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Research on a Review of Derivative Methods of Classical Hydrothermal/Solvothermal Synthesis Methods, Taking SnO_2/r-GO Gas-sensitive Composite Materials as Examples

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ABSTRACT

Against the backdrop of accelerating global industrialization, the significance of gas sensors in environmental pollution monitoring, energy security, and medical diagnostics has become increasingly prominent. Metal oxide semiconductors, such as SnO_2, have emerged as core materials due to their low cost and high sensitivity, yet they face limitations such as insufficient room-temperature sensitivity and humidity interference. The introduction of two-dimensional materials like graphene offers novel approaches to enhance gas-sensing performance, improving sensor capabilities through mechanisms such as heterojunction formation and increased adsorption sites. The hydrothermal/solvothermal synthesis method has become a mainstream approach for preparing SnO_2/r-GO composites, owing to its operational simplicity and material uniformity. This paper primarily reviews its derivative methods, including the "separate preparation and subsequent synthesis method" and the "co-preparation method." Research indicates that these methods can significantly enhance the sensor's response value, selectivity, and stability towards target gases.

KEYWORDS

Gas Sensor; MOS; SnO_2; Graphene; Heterojunctions.

1. INTRODUCTION

In the context of the accelerated global industrialization process, issues such as environmental pollution, energy security, and public health have become increasingly prominent, and the demand for precise monitoring of various gases has become a key challenge for the sustainable development of society. Metal oxide semiconductors, with their characteristics of low cost, simple preparation, high sensitivity, and strong compatibility, have become one of the core materials for fabricating gas sensors[1]. Their working principle is based on the dynamic adsorption and desorption processes of gas molecules on the semiconductor surface, and these physical and chemical behaviors are determined by the microstructure and macroscopic properties of the material[2,3]. To date, researchers have studied SnO_2, ZnO, and TiO_2 based gas-sensitive sensors[4-6]. Among them, SnO_2 has gained favor due to its high sensitivity, fast response speed, and low cost, and has a long development history with multiple research branches. At the same time, it faces bottlenecks such as insufficient sensitivity at room temperature and significant humidity interference, making it difficult to meet the detection requirements in complex scenarios[7].

In recent years, the rapid development of two-dimensional materials has provided new ideas for the preparation of high-performance composite gas-sensitive sensors. For example, graphene and its derivatives, two-dimensional metal oxides, transition metal sulfides, etc. have been used to improve

the performance of gas-sensitive sensors. Among them, graphene has been favored by researchers due to its unique advantages such as ultra-high carrier mobility and extremely large specific surface area[8]. The combination of the two to prepare nano-sized gas-sensitive materials with a microcrystalline size and active sites that are as dispersed as possible can significantly improve the gas sensing performance and selectivity[9]. Currently, the methods for preparing graphene/metal oxide two-dimensional gas-sensitive materials include hydrothermal/solvent-based methods, chemical vapor deposition methods, etc. Among them, the hydrothermal synthesis method is the most promising for large-scale production due to its low cost, low operating temperature, and simple process control, and has a longer development time and rich branches.

This paper will review the derivative methods of the classic hydrothermal/solvent-based synthesis method based on the preparation of SnO_2/r-GO composite gas-sensitive materials.

2. THE MECHANISM OF SNO_2 NANOPARTICLE GAS SENSING

For n-type metal oxides, the metal oxide particles in n-type semiconductor gas sensors act as receivers and sensors. They achieve the functions of receiving and sensing through contact with gas molecules. The interface between particles plays a decisive role in the overall resistance of the semiconductor[10]. The surface space charge layer model or the double Schottky barrier model are shown in Figure 1a or Figure 1b respectively[10]. In n-type semiconductors, oxygen in the air adsorbs on the metal oxide surface, captures electrons to form O^-, resulting in a decrease in surface electron concentration and the formation of a depletion layer with thickness w. The thickness of the depletion layer increases with the increase in oxygen adsorption, hindering electron conduction and causing the sensor resistance to increase. When contact is made with reducing gases, the gas reacts with the adsorbed oxygen to consume oxygen, causing the depletion layer to thin and the resistance to decrease[10]. At the contact point between adjacent semiconductor particles, a Schottky barrier is formed, and the barrier height determines the contact resistance. Gas adsorption changes the surface charge distribution, causing a change in the barrier height and thereby affecting the overall resistance of the sensor. In recent studies, researchers tend to prepare semiconductor particles as small as possible to improve gas sensing performance and selectivity[8]. For very small semiconductor particles, the thickness of the depletion layer can approach the size of the nanoparticles, and the depletion layer may extend to the entire particle, with w being greater than or equal to the particle radius r (volume depletion, Figure 1c)[10]. At this time, electron conduction is regulated by the overall electron concentration of the particles, rather than only the surface layer[10]. The traditional surface space charge layer model is no longer applicable, and the volume depletion model is needed to describe the electron behavior (Figure 1d). Conductance is proportional to electron concentration ([e]), that is, the higher the electron concentration, the greater the conductance.

