

***INVESTIGATIONS ON
NANOMETALLOPOLYMERS AND QUANTUM
DOTS WITH THEIR HUMIDITY AND GAS
SENSING APPLICATIONS***

Thesis

**Submitted for the Award of the Degree
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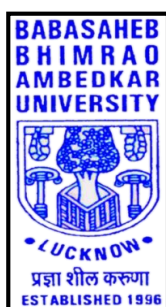
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Investigations on Nanometallopolymers and Quantum Dots with their Humidity and Gas sensing Applications

SUMMARY

Human health drew immense attention and pledged to secure health through proper control on pollution, sanitation, hygienic food, drug, disease observation, urban development, and professional health. There is an essential need to assess the safe environment for healthy living. In recent years, a variety of toxic gases such as hydrogen sulfide (H₂S), carbon monoxide (CO), oxygen depletion (O₂), carbon dioxide (CO₂), ammonia (NH₃), benzene (C₆H₆) have been released from industries and manufacturing activities from raw materials. Hence it becomes very important to control and monitor these toxic gases present in the atmosphere raising a serious concern to human life. In this direction, humidity is much important for human comfort and the environment. Water must be the most crucial element on the earth playing a vital role in the existence of human life. It exists at varying rates in the gas phase (as water vapour) in our air. The importance of the water is that without water vapour in the air, the temperature of the earth's surface would be much cooler (water molecules are the most important for greenhouse gas). However, a high amount of humidity is also leading to discomfort due to imbalance. Water is a very necessary compound for humans as well as the environment. Humidity exists in gaseous form, hence obeys gas laws. Water is a high dipolar molecule, which condenses and evaporates with the temperature of the atmosphere. These water molecules change the atmosphere according to the temperature of the atmosphere and warm air has a better capacity to clamp water than the cold air. The warm air stores a more amount of energy in the environment. Various humidity sensors have been reported based on metal oxides and based nanocomposites. Polymers are one of the attractive materials in the field of sensors. The organic polymers display unique properties due to their high

length. They are very useful for our living life, and applicable in many things, named as plastic age. Nowadays macromolecules have great interest in the field of education and research. Scientifically, the chemistry of macromolecules overlaps the disciplines of engineering science and material physics. Moreover, carbon, the main elements in chain developing organic macromolecules are oxygen, sulfur and nitrogen. The silicon, phosphorus-containing macromolecules are also very important. Organic macromolecules are limited and have some drawbacks such as low thermal stability, binding interaction, electron transfer process and healing effect. Hence it is very important to remove these drawbacks by developing a new class of material.

According to the periodic table, only hydrogen is a univalent element and can play the role to replace any groups and is incapable of generating stable macromolecules in solution. To improve the structural, electrical, photoelectrical, conductivity, stability, magnetic and thermal properties of macromolecules new class of materials are to be developed. Also, they perform in extremely interdisciplinary fields and fundamental research.

Because of these, the combination of metal ions and macromolecules provide both the active and selective properties with high stability performance. The first step regarding these materials is the successful synthesis of macromolecules containing metals or nanocrystals. The essential second step is a comprehensive study by characterization tools and the third is application. There are various combinations of metal macromolecules, organic metals, inorganic metals and semi-organic macromolecules. So the interaction of metal ions and macromolecules have numerous possibilities. Semiconductor nanocrystalline and nanometallopolymers materials are of great interest in the field of nanoscience and nanotechnology. Quantum dots (QDs) are compounds with specific characteristics; like the bandgap of QDs increases when the size of the particle decreases. Also, the colour that QDs emits is directly proportional to its size (smaller dots appear blue). Few limitations of organic metallopolymers (sensitivity, stability etc.) have motivated us towards QDs to make their nanohybrids in the future for humidity/gas sensing applications, as the humidity/gas sensors play a vital role in various fields like medical, agriculture, industries etc.

