

Investigation on Graphene functionalized with metal oxides and their sensing applications

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Nowadays environmental pollution is becoming a serious problem in the whole world and its monitoring and prevention are very challenging. Since the mid-20th century, the amplified emission of CO₂ and other greenhouse gases has been understood to be the cause of the increase in the average near-surface air and ocean temperature of the earth. The burning of fossil fuels from industries, motor vehicles, and volcanic eruption are the resources of major environmental pollutants like carbon monoxide and carbon dioxide. The continuous increase in the concentration of CO₂ in the natural environment has raised the temperature of the earth causing global warming which is further liable for the melting of glaciers and rising sea levels. Also, the excessive presence of CO₂ in the ambient environment causes suffocation and creates unconsciousness in human beings. Therefore, precise measurement and control of CO and CO₂ are necessary [1-6].

Humidity monitoring is important for weather forecasts because it is a climate variable. Hyperthermia, fatigue, skin irritation, and some respiratory disorders may be tracked through humidity concentration in breath at high humidity condition when surrounding is warmer than the body temperature. Also, monitoring of moisture levels in agriculture and during food processing is necessary. Moisture content in a fuel decides the uncertainty in its energy content hence moisture sensing of the fuel is important. Also, humidity monitoring is important for human comfort and achieving hygienic conditions [7-11].

Liquefied petroleum gas (LPG) is an inflammable gas consists of a mixture of hydrocarbon gases including both the propane C₃H₈ and butane C₄H₁₀. Ethanethiol which is a powerful odorant is added to LPG to trace its leakage. There are some other international standards like EN589, amyl mercaptane, and tetrahydrothiophene that may also be used as a substitute for Ethanethiol. LPG is used as a fuel in heating appliances and vehicles and it is a substitute for petrol and diesel. There is a large use of the LPG in the kitchen, its leakage may cause a huge burning blast, so its detection is very much important.

To overcome these above-mentioned problems, there are many sensors have been developed to detect the levels of LPG and various toxic gasses present in the environment. From

the literature survey, it has been found that currently available sensors have two major shortcomings; the first one is low sensitivity and the other one is the high operating temperature [12]. A highly sensitive gas sensor mostly works at a very high operating temperature which increases the power consumption. Room-temperature operated sensors have low sensitivity. Therefore, we have to compromise with either sensitivity or operating temperature [13]. Thin film-based sensors operated at room temperature are one of them and such type of gas sensors have been established as an emerging technology in the field of environmental sensing.

In the present work, we have focused on Graphene-based thin film gas sensors because it has extraordinary physical, chemical, structural, and mechanical properties. These are an ideal quantum system for exploring the two-dimensional physical application in nanoscale devices [14-17].

The development of portable CO₂ and LPG sensors that are robust, small-sized, have long lifetimes, are quick in response, and have sufficient sensitivity in the ambient environment is necessary and demanded to prevent explosion accidents in homes and industries for safety requirements. Solid-state CO₂ and LPG sensors using carbon nanomaterials are the most promising for the detection of such toxic gases because of their compact structure, high selectivity, low cost, and the ability of continuous monitoring. The need for reliable, cheap, and user-friendly gas sensors for the detection of CO₂ and LPG is industrially important and has led to a considerable expansion in the field of sensor research and development [18, 19]. For this reason, efforts are made nowadays by scientific research communities in leading laboratories all over the world to focus on the investigation of novel LPG, CO₂ sensitive materials suited for solid-state gas sensors. Consequently, their performances have to be improved dramatically by adopting preparation conditions and by controlling deposition processing.

Carbon nanomaterials may enhance the performance of gas and humidity sensor because of their much higher surface-to-volume ratio as compared to micro grained materials [20, 21, 22].

Room temperature gas sensing may be achieved by modifying several different material parameters with modifications available when dealing with materials at the nanoscale. First, achieving a high non-equilibrium amount of oxygen vacancies in the oxide sensor material will help in achieving this goal. A high non-equilibrium amount of oxygen vacancies would serve to

contribute to the more effective movement of charge across the sensor material. The term non-equilibrium is used here because thermodynamics at room temperature does not predict the number of vacancies required for this at low operating temperatures. Second, increasing the number of surface sites for gas interaction will help in achieving this goal; so that even a low concentration of the gas can be detected at room temperature.

The use of nanomaterials is significant for this due to their high surface to volume ratio. The third change that can be made to the sensor material is a modification of the space charge layer. This is done in order to get the maximum signal change in the presence of the target gas. Modification of the space charge layer can be done by: (i) reducing the crystallite size, (ii) changing the defect chemistry within the space charge layer and surface of the material, and (iii) changes in the particle shape may play a significant factor.

Another interesting aspect of nanomaterials that makes them a candidate for room temperature gas sensor applications is the fact that the changes in the bandgap have been reported at extremely small sizes. The possibility to modify this bandgap may be another way to improve room temperature gas sensing by changing the potential barrier energy required for charged species to conduct. Another enhanced feature of nanocrystallites is their conduction of electrons from the surface reaction. These conduction electrons have to overcome a potential barrier induced by the space charge layer.

Carbon is an exceptional and extraordinary element ever discovered. It can form different types of bonds with C atoms having varied bond lengths and strengths. This enables its presence in a variety of allotropes corresponding to the various geometries of C atoms.

