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Recent progress in Nanostructured Metal Oxides based NO_2 gas sensing in India

S. A. Vanalakar (Vhanalkar),[1*] V. L. Patil,[2] S. M. Patil,[1] S. B. Dhavale,[2] T. D. Dongale,[3] P. S. Patil[2]

[1]Karmaveer Hire Arts, Science, Commerce and Education College, Gargoti (Affiliated to Shivaji University, Kolhapur), Budhargad, Dist-Kolhapur (M.S.) 416209, India. [2]Department of Physics Shivaji University, Kolhapur (M.S.) 416004, India. [3]School of Nanoscience and Technology, Shivaji University Kolhapur, (M.S.) 416004, India.

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Review Article

ABSTRACT The metal oxides (MOs) are considered as an outstanding semiconductor material to sense several toxicants from the environment. In particular, the nanostructure containing rod, wire, and tube-like morphology of MOs were widely utilized to fabricate effective gas sensors worldwide. Out of number of toxicants, nitrogen dioxide (NO_2) is one of the extremely reactive gas, results from the burning of fuel from vehicles, power plants, and off-road equipment. The exposures to NO_2 may give rise to the development of the respiratory diseases and leads to the death. Therefore, the effective detection of NO_2 gas is the urgent need of recent era. More than 5000 research articles were published on the NO_2 gas sensing worldwide. The researchers from India also contributed a lot to detect the NO_2 gas via nanostructured MOs powder and thin films. The present article is aimed to explore the recent advances of NO_2 gas sensors based on MO nanomaterials within the country. The review begins with the general introduction of MO and NO_2 gas and followed by the broader discussion of the use of nanostructured MOs for the construction of NO_2 gas sensors. Furthermore, numerous factors like gas concentrations, working temperature, morphologies, sensor response, selectivity, etc. of MOs were discussed in the present report. The report concludes with the future directions and opportunities in the field of detection of NO_2 gas in India and world.

Keywords: Nanostructured MOS, NO_2 gas sensor, Chemiresistive sensor, Research in India, Sensor response

INTRODUCTION

Gas sensors are electronic devices capable of identifying trace amounts of gas molecules present in the vicinity of it. These sensors are used to detect the toxicants, explosives and volatile gases and to measure their concentrations. Now-a-days, there is a great demand of gas sensors for various field including monitoring of environment, food security, medical diagnostics, and national security.[1] As compared to the other solid-state sensors, MO based (MO) gas sensors showed outstanding sensing response to wide variety of gases and vapors. The basic working principle of these MO sensors involve minute change in electrical conductivity resulted from the interaction between the MO and target gases in the vicinity, before and after the adsorption process. As a result of this interaction, some chemical reactions are originated at the surface of sensor and electric output signal is generated. This signal is recorded by using microelectronic devices as sensing response of material for target gas in its vicinity. Write from the first generation MO sensors based on thick films of SnO_2 reported by Taguchi in the middle of 20th century,[2,3] many researchers have contributed in the designing of advanced MO gas sensors with fascinating properties. The development of such MO gas sensors needed interdisciplinary expertise and therefore lot of research papers and patents have been published in this field in the last decade[4]. This field is just not limited up to publishing research work but also have become well-established commercial market with environmental monitoring devices used to monitor combustible gas, humidity, oxygen, volatile vapors etc. The additional fields include air cleansers assembled with an air-quality sensor. These are being fitted in either car cabins or houses and more stimulating ones as sensing toxic or harmful gases in industrial manufacturing and rush-hour traffic areas.[5]

*Corresponding Author: S. A. Vanalakar (Vhanalkar)
Tel: +91-02324-220112
Email: sharad.vanalakar@gmail.com

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According to The Lancet Commission in 2017, pollution is the major environmental root of various disease and responsible for an estimated 9 million untimely deaths in all over the world[6]. Especially polluting gases like NOx, CO, CO_2, and other volatile organic compounds (VOCs) are accountable for serious threat for human health[7]. They are directly responsible for respiratory, cardiovascular and in some cases infertility. Furthermore, such pollutants have served major cause of environmental and climatic changes such as eutrophication, disturbance of aquatic and terrestrial ecosystems, and acidification. Numerous international and national organizations (e.g. WHO, EEA, and INERIS) are continuously involved in forming global policies and protocols (e.g. Kyoto protocol in 1997, Gothenburg protocol in 1999) to reduce emissions of pollutants. As result of this considerable decrease in air pollutant concentration has been recorded between the years 2000 to 2015.[8]

Meanwhile, out of several toxicants, NO_2 is being the prominent waste and most toxic gas present in the atmosphere. The main sources of this reddish-brown gas with bitter odor are industrial and transport emissions. If the concentration of NO_2 in the environment exceeds above 200μg/m^3, it is considered hazardous for human health. It is also responsible for acid rain: a serious environmental crisis.[9] Therefore, for the environmental safety it is necessary to detect and monitor this pollutant gas on regular basis so as to take essential measures to reduce its concentration. Recently, the porous nanomaterials like MOs and flattened transition metal dichalcogenides (2D TMDs) proved to be effective candidates as NO_2 gas sensors. With little structural modifications, these materials were showing outstanding improvement in different sensing parameters such as selectivity, sensitivity, cost effectiveness, strength and lifetime of the device.[10]

MO BASED GAS SENSORS

Semiconducting MO sensors have interesting characteristic features of excellent gas response, ease operation, compactness and low price. Owing to special characteristic properties like high surface area and surface activity and opto-electronic properties, MOs-based nanostructures with small sizes, confined dimensions and tunable morphologies become best capable materials for gas sensing. The operating temperature is a significant factor for any MOs-based gas sensor. The sensing performance of MO materials prominently dependent upon operating temperature as it plays vital role in surface electrons conductivity, mobility and overall surface reaction kinetics.[11] Most of the conventional MO sensors operated at elevated temperature in the range of 150-500 °C. Such a high temperature is required to cross the activation energy threshold of surface redox reaction and to increase concentration of charge carries for better sensing response.[12] Conversely, such a high operating temperature prevents widespread applications of MO sensors by decreasing its stability, by increasing energy consumption, and also increasing risks of explosion of flammable target gases.[13]

Thus, the normal semiconductor gas sensors essentially operated at high temperature, but currently many efforts are taken on the MO sensors that can work at ambient temperature. Gas sensors differ widely in dimension, variety, and sensing capability. Mostly, they are the part of a bigger implanted system, such as hazmat and security systems. They are generally connected to a sound alarm or interface. As gas sensors are constantly come in contact with open air and different corrosive gases, they have to be calibrated quite often as compared to other gas sensors. Every single type of gas sensor has its own physical properties and sensing mechanism depending on desirable gas sensing environment and functions of those gas sensors. The most widely used gas sensors for sensing smoke and other several types of toxic vapors are MO gas sensors. Such MO based sensors employ a chemiresistors. This chemiresistor reacts with target gases through various physic-chemical ways and generate measurable sensing response. MO based gas sensors either increase or decrease their electrical resistance when they come into contact with oxidizing or reducing gases. Majority of domestic use smoke detection sensing devices are composed of MO based sensors.[14]