Chapter 1 deals with the fundamental concept of sensors and their comparison with the existing sensing devices. Also, it briefly introduces the classification of humidity,

gas and photosensors, underlying mechanism and their performance index. A detailed overview of current sensor materials (metal oxide polymer composite, metallopolymers and quantum dots), with strategies of enhancing the performance with its characteristics of sensing materials has been deliberated here. The sensing mechanism and responsible parameters of sensing material are also discussed in detail. The chapter concludes with the concept of metallopolymers and quantum dots materials for humidity, gas and photosensors and the plausible solutions to overcome the present challenges. The wide literature review of the past decade has been also tabulated. The multipurpose applications of impedance and capacitance-based humidity sensors have also been explained in this chapter. The orientation of work, aims and objectives of the present research work are well described at the end of the Chapter. The inclusive study of the recent accomplishments in the field of sensors is done. Presently, the enthusiastic technological advancement of modern appliances raises a serious concern of environmental deterioration and release of toxic gases bad for human health. There is a deep urge to develop highly sensitive gas and humidity sensors for the precise monitoring of toxic gases and humidity present in the atmosphere. Recently, a variety of such sensors are available for the industry as well as domestic applications.

The schematic representation of Chapter 1 is shown in Fig. 1 where fundamentals, introduction, hierarchical development and significance of the sensors are epitomized.

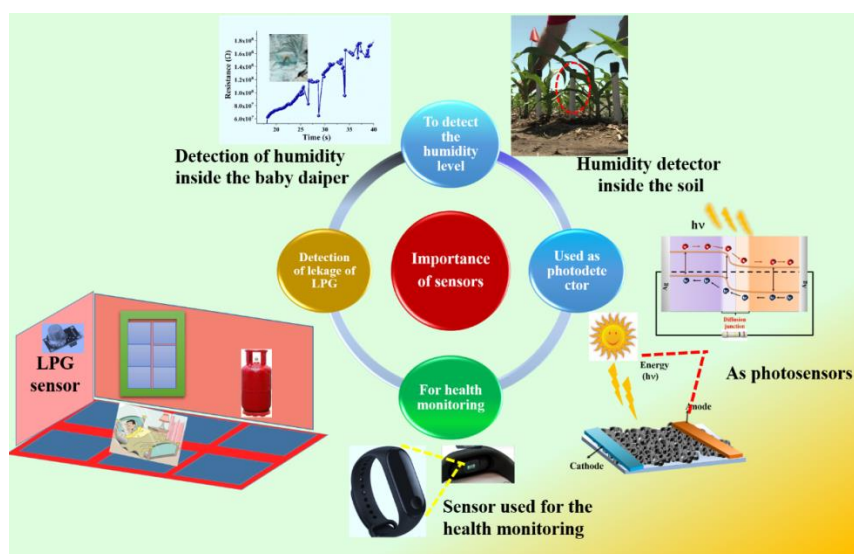


Figure 1: Schematic diagram of the present environmental challenges and need of the sensor.

Chapter 2 presents the nanostructured $\text{Ni}(\text{NO}_3)_2(\text{AAm})_4 \cdot 2\text{H}_2\text{O}$ thin film as an electrical humidity sensor. For this purpose, films were prepared by spin-coating method to investigate the adsorption/desorption of humidity at room ambient. The sensing element was investigated through X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM) and UV–Vis absorption techniques. XRD exhibited the crystalline nature of the nanomaterial thin film and evaluated the average crystallite size as 30 nm. SEM showed highly porous nanostructures of different sizes distributed throughout the film. The energy bandgap of the film was estimated as 4.03 eV. TEM image revealed the nanoparticles having sizes in the range of 4-21 nm and SAED confirmed the crystalline nature of the material. The film was further employed for humidity sensing which gave a maximum average sensitivity of $37.79 \text{ M}\Omega/\% \text{RH}$ at room temperature. The graphical abstract regarding this chapter is presented in Fig.2.

