MAGNETIC METAL OXIDE NANOPARTICLE DECORATED REDUCED GRAPHENE OXIDE (rGO) FILM FOR CHEMIRESISTIVE VOLATILE ORGANIC COMPOUND (VOC) SENSING

A dissertation submitted in the partial fulfilment of
the requirement for the degree of
Bachelor of Science
in
Chemistry

Submitted by:
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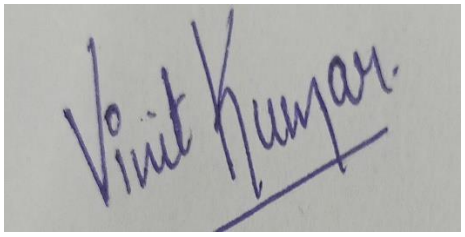
May 2024

DECLARATION

I declare that the thesis entitled "**MAGNETIC METAL OXIDE NANOPARTICLE DECORATED REDUCED GRAPHENE OXIDE (rGO) FILM FOR CHEMIRESISTIVE VOLATILE ORGANIC COMPOUND (VOC) SENSING"** has been prepared by me under the supervision of **Dr. Tridib Kumar Sinha and Dr Sushil Kumar** from **Department of CHEMISTRY, School of Engineering, University of Petroleum & Energy Studies (UPES), Dehradun, India.**



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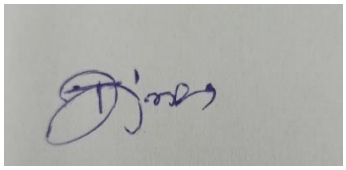
I certify that **VINIT KUMAR** has prepared his project entitled "**MAGNETIC METAL OXIDE NANOPARTICLE DECORATED REDUCED GRAPHENE OXIDE (rGO) FILM FOR CHEMIRESISTIVE VOLATILE ORGANIC COMPOUND (VOC) SENSING"** for the award of **B.Sc. (Hons) CHEMISTRY,** under my guidance. He has worked at the **Department of CHEMISTRY, School of Engineering, University of Petroleum & Energy Studies (UPES), Dehradun, India.**



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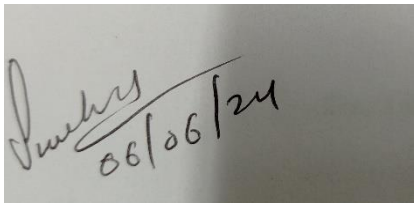

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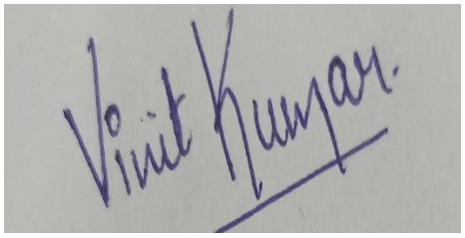

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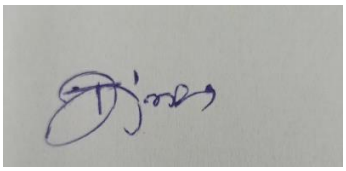

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Abstract

In this research work, attempts have been taken to develop room temperature sensor for volatile organic compound (VOC) detection. As per the market availability, the efficient commercial sensors are mostly made of metal oxides which are in general high temperature operative. In the high temperature environment, the VOCs are supposed to be unstable. On the other hand, the metal oxide-based sensors because of their high temperature requirement are considered as energy intensive. Nowadays, VOCs are being considered as marker for environment, industrial quality control, quality of food, beverage and agriproducts, health quality, etc. Thus, it becomes imperative to develop room temperature (RT) VOC sensors. In this concern, choice of metal oxides because of their abundant environmental stability, are always a better choice. The nanoparticles, because of their enhanced surface area have shown their prominent efficiency especially in sensing technology. Considering the temperature dependent tuneable electrical properties, herein magnetic metal oxide nanoparticles (MNPs) have been introduced for RT VOC sensing. Apart from the electrical tunability, the orbitals having unpaired electrons are expected to interacting with the VOC molecules mainly having non-bonded or π-bonded electron rich functional groups. In this present investigation, the RT VOC sensor was made of the composites of MNPs (e.g., Fe_3O_4) with in situ synthesized rGO to enable the sensor to be chemiresistive and RT operative. It is noteworthy to mention that the optimum capacity of the in-house fabricated sensing system (to check the sensor's performance) is limited to maximum of 1 mega Ohm (MΩ). The resistance of only Fe3O4 in its pellet form has been observed to be ≥ 1.5 MΩ. For both the elevated (e.g., 50 °C) and lower (e.g., 10 °C) temperatures, the resistance has been observed to be higher than that of the RT. However, formulating composite with only 2.5 wt % of rGO, the composite pellet i.e., rGO/Fe_3O_4, exhibit a resistance of few kΩ at RT which is considered ideal for VOC sensing. The composite has been observed to exhibit good RT sensitivity (with fast sensing and recovery) particularly towards the protic polar VOCs. The minor drift during the 1st response cycle may be considered as future scope that is supposed to resolve with applying minor temperature.

Key aspects covered:

- The interaction between VOCs and MNPs.
- Strategies for formulating the rGO/MNPs composites to ensure RT sensitivity, enhanced selectivity towards specific VOCs, and fast recovery.

Challenges and future directions:

- Optimizing sensitivity and selectivity of MNP-based VOC sensors.
- Developing robust regeneration methods for reusability.
- Miniaturization and integration for portable sensing devices.

This research area holds promise for developing VOC sensors with improved sensitivity, selectivity, and ease of use in various applications, including environmental monitoring, air quality control, and medical diagnostics.

ACKNOWLEDGEMENT

I would like to extend my sincere appreciation to my supervisor, Dr. Tridib Kumar Sinha, and co-supervisor, Dr. Sushil Kumar, for their unwavering guidance, support, and challenging assignments throughout this dissertation. Their expertise and patience have been instrumental in the successful completion of this thesis.

I am grateful to all the faculty members of the Department of Chemistry for their assistance and support. I am thankful to UPES for providing me with the opportunity to explore new horizons and for the resources and backing they have offered. I would like to express my thanks to the laboratory staff for their consistent support and provision of necessary resources.

I also extend my gratitude to the panel members for their valuable insights and feedback. Lastly, I am deeply thankful to Ph.D. scholar Shreshth Dobriyal, my friends, and my family for their unwavering love and support throughout this journey.