Requirements of a Humidity, LPG, and CO₂ Sensor

Liquefied petroleum gas is one of the most hazardous gases due to its inflammable and explosive nature which presents much harm to humans as well as the environment. Also, it is one of the extensively used gases. Therefore the leakage of LPG is a serious problem. So nowadays the LPG sensor has become a very interesting topic because of the fundamental research as well as industrial applications.

CO₂ is one of the major pollutants which causes so many problems and so hazardous for us. Hence, it is crucial to detect it in its early stages of leakage and to perform active suppression.

Humidity refers to the water in gaseous form whereas moisture refers to the water in liquid form. Therefore, humidity obeys gas laws. Humid air being less dense is one of the fundamental biotic factors that decide the habitat of a particular plant or animal. Humidity measurement is required for climate monitoring, human health, food preservations, packaging, weather forecasts etc.

For designing a robust gas/humidity sensor, the sensor material should possess the following qualities given as under:

- (i) The material should have high sensitivity over a wide range of humidity and temperature.
- (ii) It should quickly respond to any fast changes in the ambient.
- (iii) The sensor material should have a rapid response to the variation of gas concentration and good reproducibility of the electrical signal.
- (iv) The sensitivity should be independent of the ambient temperature.
- (v) The material should not react with any chemical contaminants present in the application ambient.
- (vi) It should show stable characteristics for a long time.
- (vii) The construction of the sensor should preferably be simple using IC technology and of low cost.
- (viii) The device should be operated at low voltage.

The present thesis is divided into seven chapters. **Chapter 1** contains the introductory part of the synthesis of Graphene, its functionalization, and application in various fields with the inclusion of electrochemical sensors such as humidity and gas sensors. A detailed investigation including synthesis, characterization, and application as an impedance-based moisture sensor at optimized frequency is reported in **Chapter 2**. In **Chapter 3**, the synthesis of the Graphene oxide (GO) and its functionalization with iron oxide nanoparticles is presented, and also this chapter deals with the development of a capacitance-based humidity sensor at room temperature. **Chapter 4** describes the design and fabrication of LPG and humidity sensor based on the Graphene functionalized with CuO/SnO₂ nanocomposites. **Chapter 5** reports the synthesis of Graphene oxide functionalized with poly palladium acryl amide SnO₂ nanocomposite and its application as LPG and humidity sensor at room temperature and also the effect of the humidity on the LPG sensor. **Chapter 6** presents the effect of CO₂ gas on the resistance of Graphene@CuO nanocomposite based thin film at room temperature. The variation in the

impedance of the thin film with adsorbing and desorbing humidity is also investigated in this Chapter. A comparative study of the synthesis, characterization with humidity and gas sensing properties of modified Graphene, and Graphene functionalized with different nano-metal oxides are summarized in Chapter 7. This chapter also gives the guidelines for further research work in the field of gas and humidity sensing.

Chapter 1:

This chapter consists of the basic introduction of different types of carbon nanostructures and their roles in a different type of sensing application and also provides detailed information for the synthesis of Graphene and its functionalization. However, obtaining high sensitivity, good reproducibility, negligible ageing effect with fast response, and recovery time at room temperature is a major concern. Carbon nanomaterials symbolize an increasing asset in numerous productions, particularly due to their sharp chemical, physical, and electronic properties, and also have versatile applications like environmental monitoring, health, and personal care, energy, water treatment, etc. A detailed description of the sensors, their attributes, and types along with their application in various fields has been also reported in the present Chapter.

Chapter 2:

This chapter deals with the fabrication of the modified Graphene (m-Graphene) through the thermal reduction method and also it focuses on impedance-based humidity sensors at optimized frequency. The thin films of m-Graphene were investigated with SEM and UV-visible spectroscopy for surface morphological and optical absorption characterization. In the SEM micrographs, planar sheet-type structures oriented in different directions were observed. A dilute solution of the sample was used for the particle size analysis. The average thickness, size in the plane and the diagonal of the Graphene sheet were found as 180 nm, 627 nm and 5560 nm respectively. The BET, FTIR and Raman spectroscopy were carried out with the help of the powdered sample. The graphical abstract of this chapter is shown in Fig.1. The analysis of Raman confirmed that the material has many defects that may be responsible for the humidity

sensing. Variations in impedance of the sensing element with increasing %RH were recorded at 100 Hz, 1 kHz and 10 kHz respectively. The data were plotted and shown in Fig.2. The sensor exhibited better sensitivity at 100 Hz.