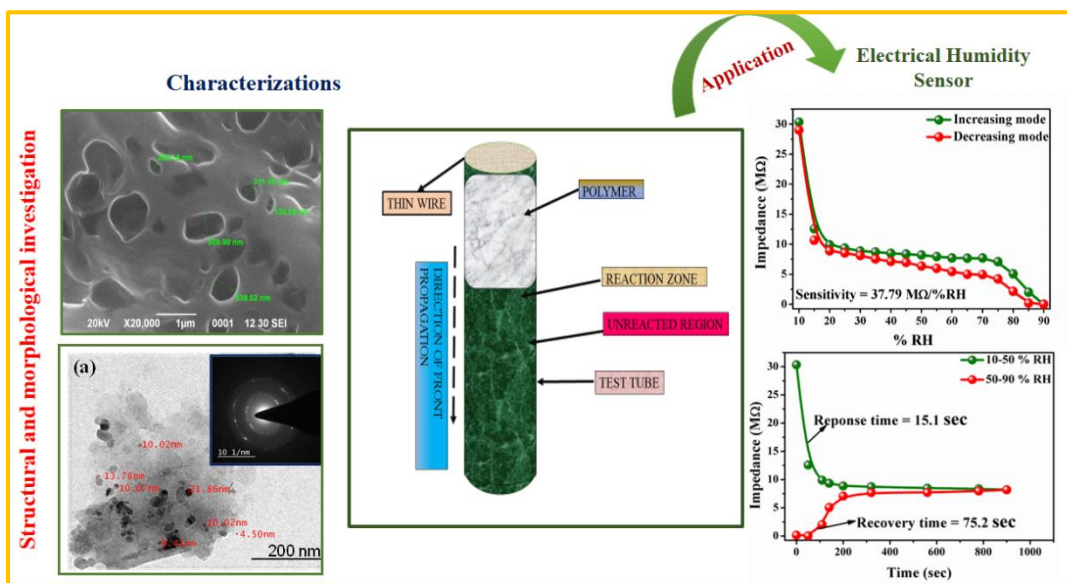


Figure 2: Display of the synthesis techniques i.e. frontal polymerization (mid) characterizations (left) and application as humidity sensor (right) as followed to obtain nanostructured $\text{Ni}(\text{NO}_3)_2(\text{AAm})_4 \cdot 2\text{H}_2\text{O}$ based humidity sensor

Chapter 3 includes the solvent-free synthesis of nanocomposite for humidity sensors which are gaining immense interest to fulfil the criteria for its practical application. Further, it describes the development of CdS/polyacrylamide nanocomposite as a flexible humidity sensor, which has been fabricated by the facile spin-coating technique on PET (polyethylene terephthalate) substrate. The morphological, optical

and crystalline nature of CdS/polyacrylamide nanocomposite were investigated using SEM, TEM, XRD, DSC, FTIR, UV-Visible, and Particle size analysis. Characteristic surface area and pore volume were estimated using BET analysis whereas the hydrophilicity of the sample was investigated using contact angle measurement. As fabricated sensor showed an outstanding humidity sensing response with good linearity in the humidity ranging from 11% to 95% RH. The average sensitivity of nanocomposite was calculated as 125.57 nF/%RH and may be utilized for moisture detection of the commonly used baby diaper with alarm. The simulated & optimized computer model of the synthesized composite (CdS & Polyacrylamide) using density functional theory was performed and analyzed. HOMO-LUMO and correspondingly obtained other electronic parameters were studied, which revealed that the bandgap of the synthesized composite material tends to decrease upon increasing the level of water molecules owing to display better humidity sensing mechanism, and therefore it is applicable for humidity sensors. These results show the establishment of the crucial fact that the developed composite material is suitable for moisture absorbent behaviours. The graphical abstract regarding this chapter has been shown in Fig.3