TABLE OF CONTENTS

Chapter 1

INTRODUCTION

1.1. Why is VOC sensing required?
1.2. Traditional Processes of VOC Sensing.
1.3. Metal oxides very stable?
1.4. The presence of unpaired electrons in metal oxide nanoparticles plays a significant role in enhancing VOC sensing.

Chapter 2

SYNTHESIS OF MAGNETIC METAL OXIDE NANOPARTICLES

2 Synthesis of magnetic metal oxide nanoparticles.
2.1 Technique of synthesis
2.2 Synthesis of Fe_3O_4 magnetic nanoparticles
2.3 XRD data of Fe_3O_4 nanoparticle
2.4 FTIR data of Fe_3O_4
2.5 Synthesis of magnetic Ni-doped Fe_3O_4 nanoparticles
2.6 XRD data of Ni-doped Fe_3O_4
2.7 FTIR data of Ni-doped Fe_3O_4
2.8 Synthesis of magnetic Co dopped Fe_3O_4 nanoparticles
2.9 XRD data of Co dopped Fe_3O_4

CHAPTER 3

FORMULATION OF rGO/Fe_3O_4 NANOCOMPOSITE AND DEVICE FABRICATION THROUGH PELLETIZATION AND ELECTRODING

3.1 Formulation of rGO/Fe_3O_4 nanocomposite
3.2 Palletisation of the composite and electroding

CHAPTER 4

FORMULATION OF rGO/Ni-DOPPED Fe_3O_4 NANOCOMPOSITE AND PELLETIZATION

4.1 Doping reduced graphene oxide on $NiFe_2O_4$ nano powder.
4.2 Palletisation of r-GO dopped Fe_3O_4 nanoparticles.

CHAPTER 5

RESULT AND CONCLUSION

5.1 Sensing of VOCs (Methanol, Ethanol, Acetone, and Ammonia.) Using pure Fe_3O_4 pellet.
5.2 Results and Discussion of VOC Sensing.

ABBREVIATION

GO: - ***GRAPHENE OXIDE***

r-GO: - ***REDUCED GRAPHENE OXIDE***

MO: - ***METAL OXIDE***

MMO: - ***MAGNETIC METAL OXIDE***

VOC*: -* *VOLATILE ORGANIC COMPOUND*

LIST OF FIGURES

Fig 1: ***Fe_3O_4 nano powder***
Fig 2: ***Fe_3O_4 nano powder attracts toward the magnet.***
Fig 3: ***XRD data of Fe_3O_4 nanoparticle***
Fig 4: ***FTIR data of Fe_3O_4***
Fig 5: ***$NiFe_2O_4$ nano powder***
Fig 6: ***$NiFe_2O_4$ nano powder attracted towards the magnet.***
Fig 7: ***XRD data of $NiFe_2O_4$***
Fig 8: ***FTIR data of $NiFe_2O_4$***
Fig 9: ***XRD data of cobalt-doped nanoparticles***
Fig 10: ***Powdered Fe_3O_4***
Fig 11: ***r-GO Doped Fe_3O_4***
Fig 12: ***r-GO doped Fe_3O_4 Pellet***
Fig 13: ***$NiFe_2O_4$ powder***
Fig 14:*r-GO doped $NiFe_2O_4$*
Fig 15: ***Palletisation: r-GO doped Fe_3O_4 nanoparticle.***
Fig 16: ***VOC Sensing Machine***
Fig 17: ***VOC Sensing of Methanol***
Fig 18: ***VOC Sensing of Ethanol***
Fig 19: ***VOC sensing Result of Acetone***
Fig 20: ***VOC Sensing of ammonia***
Fig 21: ***VOC Sensing of DCM***

CHAPTER 1

INTRODUCTION

Volatile organic compounds (VOCs) are gaseous pollutants that vaporize at room temperature and are emitted by various sources, including industrial processes, construction materials, and cosmetic products. Exposure to VOCs can result in serious health issues, such as respiratory problems, inflammation, and the development of certain cancers. Therefore, it is imperative to develop effective VOC detection systems for environmental monitoring and air quality control.

Metal oxide nanoparticles have shown promise for VOC sensing due to their large surface area, high sensitivity, and ease of mass production. However, conventional metal oxide sensors typically require high operating temperatures (over 150°C) to achieve optimal performance. This high-temperature operation results in increased power consumption and limits their use in portable and low-power devices. This study focuses on the synthesis, characterization, and VOC sensing properties of magnetic metal oxide nanoparticles. We anticipate that this research could lead to the development of next-generation VOC sensing systems with enhanced sensitivity and reduced power requirements for use in portable and low-power devices.

1.1 Why is VOC sensing required?

Volatile organic compounds (VOCs) are chemicals with a high tendency to vaporize and a poor ability to dissolve in water, resulting in rapid evaporation into the atmosphere. They originate from a diverse array of natural and artificial sources. The need for VOC sensing derives from several crucial factors. Numerous VOCs present potential health concerns to people, even when found in small amounts. Brief exposure may cause headaches, dizziness, and respiratory and eye irritation, while prolonged exposure might result in chronic respiratory ailments, liver and kidney harm, and cancer. Some VOCs that are known to be harmful include benzene, formaldehyde, and toluene. Workers in industrial and manufacturing environments may encounter elevated amounts of VOCs. Efficient VOCs monitoring is essential to guarantee workplace safety and adherence to occupational health laws. VOCs create ground-level ozone and smog, both of which are major contributors to air pollution. Monitoring VOC levels is crucial for effectively regulating and enhancing urban air quality. Certain VOCs function as greenhouse gases, contributing to global warming. Identifying and managing VOC discharges can aid in reducing the impact of climate change. In industries such as chemical manufacturing, food processing, and pharmaceuticals, detecting VOCs is crucial for maintaining control over the manufacturing process and ensuring the quality of the final products. Identifying VOC leaks or spills can help prevent the contamination of products and maintain a constant level of product quality. VOC sensors are employed in refineries, gas plants, and storage facilities for the purpose of safety monitoring. These sensors can identify leakage and effectively mitigate the risk of potential explosions and fires. Indoor air quality is affected by VOCs emitted by many household goods, including paints, cleaning agents, building materials, and furnishings. Indoor air quality monitoring helps safeguard households from detrimental exposure and informs the creation of safer consumer goods. It is essential to ensure that consumer items adhere to safety requirements regarding VOC emissions to comply with regulations and protect consumers. Governments and environmental agencies rigorously enforce stringent rules on VOC emissions to protect public health and the environment. Precise detection of VOCs is

essential for industries to adhere to these rules and prevent financial penalties and legal consequences. The ongoing monitoring of VOC emissions allows enterprises to create efficient strategies for controlling emissions and lowering their environmental impact. VOC sensors play a vital role in environmental science and health research by examining the origins, distribution, and impacts of VOCs in different settings. Innovation is propelled by the creation of novel materials and technologies that improve the ability to detect VOCs, therefore driving sensor technology advancements and more efficient monitoring systems. VOC sensing is necessary in various domains, including health and safety, environmental conservation, industrial usage, consumer protection, regulatory adherence, and scientific investigation. Dependable and precise VOC sensors are crucial for identifying and reducing the dangers linked to VOC exposure, guaranteeing a safer and healthier environment for humans and ecosystems.