With increasing efforts of development and fabrication of MO (MO) gas sensors, the strategies are focused more on their stability, structure and low energy consumption. A large number of methods have been used to modify MO sensors for better NO_2 sensing performance. Some important of them are doping, composite formation, anchoring organic molecules on to the surface, heterojunction sensitization, oxygen vacancy modification etc.[15] It is well established that the microstructure of sensors utterly spoiled during uncontrolled high temperature post heat treatments.[16] But, the extensive efforts have been taken to synthesize morphologically feasible heterostructures of MO semiconductors for gas sensing applications by controlled heat treatments.[17] Various factors like the film thickness, binding between sensing materials and the devices, distance between the electrodes, and feasible or desirable microstructure have great influence on sensing performance. Thus, along with the high-throughput screening and the material preparation more emphasis have require to give on the sensor integration.[18]

The variation in the electrical resistance or conductivity under target gas environment is the basic gas sensing principle of a chemiresistive gas sensor. In MO based chemiresistive gas sensors are of two types, n-type and p-type. The n-type MO semiconductors such as WO_3, SnO_2, TiO_2, ZnO, and In_2O_3 are usually non-stoichiometric due to the deficiencies of oxygen in their matrices.[19] Such structural defects caused due to the oxygen deficiencies responsible for the formation of free electrons on the surface thereby offering electronic conductivities to these MO based sensors. When these MO sensors exposed to electron deficient O_2 molecules in open air, the surface of MO sensors forms an electron depletion layer (EDL). Whereas, the p-type MO including NiO, Cu_2O, $CuMO_2$ (M = Al, Ga, or In), PbO, Bi_2O_3 etc. form hole accumulation layer (HAL) on the surface. When n-type MOS is exposed to an oxidizing gas, its resistance rises. On the contrary, for reducing gases the resistance falls.[20]

Figure 1 represents the principle underlying the gas sensing mechanism of n- and p-type MO based resistive gas sensors.

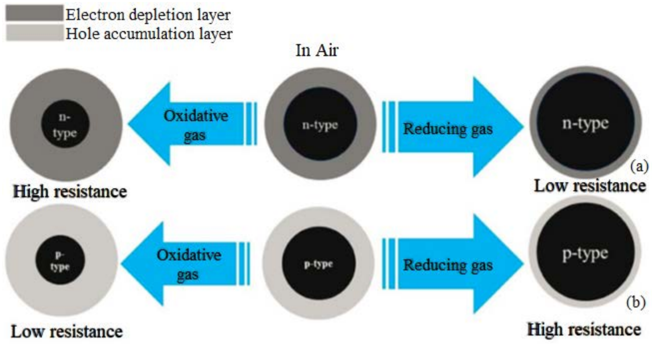

Figure (1): Representation of basic gas sensing principle for (a) n-type and (b) p-type MO based gas sensors for oxidizing gases such as NO_2 and reducing gases as well.[21]

CURRENT STATUS AND PROGRESS OF NATIONAL RESEARCH ON MO BASED NO_2 GAS SENSORS

The gas sensor is an important device used to detect toxic gases and monitor the analyte gas concentration. It plays a vital role for the safety of the environment. Therefore, number of research groups all over the globe is involving to fabricate efficient gas sensor using various materials like MOs. Among the various toxicants, NO_2 is one of the harmful gases. The long and high exposures (more than 1 ppm) of NO_2 gas may leads to the development of asthma. The higher exposure of this gas may increase the vulnerability to respiratory system and it acts as a poison. Mainly the children, old and people with asthma are mostly at high risk in the vicinity of NO_2 gas. Therefore, researchers are trying to fabricate an efficient NO_2 gas sensor using various MOs. Till-to-date (Jan. 2022), more than 5200 research articles were published on the 'MO based NO_2 gas sensor' [Source: Scopus data]. China is a leading country in the field of NO_2 gas sensing via MOs. The Scopus data shows that more than 1300 research articles on MOs based NO_2 sensor were published by Chinese authors; followed by Korean scientist with articles more than 600. Meanwhile, USA and Indian scientist were published about 500 articles each on same on NO_2 gas sensor.

In India, number of research groups are studied the MOs for the detection of gases such as NO_2. The major research groups are P. S. Patil from Shivaji University (Kolhapur), M. Tomar from University of Delhi, V. B. Patil from Solapur University (Solapur) and N. Bhat from Indian Institute of Science, Bangalore. Numerically, researchers from Shivaji University published 60 research articles on MOs based NO_2 gas sensors. The Scopus data shows that the researchers from Solapur University (50 articles), BARC (39), Delhi University (38), IISC (30) were working on the development of efficient NO_2 gas sensor using MOs. Here in this report, we are trying to summarize the research works carried out by major groups from India in the field of MOs based NO_2 gas sensor.

1 SHIVAJI UNIVERSITY, KOLHAPUR

The research group headed by Patil of Shivaji University, Kolhapur is one of the leading groups working in the field of NO_2 gas sensor in India. The group utilized number of MOs such as TiO_2, ZnO, SnO_2, In_2O_3, NiO, CeO_2, WO_3, for the detection of NO_2 gas. This research group has been working in the area of MOs based gas sensors since last 30 years. Vanalakar, V. L. Patil is the major contributor of this research group. They have published more than 50 research articles based on the development of the gas sensors. This research group mainly utilized chemical deposition techniques like spin coating, chemical bath deposition, hydrothermal, Kirkendall effect, spray pyrolysis technique, dip coating, facile successive ionic layer adsorption and reaction (SILAR) and modified SILAR to deposit the MOs. Due to the variety of the chemical method, they were succeeding to achieve exotic morphologies of MOs such as particles, rods, wires, fibres, plates, sheets, cubes, porous cubes at the nanoscale. These diverse morphology leads to the enhancement in the surface area and subsequently the enlargement in the sensing behaviour.[21]

Patil et al. prepared the ZnO nanorods (NRs) by simple and cost effective reflux method[22] within the reaction time of 6 to 9 h. They found the NRs like morphology of ZnO with an average length of about 2.5 to 4.5 µm. The spacing of the NRs constantly increases with the gradual increase in the reaction time. Furthermore, they used as synthesized ZnO NRs for the efficient sensing of the NO_2 gas. The maximum gas response of ZnO NRs based sensor was reported around 3100 % near 100 ppm NO_2 gas at 175ºC operating temperature. In the next study, they used same material, but the synthesis method was different. They used chemical bath deposition (CBD) method with variation of the concentration of the complexing agent to grow the ZnO. They found the micro-flower like morphological appearance of ZnO[23] with the average diameter of the petal of flower is about 180-300 nm in size. The ZnO micro-flowers sensor revealed the maximum gas response of about 64.50 at 150 °C for 100 ppm of NO_2 gas. The lower gas response as compared to the NRs like morphology is might be due to the less surface area available for gas adsorption-desorption. Further, the group have reported SILAR technique to synthesized ZnO thin films. As the CBD is simpler than reflux, so, to use more simplistic synthesis route than CBD, the research group used SILAR technique. They prepared porous nanograins-like morphology of ZnO using SILAR technique.[24] The gas response of such ZnO based sensor was quite impressive due to the porosity of the developed films. In the next study, using modified SILAR technique, the group was succeeded to tune the maize like morphology of ZnO. The novel maize-like morphology of ZnO showed a remarkable NO_2 gas response even at low detection limit of 1 ppm.[25] The superior gas response is due to the maize like morphology which provides the more porous nature. In this study, the group claimed invention of modified SILAR technique which use two beakers instead of conventional four beakers SILAR set up. This modified method is assumed to utilize minimum precursor concentrations. Meanwhile, they investigated the TiO_2 thin films utilizing SILAR technique to sense the NO_2 gas.[26] As like ZnO, TiO_2 also showed the grain like morphology which is prepared by conventional SILAR technique. They observed good gas sensitivity for NO_2 gas at 200ºC working temperature. However, there is no report on the designing of TiO2 thin films by improved SILAR technique.