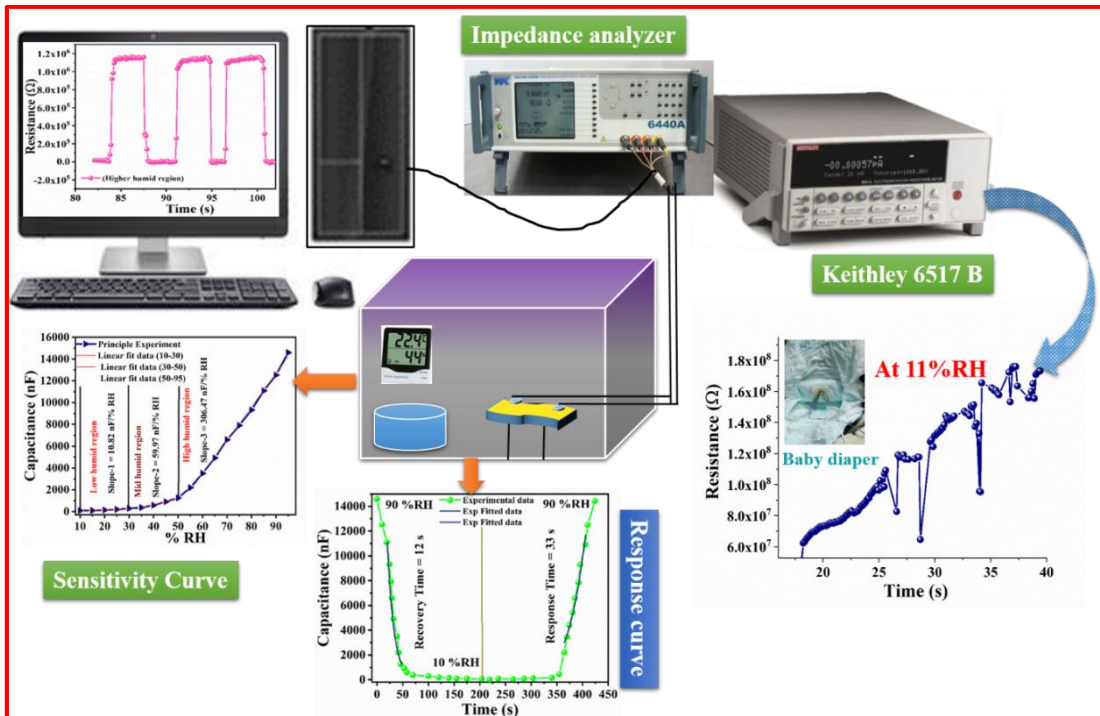


Figure 3: The schematic representation of the fabrication of flexible CdS/polyacrylamide nanocomposite and its application as a humidity sensor

Chapter 4 describes the synthesis of $\text{Cu}_{0.8}\text{Zn}_{0.2}\text{Sb}_2\text{AAM}$ -polymer nanocomposite via frontal polymerization technique with its humidity sensing and photoconductive behaviour. The surface texture was examined using scanning electron microscopy (SEM). X-ray diffraction and transmission electron microscopy (TEM) studies confirmed the crystallinity of as-fabricated nanocomposite thin film-based sensing element. The surface area, particle size, optical absorbance and thermal stability of as-fabricated thin film were successively investigated using Brunauerñ Emmettñ Teller (BET), Particle size analysis, Ultra-Violet absorption, Fourier Transform Infrared (FTIR) spectroscopy, Thermogravimetric analysis, X-ray photoelectron spectroscopy (XPS) and Differential Scanning Calorimetric (DSC) analysis. Humidity sensing characteristics were also investigated via the capacitive performance of $\text{Cu}_{0.8}\text{Zn}_{0.2}\text{Sb}_2\text{AAM}$ film with the exposure of moisture. The average sensitivity (S) of as-fabricated sensor was found to be ~ 168.477 pF/%RH at an optimum frequency of 20 Hz.