Health concerns

Acute Effects: Short-term exposure to VOCs can lead to various significant health issues. Inhaling VOCs from air fresheners, cleaning agents, and cosmetic products can cause symptoms such as skin irritation, eye irritation, burning of the throat, headaches, nausea, and difficulty in breathing. People with asthma are particularly sensitive to VOCs and may experience additional discomfort.

Chronic Effects: Persistent exposure to volatile organic compounds (VOCs) poses significant health risks. Chronic VOC exposure has been linked to an increased risk of cancer, especially leukaemia and lymphoma. Additionally, VOCs can cause damage to the liver, kidneys, and central nervous system. Children and pregnant women are particularly vulnerable to the long-term health effects of VOCs.

1.2Why Magnetic Metal Oxide nanoparticles for VOC sensing

While traditional metal oxide nanoparticles are widely used for VOC sensing, magnetic metal oxide nanoparticles offered some potential advantages.

Enhanced Sensitivity: The magnetic properties of these nanoparticles can interact with the VOCs leading to larger change in electrical conductivity compared to nonmagnetic metal oxides. This leads to a more sensitivity sensors that detect VOCs at lower concentrations.

Improved Selectivity: The magnetic properties of various magnetic metal oxides can be tuned to interact more strongly with specific VOCs. This can help increase the sensor`s selectivity, permit it to better distinguish between different forms of VOCs.

External Manipulation: An external magnetic field may be utilized to modulate the sensor response; this could provide control over sensing.

However, there are certain challenges to employing magnetic metal oxide nanoparticles for VOC sensing.

Complexity: These materials are frequently more difficult to synthesis and characterize than ordinary metal oxide nanoparticles.

Magnetic Interference: External magnetic fields can interfere with sensors responses, necessitating careful sensors design and shielding.

Limited Research: Magnetic metal oxide nanoparticles for VOC sensing are a relatively new area of study, with numerous unknowns about their long-term effectiveness and durability.

Overall, magnetic metal oxide nanoparticles show potential for building next-generation VOC sensors with higher sensitivity and selectivity. However, more study is needed to solve the issues of synthesis, characterization, and practical implementation in real-world sensing applications.

1.3 Metal oxides are very stable.

Metal oxide nanoparticles are more stable than their bulk counterparts for several reasons:

Strong ionic bonding: Metal oxides contain strong ionic bonds between metal cations and oxygen anions. These bonds are highly stable and require a significant amount of energy to break. At the nanoscale, the large surface area to volume ratio of nanoparticles further enhances this stability. The abundance of tightly bound ions on the surface hinders reactivity compared to bulk materials.

Chemisorption and Passivation: When exposed to air or other environments, metal oxide nanoparticles often form a thin layer of oxide or hydroxide on their surface through chemisorption. This passivation layer acts as a protective barrier, isolating the inner core of the nanoparticle from the environment.

Tunable properties: During synthesis, the specific composition and structure of metal oxide nanoparticles can be adjusted. This capability allows researchers to create nanoparticles with increased stability for desired uses. Additional methods to increase the stability of metal oxide nanoparticles include doping with other elements or using particular crystal shapes.

1.4 The presence of unpaired electrons in metal oxide nanoparticles indeed plays a significant role in enhancing VOC sensing.

Electron acceptors sites: Unpaired electrons in the d-block of transition metals oxide can act as electron acceptors. When a VOC molecule comes in contact with the nanoparticles' surface, these acceptors. When a VOC molecule comes in contact with the nanoparticles' surface, these unpaired electrons can interact with a cloud of VOC molecules. This interaction can alter the conductivity of the metal oxide, which is the foundation of several VOC detecting methods.

Selective Interactions: The unpaired electrons' different energy levels and orbital nature can impact VOC molecules they make contact with more successfully. This can increase the sensor`s selectivity, allowing it to distinguish between various VOCs; for example, some unpaired electrons may preferentially interact with electrons-rich VOCs, whilst others may prefer electron-deficient VOCs.

CHAPTER 2

Experimental Details

2 Synthesis of Magnetic Metal Oxide Nanoparticles.

2.1 Technique of synthesis: -

Nanoparticles can be synthesized by physical, chemical, and biological means. The technique used to synthesize magnetic nanoparticles is mentioned below in detail.

2.1.1 The co-precipitation method involves mixing aqueous solutions of soluble salts containing the desired metal ions. When a precipitation agent is added, insoluble metal hydroxides or oxides are formed, producing nanoparticles. Controlling reaction parameters such as temperature, pH, and stirring rate affects the characteristics of the synthesized nanoparticles. This method offers simplicity, scalability, and diversity. It is widely used in various industries, including biomedical sciences, magnetic material science, and nanotechnology research, due to its ability to create nanoparticles with adjustable characteristics.

2.2 Synthesis of Fe_3O_4 magnetic nanoparticles.

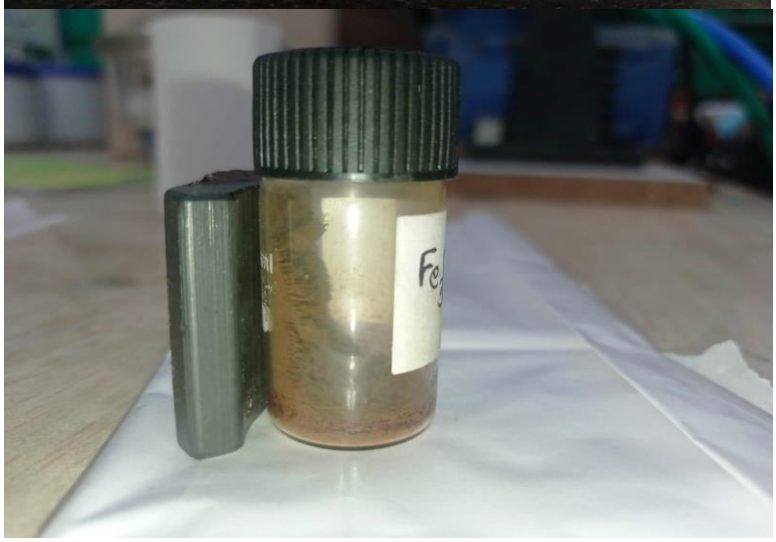
2.2.1 $FeSO_4$ and $FeCl_3$ were dissolved in 50ml beaker of water and ethanol respectively.