It is well known that, the hydrothermal method; one of the important chemical synthesis route is employed to tune the morphology of thin films at high temperature and pressure.[27] Therefore, the Patil's research group used hydrothermal method for the preparation of ZnO NRs for the detection of NO_2 gas.[28] They prepared the NR with the variation of the concentration of 'Zn' precursors in hydrothermal system[29]. The synthesized NR was highly defective which was studied by using photoluminescence spectroscopy. The defective nature of ZnO contributes to the better gas sensing capacity of about 113 towards 100 ppm NO_2 gas at 150 °C as working temperature. In order to enhance the gas response of as synthesized ZnO thin film-based sensor, the Patil's research group had undertaken the study of effect of doping element in the pure ZnO NRs by same method. They doped the 'Ni' and 'Cu' element in the ZnO NRs.[30,31] They found the drastic change in the morphology of ZnO after the doping. The copper doping adversely affects the nature of NRs, while the nickel doping leads to the nanosheets like morphology. However, due to the harmonize effect of the doping on morphology and defects; there was an improvement of the gas sensing response after doping. The beauty of the Ni doping was the detection limit for NO_2 gas was reached up to 1 ppm at 150 ℃operating temperature.

Then, Patil et al. focused to understand the effects of synthesis method on the gas sensing behaviour of MOs. They used WO_3 materials for the detection of NO_2 gas by using various methods like spin coating, spray pyrolysis techniques.[32,33] They successfully deposited WO_3 thin film by a facile spin coating method. They found the porous nature of WO_3 thin films with spherical grains. The WO_3 thin film exhibited enhanced gas sensing response of 2.04 for 100 ppm concentration of NO_2 gas at relatively optimum operating temperature 200 ℃. The porous nature and small grain size of WO_3 was found to be beneficial for the sensing performance. Furthermore, they employed spray pyrolysis techniques (SPT) to synthesize the WO_3 thin films. They deposited WO_3 thin films on to the surface of glass substrates at 400 °C by a cost effective and easy SPT. They achieved elevated gas sensing response towards NO_2 gas from as prepared extremely porous and fibrous WO_3 sample with high value of the activation energy. The gas sensor of WO_3 thin film displayed supreme response of 84.75 to 100 ppm of NO_2 gas at 150°C as an optimum operating temperature. Figure (2) shows the surface morphology of the WO_3 samples.

As the In_2O_3 is an excellent sensing element, therefore, Patil and his research group has undertaken focused research on In_2O_3 based gas sensors using chemical routes like spin coating, spray pyrolysis, hydrothermal and Kirkendall effect. Initially they reported the deposition of In_2O_3 thick films by a facile SPT at different Concentrations of spray solution. They found the nanopetal like morphology which is more effective for NO_2 gas detection. They observed the In_2O_3 a film deposited by SPT could be used for specific and efficient sensing of NO_2 gas up to lowest 5 ppm concentration. The hydrothermal synthesis of In_2O_3 nano-bricks like morphology was also reported by the group. For the

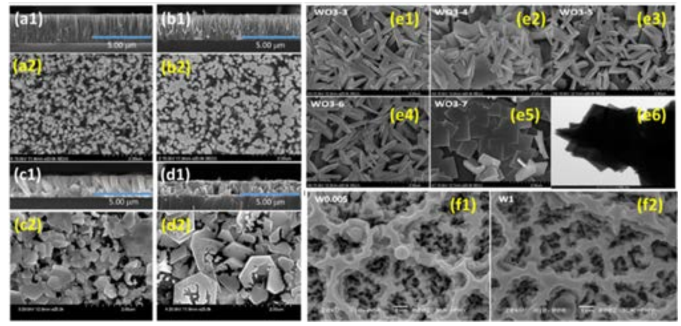

Figure 2: (a1 to d2) FESEM images of bare and Cu doped ZnO thin films (Surface and cross-sectional view), (a) bare ZnO, (b) Cu doped ZnO 1 (CZO-1), (c) Cu doped ZnO 2 (CZO-2) and (d) Cu doped ZnO 3 (CZO-3). [30], (e1 to e5) FESEM images of two dimensional WO_3 nanoplates prepared at various reaction times such as 3, 4, 5, 6, and 7 h showing effect of temperature. Here, vertically placed nanoplate changes to horizontal alignment with the rise in deposition time. (e6) to ensure the formation of nanoplate like structure the TEM image is shown[9], (f1 and f2) SEM images of highly porous and fibrous structures of WO_3 samples.[33]

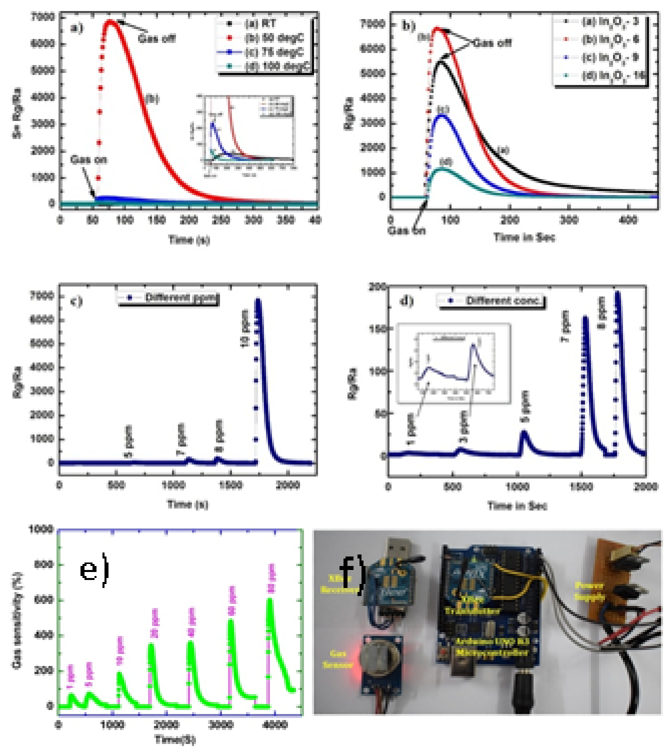

Figure 3: Gas sensing performance of different MOs towards NO_2 gas (a) deviation in In_2O_3 sensor response for the NO_2 gas with 10 ppm concentration at different operating temperatures, (b) change in In_2O_3 sensor response for the NO_2 gas with 10 ppm concentration at diverse synthesizing time, (c) and (d) In_2O_3 sensor response for different concentrations in ppm of NO_2 gas[34], (e) Gas-sensing performance of the copper doped ZnO 1 (CZO-1) thin film derived sensor towards various concentrations of the NO_2 gas (5, 10, 20, 40, 60 and 80 ppm) at 175 °C, (f) The NO_2 gas sensing device setup worked on wireless monitoring hardware. The device is fabricated by using Arduino UNO R3 microcontroller and XBee modules.[30]

