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Synthesis and characterization of polyaniline/TiO_2 nanocomposite based ammonia gas sensor

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ABSTRACT

Pure TiO_2 nanoparticles were synthesized using the sol-gel method and polyaniline (PANI), while PANI/TiO_2 (content TiO_2:25 % and 50 %) nanocomposites were synthesized by using in-situ chemical oxidative polymerization method. X-RD, FTIR spectroscopy, and scanning electron microscopy (SEM) were used for the structural analysis, elemental composition, and surface morphology. The XRD patterns of PANI/TiO_2(25 %,50 %) composites reveals that the relative intensity of diffracted peaks increases with increasing the content of TiO_2 indicating an increase of crystallinity in such case. FTIR confirmed the presence of functional groups in the nanocomposite. In SEM surface morphology observed that highly porous nanograins are formed. The average particle sizes of PANI/TiO_2(25 %) 339 nm and PANI/TiO_2(50 %) 196 nm were obtained. The resistance change response of nanocomposite material were done by using a static two-probe unit by exposure to ammonia (NH_3) gas. The maximum sensitivity of PANI/TiO_2(50 %) nanocomposite was achieved on 500 ppm (parts per million) of ammonia gas at 40 °C. The fast response time (20 s.), and recovery time (28 s.) has obtained.

1. Introduction

Hazardous gas sensors have acquired increasing importance in both industry and research due to the presence of various quantities of dangerous gases such as CO_2, H_2S, NO_2, CO, and NH_3 in the air around us. Researchers are increasingly focused on detecting these gases in the environment to ensure the safety of humans, animals, and plants. Ammonia is one of the widely used chemicals as a coolant in industries like food industries, chemicals, textiles, fertilizer, paper products, environmental monitoring, and automotive industries [1]. Serious health problems can develop in the respiratory system, skin, and lungs after prolonged exposure to ammonia gas. The National Institute for Occupational Safety and Health (OSHA) has set a maximum ammonia exposure limit of 25 ppm for 8 h and 35 ppm for 15 min [2]. The creation of inorganic decorated polymer hybrid materials at the nanoscale has drawn a lot of interest in recent decades because of their many possible uses in gas sensors, and optoelectronic devices. Because these nanoscale materials have a high surface-to-volume ratio, they significantly alter the microscopic, optical, and electrical characteristics of polymers [3]. In recent years, there has been a lot of interest in Polyaniline (PANI) due to the fabrication of its composites using inorganic particles to increase their processing ability. Well-known conducting polymer polyaniline (PANI) has increased a lot of attention lately due to its excellent optical, electrochemical, electrical and high stability [4]. The most researched class of gas sensors currently are semiconductor metal oxide (SMO) sensors. Because of their size-dependent characteristics, SMOs, which have diameters between 1 and 100 nm, are being used for gas sensing more and more. The electrical, mechanical, magnetic, catalytic, and optical properties of nanomaterials which make them unique [5]. Metal-oxides have been used in several devices, including solar cells, gas sensors for the detection of H_2, NH_3, and NO_2 gases, photosensors, transparent electrodes, antistatic coatings, and flat panel displays [6]. TiO_2 is the most promising member of the group of semiconducting metal oxides, which also include ZnO, SnO_2, WO_3, and In_2O_3. Nanocrystalline TiO_2 is one of the most appealing and extensively utilised inorganic materials for detecting gases such as LPG, NO_2, H_2, and NH_3 [5]. Composite PANI/TiO_2 materials have been created for use as gas sensor electrodes [4]. synthesis of a polyaniline titanium dioxide nanocomposite for CO and NH_3 gasses revealed that the material has a significant response for NH_3 gas in comparison with CO gas [7]. Solid-state devices based on polyaniline are economical and effective in sensors, electronics, and storage devices [8]. Conversely, PANI has been incorporated into several inorganic materials, including Fe_2O_3, ZnO, SnO_2, TiO_2, WO_2, and MoO_3 [9]. When synthesizing hybrid materials with PANI, TiO_2 is the chosen inorganic

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material because its conduction band correlates well with PANI's lowest unoccupied molecular orbital, potentially improving the material's electronic transport capabilities [10]. The redox interaction between the target gas and the sensing material has an enhanced active site in metal elements. With its significant resistance change in the presence of reducing gases like H_2, and H_2S, and oxidizing gases like NH_3, and O_2 [11]. CuO, SnO_2, Cr_2O_3, WO_3 V_2O_5, and TiO_2 are examples of metal oxides that can be used with sensors that mainly depend on resistance change responses to target gases to identify combustible, reducing, or oxidizing gases [12]. The present study shows that development of a PANI/TiO_2(50 %) nanocomposite for the study of sensing response of ammonia (NH_3) gas. The PANI/TiO_2(50 %) nanocomposites were evaluated for NH_3 gas at temperatures ranging from 20° to 70 °C. The PANI, TiO_2, and PANI/TiO_2 composite material was characterized using X-ray diffraction (XRD), Fourier transform infrared (FTIR), and Scanning electron microscopy (SEM).

