ZnO-CuO nanomaterials based efficient multi-functional sensor for simultaneous detection of humidity and mechanical pressure

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Abstract

Designing of materials is a critical factor for sensitive, stable and practically applicable humidity sensor, herein we report synthesis of ZnO-CuO mixed oxide nanoparticles and their fabrication into nanocomposite with silicon adhesive as a cost-effective humidity sensor with reliable sensitivity and stability. Between the gap of pre-fixed copper electrodes on the glassy substrate, the prepared binary oxides nanoparticles of ZnO-CuO and its silicon adhesive nanocomposites were deposited for relative humidity (RH) and mechanical pressure detection as multimodal sensor. The fabricated sensor was utilized for sensing assessment of humidity and pressure which responded in impedimetric measurements of relative humidity and pressure. The impedance of the pristine mixed oxides of ZnO-CuO decreased 35 times as relative humidity increased from 10-90% RH while 24 times decrease was observed in nanocomposite sample. Capacitances increased by a factor of 125 in pristine mixed oxides of ZnO-CuO and 80 times in nanocomposite at 100 Hz working frequency with the relative humidity (RH) increase in 10 to 90% RH range. The impedance of the sensors displayed strong dependence on the mechanical pressure as well. Similarly, the impedance of the material decreased in pristine samples by 3.8 times when a mechanical pressure of 11.0 KN/m$^2$ was applied on the fabricated sensor while capacitance in pristine samples increased by 4.61 times at the same pressure. In the composite samples impedance decreased 1.38 times and capacitance increased under effect of the pressure accordingly on 1.47 times.

Introduction

Recently great attention has been paid towards the development of multifunctional sensors which can sense different environmental parameters simultaneously for electronic devices, food preservation, day-to-day life and environmental applications [1-3]. Sensing various parameters selectively in environment like temperature, relative humidity and pressure make the sensors ideal. The main reason which attracts the researchers’ interest in the field of new multifunctional sensors development is because most of the developed sensors have limitations where there is simultaneous variation of different environmental parameters [1-3], especially, humidity sensors which have several limitations like temperature, pressure and range of humidity. On the other side, humidity sensor is very crucial for the detection and monitoring of humidity because it has great influence in the areas of pharmaceutical production, hospitals, food preservation, engineering accurate electronic devices, living organisms and environment [1-3]. A very tiny amount of humidity greatly affects the materials, electronics and environment etc. Therefore, recognition, detecting, and monitoring of humidity are of great importance for different natural and artificial materials as humidity drastically affect these materials. Therefore continuous monitoring, precise measurement and controlling of humidity in environment are important. Drawbacks like limited use of the sensors at highly humid environment are associated with optical humidity sensors although optical humidity sensors have been developed and successfully demonstrated by the researchers which can investigate and measure relative humidity and pressure simultaneously [1-3]. The drawback is usually observed in real-time variations in pressure and analyte concentration making its application limited [1-3]. Therefore,
multifunctional humidity sensors are of great importance for the detection of humidity at different conditions and different environmental parameters, specifically at different pressure. Hence, development of efficient humidity is of noticeable significance for monitoring and controlling of humidity even with pressure variation [4-6].

Several active sensing materials including metal oxide nanoparticles, semiconductors, polymers and hybrid composites have been fabricated successfully in commercially available humidity sensors [7-13]. Nanoparticles based humidity sensors have proved their multimodal functionality with higher sensitivity and diversity in structure, therefore metal oxides nanomaterials have been utilized in several every day and industrial monitoring sensors [14-21]. Molecular size and structures of metal oxide nanomaterials determine the performance of the sensors, making the choice of metal oxide based nanomaterial important in all kinds of sensors using different working principle. Numerous properties like sensitivity in a wide range of measuring analyte, short response and recovery times, stability (both chemically and physically) and inerntness towards other chemicals are the key determinants of the suitability of the materials utilized as sensing elements in the sensors. Several kinds of metal oxide nanomaterials and their nanocomposites have been used as sensing elements in the humidity sensors, like CuO, TiO$_2$, SnO$_2$, ZnO, Fe$_2$O$_3$, CeO$_2$-Co$_3$O$_4$, Co$_3$O$_4$, CeO$_2$/SnO$_2$, ZnO/TiO$_2$, Fe$_2$O$_3$-Co$_3$O$_4$, SnO$_2$/ZnO, LaFeO$_3$, SnO$_2$–Co$_3$O$_4$ etc. [7-21]. Most of the reported sensors have drawbacks associated with them like poor chemical and physical stability and long response/recovery times. Shape of the nanomaterial sensor e.g. nanowires, nanoboxes, nanorods and nanoflowers etc. could further improve their sensing performances. Properties like thermal, chemical and physical stability as well as higher sensitivity and resistance to poisonous chemicals make porous ceramic nanomaterials and metal oxide nanomaterials suitable for active sensing elements for humidity sensors [7-21]. Generally, adsorption of humidity (H$_2$O molecules) on sensing material's surface cause change in the electric signal and this adsorption of humidity strongly depend on the surface area as well as porosity of the sensing materials. The adsorption of humidity can be varied by variation in surface area of the sensing materials. As nanostructure materials have high surface area and have more active sites exposed to humidity [10-12], causing enhancement of the electrical signal by changing the intensity of charge carriers, piezoelectric output and screening effect [11-15].

