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A Review of Zinc Oxide (ZnO) Nanostructure Based Humidity Sensor

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Abstract

Numerous metal oxide nanomaterials, such as titanium dioxide (TiO_2), tin oxide (SnO_2), and zinc oxide (ZnO), are highly suitable for the fabrication of effective humidity sensors. Comparatively, ZnO is considered as metal oxide with the highest potential due to its unique properties, such as the enormous excitation binding energy of 60 meV, a direct wide bandgap (3.37eV), and the ability to be synthesized and grown at low temperatures. To further enhance the sensing performance of ZnO structure for humidity sensing, parameters such as the morphology and crystallinity of the ZnO structure can be optimized through control of synthesis conditions, such as precursor concentration, reaction time, temperature, and pH. Although various fabrication and characterization of ZnO nanostructures composited with other metal oxides have been published, there are insufficient investigations that highlight the performance of humidity sensors using ZnO alone. Therefore, this study provides a comprehensive analysis of the application of ZnO nanostructure in developing humidity sensors. The discussion in this review includes a summary of the recent development of humidity sensors and the parameters used to measure their sensing performances such as doping and compounding method. The study also highlighted the unique features of ZnO and the numerous methods used to synthesis ZnO, including sol-gel immersion, two-step solution, hydrothermal synthesis, and spin-coating process. In short, the intriguing development of ZnO-based humidity sensors would offer an alternative option to employ effective humidity-sensing devices in thin-film solar cells and ultraviolet (UV)-based applications.

Keywords: *Nanotechnology, Metal oxide, Relative humidity, Sensitivity, Methods.*

1. INRODUCTION

Humidity sensors serve a crucial function in a diverse range of industries and numerous applications, and their market potential has recently contributed a substantial portion to the global economy [1-2]. The performance of humidity sensors has dramatically enhanced over the years in various industrial applications, including the manufacturing of semiconductors [3-5], such as controlling the humidity in wafer processing sectors [6], in the medical sector [3-5], such as monitoring human respiration in ventilators, incubators, and biological products [7] and in the agricultural sector [3-5], such as monitoring soil moisture [7]. A humidity sensor assists in determining the moisture content in the air since it quantifies and reports the data using relative and absolute humidity values. An ideal humidity sensor should possess high sensitivity, low hysteresis, thermal stability, quick recovery, and response time [1, 8]. To date, various humidity sensor configurations have been developed, including humidity-sensitive resistors and humidity capacitors.

Metal oxides are the ideal nanomaterials for the advancements and fabrication of exceptional humidity sensors with the desirable features, as stated earlier, due to their extensive specific surface area [1-2, 8]. A huge surface area is required to produce a suitable sensing device [3], as the large surface area offers greater surface contact with the surrounding area, boosting the rate of reactivity. In addition to the ability of nanostructured materials to produce more effective catalysts [30], a large surface area also allows the target compound to be more exposed and easily detected, even at low concentrations. Thus, a larger surface area produces a more efficient and powerful nanostructured material.

Among the various metal oxide materials that are highly appropriate to fabricate high-quality humidity sensors are titanium dioxide (TiO_2), tin oxide (SnO_2), and zinc oxide (ZnO) [1, 5, 8], with the latter being the top candidate [3-4, 8-9]. According to past literature, ZnO is a metal oxide with great potential for many applications, such as electronics, humidity sensors, ultraviolet photoconduction, and ethanol sensors [10,13]. The broad utilization of ZnO in numerous technical domains is thanks to its outstanding characteristics, including a high excitation binding energy of 60 meV, straight wide bandgap (3.37 eV) [3-5, 9-10, 30], inherently non-toxic and affordable [1, 5, 9, 12], and able to be developed at low-temperature conditions [1, 4, 11].

Different synthesis methods of ZnO have been proposed, such as sol-gel immersion, two-step solution, hydrothermal synthesis, and spin-coating [5, 30]. Several researchers introduced other parameters in their synthesis procedure, such as doping with aluminum (Al), copper (Cu), or indium (In) [9, 12, 14], to enhance the nanostructure of ZnO and thus its efficacy as a humidity sensor [1, 5, 9, 11]. The addition of doping elements significantly impacted the efficiency of ZnO as a humidity sensor and its concentration, surface morphology, structural, electrical, and optical features [10, 15]. Therefore, this study presents a comprehensive review of the recent findings on the synthesis of ZnO.