2. Materials and methods

Materials used for synthesis are titanium isopropoxide (TIPP, 97 %) Methanol (99.8 %) Aniline (99.5 %, AR Grade), Ammonium persulfate ($(NH_4)_2S_2O_8$,98 % AR Grade), hydrochloric acid (HCl) (35.4 %, AR Grade), and Sulphuric acid (96 %) was used for the synthesis of the PANI, TiO_2, and PANI/TiO_2 hybrid materials. Deionized water was used for the hydrolysis of the solution. All the materials are AR grade and used for synthesis.

2.1. Synthesis of Tio_2

Titanium isopropoxide has been utilized as a source of titanium to create TiO_2 through the sol-gel method. A white powder was produced by adding 3.7 ml of titanium isopropoxide to 50 ml of methanol and rapidly stirring the mixture at 50 °C for 1 hour. TiO_2 nanopowder was obtained by annealing the powder for one hour at 600 °C in a tubular furnace [13].

2.2. Synthesis of polyaniline (PANI)

The synthesis of polyaniline was carried out using the chemical oxidation polymerization method. Ammonium Persulphate (APS) acts as an oxidizing agent while hydrochloric acid (HCl) acts as a catalyst in the polymerization process of aniline. For the synthesis, 2 ml of aniline was added to a 50 ml solution of 1 M HCl in distilled water. This mixture was placed on a magnetic stirrer. Next, a second beaker was prepared with 50 ml of distilled water, 4.9984 gm of Ammonium Persulphate (APS), and 1 M HCl. This solution was then added drop by drop into the previous solution. The solution begins to exhibit a greenish colour. To ensure the completion of the reaction, it is necessary to maintain the temperature within the range of 0 to 8 °C using an ice bath for a duration of 5 to 6 h. The resulting solution was then filtered. The product was washed with 1 M HCl, followed by distilled water, until the wash solution became colourless. The solution was then vacuum-dried in an oven at 80 °C for 12 h. Finally, obtained polyaniline powder [13].

2.3. Synthesis of PANI/TiO_2 nanocomposites

The PANI/TiO_2 (25 %, and 50 %) nanocomposites were synthesized by using in-situ chemical oxidation polymerization of aniline in the presence of TiO_2. The weight percentage of TiO_2 to aniline ranged from 25 % to 50 %. Divide the solution of 0.2 M H_2SO_4 in 100 ml of deionized water into two equal parts in a 250 ml beaker. A portion of a 50 ml solution, consisting of 0.2 M aniline and TiO_2(25 % and 50 %) to the ratio of aniline was introduced into the mixture and subjected to ultrasonication for 35 min. Another portion of the 50 ml solution, which contains 0.2 M ammonium persulphate (APS), was mixed and added dropwise while rotating the Aniline and TiO_2 solution. the mixture was subjected to magnetic stirring for approximately 5 to 6 h during the reaction, while maintaining a temperature of 0 to 8 °C using an ice bath. This was done to enhance the yield of the reaction. Upon combining the reactants, the solution initially exhibits a greenish colour, which then transitions to violet colour. The composite solution was allowed to sit overnight and then filtrated using distilled water and absolute ethanol until the filtrate became colourless. Ultimately, the solution underwent a drying process for 12 h in an oven set at a temperature of 80 °C [14].

3. Characterizations

The synthesized material was subjected to structural investigation using an X-ray diffractometer (X-RD) (Rigaku Mini-Flex 600, Rigaku Japan) with a wavelength of 1.5406A°. The study of the functional group is conducted by FTIR which frequency range from 400 cm^{-1} to 4000 cm^{-1}. using (Model: Spectrum 400FT-IR/FIR, Spectrometer. Perkin Elmer, USA). The morphological study of material was carried out using scanning electron microscopy (SEM). (SEM Model - Carl Zeiss EVO-18) and average particle size can be determined using image-j software. The gas sensing performance was evaluated using a static gas-detecting device equipped with a two-probe system and a temperature controller. Resistance change response was studied within a few ohms to 10 MΩ.