Another property of metal oxide semiconductors is the increase conduction with the decrease in thermoelectric coefficient with increase of pressure and can be used for the fabrication of pressure transducers [13, 20, 22]. To overcome these issues and develop new humidity and pressure sensor capable of excellent output performance, our research group intended to investigate sensing properties of ZnO-CuO based nanomaterials’ and their silicone adhesive nanocomposites for measuring humidity and pressure.

In the present work, a multimodal relative humidity and pressure sensor is described based on the ZnO-CuO nanomaterial and its composite with silicon adhesive. Metal oxide nanomaterials have the advantages of sensing applications for environmental monitoring due to its economic synthesis and processing [12, 15, 22] with quick response in humidity sensing [10-12, 22], pressure sensing [13, 20] and...
temperature monitoring [14, 16, 17]. In the present work we investigated ZnO-CuO (both pristine and its silicone adhesive nanocomposites) as an active sensing element for sensing relative humidity and pressure.

2. Experimental

2.1. Materials

All chemicals like salts of tin and manganese nitrate (Sigma Aldrich), tin chloride (Sigma Aldrich), 99% ethanol C$_2$H$_5$OH (Sigma Aldrich), sodium hydroxide (Sigma Aldrich) and other chemicals were purchased of analytical grade from Sigma Aldrich and were used without further purification.

2.2. Preparation of ZnO-CuO nanomaterials and of their nanocomposite with silicone adhesive

Zinc and copper nitrates were dissolved in distilled water in same molar ratio (0.1M:0.1M) and increased the pH (pH = 10) of the mixed salts solution by drop wise addition of 0.5 M NaOH solution. The highly basic solution was kept on heating plate for continuous stirring at 60 ºC. After 6 hours the hot solution was cooled down and the precipitate of mixed oxides was collected by discarding the supernatant. The precipitate was washed several times with water-ethanol mixture in order to remove any impurity as well as unreacted precursors. The cleaned precipitate was transferred to oven for drying at 50 ºC followed by grinding into fine powders. The powder product was further calcined at 500 ºC for five hours. The calcined powdered nanomaterial was stored in inert plastic vials which were pre-cleaned and dried for characterization and application purpose.

The above stored final product was mixed in 1:1 weight ratio with silicone adhesive for nanocomposite preparation and was fabricated for measuring relative humidity.

2.3. Characterization:

JEOL Scanning Electron Microscope (JSM-7600F, Japan) studies were carried out for morphology investigation while computer controlled ARL service diffractometer was used for crystal structure studies of the synthesized nanomaterials. FT-IR spectrum in the range of 400 to 4000 cm$^{-1}$ was recorded by PerkinElmer (spectrum 100) FT-IR spectrometer.

2.4. Fabrication of humidity sensor

Space between preliminary deposited copper electrodes on glass substrate by vacuum evaporation method was filled with nanocomposite by drop casting method and was dried at 40 ºC for 4-5 hours (Figure 1). The distance between the 6-10 mm wide copper electrodes was found about 30-40 µm and thickness of the nanocomposites was found 30 µm by measuring with the help of micrometer. A second sensor with the same thickness of sensing element and gap of copper electrodes as stated in previous sensor was prepared by the pristine ZnO-CuO nanomaterial encapsulated in porous membrane and was utilized without any additional treatment.
Figure 1 describes the plan of the sensors used for testing the relative humidity sensation in humidity testing chamber which we developed in our material testing laboratory. Humidity chamber used for humidity sensing is shown in Figure 2, in which humidity level is raised by introducing humidity by bubbling nitrogen gas in water chamber and lowered by purging dry nitrogen gas in the chamber. Humidity in the humidity chamber is measured using TECPEL 322 Humidity-Temperature meter while MT 4090 LCR Meter is used for measuring impedance and capacitance. Both measurement of humidity and pressure were carried out at 21 °C.