2.2.2 Mix both the solution and stir. The final solution Ph was maintained with the addition of NH_4OH (PH 11)

2.2.3 The sample was washed to remove unreacted precursors.

2.2.4 The washing step was carried out with pure water continuously until the solution pH reached 7.0 ± 0.2.
2.2.5 Finally, the sample (Fe_3O_4 Nanoparticle) was washed by absolute ethanol and dried in an oven for 5 hours at 75C. and calcinated at 700^0C.

Fe_3O_4 nano powder



Fe_3O_4 nano powder attract towards magnet

2.3 XRD data OF Fe_3O_4 nanoparticle

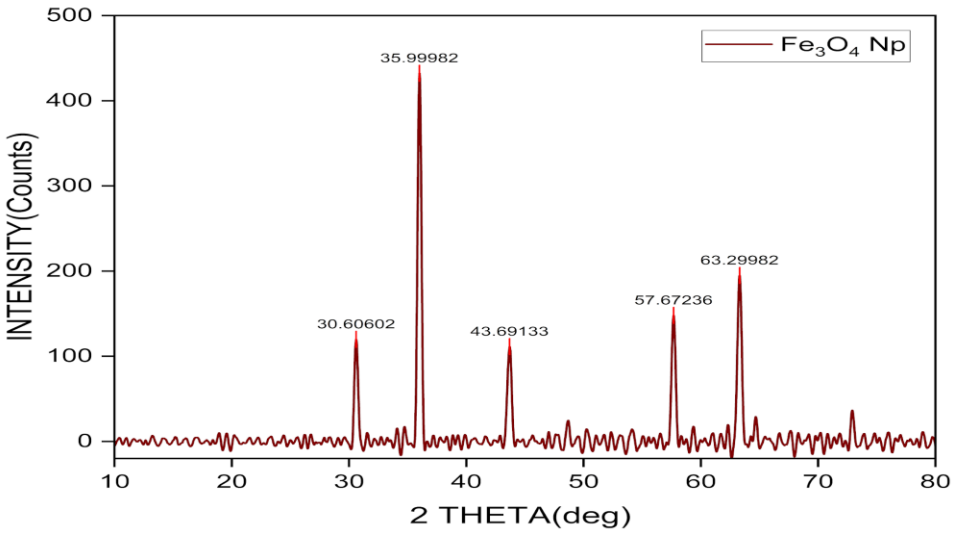


Fig.3: XRD data OF Fe_3O_4 nanoparticle

XRD, or X-ray Diffraction, is a powerful technique for studying the crystal structure of materials. Here is a breakdown of the underlying principle:

Crystal as a diffraction grating:

- Crystalline materials have a highly ordered arrangement of atoms in a repeating unit cell. This arrangement acts like a three-dimensional diffraction grating for X-rays.
- X-rays have wavelengths on the same order of magnitude as the spacing between atoms in crystals.

Constructive interference:

- A monochromatic (single wavelength) X-ray beam is directed towards the crystal.
- The X-rays interact with the electron cloud surrounding each atom in the crystal lattice.
- These interactions cause the X-rays to scatter in various directions.

Bragg's Law and diffraction pattern:

- The scattered X-rays may interfere with each other constructively or destructively.
- Constructive interference occurs only when the scattered X-rays are in phase and satisfy Bragg's Law.
    - Bragg's Law relates the wavelength of the X-rays (λ), the distance between atomic planes in the crystal (d), and the angle of incidence (θ) of the X-rays to the scattered beam.
- The diffracted X-rays are detected, and their intensity is plotted versus the scattering angle (2θ). This plot is called the X-ray diffraction pattern.

Extracting information from the pattern:

- The positions and intensities of the peaks in the diffraction pattern provide information about the material's crystal structure.
- By analysing the pattern, scientists can determine:
    - Crystallographic unit cell parameters (size and shape of the unit cell)
    - Arrangement of atoms within the unit cell
    - Presence of different crystal phases in a polycrystalline sample

XRD is a non-destructive technique:

- Unlike some other methods, XRD does not require damaging the sample. This makes it valuable for analysing precious or sensitive materials.

Overall, XRD is a versatile tool for researchers and scientists to understand the atomic-level organization of crystalline materials.

2.4 FTIR data of Fe_3O_4

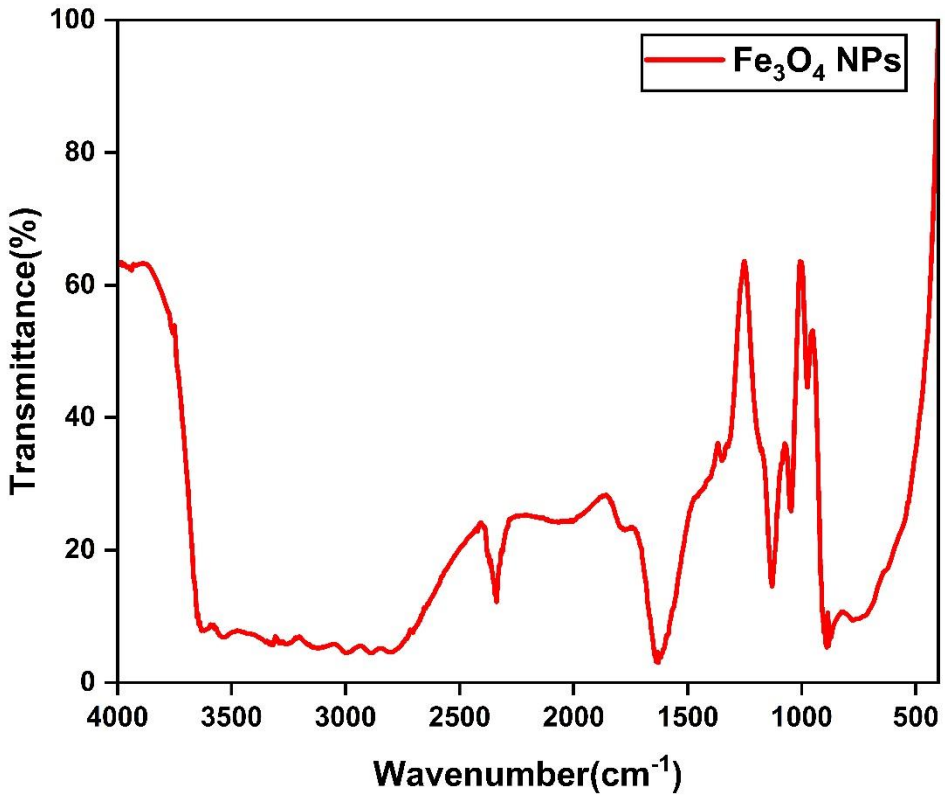


Fig.4: FTIR data of Fe_3O_4

FTIR stands for Fourier Transform Infrared Spectroscopy. It is an analytical technique used to identify organic and inorganic compounds. The basic principle of FTIR relies on molecules absorbing infrared (IR) light at specific frequencies. These absorption frequencies correspond to the vibrational modes of the bonds within the molecule.