2. HUMIDITY SENSING ANALYSIS

Humidity is defined as the concentration of water vapor in the air or other related gases. It can be expressed in various ways, where the expression unit varies depending on the measurement technique applied. Some of the commonly used expressions include “Relative Humidity” (RH) and “dew/frost point” (D/F PT). Depending on the unit of measurement, humidity sensors are categorized as RH or absolute humidity (moisture) sensors.

Relative Humidity refers to the ratio of water vapor in a water-air mixture relative to the maximum (saturated) moisture content it can withstand at a given pressure and temperature [16]. RH is a relative measurement since it depends on the temperature. An RH sensor measures the RH and is expressed as a percentage using Eq. (1) [6, 17-19]:

Relative Humidity (RH):

$$S = \left(\frac{R_a - R_{rh} \times 100}{R_a}\right) \qquad (1)$$

where:

S → Sensitivity

R_a → Resistance at a minimum humidity level

R_{rh} → Resistance of the elevated humidity

The current values of the response curves from Ohm’s law (V = IR) are used to calculate the resistance value [1]. In addition, the sensitivity value derived from the I-V measurements is utilized to assess the performance of the humidity sensor, as illustrated by Eq (2) [2]:

$$S = \frac{I_{90}\ RH\%}{I_{40}\ RH\%} \qquad (2)$$

where

S → Sensitivity

I_{40}RH% → The sensor’s current under dry conditions of 40 RH%
I_{90}RH% → The sensor’s current at 90 RH%

Humidity sensor performance is determined by two critical parameters: reaction (adsorption) and recovery time (desorption), and sensitivity (S). The time taken that was required to stabilize the total transient current and reach 90% of the total resistance change after the humidity level is adjusted from a low RH (40%) to a high RH (90%) environment is referred to as reaction time. The recovery time, on the other hand, is determined by changing the humidity level from a high RH (90%) to a low RH (40%) environment.

It is necessary to use materials with a large surface area in order to enhance the effectiveness of humidity sensors. This is because larger surface areas allow for more surface contact between water molecules and the detecting element, resulting in a higher carrier density and a greater number of carriers, enhancing the humidity sensor's sensitivity [20-22]. Accordingly, it has been reported that using materials with a large surface area can increase the adsorption of water molecules on the surface of the sensor [20].

Furthermore, the electrostatic force from the high charge carrier density is strong enough to break one of the water (H_2O) molecules' hydrogen bonds (O-H) and form a stable chemical bond between the metal (M^+) and the hydroxyl ions (OH^-)[3]. In general, protons (H^+) and OH^- are assumed to be rapidly dispersed due to surface collision or self-ionization of water molecules, resulting in an initial ion separation (H^+, OH^-) based on Eq. (3):

$$H_2O <=> H^+ + OH^- \quad (3)$$

At low humidity, proton hopping between chemisorbed OH^- generates the conductivity. However, the electrostatic field can ionize the physisorbed water molecules at higher humidity and produces a huge amount of hydronium ions (H_3O^+) as charge carriers. The primary source of charge carriers is protons produced during the hydration of H_3O^+. At higher humidity levels, proton hopping between neighboring water molecules increases, and the charge carrier transit obeys the Grotthuss mechanism [5, 20].

Notably, when relative humidity (RH) rises, so does the sensitivity of the sensor. The proton conduction responses at low (LH) and high (HH) humidity levels are expressed using Eqs. (4) and (5) [5, 20]:

$$H_2O \Leftrightarrow H^+ + OH^- \text{ (LH)} \quad (4)$$

$$2H_2O \Leftrightarrow H_2O^+ + OH^- \text{ (HH)} \quad (5)$$

The principle operation of a ZnO-based humidity sensor is based on the Grotthuss chain mechanism [23-24], as shown in Figure 1, where the reaction causes water molecules to bind to the ZnO surface, as expressed in Eqs. (6) and (7) [5, 15, 21-22]:

$$2H_2O \rightarrow H_3O^+ + OH^- \quad (6)$$
$$H_3O+ \rightarrow H_2O + H^+ \quad (7)$$