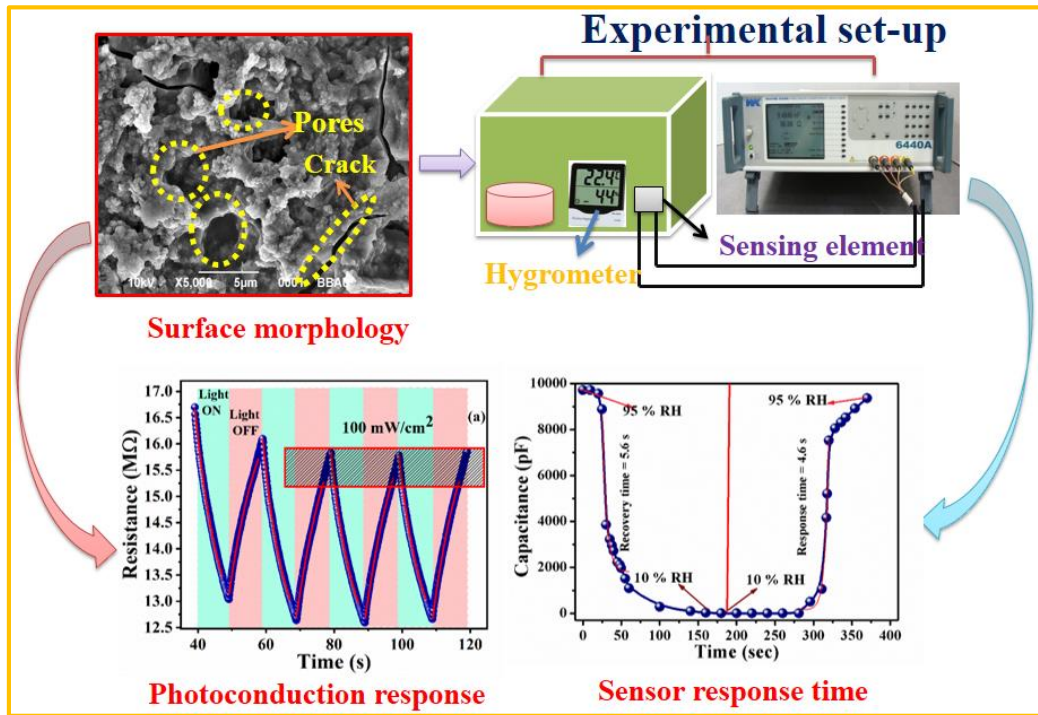


Figure 4: Surface morphology, sensing set-up, photoresponse curve, response and recovery curves of $\text{Cu}_{0.8}\text{Zn}_{0.2}\text{Sb}_2\text{AAM}$ nanocomposite.

The sensor exhibited a rapid response and recovery time of 4.6 sec and 5.6 sec with persistent stability of 96% after 60 days. Thus, $\text{Cu}_{0.8}\text{Zn}_{0.2}\text{Sb}_2\text{AAM}$ polymer-based capacitive humidity sensor ensures its potential towards reliable and safer use in the

detection of humidity levels as well as a photoconduction. In this chapter, the capacitance-based humidity sensor shows very few response recovery times as compared to $\text{Ni}(\text{NO}_3)_2(\text{AAm})_4 \cdot 2\text{H}_2\text{O}$. The graphical abstract shown in Fig.4 represents the morphology and sensing set-up including the response curve of humidity and photo-detector.

Chapter 5 reports the sustainable, cost-effective and rapid synthesis of gram derived carbon quantum dots (GCQD) employed as capacitive humidity sensor. Synthesis of GCQDs C-1 & C-2 at different carbonization temperatures has been performed by the pyrolysis method. The crystallinity of as prepared GCQDs were confirmed by XRD and TEM analysis revealing the average particle size 5.5 and 2 nm for C-1 & C-2 respectively. The optical properties of samples were further analysed by UV–visible and photoluminescence spectrophotometer which gave the bandgap as 3.82 and 4.20 eV. Compositional analysis of GCQDs was confirmed by using XPS analysis. GCQDs were subjected to a sensing test at frequency 20 – 2 MHz.