Here's a breakdown of the working principle of FTIR:

1. **Infrared radiation source:** A broad beam of IR light is emitted from a source.
2. **Michelson interferometer:** The IR light beam is directed into a Michelson interferometer. This instrument splits the beam into two paths. One path is fixed, while the other path has a moving mirror. The difference in the path lengths of the two beams creates an interference pattern, called an interferogram.
3. **Sample:** The interferogram then passes through the sample. The chemical bonds within the sample absorb specific frequencies of IR light. This absorption weakens the intensity of these frequencies in the interferogram.

4. **Detector:** The modified interferogram is detected by a detector, which converts the light into an electrical signal.
5. **Fourier transform:** The computer then performs a mathematical operation called a Fourier transform on the interferogram. This process converts the interferogram from the time domain (intensity vs. time) to the frequency domain (intensity vs. frequency). The resulting spectrum is a plot of the intensity of the IR light versus the frequency.
6. **Analysis:** The final FTIR spectrum is a fingerprint of the sample. By comparing the spectrum to a library of reference spectra, scientists can identify the functional groups and chemical bonds present in the sample.

The advantage of FTIR over traditional IR spectroscopy is that it collects all the IR frequencies simultaneously. This allows for faster and more accurate analysis. FTIR is a widely used technique in many fields, including chemistry, biology, materials science, and forensics.

2.5 Synthesis of magnetic $NiFe_2O_4$ nanoparticles

2.5.1 The synthesis of Fe-doped Nickel-oxide nanoparticles, Nickel nitrate, and Iron nitrate were used as precursors.

2.5.2 A 0.5M Nickel nitrate and 0.05M Iron nitrate 1M Aqueous sodium hydroxide solution was added as precipitating agents.

2.5.3 The mixture was stirred for 2 hrs at room temperature. The resulting precipitate was washed to remove the Na ions and dried in air at 80ºC.

