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A Recent Progress of Graphene Oxide/Metal Oxide Composite for Humidity Sensor Applications

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ABSTRACT

In this article, we have reviewed current research findings on a unique material known as graphene oxide (GO), which is a by-product of the oxidation of graphene, to assess its use as potential carbon material in metal oxide composites-based humidity sensors. Due to its exceptional mechanical, electrical, and optical properties, graphene oxide (GO) has garnered significant interest in numerous scientific and technological domains. A GO/metal oxide-based humidity sensor deposition has been made using various nanoparticles that offer an excellent interface and low defect levels in the heterostructure. This paper summarises recent developments in GO and GO/metal oxide composites for sensing materials. We begin with the principles of humidity sensing, the synthesis of graphene materials, and the deposition method of GO and GO/metal oxide composites. Moreover, the characterization, such as structural, morphology, optical, and electrical properties, was discussed based on X-ray diffraction measurement (XRD), Field emission scanning electron microscopy (FESEM), photoluminescence (PL), and current voltage (IV characteristics). This review also highlights sensor properties such as sensitivity, selectivity, stability, response time, and detection limits. Most of the findings also revealed that the highly effective sensing mechanism was used to detect respective characterizations with high sensitivity and stability. The present review is focused on exploring the synthesis, deposition, and characterization of GO and GO/metal oxide composite-based humidity sensors.

Keywords: *Graphene, graphene oxide, metal oxide, composites, humidity sensor*

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INTRODUCTION

Carbon-based nanomaterials, such as graphene and carbon nanotubes, have recently attracted much attention from researchers due to their potential as humidity-based sensing materials [1]. Understanding the fundamental mechanism in sensing materials is critical in developing sensing material, focusing on the sensor's sensitivity, response speed, and stability. Since the Nobel prize-winning discovery of graphene as novel material, various branches of pure science, applied science, and engineering have used its exceptional properties in their respective fields [2 – 4]. Graphene has a unique two-dimensional, single-layered structure with a honeycomb-like structure of atoms connected by sp^2 bonds, resulting in extraordinary properties. Graphene has excellent electrical conductivity, high charge carrier mobility at room temperature, good optical properties, and a large surface area, making it more advantageous for humidity-sensing applications [2, 5 - 8]. The potential and rising popularity of graphene-based materials, such as pristine graphene (G), graphene oxide (GO), and reduced graphene oxide (rGO) as it, shows a significant number of publications as promising materials for humidity sensing applications [9, 10].

In addition, metal oxides, such as titanium dioxide (TiO_2), tin oxide (SnO_2), zinc oxide (ZnO), and iron oxide (Fe_2O_3), were reported suitable for producing high-quality humidity sensors [11, 12]. In addition, these metal oxides can also be used for other sensor applications, such as formaldehyde and ethanol sensors [13]. Different types of humidity sensors have been reported, and they are based on a capacitive, surface acoustic wave (SAW) quartz crystal microbalance and mass spectrometry. The resistive humidity sensor, on the other hand, has an advantage over all the other humidity sensors due to its low cost of manufacture, ease of integration with the CMOS (Complementary Metal Oxide Semiconductor) platform, and simple and efficient operation [14, 15].

Recent advances in research demonstrated that the modification of graphene oxide with metal oxide nanoparticles shows an effective method for constructing high-performance sensors. GO, which acts as a highly conductive sp^2 carbon atom film, is an anchor to promote the electron transfer in the metal oxide nanoparticles, thus leading to a better sensing performance. This review will briefly discuss the recent progress in composited graphene oxide/metal oxide-based humidity sensors, which focuses on four main parts: i) Humidity sensing mechanism, ii) preparation of graphene oxide material, iii) sensing properties of pristine GO and GO/Metal oxide composite based humidity sensor and iv) recent progress in the humidity sensor based on GO and metal oxide ternary composite material.

Humidity Sensing Mechanism

The amount of water vapor in the air, or humidity, impacts human comfort and industrial manufacturing processes. The presence of water vapor affects numerous physical, biological, and chemical processes. When subjected to various atmospheric humidity levels in the surrounding environment, humidity sensors are a device that detects and measures the physical and electrical properties of sensitive elements, such as water molecule adsorption or desorption, as shown in Figure 1.

As a result of its significant technical applications in agriculture, medical instrumentation, and industrial process control systems, humidity sensors have recently attracted a lot of study attention [16 – 18]. The characteristics of the sensing material, such as porosity, surface area, pore size distribution, and

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shape, directly affect a humidity sensor's performance [19]. These sensors should respond consistently, be highly sensitive, respond quickly, exhibit minimal hysteresis, be chemically and physically stable, operate over a wide range of humidity, and be inexpensive. Relative humidity (RH), dew/frost point (D/F PT), and parts per million (PPM) are the most often used units for measuring humidity [20].