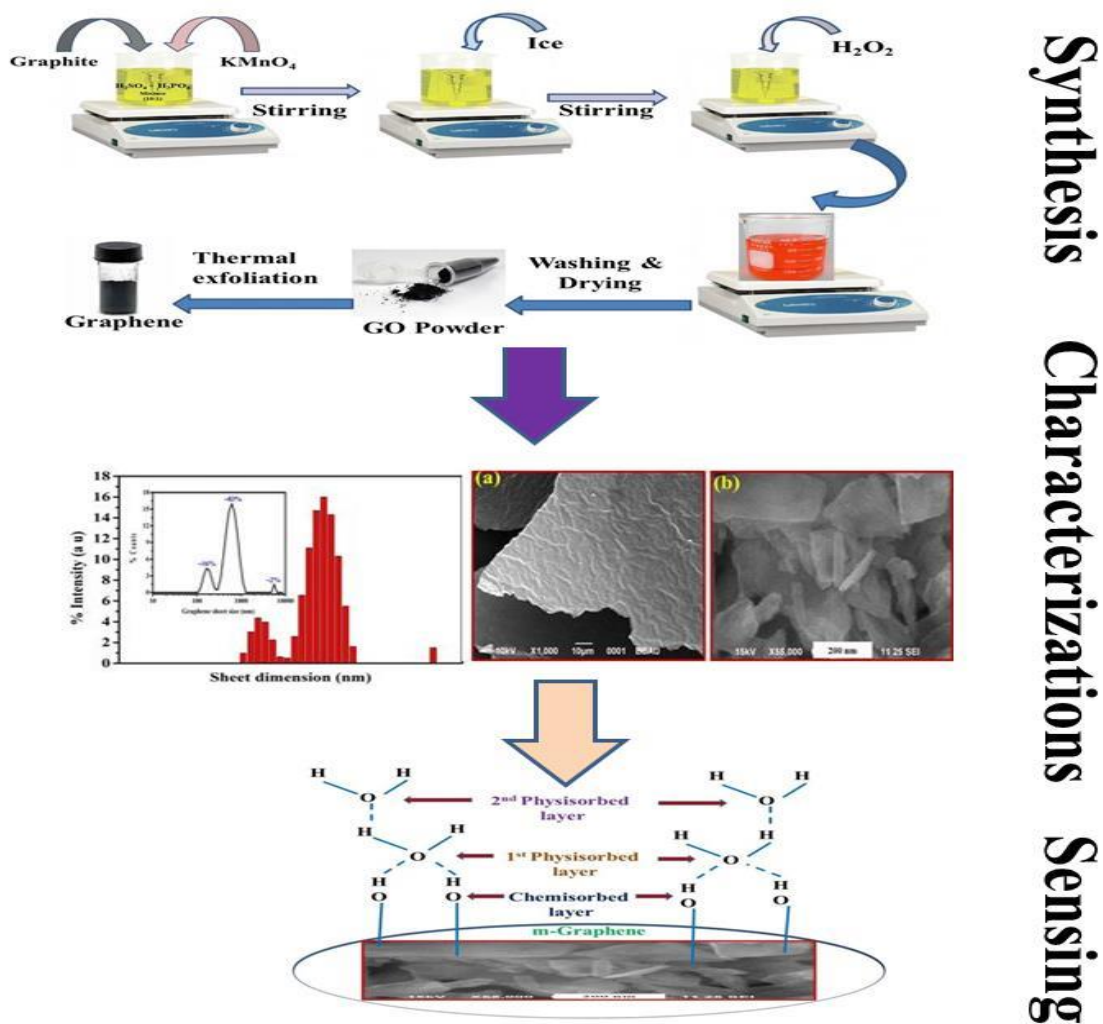


Fig. 1 Graphical abstract of Chapter 2

At the optimized frequency of 100 Hz, the entire experiment was investigated for the repeatability and long term stability of the humidity sensor. The response-recovery time of the sensor was also calculated during the adsorption and desorption of the humidity. The m-Graphene based humidity sensor exhibited the maximum sensitivity as 2.51 $\text{M}\Omega/\% \text{RH}$ at an

optimized 100 Hz frequency. The other sensing attributes as repeatability, response, and recovery time were found as 96.05%, 24, and 13 s respectively at room temperature.