synthesis of In_2O_3 bricks, they used the L-alanine as a green product. They got the bricks like morphology with size about 1.5 μm with a surface area of about 17.365 m^2/g and average pore size is about 21 nm. The sensor performance was about 600 units for the NO_2 gas with 100 ppm concentration and operating temperature of about 100℃. Figure (3) shows the sensing behaviour of In_2O_3 thin films. Further, they designed the hollow In_2O_3 microcubes by Kirkendall effect for the NO_2 gas detection. The ionic exchange is beauty of Kirkendall effect to make the hollowness of In_2O_3 microcubes. On this aspect they investigated the NO_2 gas sensing application. They obtained the sensing response 1401 for the NO_2 gas with 100 ppm concentration at identical optimized temperature. Furthermore they prepared porous In_2O_3 nanocubes by using same experimental parameters with using the Cetyl Trimethylammonium Bromide (CTAB) as a surfactant which helps to control the size of the nanocubes. They obtained sensing response is about 3450 for the NO_2 gas with 100 ppm of concentration at 100^o C operating temperature.[34,35] Again, on the same time Patil et al. worked on the chalcogenides materials. They investigated various chalcogenides including PbS, CdS etc. as a NO_2 gas sensing elements.[36] They synthesized PbS and CdS by chemical route and got the exotic morphology like grain, mesh like morphology for the NO_2 gas detection. Overall Patil and Vanalakar's research group worked on various materials prepared by chemical techniques with exotic morphology for the efficient NO_2 gas sensing. Figure (3) shows the gas sensing response of MOs prepared by this group. They efficiently detected ppm to ppb level concentration of NO_2 gas at comparatively optimum operating temperature.

2 University of Delhi, Delhi

The research group headed by M. Tomar in association with V. Gupta of University of Delhi is also working in the designing of NO_2 gas sensor making use of semiconductor MOs and their composites. Various morphologies have been developed by this group using different synthesis strategies. Initially, they have utilized RF sputtering method for the deposition of SnO_2 thin films having grain like morphology. During the deposition, they have used Sn metal as a target. The grain size of the SnO_2 film was found to be in the range of 16 to 80 nm and it was decreases as the increase in the sputtering pressure. This as synthesized SnO_2 grain acquired excellent NO_2 gas response of 2.9 x 10^4 at 50 ppm concentration of gas and at optimum temperature 100^oC. Also, lowest detection limit of 1 ppm has been reached by this group based on SnO_2 grain like morphology.[37] Figure (4) indicates the schematic presentation of SnO_2 thin film gas sensor and their gas sensing performance. Further, the trace level detection of $NO_2$was reported by the same research group. The same experimental condition has been followed for the synthesis of SnO_2 micrograins. The gas response of 1.4 x10^4 for 10 ppm of NO_2 gas at 100^oC working temperature was reported by this group in another report.[38] Further, they have prepared SnO_2 nanocluster by using sol gel method. The diameters of nanocluster were found to be in the range of 150 to 200 nm in the size. The beauty of this work was to obtain the gas sensing properties at room temperature and at ppb level detection. They observed the gas response of SnO_2 nanocluster of about 104 towards NO_2 gas for 100 ppb concentration.[39]

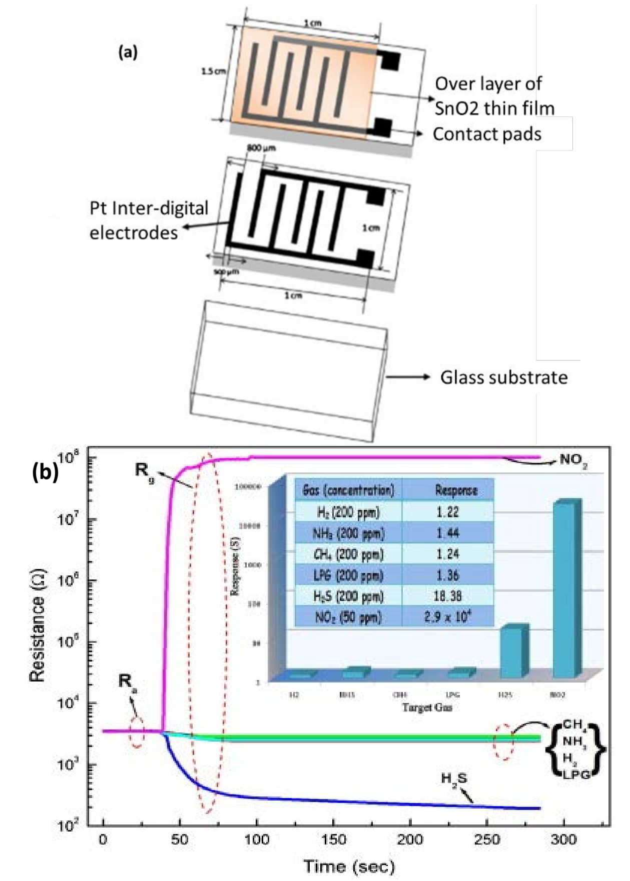

Figure 4: (a) Schematic representation of gas sensor based on SnO_2 thin film and (b) sensing performance of gas sensor based on SnO_2 thin films.[37]

In the attempt to increase the NO_2 gas response of SnO_2 thin films, they have composited SnO_2 with a mutliwallled carbon nanotubes (MWCNT). The chemical solution deposition technique was used for the preparation of composite. Once again, the nanoclusters like morphology of MWCNTs–SnO_2 hybrid was obtained by this group. However, the composite drastically enhanced the gas response. They reported the response of 1070 for 100 ppb NO_2 gas at the room temperature.[40] In the series of composites, they prepared hetero-interface of TeO_2/SnO_2 with the help of RF sputtering method. The nanocluster in the form of micro disc of TeO_2 has been uniformly distributed all over the surface of the thin film of SnO_2 material. This distribution causes more roughness of the composite, which is good for effective adsorption of NO_2 molecules on to the surface. They reported the enhanced gas sensing response (2.26 x 10^4) for NO_2 gas with 100 ppm concentration at operating temperature of about 90℃.[41] Figure (5) reveals the schematic representation of gas sensor based on SnO_2 thin film structure situated on Pt IDEs, and the sensing performance towards 2000 ppm of NO_2 gas at 90℃as an operating temperature. Image in inset indicates the deviation in the resistance of the sensor with respect to change in time after its exposure to NO_2 gas of varying concentration. Furthermore, they used same strategy to enhance the gas response for various MOs. They were prepared number of hetero-structure like WO_3-