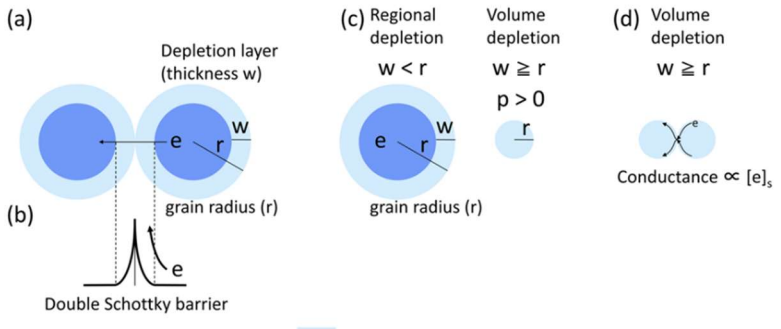

Figure 1. Diagrams illustrating the reduction of electrons in oxide grains and the resistance encountered during grain contact. (a) Space charge layer model, (b) double Schottky barrier model, (c) regional and volume depletion model, (d) surface conductive grains contact mode[10]

3. MECHANISM OF GRAPHENE-BASED MATERIALS FOR IMPROVED GAS SENSING PERFORMANCE OF SNO_2

Tyagi and his team were the first to discover that graphene can detect adsorbed gas molecules at room temperature[11]. The two-dimensional structure of graphene makes the electron transmission through graphene highly sensitive to the adsorption of gas molecules[12]. As the first discovered and applied two-dimensional material, graphene has unique advantages of high electrical conductivity and electron transmission rate due to its two-dimensional nature[11-14]. It has a series of materials with different properties, such as rGO and GO[15,16]. Taking rGO as an example, its electrical conductivity is as high as 6000 S/cm, providing an efficient transmission path for charge carriers. Pure SnO_2, due to its wide band gap at room temperature, requires high-temperature excitation for electron transition. Implementing rGO can circumvent the process of high-temperature excitation via tunneling or direct interface conduction, consequently lowering the operational temperature. Concurrently, rGO's specific surface area attains 2630 m^2/g. This, in conjunction with MOS nanoparticles, markedly enhances the sites for gas absorption and prevents SnO_2 nanoparticles from aggregating. The large number of defects resulting from lattice mismatch can also increase the gas adsorption sites and enhance the reaction activity. The functional groups such as hydroxyl and carboxyl groups on its surface can enhance the adsorption of polar gases, reduce the activation energy of physical adsorption, and enable the reaction to proceed at low temperatures. In addition, the thermal conductivity of rGO is as high as 5000 W/m·K, which can quickly dissipate the heat released during adsorption and prevent the performance decline of MOS due to local overheating.

When two different materials are combined, different types of heterojunctions are formed: p-n heterojunction, n-n/p-p homo-junction, or Schottky heterojunction. For GO-SnO_2, graphite oxide is a p-type material with a work function of 5.82 eV; SnO_2 is an n-type semiconductor with a work function of 4.55 eV[2]. When GO comes into contact with SnO_2, electrons transfer from SnO_2 (n-type) to GO (p-type). This process leads to the formation of an electron depletion layer on the surface of SnO_2, reducing the electron concentration and forming a p-n heterojunction at the interface, resulting in an increase in resistance. After contact with air, oxygen molecules capture electrons on the surface of SnO_2 to form chemical adsorption oxygen. Due to the existence of the p-n junction, the depletion layer width is usually wider, making it more sensitive to changes in carrier concentration.

4. CATEGORIES OF DERIVATIVE METHODS OF THE CLASSIC HYDROTHERMAL/SOLVENT THERMAL SYNTHESIS METHOD

We can clearly see that the current mainstream methods for preparing SnO_2 mainly rely on chemical methods using chlorine-stabilized tin as the base[17-21]. The preparation of graphene is more diverse, including the Hummers method and CVD, and many researchers choose to use the Hummers method or its improved versions to prepare graphene[22]. At the same time, some researchers use chemical vapor deposition to prepare it. The hydrothermal/solvent thermal synthesis method has the advantages of convenient operation and uniform and excellent properties of the prepared materials, and is the mainstream method for synthesizing SnO_2/graphene.

4.1. Separate Preparation before Re-composition

Researchers usually first prepare SnO_2 particles and RGO nanosheets separately, and then combine them through methods such as solution mixing and heating.

