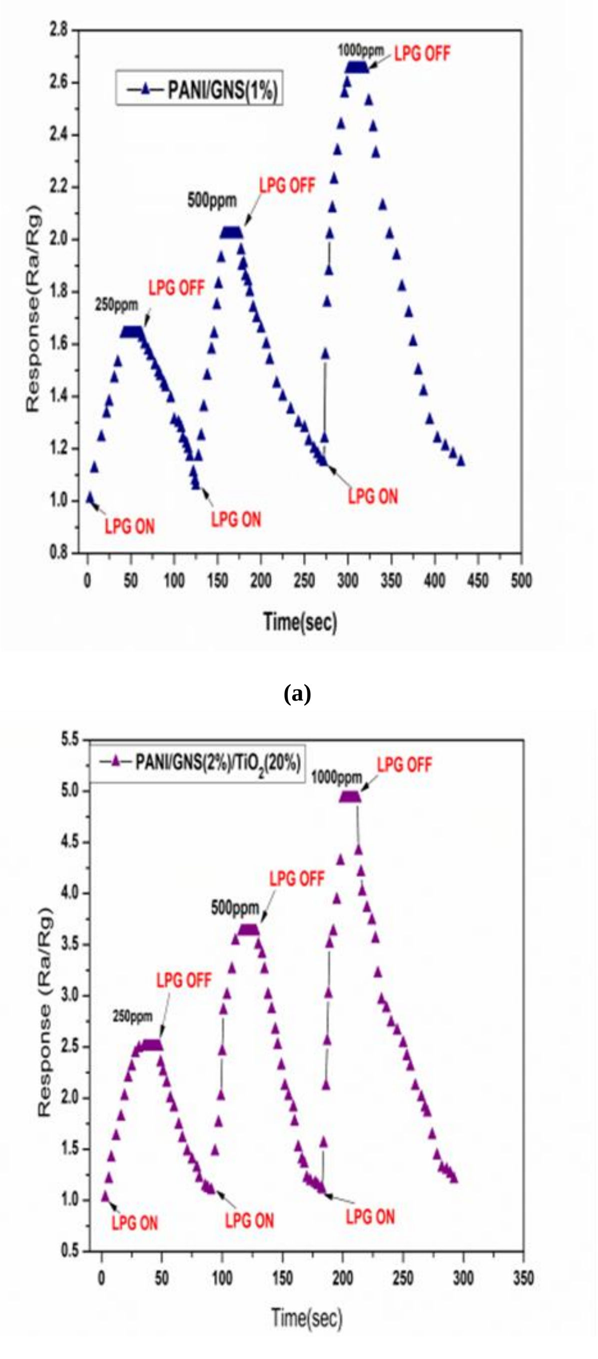


(a)

Figure 5: Sensitivity graph of **(a)** PANI/GNS (1%) **(b)** PANI/GNS (2%)/TiO$_2$(20%).

The data in Figure 5 shows the relationship between percentage sensitivity and temperature for the PANI/GNS (1%) and PANI/GNS (2%)/TiO$_2$ (20%) nanocomposite. The resistance of materials was measured in the kΩ to 10 MΩ range. At higher temperatures, the sensitivity was observed to increase before reaching its peak and subsequently decrease at higher temperatures [28]. In Figure. 5 (A), it can be observed that the sensitivity increases initially up to 39oC and then decreases. The PANI/GNS (1%) nanocomposite exhibits maximum percentage sensitivity of 164%, 202%, and 265% for concentrations of 250 ppm, 500 ppm, and 1000 ppm of LPG respectively. In contrast to the PANI/GNS (1%) nanocomposite, figure 5(B) the PANI/GNS (2%)/TiO$_2$ (20%) nanocomposite exhibits improved sensitivity, initially increasing up to 37°C and then declining. They exhibit maximum percentage sensitivity of 250%, 363%, and 493% for concentrations of 250ppm, 500ppm, and 1000ppm, respectively. In the case of the PANI/GNS (2%)/TiO$_2$(20%) nanocomposite, a similar nature was observed, but the sensitivity factor is higher compared to PANI/GNS (1%) for the range of ppm. and temperature range slightly improved which is around 37°C.