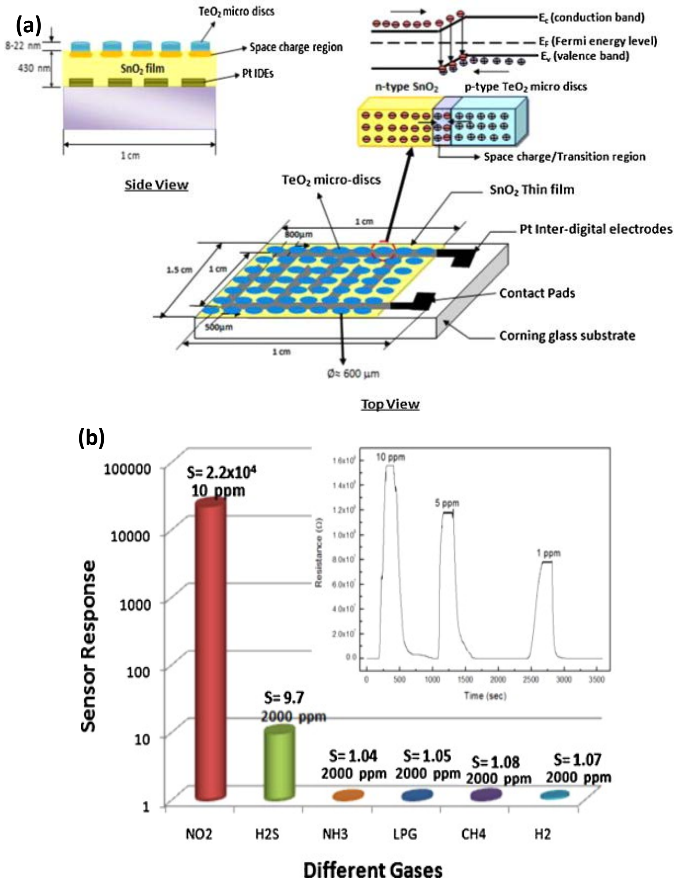

Figure 5: (a) Schematic representation of gas sensor based on SnO_2 thin film structure on Pt IDEs, (b) selectivity of hetrostructured TeO_2/SnO_2 sensor with TeO_2 size 18 nm to the different hazardous gases (2000 ppm) at 90 °C as an operating temperature. Image in the inset indicates the deviation in the resistance of sensor after exposure to various concentrations of NO_2 gas with respect to time.[41]

SnO_2, ZnO-SnO_2, etc.[42-44] The WO_3-SnO_2 nanocluster showed highest NO_2 gas sensing response of about 5.14 x 10^4 for 10 ppm gas at an operating temperature 100℃. This is attributed to the insignificant difference in the value of work function of the WO_3 and SnO_2 that is 4.41 eV and 4.18 eV respectively.

In addition, this group observed the doping effect on the gas sensing response of MOs towards NO_2 gas. The SnO_2 thin film was doped with various elements such as platinum (Pt), silver (Ag) and cupric oxide (CuO) by using chemical route. Among all the doped elements, Pt doped SnO_2 was obtained maximum gas response for 20 ppm concentration of NO_2 vapours at an optimum temperature of about 90°C with high response value of about 1.83×10^2.[45] Apart from SnO_2, this research group studied gas sensing behaviour of CuO, ZnO and some chalcogenides like CdS to sense the NO_2 gas.

3 Indian Institute of Science, Bangalore

The research group of N. Bhat from Indian Institute of Science, Bangalore, India is also working in the field of NO_2 sensor using MOs and its composites. They have published more than 10 research articles on the detection and monitoring of NO_2 gas sensor. In their report, they have designed and developed the envirobat device for the detection of various gases from the air pollutants.[46] Then, clad etched fiber Braggs grating (eF BG) coated with reduced graphene oxide (RGO) was synthesized by this group. The different characterization like RAMAN, SEM has been used for the confirmation of RGO materials. Furthermore, they have studied NO_2 gas sensing performance of these RGO coated eFBG at room temperature. The measurement of NO_2 gas sensing was observed via variation in refractive index of RGO after injecting the NO_2 gas molecules. They got interesting results for NO_2 gas sensing application and detection limit up to 0.5 ppm.[47] Figure (6a) shows the schematic representation of mechanism of NO_2 gas sensing on RGO coated eFBG and Figure (6b) is the SEM micrograph of eFBG sensor displaying homogeneously coated RGO in the region of eFBG

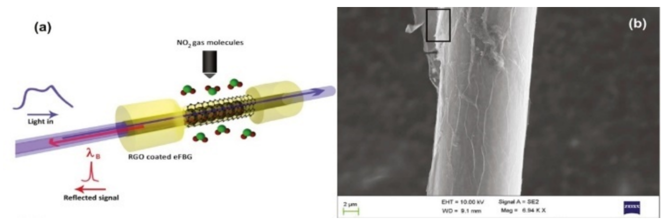

Figure 6: (a) schematic representation of mechanism of NO_2 gas sensing on rGO coated eFBG. (b) the SEM micrograph of eFBG sensor displaying homogeneously coated rGO in the region of eFBG[47]

Recently, they have focused on the simulation, fabrication and sensing characteristics of single chip sensor array by using different MOs semiconductors such as ZnO, 1% Ag doped-$BaTiO_3$-CuO, WO_3 and V_2O_5 etc. The PECVD technique was employed for the preparation of these MOs for the detection of NO_2 gas. The spherically shaped grains like morphology with size varied from 35 to 60 nm have been reported by this group. Based on these properties, they found gas sensitivity of about 1948 % for 0.9 ppm gas concentration at 150℃ operating temperature.[48] Then, this research group developed a gas sensor device on a flexible acetate substrate and the four elements have been patterned with 100 nm gap by using sputtering and PECVD method. The gas sensitivity through such device was observed about 74.8 % for 3 ppm of NO_2 gas at 300ºC operating temperature and detection limit was reached up to 100 ppb.[49] Further, this group synthesized highly porous NiO thin films via microwave method which displayed the NO_2 gas sensing with a gas sensitivity of about 51% at 25ºC operating temperature. In this study also the limit of detection was upto 100 ppb.[50] The recent study of this research group reported the gas sensitivity towards NO_2 gas is about 2301 % for 3 ppm gas concentration.[51] Meanwhile, RF magnetron sputtering technique is employed for the preparation of porous grain like WO_3 thin films. These films rendered 8.4 % gas sensitivity for NO_2 gas with lowest concentration of about 3 ppm.[52]