2.5.4 This dried sample was grinded and calcinated at 400ºC and 700ºC for 2 hours.

$NiFe_2O_4$ nano powder

$NiFe_2O_4$ nano powder
Attracted towards magnet

2.6 XRD data of $NiFe_2O_4$

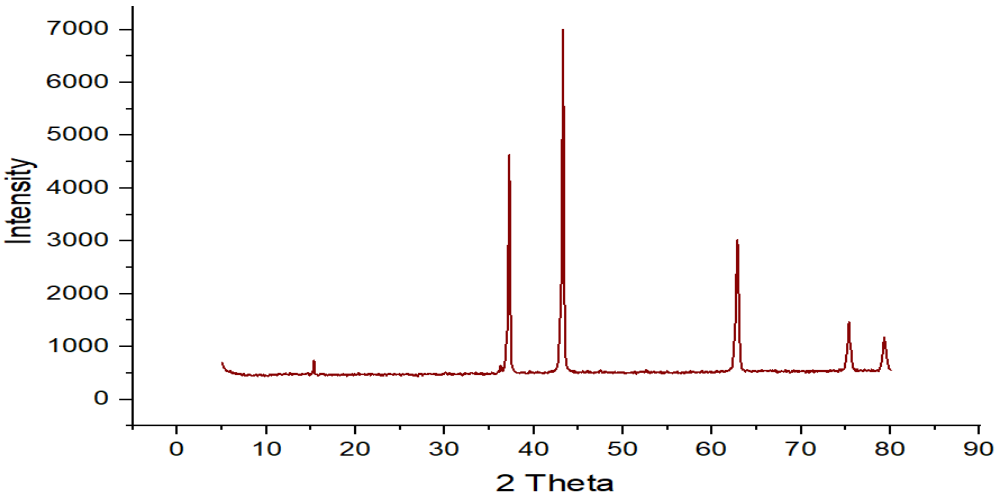


Fig.7: XRD data of $NiFe_2O_4$

2.7 FTIR DATA OF $NiFe_2O_4$

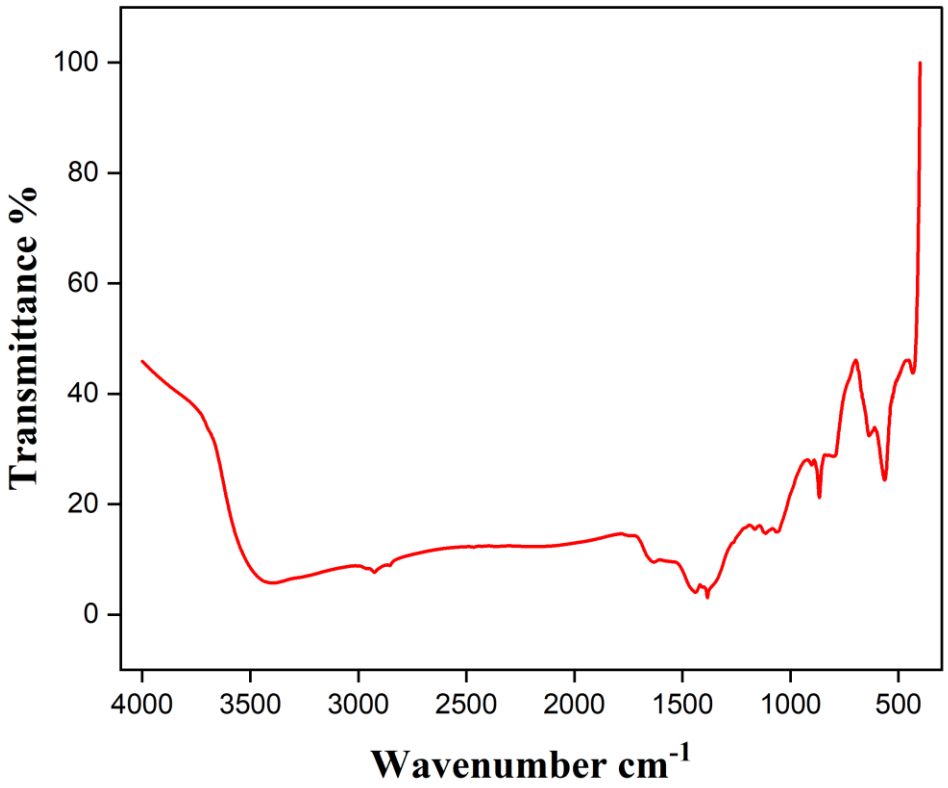


Fig.8: FTIR DATA OF $NiFe_2O_4$

2.8 Synthesis of magnetic Co doped Fe_3O_4 nanoparticles

2.8.1 Dissolve 9 grams of $FeCl_3.6H_2O$ in 80 ml of distilled water and stir at room temperature.

2.8.2 Then, add a small amount of $NaBH_4$. After 2 hours of stirring, 9 grams of $CoCl_3.6H_2O$ were added to the solution, and the synthesis temperature was increased to 90^0C.

2.8.3 The colour of the solution was changed after adding the cobalt precursor, and pH = 5.5 was maintained during the synthesis

2.8.4 The product was evaporated and dried at 200^0C for 4 hours, cooled to room temperature, and finally calcinated at 500^0C for 4 hours.

2.9 XRD data of Cobalt doped nanoparticles

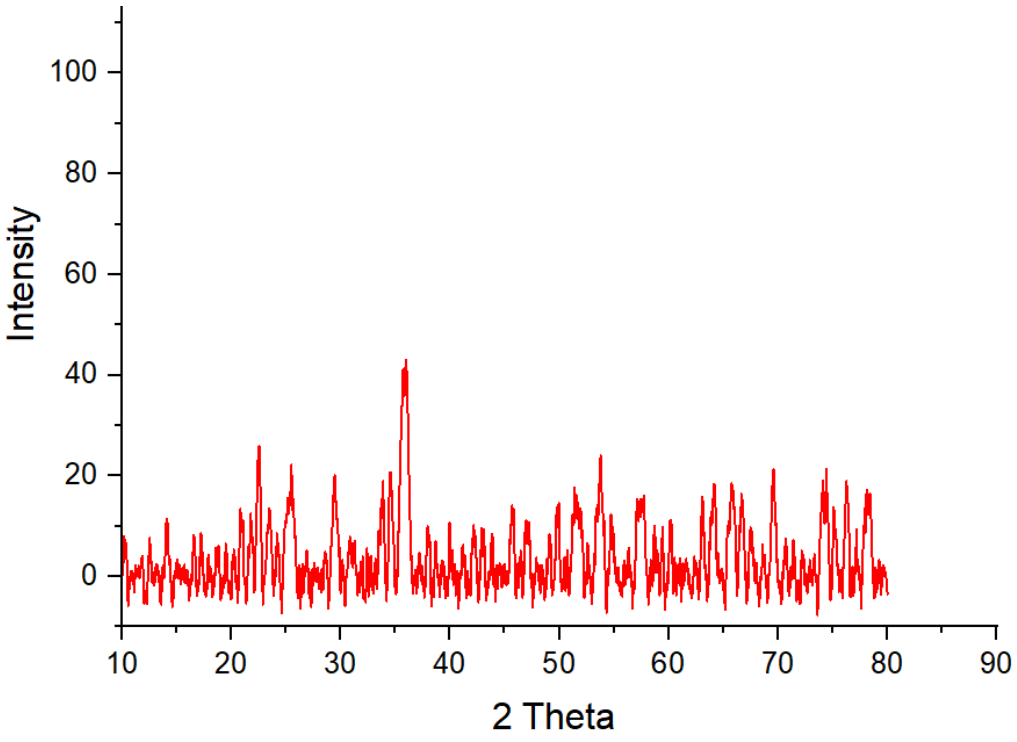


Fig.9: XRD data of Cobalt doped nanoparticles

CHAPTER 3

Formulation of Reduced Graphene Oxide on Fe3O4 Nano powder and Palletisation

3.1 The reduced graphene oxide formulation process on Fe_3O_4 nanoparticles occurs at 60-70^{0}C.

3.1.1 Take 500 milligrams of nanoparticle powder.

3.1.2 Add 1ml graphene oxide solution (containing 13 mg of graphene) and heat it for a few minutes at 60^{0}C.

3.1.3 Observe the colour change.

3.1.4 Then add 500 microliters of hydrazine hydrate and mix well. Heat the compound and observe the colour change (Brown to Black) until completely dried.

Fig 10: Powder Fe_3O_4

Fig 11: r-GO doped Fe_3O_4.

3.2 Palletisation of r-GO doped Fe_3O_4 nanoparticles.

3.2.1 Take about 500 milligrams of r-GO doped powder and grind that powder in mortar and pestle.

3.2.2 Use FTIR dye to make pellet and apply 8-10 tons of pressure for better results.

3.2.3 carefully remove the pellet and store it in a vacuum desiccator.

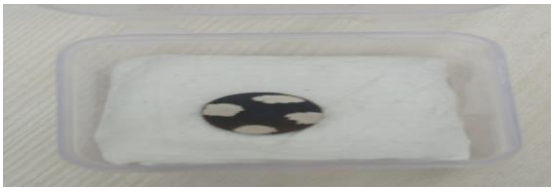


Fig12: r-Go doped Fe_3O_4 Pellet

CHAPTER 4

FORMULATION OF REDUCED GRAPHENE OXIDE ON $NiFe_2O_4$ NANO POWDER AND PELLETISATION

4.1 The process of coating reduced graphene oxide on NiFe2O4 nanoparticles takes place at 60-70^{0}C.

4.1.1 Take 500 milligrams of nanoparticle powder.

4.1.2 Add 1 ml graphene oxide solution (containing 13 mg of graphene) and heat it for a few minutes at 60^{0}C.

4.1.3 Observe the colour change.

4.1.4 Then add 500 microlitres of hydrazine hydrate and mix well. Heat the compound and observe the colour change (Brown to Black) until it is completely dried.

Fig.13 :$NiFe_2O_4$ powder

Fig 14 : r-GO dopped $NiFe_2O_4$

4.2 Palletisation of r-GO doped Fe_3O_4 nanoparticles.

4.2.1 Take about 500 milligrams of rGO doped powder and grind that powder in mortar and pestle.

4.2.2 Use FTIR dye to make pellets and apply 8-10 tons of pressure for better results.
4.2.3 Take out the pellet carefully and store it in a vacuum desiccator.