3.1. X-ray diffraction analysis

The XRD pattern of the pure PANI, TiO_2, and PANI/TiO_2(25 %, and 50 %) nanocomposite as shown in Fig. 1 An advanced diffractometer was used to acquire XRD patterns of PANI/TiO_2 nanocomposite samples, which confirming their crystalline nature across the 2θ range of 10° to 90° for pure PANI, the characteristic peaks have appeared at 2θ= 15.24°, 20.78°, and 25.26° which correspond to (110), (002) and (112) crystal planes. which shows the semi-crystalline nature of material. The crystallinity of PANI can be characterized by the recurring presence of benzenoid and quinoid rings in the PANI chains [4]. The nanocrystalline form of titanium dioxide (TiO_2) exhibits a distinct and prominent peak at 2θ=25.28° the strong diffraction peaks observed at approximately 2θ values of 25.28°, 37.81°, 47.99°, 53.95°, 55°, 62.9°, and 75° correspond to the crystallographic orientations of (1 0 1), (00 4), (2 00), (1 05), (211), (2 04), and (215). The individual peaks observed in the TiO_2 sample may be accurately matched to the anatase phase (JCPDS-ICDD card: 21–1272) [15]. Which indicates its tetragonal structure. In the XRD spectrum of PANI, the peaks at 2θ = 25.26° and TiO_2 2θ = 25.28° correspond to periodicities that are perpendicular and parallel to the PANI chain direction, respectively [16]. The anatase TiO_2 characteristic peaks are observable in the PANI/TiO_2(25 %, and 50 %) nanocomposite. The X-ray diffraction (XRD) pattern of PANI/TiO_2 composites indicates the existence of titanium dioxide within the polyaniline matrix. The diffracted peaks of these composites exhibit an increased intensity as the TiO_2 content increases. The patterns show sharp and well-defined peaks. which indicate the crystalline nature of the synthesized materials [3].

3.2. Fourier transform infrared (FTIR) analysis

The spectroscopic study of PANI, TiO_2, and PANI/TiO_2(25 % and 50 %) nanocomposites was conducted across a range of 4000 cm^{-1} to 400 cm^{-1}. The FTIR spectroscopy analysis of PANI shown in Fig. 2. (A) reveals several characteristic peaks. the peak at 505cm^{-1} is C–H out of plane bending vibration. The peak at 811 cm^{-1} is attributed to the N–H out-of-plane bending. The peak observed at approximately 1312 cm^{-1} corresponds to the stretching vibrations of the C–N bond. The peak at around 1150 cm^{-1} represents the stretching vibrations of the C–H bond. Additionally, the peaks observed at approximately 1515 cm^{-1} and 1577 cm^{-1} are attributed to the stretching vibrations of the C = N bond in the benzenoid and quinoid rings, respectively. Finally, the peaks observed

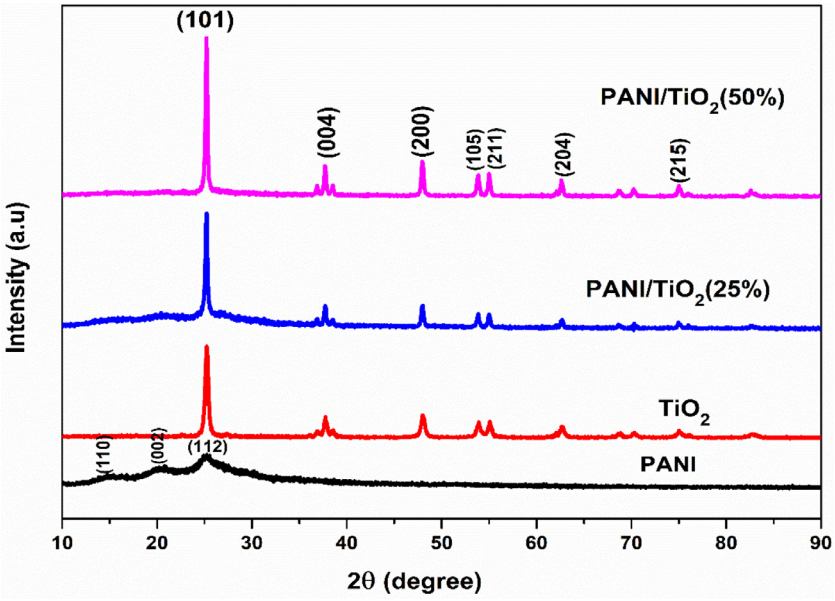

Fig. 1. The X-Ray diffraction pattern of PANI, TiO_2, and PANI/TiO_2(25 %) and (50 %).

in the range of 3605–3728 cm^{-1} are associated with the stretching vibrations of the N–H bond in the aromatic amine. In Fig. 2(B) the TiO_2 band is observed at a frequency of around 415 cm^{-1} under intense vibration. Additionally, the frequency of 1637 cm^{-1} coincides with the stretching frequencies of antisymmetric Ti-O-Ti and O–H bonds. In Fig. 2(C) the characteristic FTIR peaks of PANI were found to shift to a higher wavenumber in the PANI/TiO_2 composites. this result shows that the effect has been attributed to the interaction of TiO_2 particles with PANI molecular chains. The FTIR analysis reveals the existence of titanium dioxide (TiO_2) within the polyaniline (PANI) matrix.