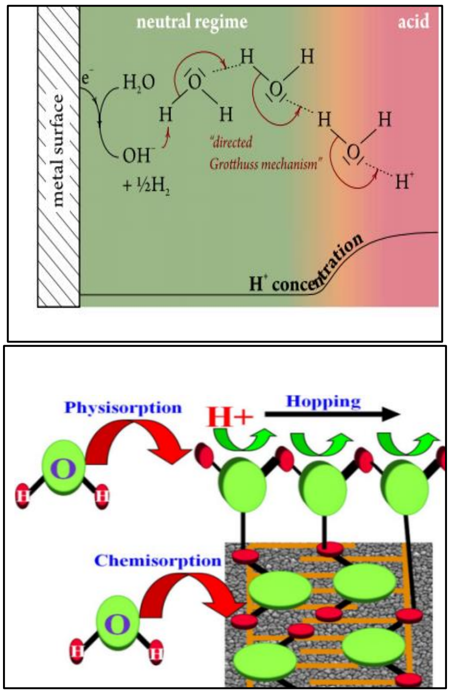

Figure 1. *Grotthuss mechanism in a ZnO-based humidity sensor* [23-24].

3. HUMIDITY SENSOR USING VARIOUS SEMICONDUCTING MATERIALS

Recently, the demand and popularity of humidity sensors have increased, especially in factories, food processing plants, medical facilities, and chemical storage facilities [7, 15, 24]. In view of this, it is vital to fabricate temperature-stable sensors with high sensitivity to changes in ambient humidity. It is necessary to have a large surface area in order to produce good humidity sensors as it is one of the primary criteria that determines how well humidity sensors operate [22]. The large surface area and high electron concentration assist in detecting the moisture level by providing a more significant number of active sites for water molecules to be adsorbed.

Over the past years, many researchers have developed various sensing devices based on nanostructured metal oxide semiconductors due to their superior chemically reactive surface, mechanical endurance, and thermal stability. Additionally, recent breakthroughs have significantly increased the performance of humidity-sensing devices. Researchers have also successfully fabricated humidity sensors using different types of semiconductors. Li *et. al*. [1], for example, used the solvothermal method to create a high-performance ZnO/SnO_2-based humidity sensor. Complex Impedance Spectrum was used to examine the humidity-sensing mechanism (CIS). The ZnO/SnO2 composite had a relatively large specific surface area, which enabled it to take in a lot of moisture, boosting its sensor performance. Consequently, the ZnO/SnO_2 humidity sensor achieved a higher and more rapid response and recovery speed, lower hysteresis between 11% and 95% RH, and improved linearity. Thus, the ZnO/SnO_2 humidity sensor analysis offered a novel strategy for developing high-performance humidity sensors.

Furthermore, Saidi *et. al.* [13] employed the spin-coating approach to developing a humidity sensor with aluminium (Al)-doped ZnO-coated films at room temperature. The study assessed the surface area and nanoporous architectures in order to comprehend the sensor's sensing mechanism. Experiments revealed that the sensitivity of the Al-doped ZnO-coated film was much greater than that of the Al-doped ZnO nanorod arrays [13], demonstrating the considerable potential of Al-doped ZnO-coated films in humidity sensor applications.

Besides, Algun *et. al.* [15] evaluated the properties of humidity sensors comprised of co-doped Al and fluorine (F) ZnO (AFZO) nanostructured thin films with varying Al concentrations. AFZO nanostructured thin films produced by the sol-gel technique (both for synthesis and coating) demonstrated uniform and homogeneous surfaces with a preferred orientation along the (002) plane [15]. In addition, as the concentration of Al grew, the grain size decreased. In short, the findings highlighted the promising application of AFZO nanostructured thin films of high-performance humidity sensors.