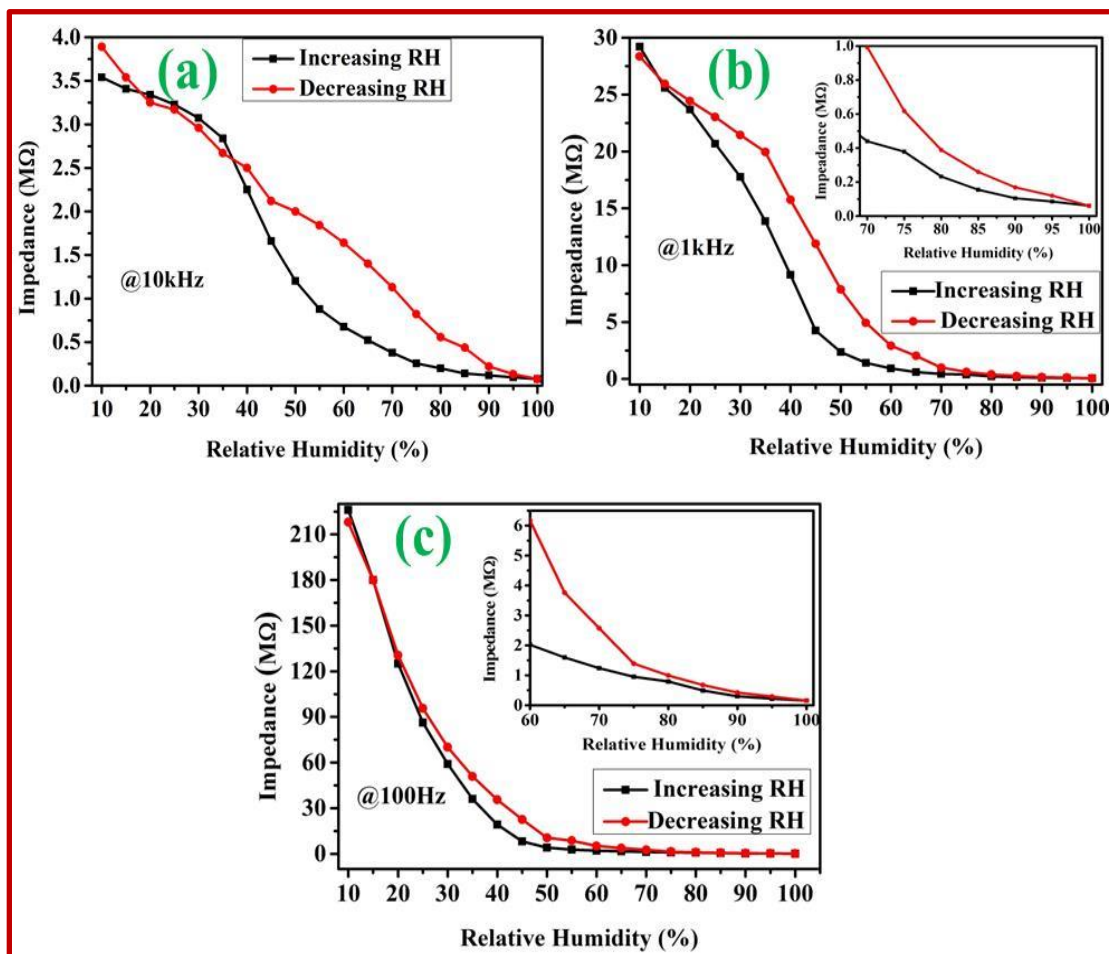


Fig. 2 Variation in impedance with humidity at 10 kHz, 1 kHz, and 100 Hz frequency

Chapter 3:

This chapter details the development of a capacitance-based humidity sensor along with the synthesis of the five-layered Graphene Oxide (GO) and GO functionalization with iron oxides (Fe-GO) by chemical route. The thin film of Fe-GO was employed to observe the humidity sensing potential at an optimized frequency (as optimized in **Chapter 2**). The synthesized materials were characterized by SEM, EDX, BET, XRD, particle size analysis, UV-Vis. spectroscopy, FTIR, and Raman spectroscopy. The graphical abstract of the Chapter is shown in Fig. 3. The SEM micrographs of Fe-GO deal with a unique flower-like structure which

is shown in Fig. 4. Such type of SEM micrographs in the case of the Graphene is not reported till now. Variations in capacitances of thin-film based Fe-GO humidity sensor on the exposure of humidity were recorded. Sensitivity and response-recovery times were calculated, further, the experiment was also repeated after some time to check the repeatability and long-term stability. The humidity sensing attributes as average sensitivity, response and recovery time were found as 5.18 pF/%RH, 31 s and 11 s respectively in the humidity range 10-95 %RH at room temperature. The sensor had the same average sensitivity after 3, 7 and 21 days.

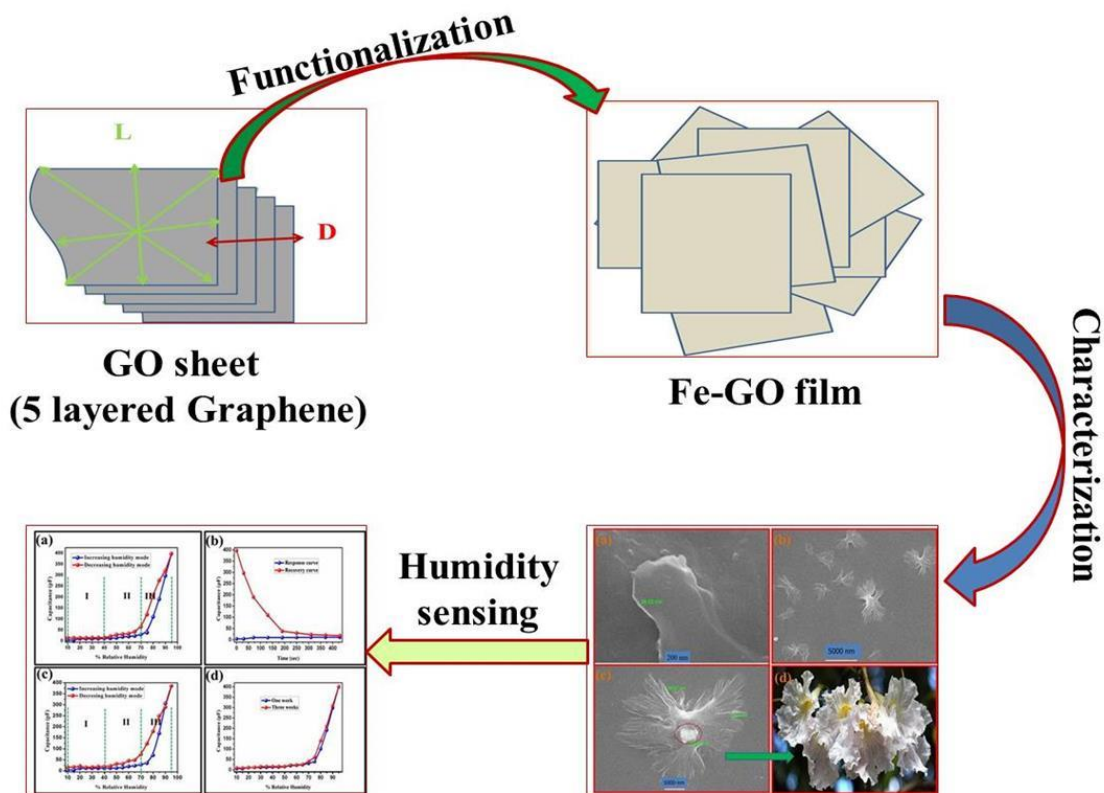


Fig. 3 Graphical abstract of Chapter 3

Chapter 4:

This chapter describes to design and fabricate highly selective and sensitive room temperature operated LPG and humidity sensors using a thin film of Graphene functionalized with CuO/SnO₂ nanocomposites (GCS). The GCS was synthesized with the hydrothermal technique. The synthesized material was under observation with SEM, XRD, UV-Visible

spectroscopy, particle size analyzer, FTIR, and Raman spectroscopy for characterization purposes. The metal nanoparticles are attached with the Graphene sheet at the side edges, corners, and in the basal plane which is confirmed with SEM micrographs. These metal nanoparticles form the metallic dangling bonds which are useful in the sensing application.