4.1.1. PANI /SnO_2/rGO Tri-composite

Satvik Kulkarni prepared and compared the gas sensing properties of PANI, PANI/SnO_2, and PANI/SnO_2/rGO[20]. First, an aqueous solution of aniline and ammonium persulfate was dissolved in hydrochloric acid, and then an ammonium persulfate solution was added and stirred to obtain a

dark green solution. After filtration, the solution was washed with double-distilled water and acetone to remove the ammonium persulfate, resulting in polyaniline nanofibers PANI. The enhanced Hummers technique was employed to create the oxidized graphene. Subsequently, hydrazine hydrate served as the reducing agent, followed by further reduction of the synthesized oxidized graphene through chemical reduction. Furthermore, $SnCl_4 \cdot 5H_2O$ was combined with double-distilled water and then introduced with ethylene glycol to create a network of polymers. Finally, ammonia water was added to obtain a white gel, which was filtered and washed, and then heated at 150℃to remove excess water. Ultimately, the substance underwent calcination at 400℃, resulting in the production of tin oxide. When preparing the composite material, aniline and ammonium persulfate were dissolved in hydrochloric acid solution, and then SnO_2 nanoparticles and rGO nanosheets were added to the solution containing aniline in the hydrochloric acid solution and stirred evenly. Finally, the ammonium persulfate solution was slowly added to the mixed solution containing aniline and the nanomaterials, and the solution was stirred for 2-3 hours continuously. The solution gradually turned black green. After filtration and washing, PANI/SnO_2/rGO (PSR142) was obtained, where the ratio of tin oxide and reduced oxidized graphene was 40% and 20% by weight. The samples without rGO were denoted as PS10, PS20, and PS40, representing the weight percentage of SnO_2. The PANI/SnO_2/rGO obtained in the experiment had a fibrous structure of polyaniline wrapped by tin oxide, and the nanoparticles were deposited around the reduced oxidized graphene. The tests found that the samples had excellent selectivity for NH_3, and the recovery time of the PANI/SnO_2/rGO nanocomposite material was the shortest, at 80 seconds under a 10 ppm ammonia gas concentration condition. This was because SnO_2 formed a heterojunction with PANI, promoting electron transfer. The introduction of rGO enhanced the conductivity and gas adsorption capacity of the composite material through the π electron cloud conjugation with PANI, and the mutual nesting of the nanoparticles and rGO prevented the transformation of graphene sheets into graphite layers, fully utilizing its specific surface area and accelerating the release of gases. The researchers also found that for PS, the more SnO_2 was doped, the greater the gas response value, but an increase in SnO_2 content would lead to an increase in the recovery time. This might be due to the excessive doping of SnO_2, which filled the fibrous structure of PANI, resulting in excessive agglomeration.

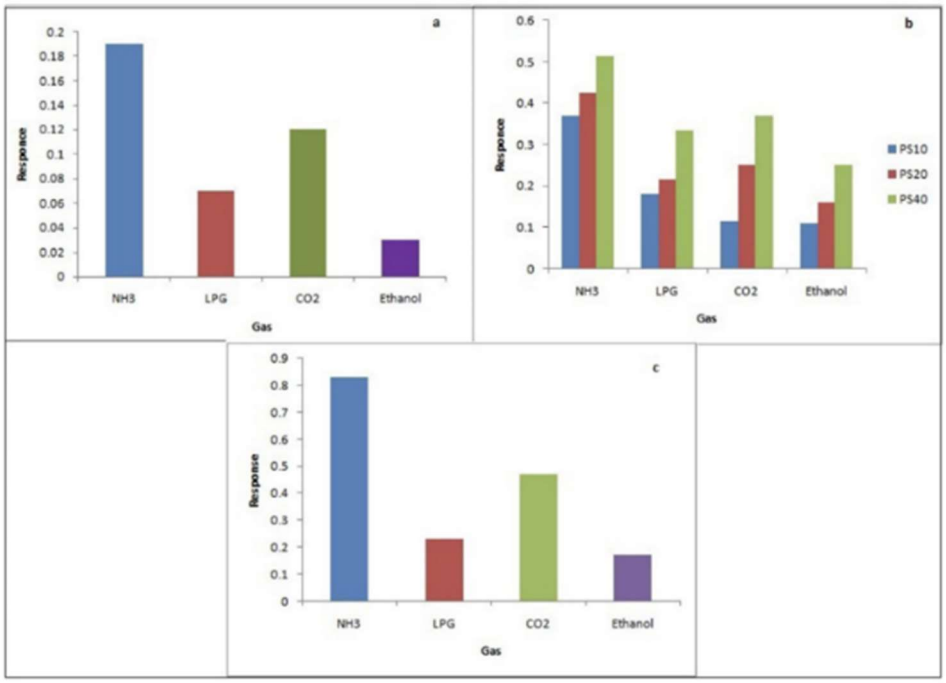

Figure 2. Detailed examination of a) PANI, b) PANI/SnO_2 nanocomposites, and c) PANI/SnO_2/rGO nanocomposite[20]