3.3. Scanning electron microscopy (SEM)

SEM images of pure PANI, pure TiO_2, and PANI/TiO_2(25 %,50 %) nanocomposite are shown in Fig. 3. Pure PANI is a spherical grain with well-interconnected particles that create a net-like structure, although it is not homogeneous throughout and has an average particle size of 97nm. The structure of the TiO_2 nanoparticles is nearly spherical, consisting of tiny grains spread randomly with an average particle size of 51 nm. At concentrations of TiO_2 (25 and 50 wt.%), composites it is observed that highly porused nanograins are formed, which help to increase the active sites and adsorb more oxygen on the surface of PANI/TiO_2 and immobilization of gas molecules in pore channels. Exhibit the average particle sizes of PANI/TiO_2(25 %) 339 nm, and PANI/TiO_2(50 %) 196 nm, respectively. When the concentration of TiO_2 in the PANI matrix increases, the particle size of the composite decreases. The scanning electron microscopy (SEM) images showed that the addition of dopants to titanium dioxide (TiO_2) significantly affected the shape of the polyaniline (PANI) matrix [17]. It shows that the TiO_2 nanoparticles have a nucleus effect on the polymerisation, which causes a uniform PANI shell around them [26]. The SEM images indicate that TiO_2 doping has a significant impact on the morphology of PANI particularly as TiO_2 content increases.

4. Experimental setup of gas sensor

The static gas sensing unit consists of 2 probe systems separated by distance (1mm), temperature controller and chamber ss 304 with PID temperature controller. The metallic connections of the composite pallet (thickness 1.93 mm and 11 mm diameter) were placed within a test chamber with a defined volume. A predetermined quantity (equivalent to 100, 250, and 500 parts per million(ppm)) of NH_3 gas was introduced into the experimental chamber. Electrical leads were taken out from the chamber to monitor electrical parameters. and the resistance of the pallet was recorded with temperature. The resistance variation was measured by Keithley 2000 Mustimeter. The change in resistance of the sensing material was monitored at different temperatures during exposure to both clean air and ammonia gas. Obtain the sample's resistance within a range of a few ohms to 10 MΩ, using an adjustable scale that depends on the temperature and gas concentration. The resistance variation was measured within the kilo-ohm range. The Sensitivity (%) was defined as:

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

where R_{gas} and R_{air} are the resistance of the sensing material in gas and clean air, respectively [19].

4.1. Gas sensing mechanism

Gas sensing mechanisms mainly involve three processes: charge transfer, desorption and adsorption. According to the electron depletion region theory, electrons are present on the metal oxide surface. These are trapped from the conduction band by absorbing oxygen molecules from the air and form various ionised forms (O_2^-, O^-, and O^{2-}), which lowers the electrical conductivity of the sensor. Increasing the active sites on the surface material is crucial for absorbing more oxygen and improving the sensing properties. This can be achieved with nanostructures and pores in the sensing material [1]. Several n-p semiconductor connections are predicted to enhance the development of diverse gas molecule adsorption sites on the PANI surface. Oxygen, being an electron acceptor, minimises the electron-withdrawing sites on the PANI chain, increasing oxygen adsorption on the PANI surface [28]. The gas-sensing process is based on the theory that when gas-sensitive materials react with oxidizing or reducing gases, they change their electrical resistance [20]. The increase in the surface area of the polymer due to the presence of the nanoparticles resulted in a better sensitivity. The inclusion of metal oxide nanoparticles preserved the PANI in its reduced state, causing it to transform into a substrate that is readily oxidized and promoting electronic transmission with the analyte [21]. When NH_3 comes in contact with the PANI/TiO_2 composite, the NH_3 molecules take protons from the PANI. This makes an ammonium ion (NH_4+) with better energy at the N^+-H adsorption point. This causes the resistance to rise,