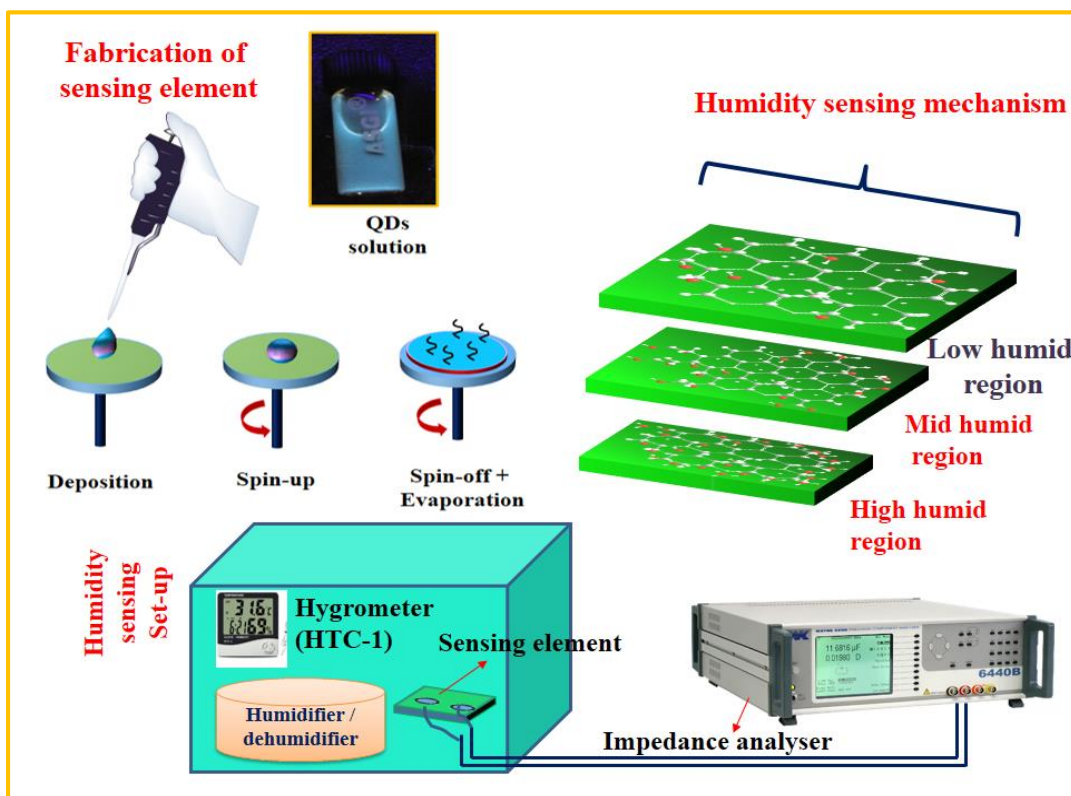


Figure 5: Fabrication of sensing element, humidity sensing mechanism and sensing set-up to fabricate GCQDs based sensor.

The C-2 gave an enhanced sensing performance with an average sensitivity of 254.861 pF/%RH. Also, the sensor showed rapid response and recovery times of 7.3 & 4.7 sec respectively. Theoretical modelling has been performed to further look into the insight of interaction between functional group of GCQDs and water vapour molecules. Transient parameters such as charge density, HOMO-LUMO gap, electron clouds and H-bonds were calculated using B3LYP hybrid functional at 6-31g diffused and polarised basis set. This study provides insight chemistry of GCQDs with water towards an efficient humidity sensor development. In this chapter, the experimental results correlate with the theoretical results. This proves that the sensing behaviour of the materials also depends on the functional group of the material. The graphical abstract of this chapter is shown in Fig. 5.

Chapter 6 reports the novel material synthesis of Indium selenide (InSe) quantum dots, via a one-pot route without any phosphine solvent. TEM analysis reveals that the particles were spherical and most of them were within the size range of 4-6 nm. The particle size distribution curve shows that the average particle size of InSe QDs is ~13 nm. Optical characterization displays that InSe also had the luminescence property. The excitation wavelength at 370 nm shows a strong broad emission peak ranging from 400 to 700 nm, with two max positions at ~ 405 and 432 nm. The UV-Visible spectra confirmed the absorption spectra of the quantum dots and also increases the optical band gap as compared to the electronic bandgap. The estimated optical energy band-gap is 2.20 eV (inset). Here, the bandgap increases with a decrease in the size of quantum dots. The TRPL (Time-resolved photoluminescence) of InSe QDs was also calculated as 6.41 ns. These InSe quantum dots were used as gas and humidity sensors, for this, we have fabricated the sensing element by a spin coating method. Our results show that InSe quantum dots are able to perform as a well-performing humidity sensor. The average sensitivity of the sensing element was found as 8.89 MΩ/%RH. Response and recovery times of the InSe quantum dot were calculated as 5.3 sec & 12.4 sec. From the ageing curve, we can say that our sensor is quite stable and 99.2 % reproducible.