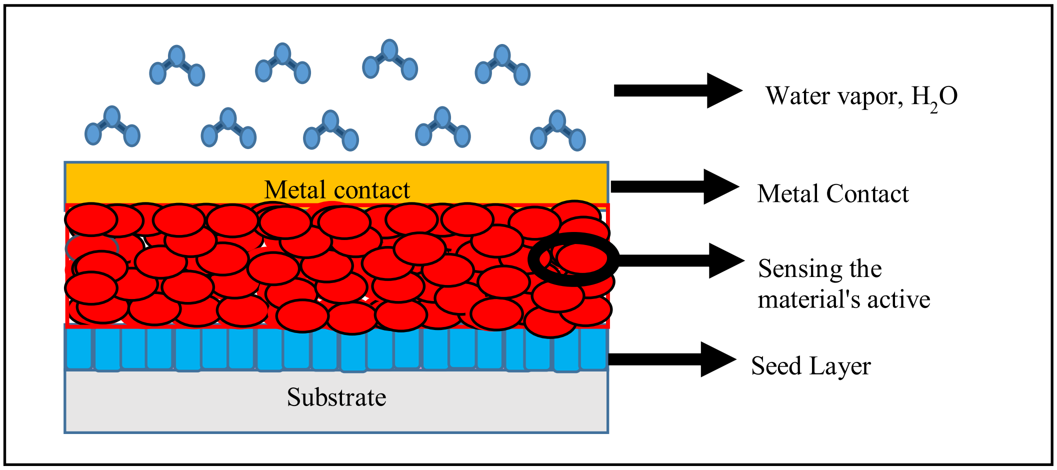

Figure 1: Schematic diagram of a humidity sensor

Preparation of Graphene Oxide Material

Various techniques can be used to create graphene, including mechanical exfoliation, chemical vapor deposition, the enhanced Hummer's process, reduction-oxidation, and ultrasonic stripping. This review paper focuses on two infamous synthesis methods: chemical vapor deposition (CVD) and Hummer's method.

Chemical vapor deposition (CVD), which has evolved into a primary thermal deposition method for graphene synthesis, is one of the numerous recently developed to provide high-quality, large-scale graphene production [21]. This method helps transfer graphene films for device applications with fewer flaws or wrinkles to an insulating substrate. Using CVD, graphene can be produced from several carbon sources, such as a gas, liquid, or solid precursor. The method of graphene formation is significantly influenced by temperature, metal substrate type, gas pressure, concentration, and kind of carbon precursor. However, graphene generated by CVD is often polycrystalline [22].

Brodie first showed the synthesis of GO in 1859 by adding potassium chlorate to a graphite solution in fuming nitric acid. Staudenmaier used fuming nitric acid and concentrated sulfuric acid to advance this technique in 1898. He then gradually added chlorate to the reaction mixture. A streamlined and revised methodology for manufacturing highly oxidized GO was produced due to this minor procedure adjustment. In 1958, Hummers published a study describing an alternate process for synthesizing graphene oxide using potassium permanganate ($KMnO_4$) and sodium nitrate ($NaNO_3$) in concentrated hydrogen peroxide (H_2SO_4). On the other hand, the Hummers approach frequently necessitates several procedures, difficult and time-consuming experimental time, and heat management

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for GO preparation [23].

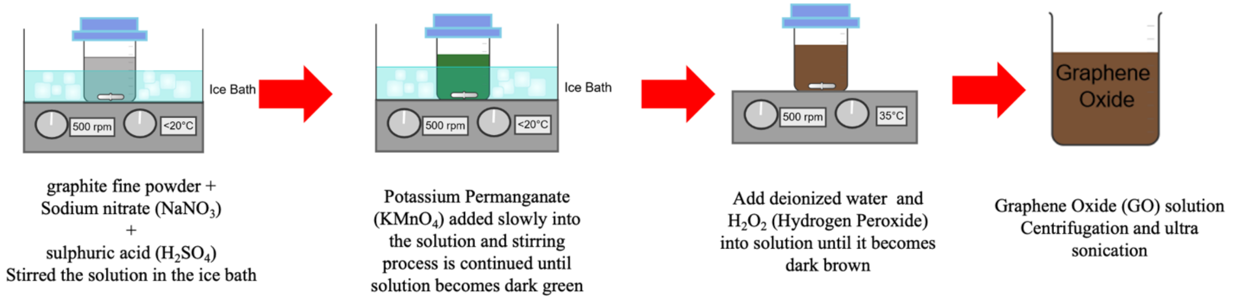

Figure 2: General Hummer's method to synthesize graphene oxide

Sensing Properties of Pristine Go and Go/Metal Oxide Composite-Based Humidity Sensor