3. Response (R_s) and Recovery Time (R_t)



(a)

(b)

Figure 6: (a) Polyaniline (PANI)/Graphene (GNS) (1%) **(b)** PANI/GNS (2%)/TiO$_2$ (20%)

Response (R_s) and recovery time (R_t) is another crucial factor of gas detection. It is the time required by the material to transform its initial resistance to 90% of its original value [29]. Figure 6 illustrates that the nanocomposite material exhibited a notably high and rapid response to LPG concentrations of 250 ppm, 500 ppm, and 1000 ppm. The samples demonstrated a consistent and attainable change in resistance over numerous cycles. Response and recovery times for PANI/GNS (1%) for 1000 ppm LPG were found to be 28 sec and recovery time 109 sec. In PANI/GNS (2%)/TiO$_2$(20%) for 1000 ppm, the response time are 16 sec and the recovery time is 83 sec. The response and recovery times exhibit an inverse correlation with the LPG concentration. The response and recovery time in ternary nanocomposite material is faster than in binary nanocomposite material. The graph indicates that increased concentrations of LPG resulted in prolonged recovery times. This is likely attributable to the denser characteristics of LPG [30].

4. Sensitivity vs Concentration of LPG Gas

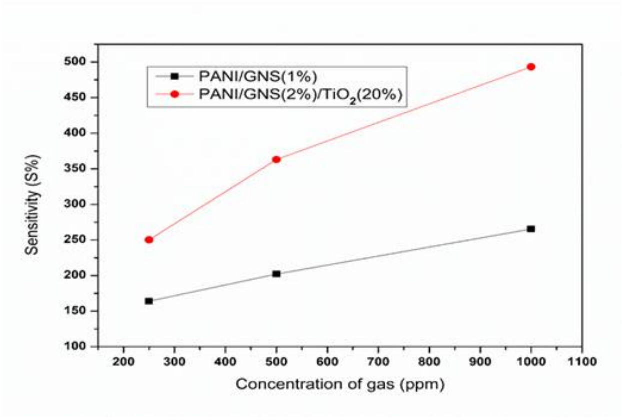


Figure 7: Graph displays the sensitivity versus concentration of LPG gas for the nanocomposites PANI/GNS (1%) and PANI/GNS(2%)/TiO$_2$(20%).

An analysis of the graph reveals that the sensitivity factor of the PANI/GNS (1%) and PANI/GNS(2%)/TiO$_2$(20%) nanocomposite increases as the concentration of LPG rises. However, the sensitivity of PANI/GNS (1%) nanocomposite material is lower compared to PANI/GNS(2%)/TiO$_2$(20%) nanocomposite material. This difference can be attributed to the higher concentration of LPG.

Conclusions

Highly crystalline PANI/GNS (1%) and PANI/GNS(2%)/TiO$_2$(20%) nanocomposites were successfully synthesized by the chemical oxidation polymerization method and TiO$_2$ were synthesized by the sol-gel method. FTIR verifies the presence of functional groups in the nanocomposites. By embedding a layer of PANI on functionalized graphene (GNS) and TiO$_2$ nanoparticles. SEM analysis produces highly porous nanostructured materials that result in a nanocomposite with an increased surface area. In the case of the PANI/GNS(2%)/TiO$_2$(20%) nanocomposite, the sensitivity is higher as compared to PANI/GNS (1%) in terms of concentration of LPG and temperature. To obtain 1000 ppm LPG at room temperature (37°C), composite nanomaterials of PANI/GNS(2%)/TiO$_2$(20%) had a response time of 16

Table 2. Compares the data with some other reported work for detection of the LPG.

S. No.	Materials	Methods of Synthesis	Operating temperature	LPG concentration (ppm)	Response time (sec) &, Recovery time (sec)	References
01	Polyaniline/ZnO	Chemical bath deposition	Room Temperature	1040 ppm	100 and 150	[1]
02	PANI/MWCNT	Electrospinning method	Room Temperature	1250 ppm	13 and 80	[6]
03	PANI/TiO$_2$	Chemical bath deposition and electrodeposition	Room Temperature	1000 ppm	140 and 180	[23]
04	PANI/Cu$_2$ZnSnS$_4$	Electrodeposition method.	Room Temperature	750 ppm	120 and 125	[24]
05	PANI/CNT/V$_2$O$_5$	Chemical oxidation polymerization method	Room Temperature	50 ppm	20 and 15	[25]
06	PANI/ZnO	Electrospinning method	Nearly Room temperature	1000 ppm	110 and 185	[28]
07	Polyaniline/Nb$_2$O$_5$	Chemical oxidation polymerization method	Room Temperature	500 ppm	30 and 50	[31]
08	**Polyaniline/GNS/TiO$_2$**	**Chemical oxidation polymerization method**	**Room Temperature**	**1000 ppm**	**16 and 83**	**Present study**

seconds and a recovery time of 83 seconds. The response and recovery times vary with the concentration of LPG. Compared to binary PANI/GNS (1%) nanocomposite materials, ternary nanocomposite PANI/GNS (2%)/TiO_2 (20%) materials showed better sensitivity and response and recovery time.

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