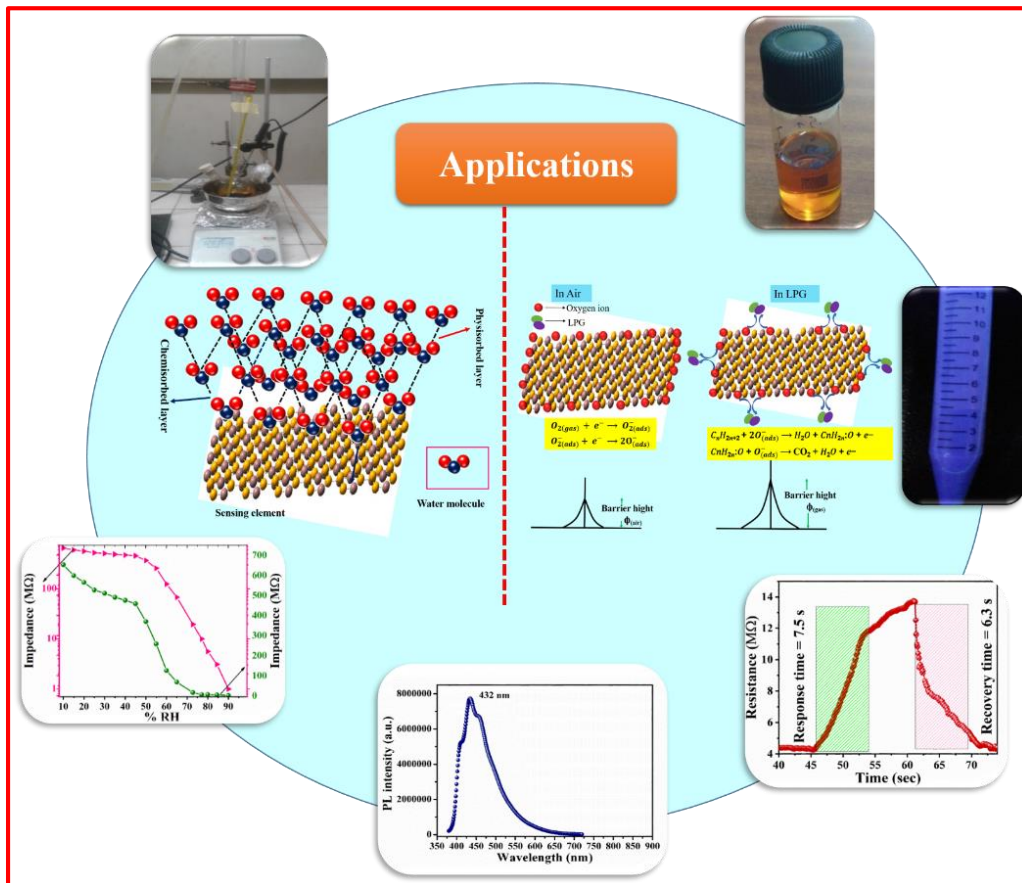


Figure 6: The graphical abstract for InSe quantum dot based sensor.