2.5. Fabrication of pressure sensor

Pristine ZnO-CuO nanomaterial and its silicone adhesive nanocomposite was fabricated for pressure sensing measuring impedance and capacitance with variation in pressure as shown in schematic diagram (Figure 3). 0.5 mm thick layer of pristine ZnO-CuO nanomaterial and its composite with silicone adhesive was sandwiched between two stainless steel plates, one the base and the other piston in a 10 mm cylinder. The cylinder was cover by rubber ring so as to minimize effects of dust and humidity from the environment. When pressure was applied on the upper piston stainless steel cover, the sample was pressed resulting variation in the impedance and capacitance in the encapsulated nanomaterial or nanocomposite layer. The pressure load was applied in the range of 0-11 KNm$^{-2}$, while variation in impedance and capacitance was measured using MT 4090 LCR meter (MOTECH) at 100 Hz working frequency.

Detailed arrangement and function is described in our previous article [23] and the schematic diagram is given in Figure 4.

3. Results And Discussion

3.1.1. Morphology studies (FESEM)

The morphology of ZnO-CuO mixed oxides were analyzed by FESEM and the images are shown in Figure 5 (a, b). FESEM images are very clear and indicate that the morphology of ZnO-CuO mixed oxides looks like rods made of sheets. It is not complete rod nor sheets. It gives outlook like rods and sheets. This indicates that the materials initially grown in the form of sheets and further several sheets merged or aggregate together and gave rods like morphology. It is clear from FESEM, that ZnO-CuO mixed oxides is grown in irregular, dispersed and different size of rods whose diameter is in the range of 80 -300 nm.

3.1.2. Energy dispersive spectroscopy (EDS)

The elemental composition of the ZnO-CuO mixed oxides was investigated by analyzing EDS. EDS spectrum (Figure 6) of ZnO-CuO mixed oxides clearly shows peaks at 0.5, 0.9, 8.0, 8.6 and 8.9 keV which are related to O, Zn and Cu. Peaks for Zn, Cu and O present in the EDS spectrum, verifies the existence of zinc, copper and oxygen in the mixed oxides nanorods. Further EDS analysis shows almost same weight % composition which suggest that Zn and Cu exist almost in quantity. The EDS spectrum does not show
other peaks related to impurities which suggest that the synthesized mixed oxides nanorods are composed of ZnO-CuO.

### 3.1.3. Phase and compositional study (XRD)

The XRD pattern obtained for the synthesized product showed diffraction lines which correspond to both ZnO and CuO (Figure 7). Fourteen diffraction lines were identified for synthesized product (ZnO-CuO). The 7 diffraction lines were observed with strong intensities (100, 002, 101, 102, 110, 103, 200, 112, 201 and 004) were used for further crystallographic characterization. The superstructure lines related to ZnO are clearly visible and these superstructure diffraction lines appear in the XRD pattern at 2θ = 31.89, 34.46, 36.40, 47.71, 56.64, 63.01, 66.41, 68.09, 69.29, and 72.69 and a face centered cubic structure can be indexed for ZnO [24]. The XRD pattern also observed a fairly large number of diffraction lines with poor or very poor intensities in the synthesized product. The diffraction lines with low intensities (marked with the *) present in the XRD patterns of the synthesized product are due to the presence of CuO which suggests that the prepared nanomaterial also comprise CuO. Peaks beyond ZnO in the XRD spectrum are diffraction lines which have poor intensities (marked with the *) and are assigned to CuO [25]. Some of the peaks related to CuO are overlapped with ZnO peaks. XRD pattern contains diffraction peaks for both ZnO and CuO which suggest that the prepared nanomaterial is made of zinc and copper mixed oxide. Thus, it is concluded that the synthesized ZnO-CuO nanomaterial contained two different oxide nanomaterials.

### 3.1.4. Compositional study (XPS)

In order to investigate the chemical composition of ZnO-CuO mixed oxides, XPS analysis was conducted for the prepared nanomaterial, as illustrated in Figure 8. The XPS spectrum displays clear peaks for O, Cu and Zn, indicating that the synthesized mixed oxides are composed of Cu, Zn and O. The XPS spectrum shows a peak at 532.3 which is related to oxygen. This peak specified the existence of oxygen in the synthesized product [26]. Further the XPS spectrum exhibit peaks for Cu and Zn which appeared at 935.0 (Cu 2p$_{3/2}$), 954.6 (Cu 2p$_{1/2}$), 1023.1 (Zn 2p$_{3/2}$) and 1046.3 (Zn 2p$_{1/2}$) eV. Thus the peaks of both Cu 2p$_{3/2}$ and Cu 2p$_{1/2}$ binding energy for the synthesized product were found to be around 935.0 and 954.6 eV, respectively. These findings were well-agreed with the CuO in the literature [26]. From Figure 8, it is obvious that the peaks related to Zn 2p$_{3/2}$ and Zn 2p$_{1/2}$ binding energy of synthesized product existed at 1046.3 and 1023.4 eV, respectively and the obtained results were well matched with the literature [27]. XPS compositional analyses revealed that the synthesized product contains both ZnO and CuO and these two phases co-exist together as ZnO-CuO nanomaterials. The overall results of XPS are consistent with the XRD data.