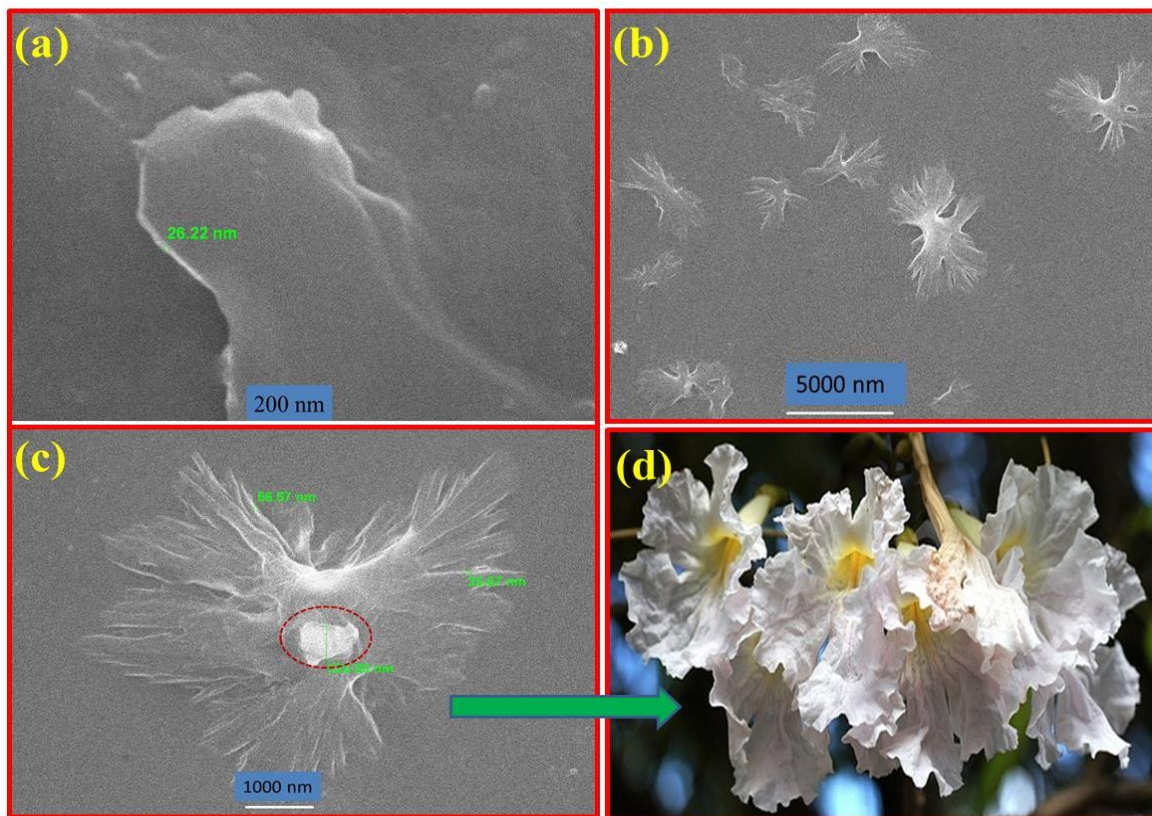


Fig. 4 Surface morphology of the (a) pristine GO and (b, c) Fe-GO (d) photograph of Gulmohar flower

The constructed sensing element was investigated with humidity and LPG sensor characterization setups. The graphical abstract of this Chapter is shown in Fig. 5. The LPG sensor was inspected with 0.5, 1.0, 1.5, and 2.0 vol.% LPG concentrations. The calculated sensor response, sensitivity, and response-recovery times along with reproducibility and selectivity of the sensor were also reported in this chapter. The % sensor response of the sensor was found maximum (11.76) for the 2.0 vol.% LPG and also the minimum response and recovery time were found as 1.7 and 1.2 s for the 0.5 vol.% LPG. The selectivity of the sensor shows good results for the LPG in comparison to the ethanol, acetone, and relative humidity, and

CO₂ gas. The effect of the relative humidity on the LPG sensor exhibited a relative increase in the sensor response of the sensor which increases with increasing the relative humidity.