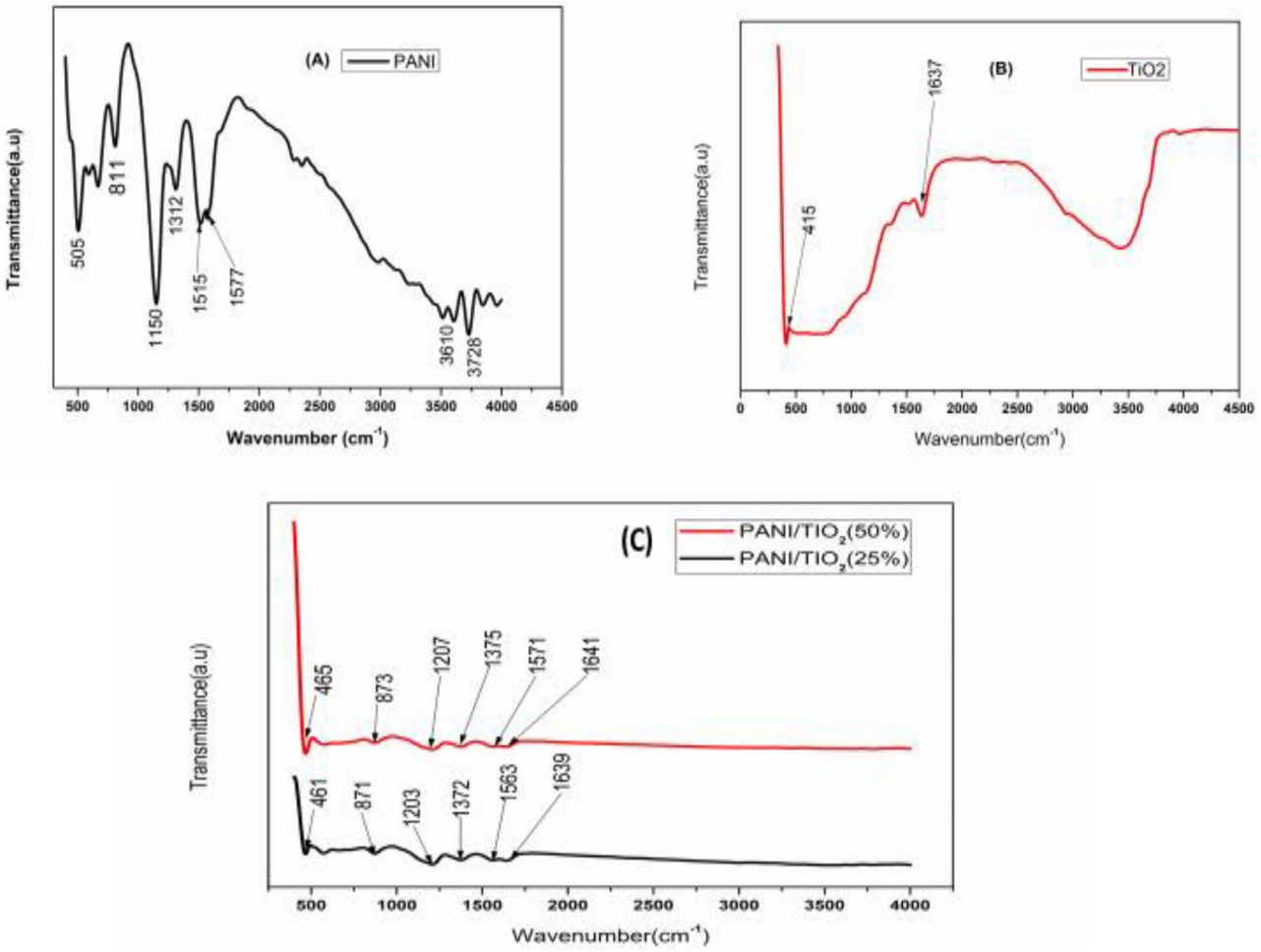

Fig. 2. Fourier Transform Infrared (FTIR) Analysis of A) PANI, B) TiO_2, and (C) PANI/TiO_2(25 %, and 50 %) Nanocomposites.

which is known as dedoping (deprotonation). In normal air, though, the ammonium ion breaks down into ammonia gas and a proton, which is then added to PANI. This makes PANI return to its original amount of doping. This is how the ammonia effect's reversibility works [19].