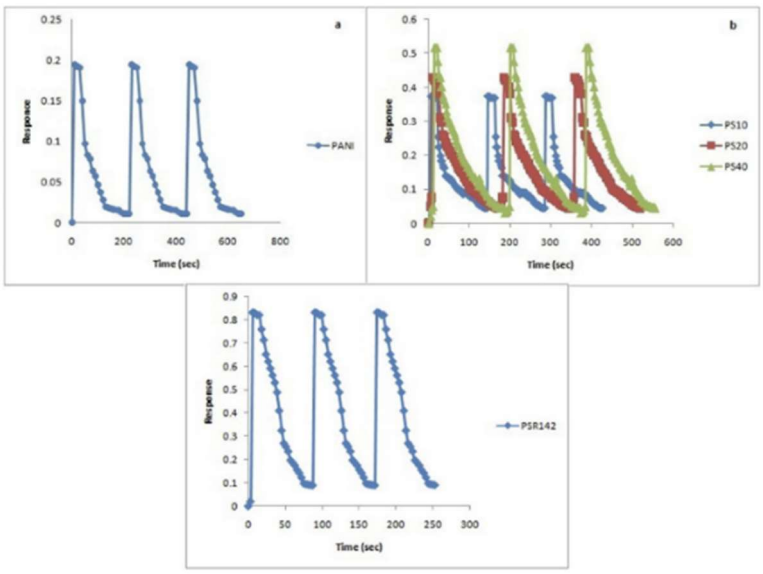

Figure 3. The reactive properties of a) PANI, b) PANI/SnO_2 nanocomposites, and c) PANI/SnO_2/rGO nanocomposite[20]

4.1.2. Separate Preparation with Metal Decoration

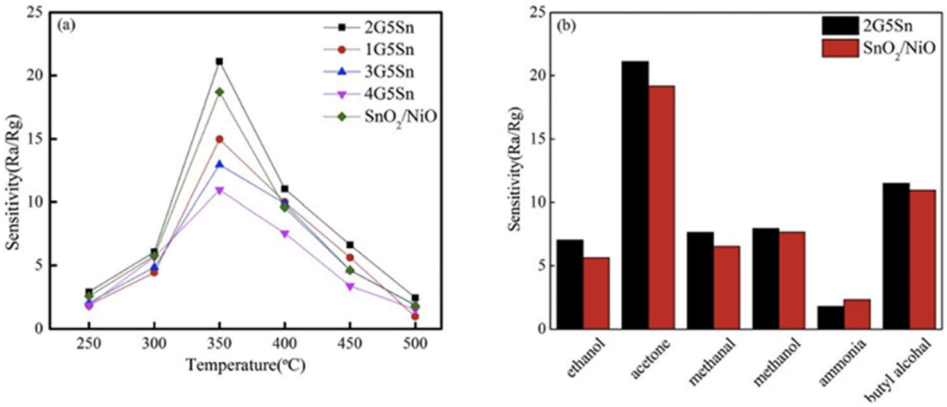

Figure 4. A visual depiction of the responsiveness of GO/SnO_2/NiO to different mass ratios in a 50 ppm acetone environment (a), accompanied by a sensitivity graph for 2G5Sn and SnO_2/NiO in a 50 ppm gas setting (b) [17]

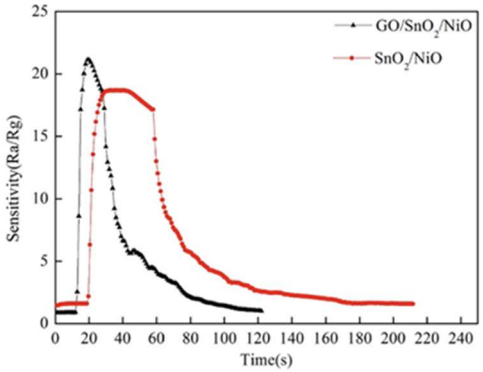

Figure 5. A visual depiction contrasting GO/SnO_2/NiO reactions within an acetone gas environment, with different concentrations at 350℃[17]

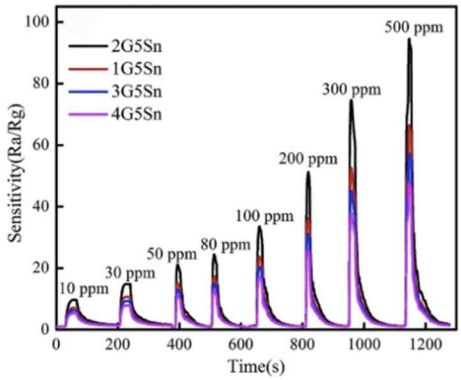

Figure 6. How GO/SnO_2/NiO and SnO_2/NiO react in an acetone environment with a 50 ppm concentration at 350°C [17]