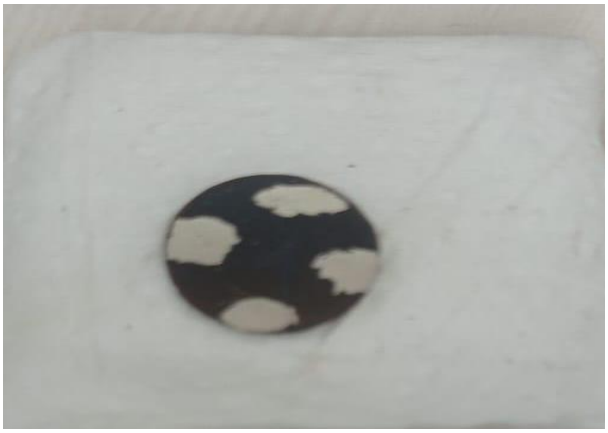


Fig15: Palletisation r-GO dopped Fe_3O_4 nano particles

CHAPTER 5

RESULT AND DISCUSSION

5.1 Sensing of Volatile organic compound using pure Fe_3O_4 pallet.

Fig.16: VOC Sensing Machine

At low temperatures, it shows high resistance (above 1 MΩ; ~2 MΩ), and our measuring instrument limitation is ≤ 1 MΩ.

At high temperature again, it shows high resistance (above 1 MΩ; ~ 2 MΩ) it acts as an insulator.

5.2 RESULT OF VOC SENSING FOR METHANOL, ETHANOL, ACETONE, AMMONIA, DCM.

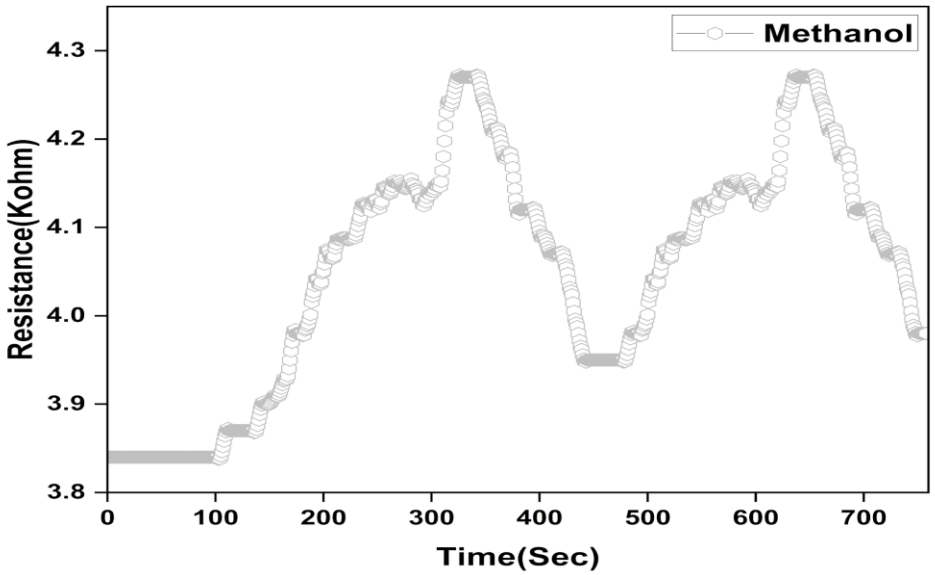


Fig.17: VOC Sensing of Methanol

The graph shows the **resistance of a sensor to methanol over time**. The resistance increases as the sensor is exposed to more methanol, and the resistance decreases as the sensor is exposed to less methanol.
Maybe the sensor material interacts with methanol molecules. It is possible that the methanol molecules block some of the pathways for electrons to flow through the sensor material, thereby increasing resistance.
Here are some of the things to note about the graph:

- The x-axis is labelled "Time (Sec)" and goes from 0 to 700 seconds.
- The y-axis is labelled "Resistance (Kohm)" and goes from 3.8 kΩ to 4.3 kΩ.
- There is a slight increase in resistance over time when the sensor is exposed to methanol.

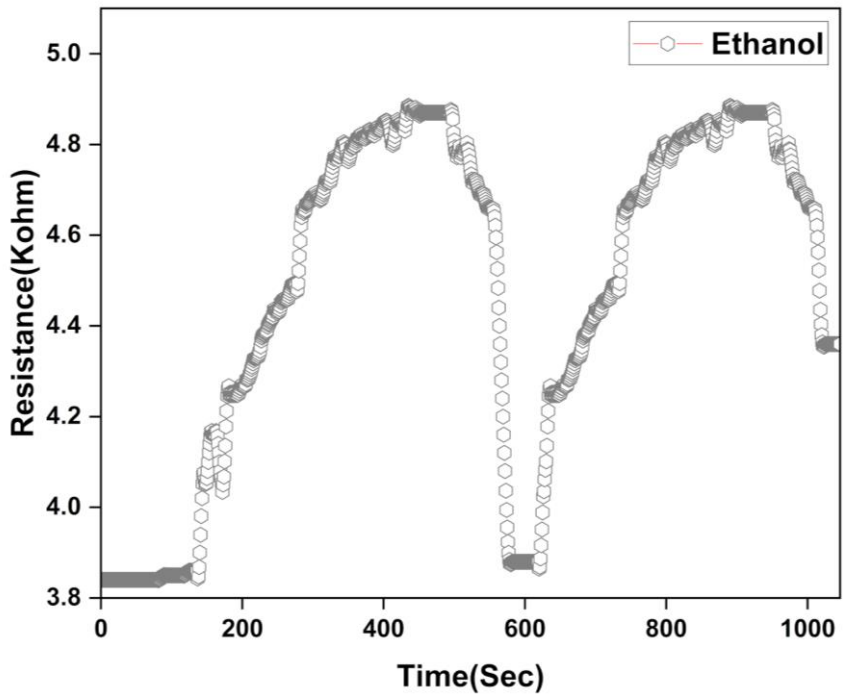


Fig.18: VOC Sensing of Ethanol

1. The x-axis is labelled "Time (Sec)" from 0 to 1000 seconds.
2. The y-axis is labelled "Resistance (Kohm)" from 3.85 kΩ to 5.0 kΩ.
3. There is a clear increase in resistance over time when the sensor is exposed to ethanol.