4 Solapur University, Solapur

The research group headed by V. B. Patil from Solapur University Solapur is one of the well-known groups in the field of NO_2 gas sensing in India. This group published more than 50 research articles on the sensing of the NO_2 gas using MOs and its composites with different polymer. Initially, they have developed the nanocrystalline ZnO thin film by using spin coating method for the detection of NO_2 gas. They got the 37.2 % response and 78 % stability for NO_2 gas at 200º C operating temperature.[53] Then, they have synthesized the SnO_2 nanostructured thin films by sol gel spin coating method. They observed the grain like morphology and the diameter of the particles varies from 5-10 nm in size. They showed the 19 % gas response for 100 ppm NO_2 gas at 200℃ operating temperature.[54] Then, by same synthesis method they have prepared the NiO nanostructure for NO_2 gas sensing application. The grain like morphology with size about 40-51 nm has been developed by this group, which showed about 23.3 % gas response for 200 ppm NO_2 gas at 200º C operating temperature.[55] Furthermore, they prepared Fe_2O_3 nanoparticles (NPs) with size in the range of 40 nm by spin coating technique. They exhibited the 17.12 % gas response for NO_2 gas at 200º C operating temperature.[56] Along with the MOs, Patil's research group developed MO-polymer composite to use the synergy of both the materials. They synthesized camphor sulfonic acid (CSA) doped PPy/a-Fe_2O_3 hybrid nanocomposites via spin coating method for NO_2 gas sensing application. The composite showed the spherical granular porous morphology which proved to be beneficial for the improvement of the NO_2 gas response based on hybrid nanostructures.[57] In addition to MOs or the composite, they also prepared the polymers such as polypyrrole (PPy) thin film for sensing applications. Furthermore, they synthesized the CSA doped polypyrrole based gas sensor via spin coating method. The porous and uniform granular morphology of the composite reveals about 52 % gas response for 100 ppm NO_2 gas at room temperature. Its shows improvement of the gas response as compared to the pure PPy samples. This improvement of the gas response might be due to the harmonious effect of CSA and PPy and their bonding.[58] The NiO-PPy, Fe_2O_3-PPy composite, WO_3 thin films and their composite was also prepared and their NO_2 sensing behaviour tested by the group. Meanwhile, the bio green synthesis of Ni element and their doping in SnO_2 thin film also reported by the group for NO_2 gas sensing. They found the spherical morphology with 6 nm of average size of Ni element and the gas response of about 40 for 100 ppm NO_2 gas at 200º C operating temperature. Here the Ni may acts as catalysts so thereby the sensing response increased noticeably.[59] Recently they are engaged in the improvement of the gas response of MO-polymer composite.

5 Cochin University

Kusumam et al.[60] from Cochin University of Science and Technology have studied the NO_2 gas sensing properties based on MOs. They have prepared the surfactants assisted ZnO NPs and nanosheets by using hydrothermal method. They fabricated less than 100 nm size of Zn NPs and 30 nm thicknesses of ZnO nanosheets with proper spacing. Furthermore, 280 % gas response have been reported for 5 ppm NO_2 gas at 170º C operating temperature for nanosheets like morphology. Here, sheets like morphology reveals more oxygen vacancies which are beneficial for the adsorption of NO_2 gas and hence increases the gas response of NO_2 gas. Another group from the same university have been working on NO_2 gas sensing based on ZnO NPs thin films obtained by spray pyrolysis method.[61] The oxygen-deficient ZnO thin film has been prepared by this group. The nanocrystalline material found to show 3.32 gas response for 7 ppm NO_2 gas at 200º C operating temperature.[61] Another group has been worked on optical fiber senor based on the principle of evanescent wave (EW) absorption phenomenon. They studied the NO_2 gas sensing properties based on a coating of metallophthalocyanine (MPc) which is highly sensitive and selective to NO_2 gas.[62]

6 PSG Institute of Advanced Studies (PSGIAS), Coimbatore

The research group Dinesh et al. from PSGIAS, Coimbatore, India has been working in the field of gas sensor application based on the MOs and its composites. They prepared graphene oxide supported zinc stannate ($ZnSnO_4$) via hydrothermal method for gas sensing applications. This method successfully forms nanocubes morphology with 80 nm length. Based on this exotic morphology they studied the gas sensing properties of NO_2 gas at 30º C operating temperature. An enhancement in the gas sensing response at the low operating temperature after forming composite with graphene oxide is due to the spill-over effect.[63] Then, ultralong ZnO@Au heterojunction NRs having a diameter 60 ± 2 nm and several micrometer in length with uniformly distributed 'Au' cluster have been prepared by this group via wet chemical method. The physicochemical characterization of ZnO@Au heterojunction NRs have been studied by using different techniques. They found surface oxygen defects and zinc interstitials present at the interfaces of ZnO@Au heterojunction which is beneficial for gas sensing application. The gas response of 400 at 150℃ operating temperature based on ZnO@Au heterojunction has been studied by this group. Improvement in the NO_2 gas sensing response is might be due to the charge transfer process and spill over effect.[64] Then another group from same institute worked on NO_2 gas sensing application based on Au nanoclusters decorated TiO_2@Au heterojunction NRs and it was synthesized by using facile wet chemical method. Furthermore, they studied the NO_2 gas sensing properties of TiO_2@Au heterojunction NRs. They found the 250º C operating temperature of NO_2 gas for TiO_2@Au heterojunction NRs which is less as compared to the pristine TiO_2 gas sensor (400º C). Also, the sensor response of TiO_2@Au heterojunction NRs is linear and good for the trace level detection of NO_2 gas that is 500 ppb.[65] Some workers studied NO_2 gas sensing application based on mesoporous ZnO@Au heterojunction nanosphere which was synthesized by two-stage facile chemical method. The ZnO@Au mesoporous-spheres showed an extraordinary sensitivity and selectivity at a lower operating temperature of about 250° C as compared to pristine ZnO mesoporous-spheres (450° C) for NO_2 gas sensing application. An improvement in the NO_2 gas sensing properties of ZnO@Au mesoporous-spheres may be due to the synergetic effect of Au nanoclusters at the heterojunctions and increases the surface area and defects in the heterostructures.[66] Then, the same group reported novel nanograined ZnO/Au heterostructured nanofibers by using electrospinning method. The prepared nanofiber shows one dimensional nanostructures with more oxygen vacancies than pristine ZnO nanofibers. The ZnO/Au heterojunction nanofibers showed good gas response i.e. 98 and selectivity at low operating temperature of 350° C than pristine ZnO nanofibers (450° C). The enhanced sensing behavior of the heterostructured nanofibers are ascribed to the synergetic effect of Au nanoclusters at the interface which act as spill-over zone favoring physisorption and defect mediated sensing process.[67] Furthermore, they have been prepared the porous 1D n-p type ultra-long ZnO@Bi_2O_3 heterojunction NRs by using solvothermal method. The porous ZnO@Bi_2O_3 heterojunction NRs with high oxygen vacancies have been prepared by this group. The gas response of ZnO@Bi_2O_3 heterojunction NRs for NO_2 gas is 10 times higher than that of the pure ZnO sensor towards 500 ppb NO_2 gas concentration. This is good candidate for the detection of the NO_2 gas sensor for real time application.[68] Then, they studied the comparative study of different morphologies like nanospheres, NRs, ultralong NRs, and nanofibers of Au decorated ZnO nanostructures. Based on these morphologies they have studied the gas sensing properties of NO_2 gas at optimized temperature. They open a new area for the detection of NO_2 gas based on MOs.[69] Furthermore, they have been reported the 1D aligned gold nanoisland (GNI) anchored ZnO-based heterojunction nanofibers (HNFs) and their NO_2 gas sensing mechanism has been investigated by using operando photoluminescence studies. The nanofibers exhibited excellent sensor response (196 %) with rapid response time towards 500 ppb NO_2 gas at reduced operating temperature of about 200 °C.[70]

7 Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam

The research group from IGCAR working on nanostructured MOs based NO_2 gas sensing application prepared the Ba doped In_2O_3 via pulsed laser deposition (PLD) technique. They found the lower detection limit i.e. 0.5 ppm NO_x concentration in air at 300° C operating temperature.[71,72]