Pristine Graphene Oxide Based Humidity Sensor

Recently, graphene oxide (GO) has been showing an excellent membrane material and attracts researchers to investigate its ability in electrical and mechanical properties. In 2019, research done by Pengomai and his teams reported that GO offers extraordinary flexibility and a high surface area-to-volume ratio. GO also has hydrophilic properties making it beneficial for detecting polar gases like water molecules which are essential to enhance the sensitivity of the sensors to water [24]. In another study, Bi and co-workers fabricated the first micro-scale capacitive GO-based humidity sensor. From the response and recovery figures, they have successfully produced a quick half response and recovery time of the humidity sensor compared to the traditional sensor [25].

Moreover, some studies have shown molecular interaction between water increment and GO-charged groups by varying the pH of GO. The sensitivity and the hysteresis show an improvement in conductance measurement, thus showing the potential of GO in humidity sensor applications [26]. The capability of GO in sensing performance is attributed to the morphological structure of graphene itself. The imperfect flat, rough, and out of the wrinkled surface of GO in FESEM images shows how the GO morphology affects the sensing performances. Folds generally trap impurities that act as dopants or dispense material, affecting sensor resistance [27 – 29].

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Graphene Oxide and Metal Oxide Composite Based Humidity Sensor

Sensing technology has advanced in recent years and continues to do so in order to ensure human well-being, quality, and safety from food to air, as well as environmental protection. Metal oxides, including ZnO, TiO_2, WO_3, CuO, Cu_2O, SnO, SnO_2, and VO_2/V_2O_5, are examples of such materials that have been used in a variety of sensors, including gas, humidity, UV, and biological sensors. These materials will take on a variety of nanoscale structures, ranging from nanowires to nanospheres or nanosheets, which will directly impact their performance in sensing applications [30]. However, a single or pristine sensing material has low sensitivity, accuracy, and stability, which is essential to respond exclusively and exhibit better humidity sensing properties [31, 32]. Incorporating GO with other materials would enhance the structure's versatility and the material's excellent sensing properties [33]. The surface area and stability of humidity sensing can be improved by combining GO with active materials such as metal oxide SnO_2, TiO_2, ZnO, and CuO [34]. The graphene/metal oxide composite offers massive mechanical properties, electrical conductivity, thermal stability, and chemical resistance improvements. Combining composites will provide better performance since it combines the advantages of different materials.

A further study by other researchers shows excellent capabilities when utilizing TiO_2 nanorods with GO, allowing water molecules to be absorbed efficiently, even in a dry environment. The outcomes demonstrate that TiO_2 nanorods enhance GO film performance at low humidity levels [35]. From the schematic diagram, the presence of TiO_2 nanorod can keep the GO film from falling into the bottom of the electrode gap, thus increasing the application of the surface of GO [35]. This mechanism will make the water molecules instantly diffuse through the GO layer on both sides and increase the sensor's responsivity.

The limited performance, slow response, and recovery time of SnO_2 and graphene-based humidity sensors have motivated Xu *et al.* [36] to develop a novel composite material using the conventional electrospinning and solvent evaporation method. In their study, they successfully wrapped a GO with SnO_2/Graphene (SnO_2/G) nanocomposite, thus forming SnO_2/G-GO nanocomposite films. By employing a GO into the SnO_2/G, the response and recovery times were significantly boosted to 2s and 4s, respectively, compared to pristine SnO_2 and SnO_2/G-based humidity sensors, which took a long time to respond (longer than 10s) [36]. This practical and novel GO application gives other researchers a new idea to utilize the benefit of GO to deliver excellent and optimal sensing characteristics in the future.

Recent Progress in The Humidity Sensor Based on Graphene Oxide

The remarkable feature of graphene material, like its high surface area and excellent conductivity, has made them one of the most popular carbon-based compounds. The advancement of sensors based on GO composite materials has attracted extensive research due to simplicity, inexpensiveness, and the GO's hydrophilic sensitivity to water. To utilize the use of even more of its fascinating characteristics, graphene is engineered to yield graphene oxide and reduced graphene oxide, which improve the material's water dispersibility and make it simpler to combine with other substances to create binary or ternary composites [37]. Nowadays, a broad study explores the potential GO in ternary composites such as ternary graphene/metal oxide/ metal oxide and graphene/metal oxide/polymer-based sensors.