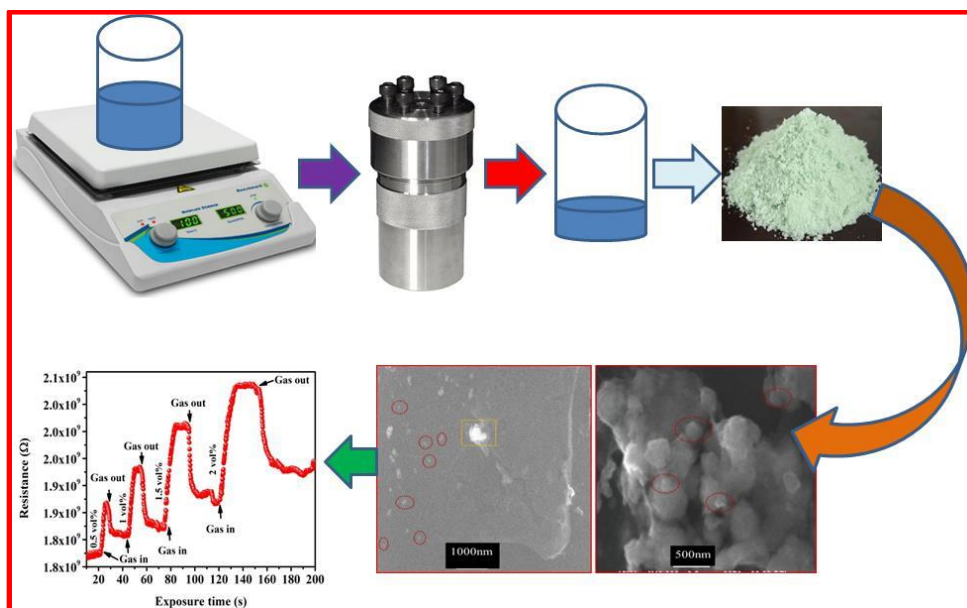


Fig. 5 Graphical abstract of Chapter 4

Chapter 5:

In this chapter, five-layered GO functionalized with Poly Pd acryl amide SnO₂ nanocomposite metallopolymer (MP-GO) based LPG and humidity sensors have been reported. The GO was functionalized with metallopolymer by using N-Methyl Pyrrolidone (NMP). The synthesized samples were investigated with SEM, XRD, Particle size analyzer, UV-Vis. spectroscopy, FTIR, Raman spectroscopy, and other characterization techniques. The fabricated sensing element was exposed to LPG and humidity in the sensing setup. The graphical abstract of this chapter is shown in Fig. 6. The sensitivity, %sensor response, reproducibility, and response-recovery times of the sensor were calculated and it was very selective for the LPG in comparison of humidity, acetone, ethanol, and CO₂ gas. The MP-GO based LPG sensor showed the maximum % sensor response (570.58) for the 2.0 vol.% concentration while the response/recovery time were found minimum (7.8/7.3 s) for the 0.5 vol.% concentration.

Chapter 6:

This chapter addresses the development of highly selective and sensitive CO₂ and humidity sensors operated at room temperature fabricated by a thin film of Graphene@CuO

nanocomposites synthesized through the Chemical Vapor Deposition technique. The CO₂ sensor characterization setup is shown in Fig. 7. For characterization purposes, the synthesized Graphene@CuO nanocomposites were under investigation with SEM, XRD, UV-Visible spectroscopy, Particle size analyzer, FTIR, and Raman spectroscopy. The calculated I_D/I_G ratio for the Graphene@CuO nanocomposites was 0.9. The fabricated sensor was under observation with CO₂, LPG, and humidity sensing characterization setups. The CO₂ sensor was inspected with 0.5, 1.0, 1.5, and 2.0 vol.% CO₂ gas concentrations. The sensor response, sensitivity, and response-recovery times along with reproducibility and selectivity of the sensor were also reported in this Chapter. The Graphene@CuO nanocomposites-based CO₂ sensor showed the minimum response and recovery times as 1.3 and 1.5 for 0.5 vol.% concentration respectively and the maximum %S.R was found as 1178.31 for the 2.0 vol.% concentration.