Another group from this institute have been working on MOs nanocomposites for NO_2 gas sensing application.[73] Firstly, they prepared the ZnO-TiO_2 nanocomposites by using wet chemical route. The structural, morphological properties of nanocomposites have been studied by using different techniques. This ZnO-TiO_2 composite reported 22 times higher NO_2 sensing response than the pure ZnO.[73] Then, NO_2 gas sensing can be carried out by using CuO NPs. The highest gas sensitivity for NO_2 was observed at 150° C operating temperature based on CuO NPs with 10-12 nm in size.[74] Then, Fe doped ZnO nanowires (NWs) have been synthesized via vapour transport method by another group. The 20-70 nm diameters of NWs have been reported by this group. They reported the NO_2 gas sensing properties at 150℃operating temperature.[75] Another report on the Al doped ZnO thin film by using Radio Frequency (RF) method is available. This material showed the defect density of electronic states of the Al^{3+} ion increases with an increase of RF power due to the increase in the thickness of the film and the crystallite size. The gas sensing performance of AZO films was studied for NO_2 at 350° C operating temperature. The sensing mechanisms of NO_2 gas has been discussed in details.[76] Then, Vanadium pentoxide (V_2O_5) have been prepared by pulsed dc magnetron sputtering. The layered structure with sharp edges of V_2O_5 has been confirmed by SEM technique. This V_2O_5 layered structure with sharp edges has more selectivity towards NO_2 gas at low operating temperature of around 50° C which is nearly equal to room temperature.[77]

8 Bharathiar University

The research group from Bharathiar University reported the $LaFeO_3$ nanostructure thin films synthesized via RF magnetron sputtering method. The effects of thickness and temperature on the structural and morphological properties of the $LaFeO_3$ nanostructures films have been studied in details. They found the nanocube like morphology with average size of 70 nm. Furthermore, they studied gas sensing properties of nanocube like morphology $LaFeO_3$ thin films. They reported the detection limit up to 1 ppm NO_2 gas at room temperature and the gas response of 29.60 was achieved.[78] Then another group from the same university worked on NO_2 gas sensing application. They reported the platinum doped indium oxide (Pt-In_2O_3) thin film which is prepared by surfactant assisted hydrothermal method. Furthermore, they studied the NO_2 gas sensing properties at 165° C operating temperature. The gas response was 2.5 for 5 ppm NO_2 gas concentration has been reported by this group.[79]

9 BARC, Mumbai, India

A group of workers from Bhabha Atomic Research Center (BARC), India with scientist N. Ramgir have furnished significant work in the field of gas sensing. This group focused on sensing hazardous gases like H_2S, NH_3, NO_2, CO, O_3, along with vapors of ethanol, acetone, and humidity by using functional MOs sensors. Ramgir et al. published more than 160+ research articles on MO based sensors out of which 12+ articles are solely on MO based NO_2 gas sensors [Source: Google Scholar]. Recently, Sinju et al.[80] developed a prototype of e-nose as multiple gas sensor based NWs of ZnO. They have modified the surface of ZnO NWs by using different sensitizers such as Ni, Cu, Au, and MgO. A post heating leads to form thin layer of heterojunctions namely ZnO-NiO, ZnO-CuO, ZnO-Au, and ZnO-MgO respectively on to the surface of NWs. The principal component analysis (PCA) along with 3D graphical analysis were used for the interpretation gas sensing results and it revealed that as prepared e-nose efficiently discriminate quantitatively as well as qualitatively between two gases H_2S and NO_2 have attempted surface modification of ZnO NWs prepared by using hydrothermal method by depositing a very thin layer of aluminium. This Al coated ZnO NWs (having 1.8 % Al) with thickness 5 nm showed high and selective response towards NO_2 gas in the low detection limit ranging from 0.5 to 5 ppm at operating temperature of about 230°C. Here, Al coating on to the surface leads to decrease in the resistance of ZnO NWs sensor film thereby improving sensor response towards NO_2 gas. The various significant attempts made by this group for the designing of MO based sensor materials for NO_2 gas sensing applications have been summarized in the Table 1.

Table 1. NO_2 gas sensing by using nanostructured MOs done by BARC scientists

MO sensor	**Sensor response**	**Detect limit (ppm)**	**Operating temp. (°C)**	**Year**	**Ref.**
CuO/ZnO composites	96 %	1	150	2021	[81]
Ni, Co, Mg, Fe deposited ZnO NWs	1.23 to 2.61 (Rg/Ra)	1	250	2020	[82]
Au incorporated ZnO NWs	41 %	1	200	2020	[83]
Zn doped CuO nanocrystal	34 %	1	150	2020	[84]
TiO_2/ZnO NW composite	7 (Rg/Ra)	5	200	2020	[85]
SnO_2/rGO composites	185 (Rg/Ra)	0.5	200	2020	[86]
SnO_2/rGO composites	4.56 (Rg/Ra)	0.5	200	2019	[87]
ZnO/CuO composite	175 %	1	150	2018	[88]
CuO nanocubes	76 %	1	150	2017	[89]

Meanwhile, the scientists from BARC including D. K. Aswal, Mameet Kaur, J. V. Yakmi, A. K. Debnath, S. K. Deshpande, and S. C. Gadkari have also contributed in the field of NO_2 gas sensing. Recently, yttrium doped SnO_2 thin film sensors were developed with varying wt. % composition of of Y from 1 to 3 % for the detection of NO_2 gas. Among different Y doped SnO_2 films, 3 wt. % Y doped SnO_2 (3YS) shown phenomenal increase in sensing response from 19 % to 1445 % towards sub-ppb (0.6 ppb) concentration of NO_2 gas. Furthermore, this sensor was highly reproducible with high stability.[90] Hybrid thin films of poly(3-hexylthiophene) (P3HT) and ZnO NWs were synthesized by drop-cast method. These films exhibited excellent selectivity towards NO_2 gas with fast response and recovery time at room temperature. The film with 1:1 weight ratio of P3HT and ZnO NW furnished good sensor response in the 0 to 10 ppm concentration of gas with high selectivity.[91] Gas sensors of ZnO material with diverse morphologies such as NWs and nanobelts developed by dielectrophoresis method showed enhanced sensitivity for sensing NO gas at room temperature. The sensor response for 40 ppm gas obtained is about 500 % with high response-recovery time.[92]

10 Other National Institutes

The work of NO_2 gas sensing by using MO based sensors done by several researchers working in the various reputed research and academic institution all over the India can be summarized in the Table 2.

Table 2. The work of NO_2 gas sensing by using nanostructured MOs based sensors done by several researchers all over the India. where, S is sensor response, DL is detection limit and T is the operating temperature.