The Lili Jiang team successfully prepared an oxidized graphene oxide/oxidized tin/oxidized nickel gas-sensitive composite material that is sensitive to acetone[17]. First, mix ion water, ethanol, and tin chloride, then vigorously stir for 20 minutes. Then add nickel nitrate, and after it dissolves, add ammonia water to obtain a solution with uniformly dispersed white precipitate. Transfer the dispersed solution to a polytetrafluoroethylene reactor, conduct a 18-hour hydrothermal reaction at 180℃, and obtain a light blue precipitate. After washing, drying, and grinding into powder, heat treat it at 400℃ in an argon atmosphere to obtain a light blue powder of SnO_2/NiO for future use. Finally, disperse the finished GO in ion water, add SnO_2/NiO, and transfer it to the polytetrafluoroethylene reactor. Perform a thermal process lasting 12 hours at 160°C, cleanse, dehydrate, and achieve a black powder-like composition of GO/SnO_2/NiO. GO/SnO_2/NiO has a mass ratio of 1:5, 2:5, 3:5, and 4:5, identified as 1G5Sn, 2G5Sn, 3G5Sn, and 4G5Sn, respectively. Post-characterization, it was discovered that the granular SnO2/NiO accumulated on GO's stratified surface and interlayers, with SnO_2/NiO particles still exhibiting agglomeration patterns. The ideal operational temperature for GO/SnO_2/NiO stands at 350°C. When maintained at a temperature of 350°C, its specificity is notable for acetone concentrations of 50 ppm. In contrast to SnO_2/NiO, GO/SnO_2/NiO exhibits a quicker response to acetone and greater sensitivity. At the same time, we can see that introducing too much GO into the composite material will reduce its gas sensing performance. In addition, the addition of NiO not only forms a pn junction with SnO_2, but also oxygen molecules will preferentially adsorb on NiO and then escape to SMO, allowing the material to react with more target reducing gas molecules, significantly improving the gas sensing performance[18].

4.2. Re-composition before Separate Preparation

The researchers added graphene to the precursor before SnO_2 was formed or when it was not fully formed, and then prepared a composite of SnO_2 and graphene in one step.

4.2.1. Microwave-assisted Hydrothermal One-pot Synthesis

The Zhenyu Wang team prepared SnO_2/rGO using the microwave heating method[19]. The GO prepared by the improved Hummer method was dispersed in deionized water. Then, $SnCl_4$-$5H_2O$ was dissolved in the resulting dispersion and hydrazine hydrate was added. Subsequently, it was placed in a Teflon container and heated using a microwave digestion system. The reaction was carried out at 160°C for 2 hours, and the precipitate was washed to obtain the sample. The resulting composite material has a loose and porous structure, is wrinkled, and SnO_2 particles are distributed between the rGO layers. The aggregation of SnO2 nanoparticles has significantly decreased, and the nanoparticles of SnO_2 which covered on the rGO sheets are uniformly dispersed, with a particle size of approximately 3-5 nanometers. This is because the sample was synthesized by a one-step microwave-assisted hydrothermal method, and the reduction of graphene oxide and the synthesis of SnO_2 were

completed simultaneously. The strong reducing agent, hydrazine hydrate, was added to convert GO into rGO. This reduction process helps to restore the conjugated structure of graphene, increase its electron mobility, and enhance the interaction with SnO_2 nanoparticles, encouraging the creation and distribution of SnO_2 nanoparticles. At the same time, the surface of the reduced rGO has certain charges or functional groups, which can adsorb the SnO_2 precursor through electrostatic interaction or chemical bonding, inhibiting particle aggregation and allowing them to uniformly disperse on both sides of the rGO layers. Furthermore, unlike the conventional heating technique that slowly moves heat from the exterior to the inside of the autoclave, this method allows for the immediate creation of the crystal nucleus without any temperature difference. In various areas, the expansion rates of particles remain consistent, with the created particles being uniformly small in size. Moreover, due to the synthesis under high pressure, the interfacial bonding force between the composite materials is stronger. Consequently, its peak reaction to 350 ppb NO_2 stands around 227.6, significantly surpassing SnO_2's response (34.6), with a notable rise in both response and recovery durations. The sensor is capable of identifying NO_2 concentrations down to 50 ppb, yielding a response rate of 2.61. Additionally, it exhibits notable consistency and specificity.

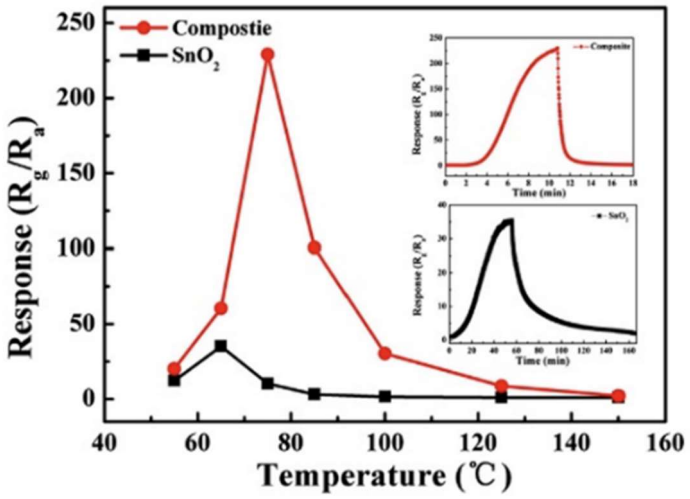

Figure 7. The reaction of sensors reliant on SnO_2/rGO and SnO_2 to 350ppb of NO_2 varies with temperature changes. The insets illustrate the temporary reaction patterns of SnO_2/rGO and SnO_2 to 350 ppb NO_2, measured at temperatures of 75℃ and 65℃, respectively.[19].