### 3.2. Humidity sensing performance

Figure 9(a,b) shows the impedance response curves of the sensors as a function of RH. The impedance response decreases as the humidity increases. The impedance shift of the sensor with pristine ZnO-CuO (Figure 9a) is evidently higher as compared to the sensor with ZnO-CuO nanocomposite (Figure 9b).
These results indicate that the response of the sensor with pristine ZnO-CuO is significantly larger than that of the sensor with ZnO-CuO nanocomposite. This highly enhanced sensing behavior of the pristine ZnO-CuO sensor can be attributed to the presence of large number of water adsorption sites which cause high sensitivity. The relationship of impedance calculated according to relative humidity of pristine ZnO-CuO nanomaterial at 100 Hz. The graph illustrates that impedance is showing inverse relationship with humidity. The sensor with pristine ZnO-CuO exhibited 35 times decrease in impedance with increase in relative humidity in the range of 10-90% RH, while sensor with ZnO-CuO nanocomposite shows 24 times decrease in impedance.

Figure 9(c,d) exhibits the capacitance relationship with variation in relative humidity level from 10-90 % RH at 100 Hz working frequency for pristine ZnO-CuO and ZnO-CuO nanocomposite. 125 fold increases in capacitance was found in pristine ZnO-CuO (Figure 9c) and 80 times in ZnO-CuO nanocomposite (Figure 9d) at 100 Hz as humidity level was increased from 10% RH to 90% RH.

Recovery and response time of the pristine ZnO-CuO were determined by changing the relative humidity level 50% to 80% RH for measuring impedance and found to be 17 and 39 seconds, respectively. Whereas for ZnO-CuO nanocomposite, recovery and response time at same conditions were 21 and 47 seconds, respectively. On the other hand, by measuring the capacitance, response and recovery times were equal to 19 and 41 seconds for pristine nanomaterial sensors and 23 and 45 seconds for nanocomposite sensors.

Scanning of pristine ZnO-CuO sensor and its silicone adhesive nanocomposite provided the base for increasing capacitance and decreasing impedance with increasing value of relative humidity which were calculated as shown in the Figure [3, 18, 28].

In order to explain the relationship between increasing the relative humidity and decreasing the impedance or increasing the capacitance from electronic point of view, we can use the following equation [3, 15, 29].

\[ Z = \frac{R}{1 + j \omega R C} \]  

where \( \omega \) stand for circular frequency

Hence, we can observe that the resistance decreased greater than increased the amount of capacitance by increasing the relative humidity impedance. An increase in the polarizability like dipolar \( \alpha_{dip} \), ionic \( \alpha_i \) and electronic \( \alpha_e \) polarizability enhances the capacitance [3, 15-18, 29]. Dipolar polarizability \( \alpha_{dip} \) is increased due to contribution from water molecules on the surface of nanoparticles enhances as well as displacement of orbital electrons also enhances the electronic polarizability. Similarly the creation of charge transfer complexes by the combination of ZnO-CuO nanocomposite and ionic polarization may be developed due to nanomaterials.

Furthermore, resultant increase in capacitance with enhancement of relative humidity can be interpreted by absorption of water molecules on the nanocomposite surface, which raises the dielectric permittivity.
of the nanocomposite owing to its higher dielectric permittivity value. Doping of water molecule on the surface of nanomaterial displace current resulting an increase in the capacitance. So the increase in charge carriers leads to increasing the polarizability. These mechanisms are explained in detail in the reported literature [15, 18, 22].

The sensitivity ($S_z$ and $S_c$) is an important parameter that can explain the increase in capacitance and fall in impedance with increasing the RH, which is given as follows [15, 18, 22].

\[
S_z = \frac{\Delta Z}{\Delta R_H} \quad (2)
\]

\[
S_c = \frac{\Delta C}{\Delta R_H} \quad (3)
\]

where $\Delta Z$, $\Delta C$ and $\Delta R_H$ refers to the variation in impedance, capacitance and relative humidity. Average $S_z$ was calculated and was found 0.55 MΩ/% and 7.19 kΩ/%, whereas the value of $S_c$ was 6.2 pF/% and 11.88 nF/% in case of pristine ZnO-CuO nanomaterial and nanocomposite, respectively.