Table 1: A chapter-wise outline of the Thesis

Chapter No.	Sensing Material	Size (nm)	Energy Band-gap (eV)	Sensitivity (MΩ/%RH)	Capacitive (pF/%RH)	Recovery Time (sec)	Response Time (sec)
Ch. 1	Introduction						
Ch. 2	Ni(NO ₃) ₂ (AAm) ₄ .2H ₂ O	4.0-21.0	4.03	0.37	94.63	75.2 5	15.15
Ch. 3	CdS/polyacrylamide	5.0-	3.50	3.89	125.57	33.0	12.50

		3.1				0	
Ch. 4	Cu _{0.8} Zn _{0.2} Sb ₂ AAM	17.3- 21.8	3.91	4.45	168.47	5.65	4.65
Ch. 5	Carbon quantum dots	2.0- 10.0	4.20	5.12	254.86	4.75	7.35
Ch. 6	InSe quantum dots	4.0- 6.0	2.20	8.89	261.63	4.32	12.21
Ch. 7	Conclusion						

The scope of Further Research Work

In future, research works for creative and efficient devices, the understanding about the role of nanodimensional material at room temperature applications would be desirable. This includes a combination of the recovery aspects onto the surface of the sensing element.

- In the present work, the InSe quantum dots has been optimized to improve the sensing parameters, due to the high surface to volume ratio while there is scope for integration of modifiers like of similar radius or some other less toxic quantum dots for reducing the response and recovery times of humidity and gas sensor.
- An effort may also be made for the synthesis of reported materials using other methods and their versatile sensing applications.
- The dielectric behaviour of these nanometallopolymers and quantum dots may be interesting. The effects of other gases on room temperature on the thin films may be investigated in future.
- Detailed analysis of the evolution of the surface reactions concerning temperature needs to be carried out, in order to exactly understand the reaction products from the surface interaction.

- The theoretical investigation may be very helpful to investigate the interaction between the surface of the sensing element and gases.

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LIST OF PUBLICATIONS

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1. **Priyanka Chaudhary**, Dheeraj Kumar Maurya, Samiksha Sikarwar, B.C.Yadav, G. I. .Dzhardimalieva, Rajiv Prakash, Development of nanostructured nickel reinforced polyacrylamide via frontal polymerization for a reliable room temperature humidity sensor European Polymer Journal. 112 (2019) 161–169.
2. **Priyanka Chaudhary**, Dheeraj Kumar Maurya , Ravi Kant Tripathi , B.C. Yadav, Nina D. Golubeva , Evgeniya I. Knerelman , Igor E. Uflyand, Gulzhian I. Dzhardimalieva Synthesis of $\text{Cu}_{0.8}\text{Zn}_{0.2}\text{Sb}_2$ -polyacrylamide nanocomposite by frontal polymerization; highly sensitive for moisture and photo detector performance, Materials Advances 1 (2020) 2804-2817.
3. **Priyanka Chaudhary**, Dheeraj Kumar Maurya, Anwesh Pandey, Arpit Verma, Ravi Kant Tripathi, Sudhir Kumar, B. C. Yadav, Design and development of flexible humidity sensor for baby diaper alarm: Experimental and theoretical study, Sensors & Actuators: B. Chemical (Minor revision).
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Papers presented in International Conferences/Proceedings

1. Training School on Instrumentation and Characterization (TraSIC) May 24-25, 2018, BBAU Lucknow, U.P. India

2. National Seminar on Nano Science and Nano Biotechnology, 25th – 26th February, 2017, DAVC, Kanpur.

3. 4th Lucknow Science Congress (LUSCON-2017), 3rd – 4th March, BBAU, Lucknow.

4. International Conference on Renewable Energy for Sustainable Environment: Challenges and Remedies, 20th – 21st March, 2017, SMVDU, Jammu.

5. International Conference on Nanoscience and Nanotechnology (ICNN-2013), 24th – 26th September, 2017, BBAU, Lucknow.

Achievements

- ❖ **“Best Poster Presentation Award”** in National Seminar on Nano science and Nano Biotechnology, Feb. 25-26, 2017, **DAV College, Kanpur.**
- ❖ **“Best Poster Presentation Award”** in 4th Lucknow Science Congress (LUSCON-2017) 3rd & 4th March 2017.
- ❖ **“Best Poster Presentation Award”** in International Conference On Renewable Energy for Sustainable Environment: Challenges and Remedies, March 20-21, 2017, **SMVDU, Jammu.**