In one study, Li *et. al.* [17] prepared a humidity sensor using silver (Ag) modified ZnO (Ag/ZnO) nanoparticles via the hydrothermal technique and analysed the influence of various Ag concentrations on the performance of the humidity sensor. It was found that uniformly distributed Ag particles on ZnO increased the number of active surface areas and oxygen vacancies, allowing the surface-modified sensor to capture more water molecules and accelerate the water degradation to form conductive ions. As a result, the Ag/ZnO nanoparticles effectively enhanced the performance of the humidity sensor with higher a response (151,700%), low hysteresis (3%), good linearity, and short response/recovery time (36/6 s) [17].

In another study, Ismail *et. al.* [25] developed a humidity sensor with a unique configuration using crystalline Sn-doped ZnO nanorod arrays and rutile SnO_2 ($SnZO/SnO_2$) nanowire via a two-step solution immersion approach. The study revealed that the fabricated $SnZO/SnO_2$-based humidity sensor demonstrated significant stability and exceptional sensing performance compared to undoped SnZO sensors [25].

Table 1. *Semiconductor materials and methods used to fabricate humidity sensors and their performance based on past studies.*

Materials	***Performance***	***Synthesis method***	***Ref.***
ZnO/SnO_2	*Lower hysteresis between 11% and 95% RH*	*Solvothermal*	*[5]*
Al-doped ZnO-coated/Al-doped ZnO nanorod	*7.38 (Sensitivity ratio at 40–90% RH)*	*Spin-coating*	*[6]*
AFZO nanostructure	*247 (Sensitivity ratio at 40–90% RH) for AFZO–010*	*Sol-gel*	*[7]*
Ag/ZnO nanoparticles	*151,800% RH at 11–95%, Smaller lag error (3%) Fast recovery time (36/6s)*	*Hydrothermal*	*[8]*
SnZO nanorod /SnO_2 nanowire	*67.8 (Sensitivity ratio at 50–90% RH)*	*Two-step immersion*	*[9]*

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Table 1 summaries the semiconductor materials and synthesis methods employed to fabricate excellent humidity sensors in past studies. It can be seen that sensitivity ratio is the key parameter of humidity sensor as it help indicate how much electrical or optical responses change with humidity. Higher sensitivity ratio provide more responsive sensor to the humidity changes.

Based on the table, we can see that different materials and synthesis method lead to various levels of sensitivity ratio and performance. For example, Ag/ZnO material synthesis by using hydrothermal method [17] shows better response (151,700%), good linearity, low hysteresis (3%), and short response/recovery time (36/6 s), making it a promising high performance humidity sensor.

On the other hand, the sensitivity of the Al-doped ZnO coating synthesis via spin coating improved compared to that of the Al-doped ZnO nanorod arrays [13], with values of 7.38 at 40% to 90%RH, but, the sensitivity ratio is relatively lower than other material listed in the table. However, the spin-coating method used to synthesize this material is simple and low-cost, making it an attractive option for mass production of humidity sensors.

In conclusion, it can be seen that the different choices of material and the various synthesis method for humidity sensors all depends on the specific requirements of the application, such as sensitivity, response time, cost, and stability. The table above provides a useful starting point for other researchers to select ideal materials and synthesis methods for their humidity sensing needs.

4. DEVICE CONFIGURATION

Metal oxides have been utilized to produce high-quality humidity sensors over the past many years, given their remarkable properties, including high sensitivity, strong adsorption and desorption, relatively cheap, and ease of fabricating. Despite the numerous publications on the fabrication and characterization of ZnO nanostructures composited with other metal oxides, very few reports focused specifically on the development of humidity sensors using ZnO alone. The following section presents several past studies that implemented different methods and semiconducting materials to develop humidity sensors with various configurations.

Previously, Subki *et. al.* [5] fabricated a humidity sensor with pristine ZnO and Al-doped ZnO (Al:ZnO) nanostructures via a simple low-temperature ultra-sonicated immersion technique. The flexible Al:ZnO nanostructured-based was first developed using a simple brush printing technique on cellulose filter paper as the substrate and transparent paper glue as the binder before the component was subjected to immersion at varying durations of 2–5 hours. The chemical, structural, electrical, morphological, and humidity-sensing characteristics of pristine ZnO and Al:ZnO nanostructures were assessed using Field Emission-Scanning Electron Microscope (FESEM), High-resolution Transmission Electron Microscopy (HRTEM), X-ray Diffraction (XRD), X-ray Photoelectron Spectroscopy (XPS), Energy-Dispersive Spectroscopy (EDS), a humidity-measuring system, and a two-probe I-V measurement system. Based on the results, the fabricated Al:ZnO-4 h nanostructured-based flexible humidity sensor outperformed all other samples with the highest sensing response and sensitivity to humidity changes [5]. Figure 2 illustrates the procedure for humidity sensor fabrication.