4.2. Sensitivity of PANI/TiO_2(50 %)

The variation of percentage sensitivity of PANI/TiO_2(50 %) nanocomposite is shown in Fig. 4. The above figure indicates that percentage sensitivity. First, it rises to a maximum value for a temperature of about 40 °C and falls as the temperature increases. A similar variation is observed for 100 ppm, 250 ppm, and 500 ppm of Ammonia gas. ppm stands for parts per million, which expresses the concentration of ammonia (NH_3) gas. The maximum sensitivity of PANI/TiO_2(50 %) for 100 ppm, 250 ppm, and 500 ppm is observed as 117 %, 215 %, and 312 % respectively. The sensitivity increased with temperature, reaching the maximum sensitivity at a specific temperature and then decreasing at higher temperatures. The process happens when the doping of TiO_2 in the polymer matrix increases the band gap and decreases electrical conductivity while enhancing the sensing properties. the physical properties of conducting polymers strongly depend on their doping levels. The doping levels of conducting polymers can be easily changed by chemical reactions with different metal oxide-based materials at room temperature [18]. Most conducting polymers doped/undoped occur through redox processes, allowing electron transfer to modify their doping level. The electron transport processes can cause changes in the sensing material's resistance and work function. When NH_3 and other redox-active gases were put on the PANI/TiO_2 nanocomposite, it resulted in the removal of electrons from the aromatic rings of PANI enhancement of both the doping level and electric conductance of a p-type conducting polymer [27]. An opposite process will occur when detecting an electro-donating gas, The PANI/TiO_2 composite sensor's response at higher temperatures is reduced compared to room temperature, possibly due to exothermic NH_3 adsorption [19]. The NH_3 sensitivity of PANI/TiO_2 is initially high, but it decreases progressively with the saturation of NH_3 adsorbed on the PANI surface [28].

4.3. Response and recovery time

Another important parameter of the gas sensor is Response and Recovery time. After studying the temperature corresponding to maximum sensitivity, all the above nanocomposites are analyzed for variation in response to the time interval for 100 ppm, 250 ppm, and 500 ppm of Ammonia gas (NH_3) inserted into the chamber. The variation of response of the nanocomposite is shown in Fig. 5 The figure shows that the response time and recovery time of PANI/TiO_2(50 %) are about 20 s and 28 s respectively. It is further clear that the composite material shows fast response and recovery time nearly at room temperature.

4.4. Variation of the sensitivity

Percentage Sensitivity of PANI/TiO_2(50 %) Composite under study Fig. 6. indicates that PANI/TiO_2(50 %) nanocomposite has a good impact on the sensing mechanism of ammonia gas due to increasing the concentration of gas (Table 1).