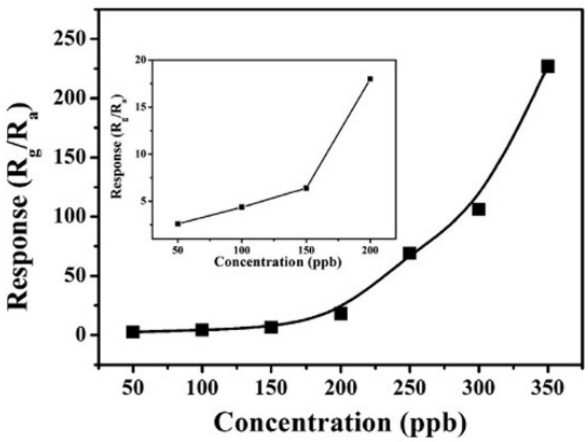

Figure 8. Sensor data that reacts to varying NO2 levels at 75℃, as determined by SnO_2/rGO [19].

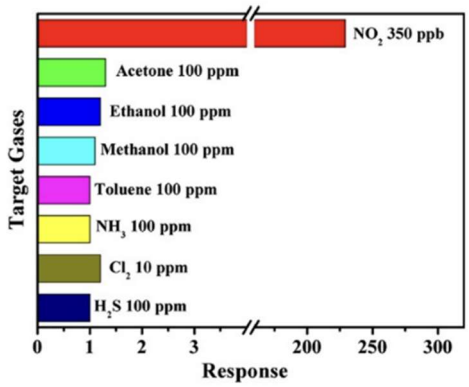

Figure 9. The SnO_2/rGO composite's reaction to various gases was measured at a temperature of 75°C. [19].

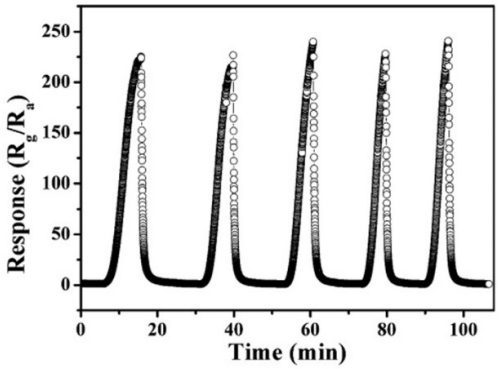

Figure 10. The set of five temporary reaction stages for SnO_2/rGO in response to 350ppb NO_2 at 75°C.[19].

4.2.2. Simultaneous Preparation with Metal Modification

Ramji Kalidoss prepared the GO-SnO_2-TiO_2 ternary nano-composite material by hydrothermal method and compared it with the binary materials GO-SnO_2 and GO-TiO_2[21]. Graphene oxide was synthesized using the improved Hummers method. $SnCl_2$ and GO were added to isopropanol, and then ultrasonically dispersed. Subsequently, the $SnCl_2$ and GO were gradually added to deionized water. After stirring evenly, the dispersion was transferred to a polytetrafluoroethylene high-pressure reactor and heated at 180°C for 16 hours. The precipitate was washed to obtain GO-SnO_2. GO-TiO_2 was obtained using a similar method. The GO-SnO_2-TiO_2 ternary nano-composite material was prepared by dispersing graphene oxide in isopropanol first, and then adding stannous chloride and isopropyl titanate at the required stoichiometric ratio and stirring. After adding deionized water, it was transferred to a stainless steel high-pressure reactor lined with polytetrafluoroethylene and heated at 180°C for 18 hours. Finally, the solid product was washed to obtain the solid product. The achieved ternary composite material featured graphene sheets entirely encased in numerous SnO_2 and TiO_2 nanoparticles, ensuring thorough integration. This action hindered not just the rearrangement of two-dimensional graphene layers but also lessened the clustering of SnO_2 and TiO_2 nanoparticles. In addition, the nanoparticles were evenly distributed on the single-layer thick graphene sheets. This may be due to the closely and uniformly combined method of simultaneous preparation of the three. The gas sensing test revealed that the sensor had the best working temperature of 200°C for 5 ppm acetone. At the 200°C, the ternary sensor's reaction to minimal acetone levels was accurately linearly aligned and consistently repeatable, with a brief recovery period. Furthermore, it's evident that the ternary composite sensor exhibits strong acetone selectivity, possibly because acetone possesses a greater dipole moment, enhancing its interaction with the exposed crystal plane. Yet, as humidity levels rise, its reaction continues to be reduced.