A study done by Zhang and his co-workers [38] has constructed a quartz crystal microbalance (QCM)-based humidity sensor based on graphene oxide/ tin oxide/polymer polyaniline (GO/SnO_2/PANI). The ternary composite device reported a high sensitivity of 29.1 Hz/%RH and a short response/ recovery time (7s/2s) better than the previous study, as shown in Table 1. Even though graphene/metal oxide/polymer ternary composite can promise a high sensing performance, the selection type of polymer should be noticed before it can be used in the composite for humidity sensor applications. Sears et al. stated that the polymer's capability to absorb water molecules could affect conductivity as it strongly correlates with output frequency [39].

Another study done by Jiang *et al.* [40] found that employing GO and metal oxides compound can enhance their sensing properties. The GO/SnO_2/NiO sample showed significant improvements in response time, recovery time, and sensitivity compared to SnO_2/NiO composite. In the structural design of materials, the GO acts as a p-type semiconductor, and the metal oxide (SnO_2 and NiO) of an n-type semiconductor are combined to enhance their sensor performance. The results revealed that the response and recovery times were 5s and 150s, respectively [40]. This study employed GO as a p-type semiconductor, which is one reason the response and recovery time was slower compared to GO as n-type material. Subsequently, a ternary composite of rGO/Zno-SnO was proposed by Sen and his co-workers [41] to fabricate a volatile organic compound (VOC) sensor. The sensor showed an excellent sensing property with a fast response time (10s) and a recovery time is 100s. The developed sensor exhibits long-term stability and repeatability, shown in its dynamic response curve. Even though these ternary composites were purposely fabricated for other sensors, there are similarities in humidity sensing measurement characteristics such as response and recovery time and stability and repeatability performance. The sensing performance of sensors-based graphene oxide is summarized in Table 1.

Table 1: Summary of graphene oxide progress in sensor applications

Sensing Material	**Humidity Range**	**Sensitivity/Response**	**Response and Recovery Time**	**Reference**
GO/SnO_2/PANI	0-97 %RH	29.1 Hz/%RH	7s / 2s	[38]
SnO_2/ZnO-T / Graphene	15-95 %RH	Not given	Not given	[42]
SnO_2/rGO	11-97 % RH	15.19 - 45.02%RH	<100s/<100s	[43]
SnO_2/ G-GO	65% RH	32 M %RH	<1s/<1s	[36]
GO/TiO_2 nanorod	11.3 - 97.3 %RH	Not given	450ms/ 890 ms	[35]
GO/ZnO	11.3 - 97.3 %RH	Not given	9s / 5s	[28]
GO	0.47 - 67 %RH	0.03 % RH	60s / 120s	[44]
GO	15 - 95 %RH	Not given	10.5s / 41s	[25]
GO	10 - 90 %RH	12.3 %RH (pH 3.3) 12.3 %RH (pH 9.5)	2.2s/1.6s (pH2.8) 91.8s/11.3s (pH9.8)	[26]
rGO	11 - 95 %RH	Not given	3s / 10s	[45]

CONCLUSION

Several efficient strategies have been devised to create graphene oxide/metal oxide composite-based humidity sensors with good sensing capability. Graphene-based humidity sensors initially have low selectivity and slow delayed recovery, but they also have high sensitivity, a detecting limit, and quick response times. Researchers worldwide are still interested in graphene materials because of their distinctive electrical, physical, and chemical characteristics. Using chemical techniques like CVD and Hummers' method, graphene oxide/metal oxide composite-based humidity sensors may be produced cheaply and easily. In this paper, we reviewed the current progress in GO applications in fabricating humidity sensors, as it plays a crucial role in reacting with the humidity mechanism. The interaction between functional groups of graphene oxide incorporating metal oxide composite can increase the sensing mechanism with increasing adsorption surface. The hydrophilic nature of graphene exhibits various sensing properties, either pristine graphene or graphene-metal oxide-based humidity sensor. There is also growing interest in using ternary composite materials that have been proposed in attempts to produce a reliable, stable, and high-sensitivity sensor in a humid environment. In conclusion, developing humidity sensors will depend on identifying and understanding the best graphene material and the type of metal oxide that can be incorporated to optimize their use in high-sensitivity humidity sensors. Modifying metal oxides nanostructures such as nanorods or nanofiber has been proven to enhance sensing performance by incorporating GO films. This paper helps other researchers to have a deeper understanding of the interaction process between graphene and metal oxide for the humidity sensor mechanism.

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AUTHOR’S CONTRIBUTION

Fazlinashatul Suhaidah Zahid and Nor Diyana Md Sin carried out the literature review and wrote the article. Nur Amalina Muhamad and Mohamad Zhafran Hussin conceptualized the central idea.

CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest.

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