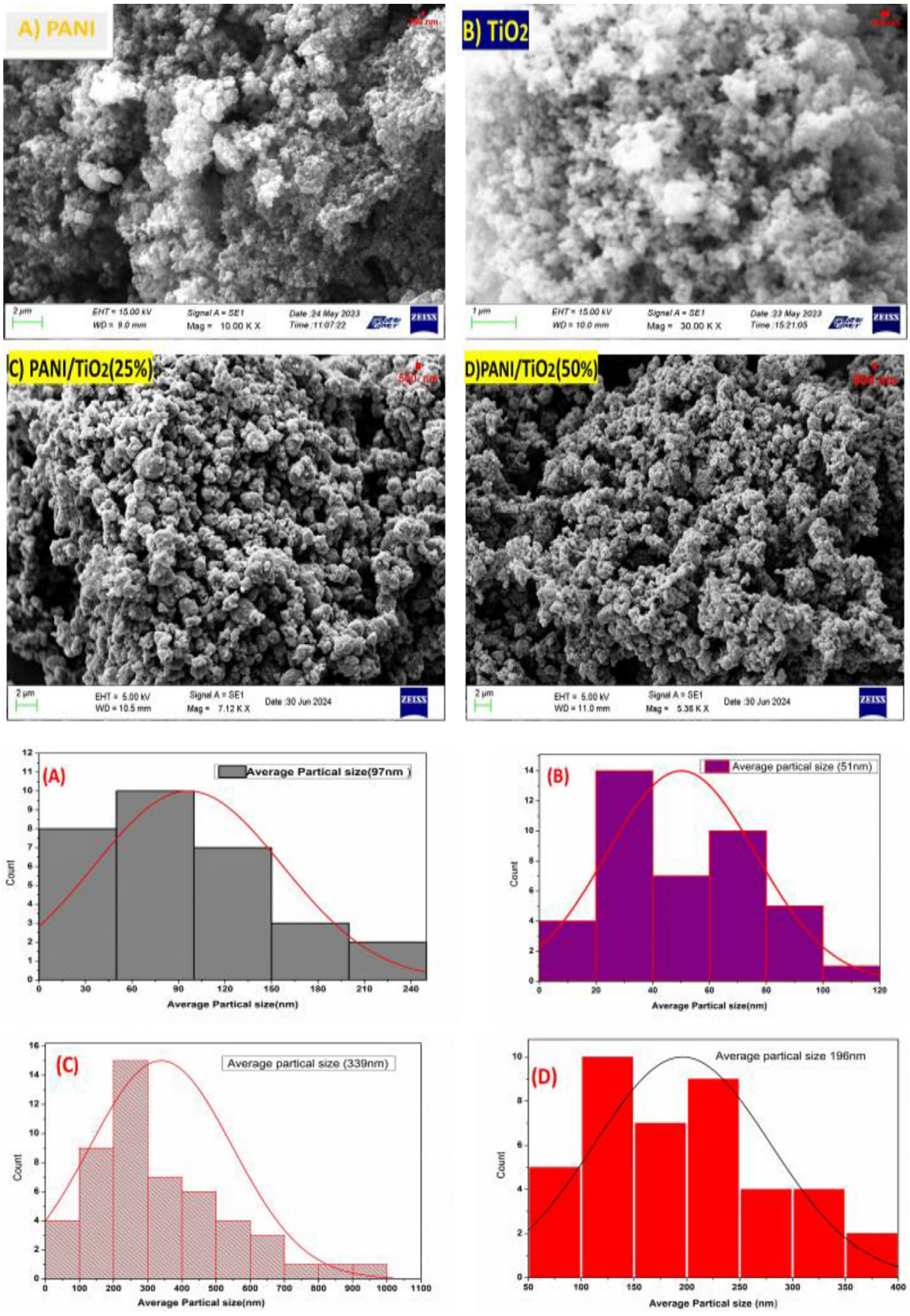

Fig. 3. SEM images of A) PANI, B) TiO_2, C) PANI/TiO_2 (25 %), and D) PANI/TiO_2(50 %) Nanocomposites.

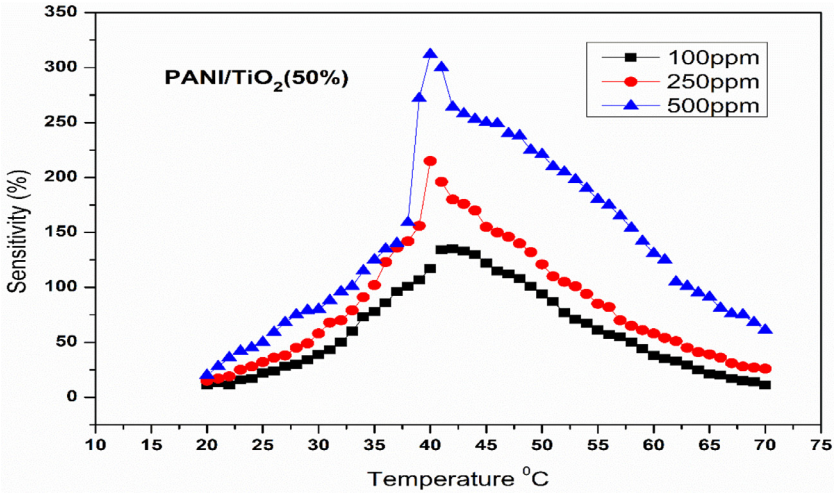

Fig. 4. Sensitivity of PANI/TiO_2(50 %) nanocomposite by exposure to parts per million(ppm) of Ammonia (NH_3) Gas.

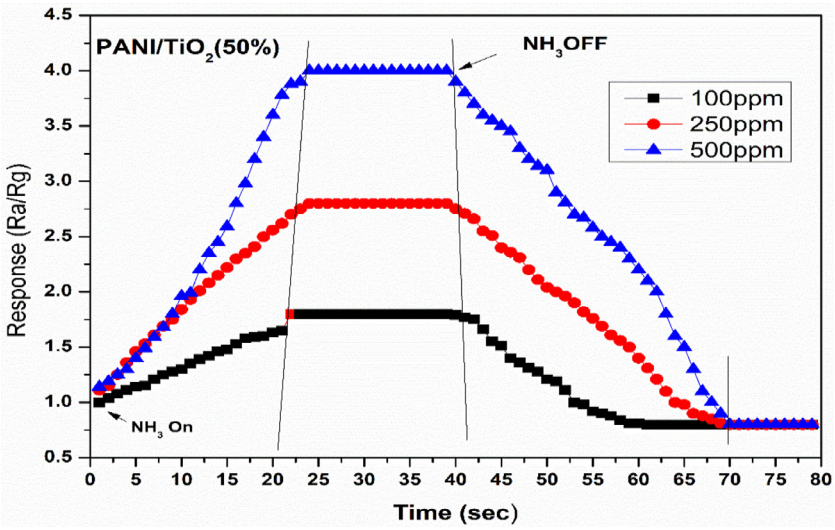

Fig. 5. Response and Recovery time of PANI/TiO_2(50 %) Nanocomposite nearly at room temperature.