MO sensor	S	DL (ppm)	T (°C)	Ref.
CuO/ZnO nanocomposites	96 %	1	150	[81]
Ni, Co, Mg, Fe deposited ZnO NWs	1.23 to 2.61 (Rg/Ra)	1	250	[82]
Au incorporated ZnO NWs	41 %	1	200	[83]
Zn doped CuO nanocrystals	34 %	1	150	[84]
TiO_2/ZnO NW composite	7 (Rg/Ra)	5	200	[85]
SnO_2/rGO nanocomposites	185 (Rg/Ra)	0.5	200	[86]
SnO_2/rGO nanocomposites	4.56 (Rg/Ra)	0.5	200	[87]
ZnO/CuO nanocomposite	175 %	1	150	[88]
CuO nanocubes	76 %	1	150	[89]

Challenges, Future prospectus and Conclusion

The standard living, high energy consumption and pollution of modern age is demanding a gas sensor with superior performance. The higher sensitivity, excellent specific gas selectivity, low operating temperature, and lower cost are the prime requirements of the sensor devices. In order to prepare desired gas sensor several strategies have been established. One of the promising strategies is the use of MOs. Ease of synthesis, lower cost, ability to sense various toxicants, simplistic sensing mechanism, higher gas response are the merits of semiconductor MOs based sensors. However, large response and recovery time as well as high operating temperature, issue of non-repeatability, stability and specific selectivity hurdles the commerciality of MO based gas sensors. In this scenario, special emphasis is needed right from selection of specific MO, synthesis route, followed by its role as sensing element. As we know that the fascinating morphologies of MOs plays very important part in gas sensing, because the gas sensing mechanism of chemiresitive type sensors mostly rely upon the adsorption-desorption of gases. The peculiar morphologies of MOs can offer efficient adsorption sites for the gas molecule interaction. There is challenge to develop morphologies suitable for higher gas sensing behavior. The high operating temperature is another major issue. However, recently researchers have developed a strategy to dope the MOs or composite them to work at lower temperature.

At the national level, a considerable research work was carried out, however, very few MOs were used for sensing NO_2 gas. The MOs such as ZnO, SnO_2, WO_3 and In_2O_3 are mainly utilized to fabricate the gas sensor. There is scope to explore another MOs as well as chalcogenides and composite to build an efficient gas sensor. In India, the research groups focused on the preparation of various morphologies of the MOs, as it helps to boost the sensing abilities. However, the research is limited up to the laboratory scale. The maximum gas sensing response of reported MO based gas sensor is far away from the commercial NO_2 sensor.

In the review report, we summarize the development of MOs to sense the NO_2 gas in India. Thousands of research articles were published on MOs as a gas sensor element and hundreds of research groups were presently working in this area. However, the aim of the present article is to explore the current developments of NO_2 gas sensors fabricated by using MO nanostructures within the country. Therefore, we reviewed research work of prominent groups within India.

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Conflict of Interest

Authors declared no conflict of interest.

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AUTHORS BIOGRAPHIES

Dr. S. A. Vanalakar is currently working at K. H. College, Gargoti, affiliated to Shivaji University, India as Head, Dept. of Physics. He worked as visiting scientist at Iowa State University, Ames (USA), Chonnam National University, Gwangju (South Korea) and Koc University, Istanbul (Turkey). He is ranked in World Top 2% Scientist published by Standford University, USA-2021. He ranked in Sci-Val Elsevier Ranking-2021. Also, he ranked in A. D. Scientific Ranking-2020 and 21. He is recipient of prestigious fellowship and awards like Raman, MCSA, Tubitak, etc. He has received about 2 crore INR as research grant from national and international funding agencies. His research interest lies in Solar Cells Materials, Materials Sciences, Nanotechnology, Thin Films, Gas Sensor, etc. He has published more than 80 research papers in various reputed peer reviewed International Journals in aforesaid research interest. He has presented more than 50 research papers in National/International Conferences and edited/published around 10 books on Basic and Applied Sciences. He is also an editorial board member and reviewer of various international journals.

Mr. Vithoba L. Patil is a doctoral student under the guidance of Prof. Pramod S. Patil, Department of Physics, Shivaji University, Kolhapur. He completed his master degree in 2013 in Physics at Shivaji University, Kolhapur. He has worked as a project fellow of a DST-sanctioned research project entitled, "synthesis and characterization of nanostructured metal oxide for gas sensor application". His main research interest is the synthesis of nanostructured metal oxides and its composites for gas sensing applications. He has published more than 50 research articles for well reputed journals. He received more than 1685 research citations for his research articles with h-index 22. He is also reviewer of various international journals.

Dr. S. M. Patil was born in Kolhapur, Maharashtra, India, in 1982. He received B. Sc., M. Sc., and Ph. D. degrees from Shivaji University Kolhapur in 2002, 2004 and 2019, respectively. He is currently working as an Associate Professor of Chemistry at Department of Chemistry, Karmaveer Hire College Gargoti, Kolhapur (Affiliated to Shivaji University Kolhapur). His research interest include nanomaterials synthesis, characterization and their applications in various fields like catalysis, gas sensing, photodegradation of hazardous compounds, biosensing, energy harvesting etc.

Ms S. B. Dhavale is a doctoral student at Department of Physics, Shivaji University Kolhapur. She has completed her M.Sc. degree in 2017 at Department of Physics Shivaji University Kolhapur. After that she has passed the UGC-NET JRF exam with ranking in India. Her main research interest is the synthesis of metal oxides for Super capacitors, Gas sensors etc. She has published more than 05 research papers in various reputed peer reviewed International Journals.

Dr. Tukaram D. Dongale is working as an Assistant Professor at the School of Nanoscience and Biotechnology, Shivaji University, Kolhapur. He did his Bachelors and Masters in the Electronics specialized in Embedded Systems and earned a Ph.D. degree from Shivaji University, Kolhapur. Furthermore, he has completed post-doctoral research work at Korea University, South Korea in the field of high-density resistive memory and synaptic devices. He has cleared SET and NET-JRF (Electronic Science) exams during his M.Sc. II year itself. He has established a research group with young and dynamic graduate and undergraduate students. His research group is working on the interface between Electronic Engineering and Material Science. His research group focuses on the synthesis, characterization, and development of micro and nanoscale devices for resistive memory, neuromorphic computing, sensor, and energy applications. His research group is currently exploring the growth and electronic properties of functional nanomaterials and thin films. Together with his students, he has published more than 95 SCI research articles and filed 03 Indian patents. Recently, he has elected as a Young Associate of Maharashtra Academy of Sciences, India.

Prof. (Dr.) Pramod Shankararao Patil is currently working as a Pro-Vice Chancellor and Senior Professor at Department of Physics Shivaji University Kolhapur. He is former Founder/Director of School of Nanoscience and Technology, and Former Head of Department of Physics Shivaji University, Kolhapur. Also, he is former Coordinator of Department of Technology and Energy Technology, Shivaji University, Kolhapur. He is former Dean of Science & Technology and he is recipient of DAAD and brain pool Fellow. He Ranked 391st in World Top 2% Scientist by Standford University, USA-2020. He Ranked 99th in India and 5889 th in world by A. D. Scientific Ranking-2021.He Ranked 2 nd in India and 147 th in world by Sci-Val Elsevier Ranking-2021. He is recipient of best teacher award in 2015. Prof. Patil received his Ph. D. degree from Shivaji University Kolhapur, India in 1990. He has been continuously engaged in the research filed more than last 30 years. His research interest is in Solar cells, Gas sensors, Nanotextiles, Electrochromism, Anitmicobial and Supercapacitors. He has published more than 475 research articles for well reputed journals. He received more than 23669 research citations for his research articles with h-index 76 and i-10 is 473.