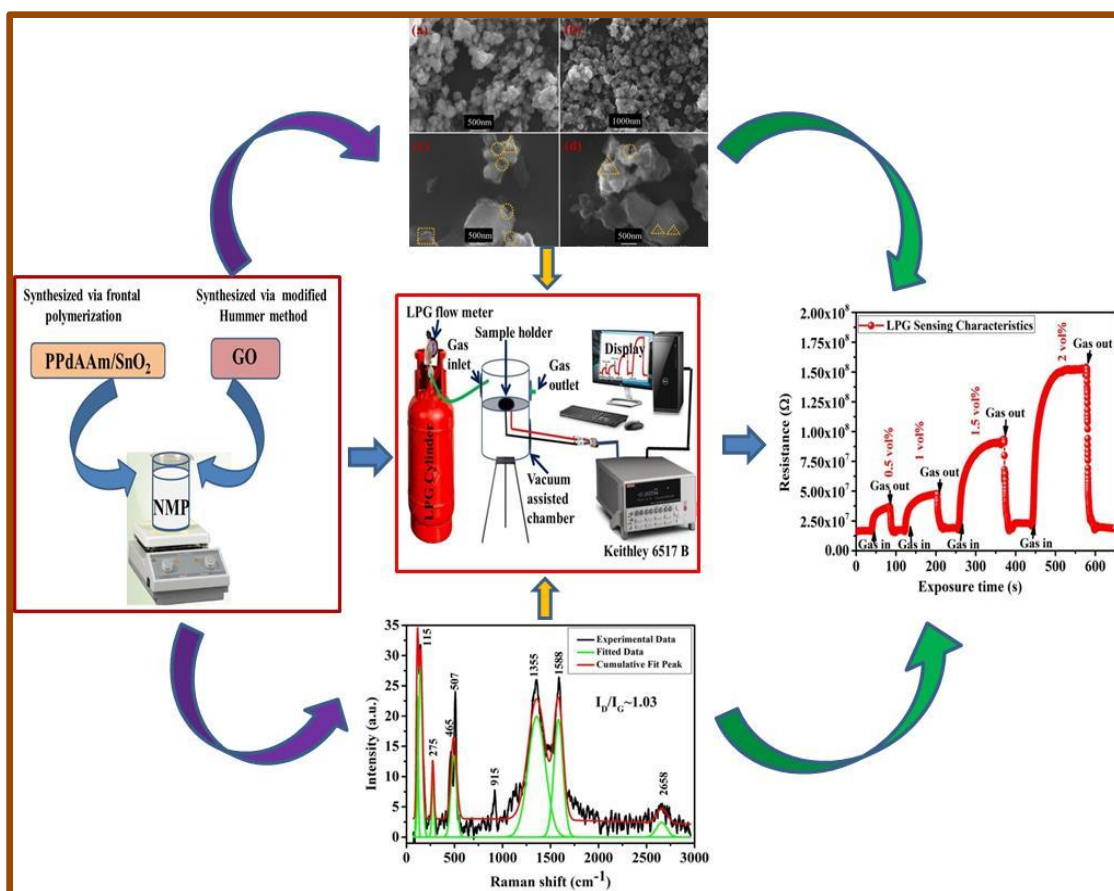


Fig. 6 Graphical abstract of Chapter 5

Chapter 7:

A study of synthesis, characterization, and sensing properties of Graphene and its nanocomposites is summarized in this Chapter. It also gives the guidelines for further research work in the field of carbon nanostructure and its nanocomposite films as efficient humidity and gas sensors.

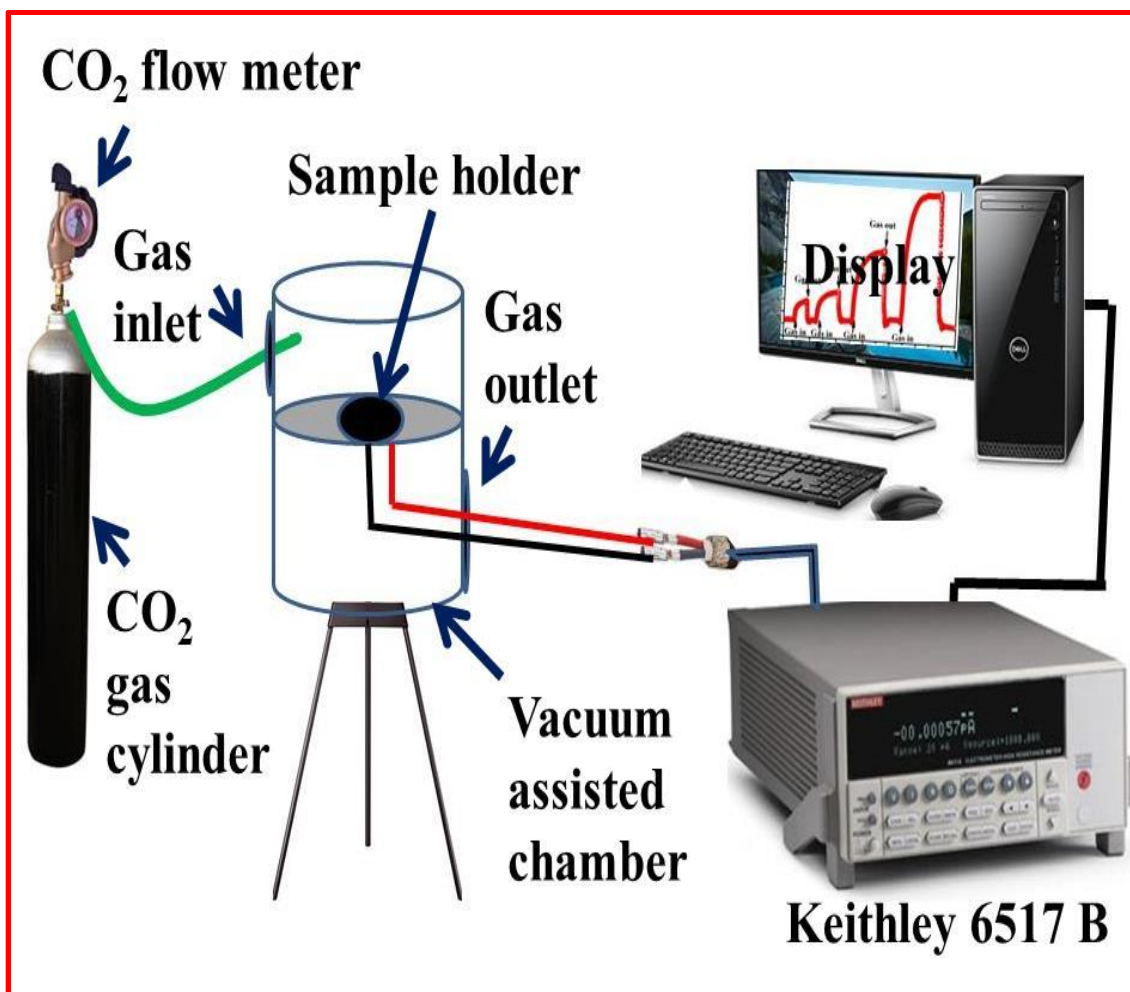


Fig. 7 Experimental CO₂ sensor characterization setup

The complete overview of the Thesis including the sensing materials, synthesis method, sensitivity, %sensor response, response, and recovery times are depicted in Table 1. The main target of our research work was to design and fabricate the different types of sensors using Graphene metal nanocomposites which would be robust, cost-effective, and more sensitive than previously reported sensors.