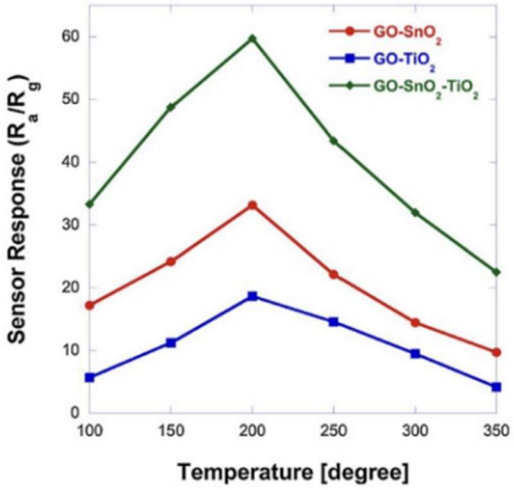

Figure 11. Data on how GO-SnO_2, GO-TiO_2, and GO-SnO_2-TiO_2 react to acetone concentrations of 5 ppm at varying temperatures.[21].

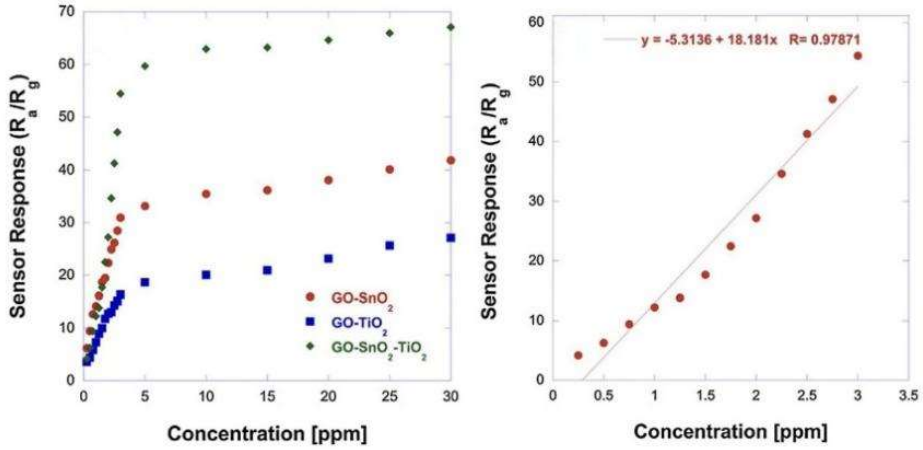

Figure 12. Curve of Linear Fitting for GO-SnO_2-TiO_2sensor in Low Concentration Levels and the Reaction Curve of Binary and 3D Nanocomposites to Acetone Levels at Ideal Operating Temperature of 200℃[21].

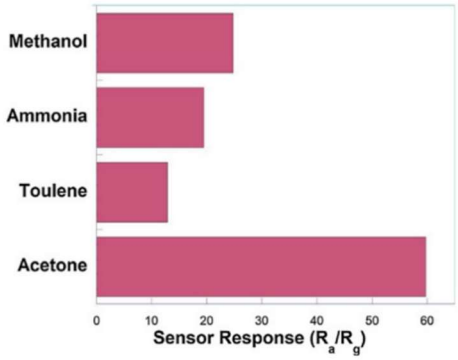

Figure 13. The reaction of GO-SnO_2-TiO_2ternary nanocomposite to varying concentrations of 5 ppm of VOCs was conducted at a temperature of 200℃.[21].

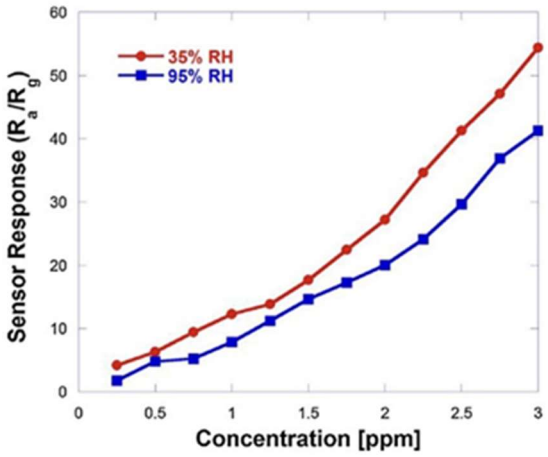

Figure 14. The reactions of GO-SnO_2-TiO_2 nanocomposite when exposed to low levels of 35% and 95% relative humidity. [21]