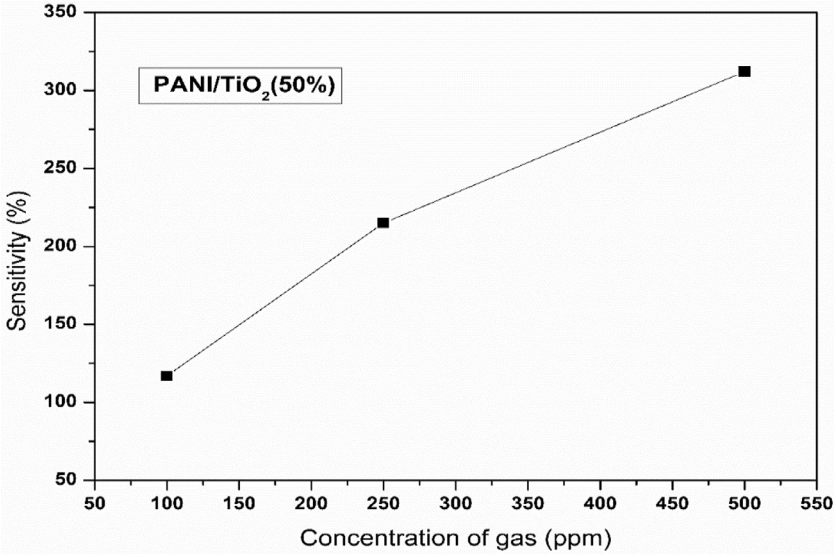

Fig. 6. Percentage variation of the sensitivity of PANI/TiO_2(50 %) due to the concentration of gas(ppm).

Table 1
Comparative study of gas sensing performance of PANI based nanocomposite with other reported work.

Sr. No	Materials and Sensing gas	Operating temperature	Method of Synthesis	Concentration of gas	Response Time, and Recovery Time	Reference
1	PANI/TiO_2 (NH_3)	Room Temperature	Spin Coating method	100 ppm	41 s, and 300 s	[7]
2	PANI/TiO_2 (NH_3)	Room Temperature	Layer-by-layer self-assembly method	5 ppm	110 s, and 1086 s	[22]
3	PANI/GO (NH_3)	Nearly Room Temperature	Nano emulsion method	1000 ppm	5 s and 87 s	[25]
3	PANI/CdS (H_2S)	Room Temperature	Spin Coating method	100 ppm	41 s, and 345 s	[8]
4	PANI/ZnO (LPG)	Room Temperature	Chemical bath deposition	1040 ppm	100 s, and 150 s	[29]
5	Pd-PANI/SnO_2 (H_2)	Nearly Room temperature	Electrospinning method	500 ppm	34 s and 63sec	[11]
6	PANI/TiO_2 (LPG)	Room Temperature	Chemical bath deposition	0.1 V%	140 s and 180 s	[23]
7	Al-SnO_2/PANI (H_2)	Nearly Room temperature	Electrospinning method	1000 ppm	2 s and 2 s	[24]
8	PANI/ZnO (LPG)	Nearly Room temperature	Electrospinning method	1000 ppm	110 s, and 185 s	[18]
9	PANI/TiO_2 (NH_3) sensing	Nearly Room temperature	Chemical oxidation Polymerization method	500 ppm	20 s, and 28 s	Present work

5. Conclusion

Chemical oxidation polymerization processes successfully synthesized a nanocomposite of PANI/TiO_2 with doping percentages of TiO_2 (25 % and 50 %) for ammonia gas detection nearly at room temperature. The XRD pattern of the PANI/TiO_2 composite shows sharp and well-defined peaks. Which indicates the crystalline nature of the synthesized materials. The FTIR confirms the presence of functional groups in the composite. Surface morphology has revealed the formation of highly porous nanograins in composites. The gas sensitivity of PANI/TiO_2(50 %) nanocomposite shows higher sensitivity (312 %) for 500 ppm of ammonia gas at 40 °C. The gas sensing results showed that PANI/TiO_2 (50 %) had very good sensing performance, a fast response time (20 s), and a recovery time (28 s) for detecting ammonia gas at 100 ppm, 250 ppm, and 500 ppm, all of which were operated nearly at room temperature.

Declaration of competing interest

The authors of this work declared that they have no financial or personal interests that could influence their findings or conclusions. Competing

CRediT authorship contribution statement

S.D. Rokade: Investigation, Writing – original draft, Formal analysis, Writing – review & editing. **D.V. Nandanwar:** Conceptualization, Supervision, Methodology, Resources. **S.B. Kondawar:** Conceptualization, Supervision, Methodology, Investigation, Resources. **P.A. Bramhankar:** Visualization, Formal analysis. **P.B. Wasnik:** Formal analysis, Software, Writing – review & editing. **M.S. Bisen:** Data curation, Software, Writing – review & editing.

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