Meanwhile, Ismail *et. al.* [11] synthesized a pristine ZnO and iron (Fe)-doped ZnO nanotip arrays (FZO) via a low-temperature sol-gel immersion process to fabricate a humidity sensor. Figure 3 illustrates the sol-gel immersion method and the growth mechanism of FZO [11].

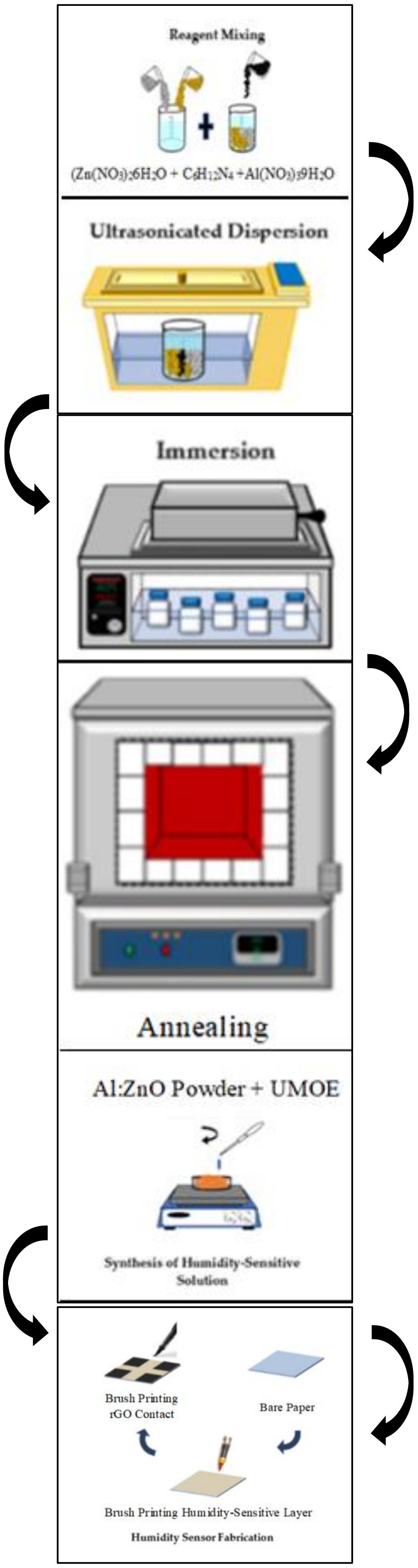

Figure 2*. The schematic illustration describing the procedures for humidity sensor fabrication* [5].

According to the study, the XRD analysis revealed that the crystallite size and lattice constant of FZO were reduced compared to that of the pristine ZnO. Moreover, the FESEM images displayed that the Fe doping led to a substantial ZnO structure alteration from a rod-like structure into a tip-like structure. Furthermore, the I-V measurement showed that the FZO film demonstrated outstanding electrical properties, including a resistance value of 12.34 MΩ. The FZO was also suitable for multifunctional uses, particularly as an ultraviolet (UV) and humidity sensor.