Table 1: A chapter-wise sketch of the Thesis.

Chapters	Materials	Synthesis Method	Disorder Parameter (I_D/I_G)	Type of sensor	Sensitivity/ %Sensor Response	Response time (s)	Recovery time (s)
Chapter 1	Introduction	-	-	-	-	-	-
Chapter 2	m-Graphene	Wet chemical method	1.55	Humidity	2.51 M Ω /%RH	24.0	13.0
Chapter 3	Fe-GO	Wet chemical method	1.53	Humidity	5.18 pF/%RH	31.0	11.0
Chapter 4	G-CuO/SnO₂	Hydrothermal method	1.09	LPG	5.4	1.6	1.2
Chapter 5	MP-GO	Wet chemical method	1.03	LPG	120.90	7.8	3.3
Chapter 6	Graphene @Cu nanocomposites	Chemical Vapor Deposition	0.90	CO ₂	133.33	1.3	2.3

From Table 1, it is clear that in comparison to the modified Graphene and Fe-GO-based humidity sensor, the later one shows better results. The reason behind it is in the Fe-GO there are more dangling bonds are present which are of the breakage of the GO sheets as well as metallic bonds in comparison to m-Graphene. The 4th and 5th Chapters are of LPG sensors which are

based on GCS and MP-GO. In the comparison of these two LPG sensors, the MP-GO-based LPG sensor shows better results. In the MP-GO based LPG sensor, the Pd plays a prominent role in enhancing the sensor response as well as shorter response-recovery times. The 6th chapter reveals the CO₂ sensor based on the thin film of the Graphene@CuO nanocomposite synthesized by Chemical vapor deposition technique. The CO₂ sensor has the ultra-high %sensor response as 1178.31 for the 2.0 vol.% concentration of the CO₂ gas and minimum response/recovery times were calculated as 1.3/2.3 s respectively. As the I_D/I_G ratio get decreasing the sensitivity is going to increase. The disorder parameters are getting decrease with the functionalization of the Graphene sheets with the metal oxides nanoparticles. After optimization, these materials may be useful for a commercialized model of sensor applicable for both indoor and outdoor detection of LPG/ humidity/CO₂. Thus different configurations/systems described in the thesis and the detailed specifications are given for each of them are expected to prove useful in fabricating sturdy, robust, and cost-effective sensors suitable for operation at room temperature.

Scope of Further Research

Future inspections that would be fruitful in further understanding the role of carbon nanomaterials for more applications in the sensor at high and below room temperature are needed. These include incorporation of the recovery aspects achieved from the incorporation of catalyst onto the surface of carbon nanomaterials being used to detect a reducing gas. Spectroscopic evaluation and surface nanoscale engineering of the sensing mechanism would be very fruitful in the selection of the material to be used for the fabrication of cost-effective sensors in the future. The future scope of carbon nanomaterials can be understood as follows:

- A comprehensive investigation of the evolution of the surface reactions for the temperature regarding to understand the reaction products from the surface interaction needs to be carried

out. Temperature Programmed Desorption (TPD) experiments give valuable information on the formation and desorption of reaction products on the surface.

- The manifestation of crystallographic defects affects the sensing mechanism of gas sensors. An in-situ XPS with gas atmosphere control would give a better idea as to what happens to the electronic structure of the carbon nanomaterials and its nanocomposite during high-temperature gas interaction and also an idea of how the shear planes grow and whether this leads to an increase in the density of states in the gap.
- Theoretical modeling on the sensing mechanism of Graphene-based humidity, LPG, and other oxidizing/ reducing gases may be undertaken for further research.
- Carbon nanostructured materials may be very useful in biomedical applications such as drug delivery systems, cancer cell detection, and treatment, etc. The related Physics may be studied.
- Graphene is a 2D material that can be used in flexible electronics to develop flexible sensors and other electronic devices.

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LIST OF PUBLICATIONS

Part of the thesis published and communicated in the refereed journals:

- **Kuldeep Kumar**, A. Singh, U. Kumar, R. K. Tripathi, B. C. Yadav, **The beauty inhabited inside the modified Graphene for moisture detection at different frequencies**, Journal of Materials Science: Materials in Electronics, 31 (2020) 10836-10845.
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- **Kuldeep Kumar** and B. C. Yadav, **Development of highly sensitive, selective and reduced response-recovery time for LPG sensor based on Graphene functionalized with CuO/SnO₂ nanocomposites**, Sensors and Actuators B: Chemical (Communicated).
- **Kuldeep Kumar** and B. C. Yadav, **Development of ultra-fast sensitive and selective CO₂ sensor based on Graphene@CuO nanocomposites**, Sensors and Actuators B: Chemical (Communicated).

Work not included in the Thesis:

- S. Singh, U. Kumar, **Kuldeep Kumar**, R. Tripathi, B. C. Yadav, K. Singh **Development of scattering based glucose sensor using hydrothermally synthesized cuprous oxide nanoparticles**, Results in Physics 15 (2019) 102772.

- P. Gupta, N. K. Pandey, **Kuldeep Kumar**, B. C. Yadav, **Structural, optical and LPG sensing properties of zinc-doped nickel oxide pellets operated at room temperature**, Sensors and Actuators A: Physical (**Accepted**).
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