These variations in impedance and capacitance with respect to alteration in relative humidity suggested that ZnO-CuO is a suitable candidate for the fabrication of humidity sensors, which can be applied in humidity meter in order to monitor humidity in the environment. Sensitivity of the humidity meter can be enhanced by measuring the impedance change in 10-50% RH range and capacitance in 50-90% RH range. The results conclude that pristine ZnO-CuO is more sensitive towards humidity at measurement of impedance comparing with ZnO-CuO nanocomposite. Whereas the nanocomposite is more sensitive to humidity at measurement of capacitance.

### 3.3. Pressure sensing performance

The relationship of pressure with impedance and capacitance for pristine ZnO-CuO nanomaterial (Figure 10a, 9c) and ZnO-CuO nanocomposite (Figure 10b, 10d) are illustrated in Fig. 9. It is obvious from the graphs that increase in pressure, impedance decreases while capacitance increases with increase in pressure. For pristine ZnO-CuO nanomaterials, average decrease in impedance of 3.8 times and average increase in capacitance of 4.6 times was calculated as pressure was increased from 0-11 KNm$^{-2}$. Similarly for ZnO-CuO silicone adhesive nanocomposite, average decrease in impedance of 1.38 times and average increase in capacitance of 1.47 times was calculated as pressure was increased from 0-11 KNm$^{-2}$.

The acquired results using ZnO-CuO pressure sensor can be interpreted based on the following ways: (i) Under the influence of the pressure inter particle distances decreased and cross-section area of their contacts increased that will lead to the decrease of the “micro” resistances and increase the “micro” capacitances. (ii) More detail explanation and quantitative calculations can be made on the base of percolation theory described in [20-22]:

\[
\sigma = \frac{1}{L Z} \quad (4)
\]
where $L$ is characteristic length, on the basis of the concentration of the sites, $Z$ is the resistance (or impedance at use of AC) of the path with the lowest average resistance. Regarding to sensor designed by ZnO-CuO material, it can be concluded that $L$ and $Z$ can be decrease by increasing the pressure, thus increasing conductivity and decreasing resistance and impedance of the samples.

4. Conclusions

ZnO-CuO nanomaterials were synthesized via low temperature method and characterized using different spectroscopic techniques. The synthesized ZnO-CuO nanomaterials and their silicon adhesive nanocomposites were fabricated to investigate for sensing capabilities of relative humidity and pressure. An increase in the amount of relative humidity increased the capacitance ($C$) and decreased the impedance ($Z$) due to enhancement of displacement current and charge carriers by adsorption of water molecules of the fabricated samples. Similarly, an increase in pressure results rise in capacitance ($C$) and drop in impedance ($Z$) in the prepared pressure samples. From the above discussion it can be concluded that variation in capacitance and impedance with change in relative humidity and pressure, pristine ZnO-CuO and silicone adhesive nanocomposite can prove a utilitarian material for sensing relative humidity and pressure in multimodal sensor.

Declarations

Acknowledgments

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**Figures**
Figure 1

Schematic diagram of the surface type ZnO-CuO based humidity sensor

Figure 2

Humidity sensing chamber
Figure 3

Schematic diagram of testing of pressure sensor
Figure 4

Schematic diagram of Pressure Sensing Assembly
Figure 5

FESEM images of ZnO-CuO nanomaterials.

| Element | Weight% | Atomic% |
|---------|---------|---------|
| O K     | 27.26   | 60.17   |
| Cu L    | 35.40   | 19.67   |
| Zn L    | 37.34   | 20.17   |
| Totals  | 100.00  |         |

Figure 6

EDS spectrum of ZnO-CuO nanomaterials
Figure 7

Powder XRD patterns of ZnO-CuO nanomaterials
Figure 8

Powder XPS patterns of ZnO-CuO nanomaterials
Figure 9

Relationship of Impedance with relative humidity for pristine ZnO-CuO (a) and ZnO-CuO nanocomposite (b), relationships of capacitance versus humidity for pristine ZnO-CuO (c) and ZnO-CuO nanocomposite (d) sensors at 100 Hz, respectively

Figure 10

Pressure Vs Impedance graph of pristine ZnO-CuO (a) and ZnO-CuO nanocomposite (b) and pressure Vs Capacitance graph of pristine ZnO-CuO (c) and ZnO-CuO nanocomposite (d) sensors at 100 Hz respectively