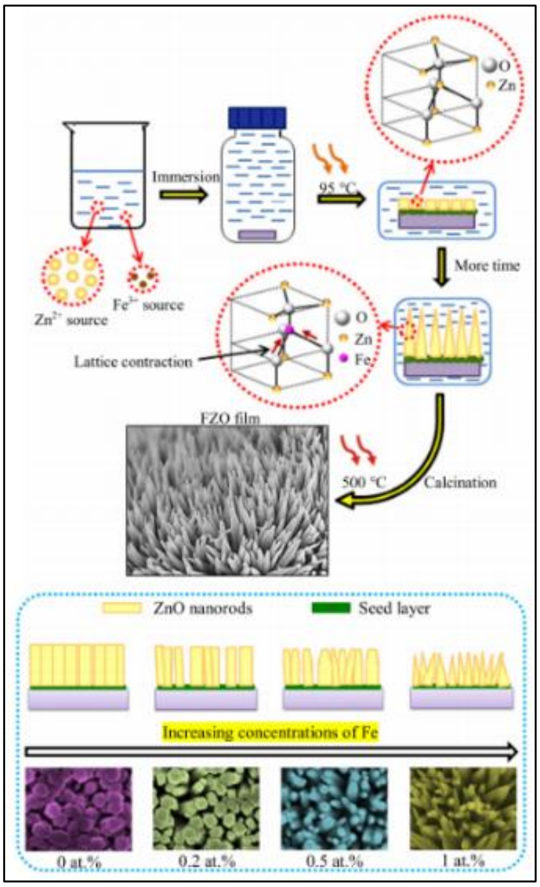

Figure 3. *The sol-gel immersion method and the growth mechanism of FZO* [11].

In another study, Young *et. al.* [26] effectively prepared a humidity sensor using pure ZnO and gold (Au) nanoparticle-modified (ANM) ZnO nanorods (ZnO-NRs) on a glass substrate via a hydrothermal approach for 6 hours at 90 °C. The optical and crystalline properties of both pristine ZnO and ANM ZnO-NRs were examined using XRD, Photoluminescence (PL), and Scanning Electron Microscopy (SEM). The findings showed that both pristine ZnO and ANM-NRs achieved high photo-to-dark current contrast ratios and fast recovery time. The ANM ZnO-NRs recorded a more impressive humidity-sensing performance than pure ZnO. Additionally, the humidity sensors can be further enhanced by incorporating Au-absorbed nanoparticles. Figure 4 shows the photoresponse of the ANM ZnO-NRs both under dark and UV conditions [26].

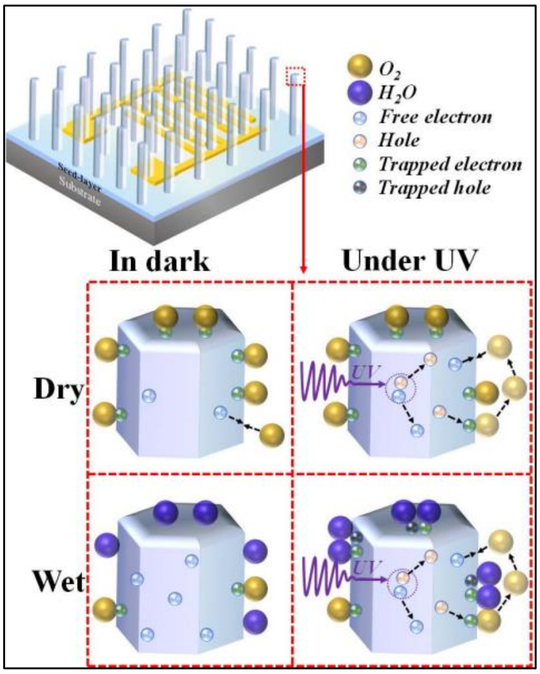

Figure 4. *The photoresponse of the ANM ZnO-NRs under dark and UV conditions* [26].

Furthermore, Kadem *et. al.* [27] investigated the fabrication of ZnO thin films using three different methods: sol-gel, spin-coating, and spray-coating. The impact of the different preparation methods on the morphological and optoelectronic properties of the thin films was analysed using SEM and Atomic Force Microscopy (AFM). Several parameters were also evaluated, including the spin times on the spin-coating, growth time, and doping. The results showed that a longer spin time led to more compact rippled-shaped features on the ZnO spin-coated layer. The findings also revealed that the use of different dopants produced varying morphological features. Therefore, it shows that these properties may be applicable in the development of humidity sensors. Figure 5 shows the three preparation methods of the ZnO thin films (a) spin-coating technique; (b) growth process; (c) spray-coating technique [27].