4.2.3. Ultrasonic-Assisted Hydrothermal Method

Aditya Choudhari prepared the rGO/SnO_2 nanocomposite material by using the ultrasonic-assisted method[23]. The graphite oxide was prepared by the modified Hummers method with the assistance of ultrasonic waves. The prepared GO was dissolved in distilled water and then ultrasonically treated. After that, $SnCl_2$ was added and ultrasonically treated again. The synthesized rGO/SnO_2 nanocomposite was centrifuged and separated. The sample was washed and dried in a 100°C oven to obtain the product. The cavitation effect caused by the generation of a large number of bubbles by ultrasonic waves enables the SnO_2 nanoparticles to be uniformly distributed on the surface of rGO, reducing the agglomeration phenomenon. Moreover, the collapse of the transient holes generated by ultrasonic waves leads to a strong shear effect and turbulence, reducing the size of SnO_2 particles to 3-5 nm. The uniform dispersion of SnO_2 nanoparticles increases the surface area of the material, making it have excellent gas sensing performance. We can clearly see that the sensor still responds to 5 ppm of NO_2 gas at 150 ℃. However, the recovery time sharply climb from 182 seconds to 509 seconds when the concentration of NO_2 growth from 40 ppm to 100 ppm. This may be due to the occupation of active sites on the material by the gas. At 150 ℃, the gas adsorption of the sensor reaches saturation at around 40 ppm, and it has excellent selectivity for NO2 at a working temperature of 150 ℃ and a concentration of 100 ppm.

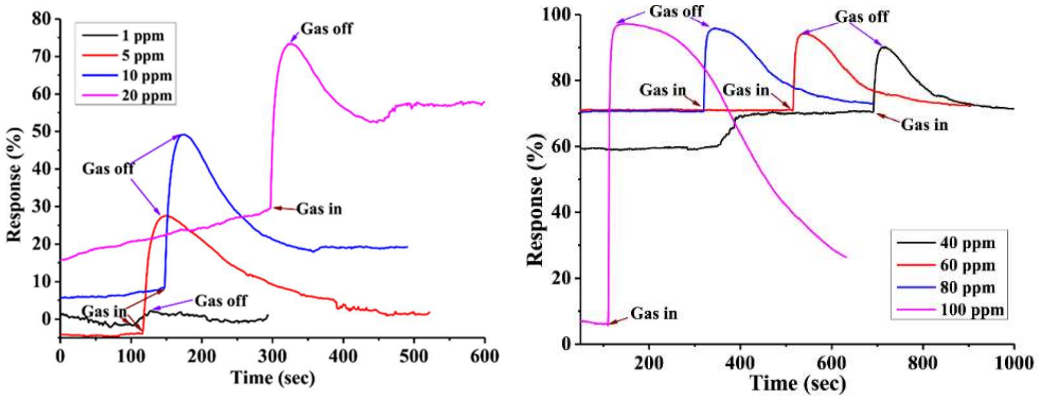

Figure 15. Response curves of rGO/SNO_2 nanocomposites prepared with ultrasonic assistance to different concentrations (ppm) of NO_2 at operating temperature of 150°C[23].

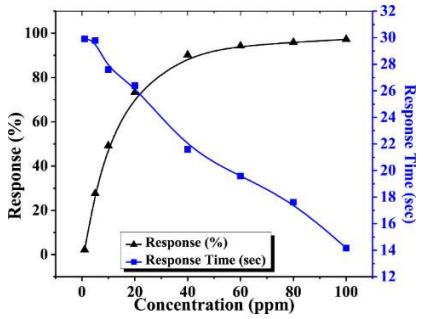

Figure 16. Response (%) and response time (s) of rGO/ SnO_2 at different concentrations (ppm) at operating temperature of 150℃[23].

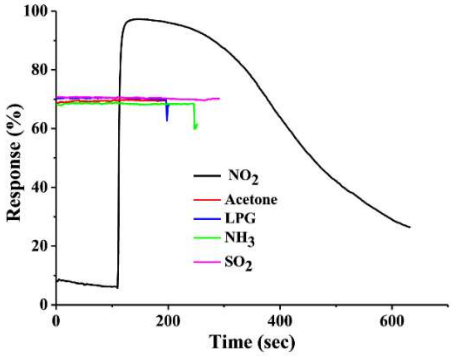

Figure 17. response curves of rGO/SnO_2 nanocomposites to sensors at operating tempera ture 150℃ and 100 ppm concentration assisted by ultrasound[23].

5. CONCLUSION

In the field of gas sensor fabrication, various hydrothermal synthesis methods based on the composite system of SnO_2 and graphene can significantly enhance the sensitivity, response speed, selectivity and durability of gas sensors. The development of society and industry determines the importance of detecting low-concentration gases. This review aims to analyze the subtle differences in the existing hydrothermal/solvent-based synthesis methods for the derivatization of SnO_2/graphene from a unique classification perspective. It is expected to promote the development of highly sensitive gas sensors to address gas sensing issues in food transportation, healthcare, indoor air control, and factory safety and construction.

From the existing improvement methods of gas sensors, we can obtain the following principles: the crystal size of metal oxides should be as small and dispersed as possible; graphene and its derivatives should avoid overlapping to increase the contact area with gases; additives for the composite structure or structural design of SnO_2 can all improve the response performance. At the same time, we also found that excessive activators will affect the adsorption and desorption speeds of gas molecules. The influence of humidity changes on gas sensors is inevitable, and the underlying mechanism of gas selectivity is still unclear. How to solve these problems in the future, and reduce costs and overcome preparation difficulties, will become the development direction of the industry.

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