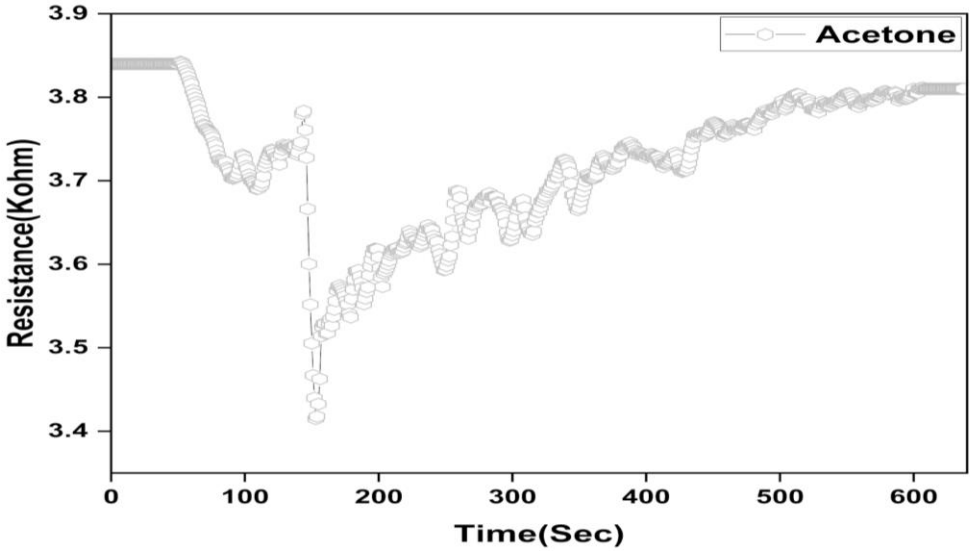


Fig.19: VOC sensing result of Acetone

The graph you sent shows a sensor's resistance to acetone over time. The x-axis is labelled "Time (Sec)" and goes from 0 to 600 seconds. The y-axis is labelled "Resistance (Kohm)" and goes from 3.85 kΩ to 3.4 kΩ. When the sensor is exposed to acetone, the resistance decreases over time.

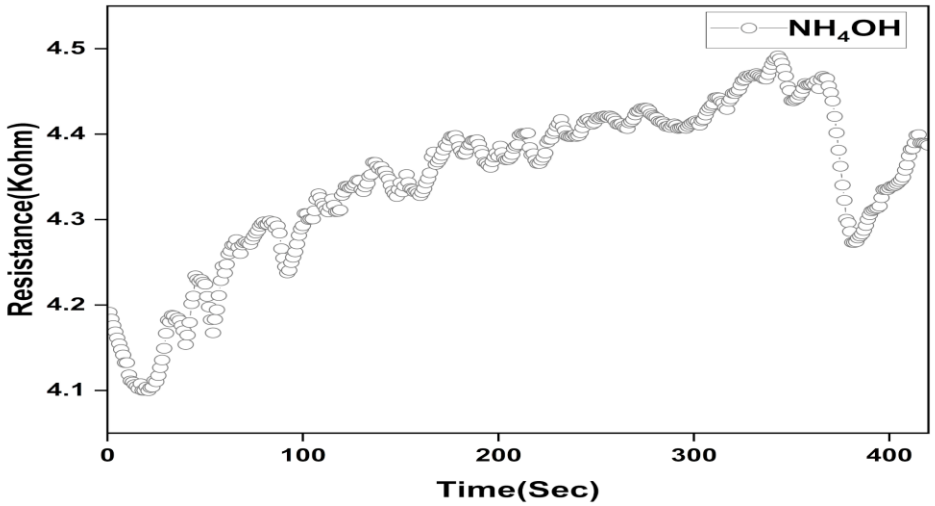


Fig.20: VOC sensing of Ammonia

- The graph you sent shows the resistance of a VOC sensor to ammonia over time. The x-axis is labelled "Time (Sec)" from 0 to 400 seconds. The y-axis is labelled "Resistance (Kohm)" and goes from 4.1 kΩ to 4.5 kΩ. There is a slight increase in resistance over time when the sensor is exposed to ammonia.
- The change in resistance is relatively small. This may mean that the sensor is not very sensitive to ammonia or that the concentration of ammonia that the sensor was exposed to was relatively low.

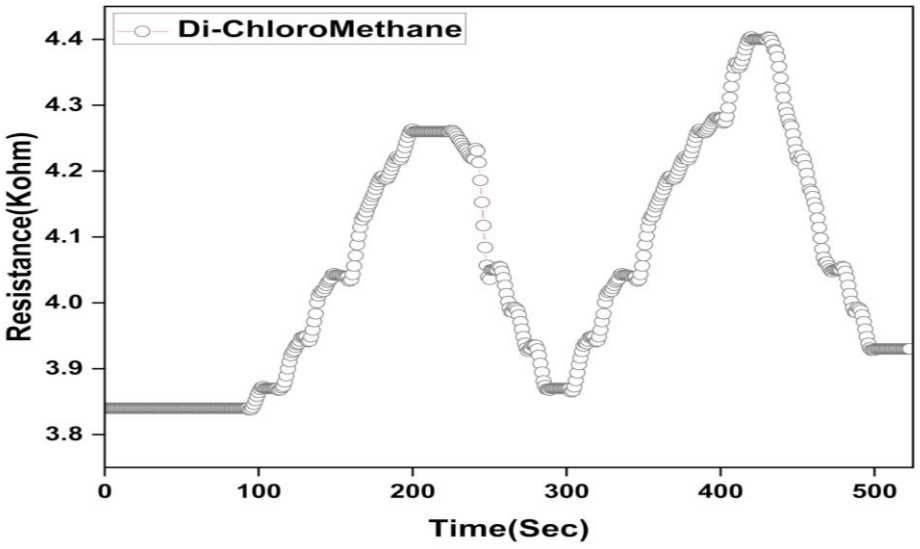


Fig.21: VOC sensing of DCM

The graph you sent shows the dichloromethane (DCM) vapor concentration measured by a VOC sensor over time. The x-axis is labelled "Time (Sec)" from 0 to 500 seconds. The y-axis is labelled "Resistance (Kohm)" and goes from 3.8 kΩ to 4.4 kΩ. There is a slight increase in resistance over time when the sensor is exposed to DCM vapor.
It is possible that the DCM molecules block some of the pathways for electrons to flow through the sensor material, thereby increasing resistance.
The change in resistance is relatively small. This may mean that the sensor is not very sensitive to DCM or that the concentration of DCM vapor that the sensor was exposed to was relatively low.