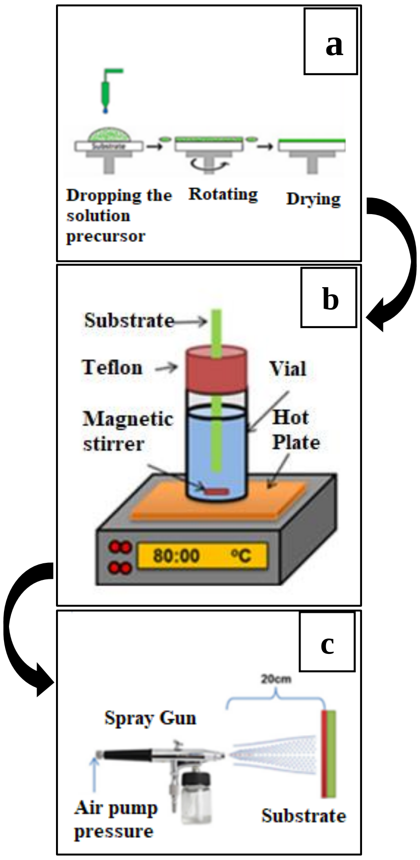

Figure 5. *The preparation of the ZnO thin films using the (a) spin-coating technique; (b) growth process; (c) spray-coating technique* [27].

Besides that, Z. Xu *et. al.* [28] developed a humidity sensing device using a low power homo-buffer layer to enhance the device's sensing performance. ZnO films were deposited on the surface of SAW devices using different sputtering power which then successfully create an island-shaped ZnO nanostructures using a simple homo-buffer layer method. The researchers evaluated the mass sensing performance of the Love-SAW sensor by adding layers of ZnO top layer and studying the effect on different wave modes. From the results it shows that the mass sensitivities of the Rayleigh and Sezawa wave modes significantly increased with the thickness of the ZnO top layer. The findings conclude that the Love-SAW sensor fabricated using the LPHBL method has the potential to be used in humidity sensing applications. Figure 6 below shows the testing system schematic used for humidity sensing [28].

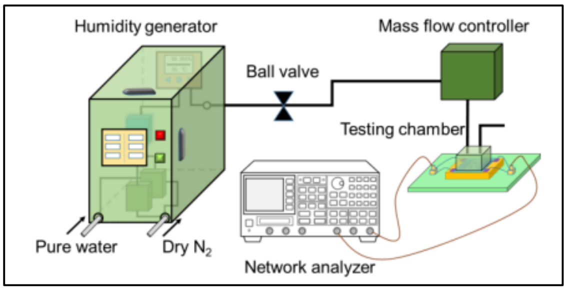

Figure 6. *The testing system schematic used for humidity sensing* [28].

M. Morsy *et. al.* [29] investigate the effect of ZnO nanorods on the humidity sensing abilities of graphene foam (GF). The researchers synthesize GF using atmospheric pressure CVD with xylene as the hydrocarbon source and use hydrothermal method to added ZnO nanorods to the surface of GF. The results were analyzed using various technique such as SEM, XRD, Raman spectroscopy, Furrier transform infrared spectroscopy (FTIR), thermal gravimetric analysis (TGA), BET surface area, and BJH pore diameter distribution. The findings shows a linear relationship between humidity sensing performance and relative humidity (RH) on the composite of ZnO/GF. Figure 7 below shows the GF humidity sensor schematic diagram [29].

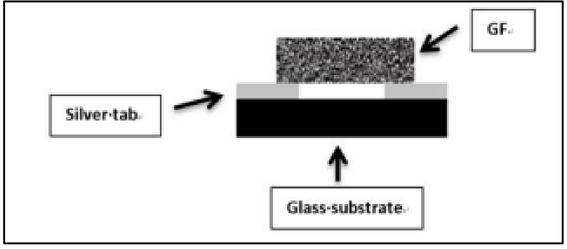

Figure 7. *The GF humidity sensor schematic* [29].

5. CONCLUSION

This paper highlighted numerous literature studies investigating and verifying the excellent performance of relative and absolute humidity sensors. While past studies have employed multiple parameters to improve the performance of ZnO in humidity sensors, including immersion time, type of dopants, sensitivity, reaction time, stability, and electrical properties, further studies are required to gain an in-depth understanding of the humidity sensing mechanism to boost the efficiency of humidity sensors in various critical applications.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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