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Green Synthesis of SnO_2/Carbon Quantum Dots Nanocomposite for Gas Sensing Application

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ABSTRACT

SnO_2/Carbon Quantum Dots (CQDs) were synthesized by a hydrothermal method using grape fruit juice. The nanocomposites (NC) were characterized by means of XRD and Gas sensing properties. The sensor devices were fabricated using SnO_2/CQDs NC as sensing materials. The effect of the CQDs content on the gas-sensing responses and the gas-sensing selectivity was investigated. In this work, the gas sensor developed is exposed to carbon monoxide polluting gas like at different temperatures to determine the optimum operating temperatures which allow obtaining the highest sensitivity for gas.

Keywords: Green synthesis, Grape fruit, SnO_2 nanoparticles, CQDs, Gas sensor

INTRODUCTION

Green synthesis is defined as the use of environmentally compatible materials such as bacteria, fungi and plants in the synthesis of nanoparticles [1]. These green and economical strategies are free of the short falls associated with conventional synthetic strategies, i.e. they are environment-friendly [2,3]. We have synthesized a carbon dot compositor with SnO_2 nanoparticles by green preparation method and gas sensors are widely used in hydrothermal processing [4]. They are one of the indispensable technologies in modern life. Materials which change their properties depending on ambient gases can be used as gas detection materials [5]. Optical or electrical signals can be used to detect gas concentrations. For an optical gas sensor, the color of the sensor varies with the concentration of gas detected [6]. For an electric gas sensor, the gas concentration can be detected either by changing the resistance of the sensor resulting from a reaction of the gas with chemisorbed oxygen on the surface of the sensing material, or by varying the output voltage by applying a temperature gradient provided by a chemical reaction to a thermoelectric sensing material [7].

Many transition metal oxides exhibit sensitivity towards oxidizing and reducing gases by varying their electrical properties: usually changes in the electrical conductance in response to environmental gases are monitored [8]. SnO_2 is currently the most widely used material for detecting of various gases due to its suitable physicochemical properties and lower cost compared to available materials for the similar applications [9]. It is sensitive to gas CO at moderate temperature. Because gas sensing procedures strongly rely on the surface chemical reactions, there is a relationship between the gas sensitivity and their surface chemical activities [10]. SnO_2 has a high reactivity to reduce gases at relatively low operating temperatures due to the easy adsorption of oxygen at its surface. The presence of a certain metal or oxides on the sensing material surface would improve its sensing properties to certain gases [11].

Carbon quantum dots (CQDs) have received much attention in recent past

decades, due to their unique physical, chemical, optical and surface properties [12]. Carbon dots are recognized as carbon, having angstrom size poly aromatic carbon core shell, surrounded by amorphous carbon domains. However, their defined structure is still a matter of debate. Instead of other nano sized allotropes of carbons, carbon dots exhibited excellent biocompatibility, photo stability and innocuousness properties [13].

Basic mechanism of oxide gas sensor

The exact fundamental gas sensing mechanisms of SnO_2 are still not fully investigated. However, the principle of operation of SnO_2-based sensors lies on detecting the electrical conductivity changes experienced by an n-type material when surface chemisorbed oxygen reacts with the reducing gases, such as H_2S, H_2 or CO. In a simpler schematization of the detection mechanism, in clean air the conductivity of SnO_2 is low because the conduction electrons are bound to surface oxygen, whereas in the presence of a gas, electrons are no longer trap to the surface states and the conductivity increases. Therefore, the adsorption of gaseous species controls the surface resistance of SnO_2. As the gas sensing properties of SnO_2 are related to the chemisorption of gas molecule on their surface, a detailed understanding of the charge transfer in a chemisorption process is very important; one can refer a review on this aspect in literature [14]. Only brief summary is presented here. In air the band structures of SnO_2 change by adsorption of oxygen molecule. At its surface, the periodicity of the crystal structure is broken, leading to the creation of unsaturated sites or dangling bonds. In this case of oxygen molecule, it picks up electrons from SnO_2 surface, and charge transfer equation can be written as,

$$\beta/_2\, O_2(\mathbf{g}) + \alpha e^- \leftrightarrow O^{-\alpha}\beta \qquad \textbf{(1)}$$

Where α can take place values 1 or 2 depending upon the oxygen reduction states, and β may have values of 1 for single atom/ionic form and 2 for molecular form of oxygen. The charge transport process between atmospheric O_2 and SnO_2 surface which results in the development of a space charged layer at the surface.

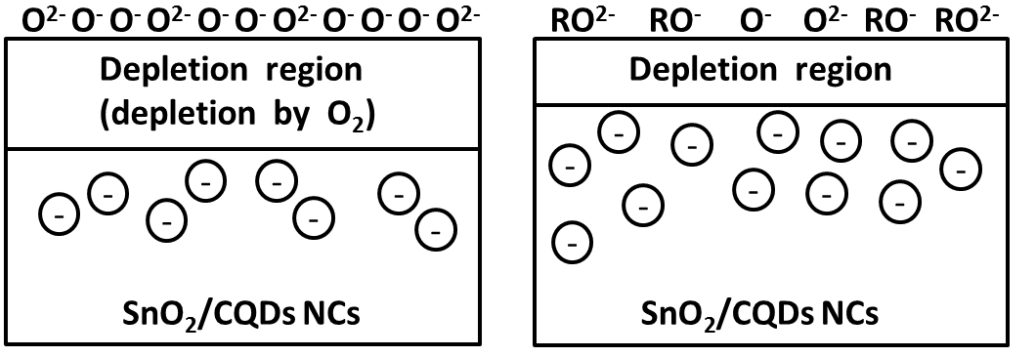

Figure 1: (a) Oxygen extract electrons from metal oxide thereby decreasing conductivity, and (b) when reducing agent (R) is present, electrons are injected into the oxide again and thus increasing conductivity.

The total charge at the surface produces an electric field, which causes a twisting of the energy bands in SnO_2. A negative surface charge bends the bands growing, i.e. pushes the Fermi levels in the band gap of SnO_2, effectively reduce the charge carrier concentration and resulting in an electron depletion zone. In other words, this is an electron trapping process at the surface, which leads to a decrease in conductivity) of the surface layer. When a reducing molecular gas adsorbs at its surface, electrons can be transferred to these molecules if the lowest unoccupied molecular orbitals (LUMO) of the adsorbatelie below the Fermi levels (acceptor levels) of SnO_2 and vice-versa electrons are donated to SnO_2 if the highest

occupied molecular orbitals (HOMO) lie above the Fermi levels of the SnO_2 (donorlevels). When a reducing gas, e.g. CO, reacts with the adsorbed oxygen to form CO_2, which can be expressed by the following oxidation reaction,

$$\beta CO(g) + O^{-\alpha}\ \beta \rightarrow \beta CO_2(g) + \alpha e^- \qquad (2)$$

The electrons released in this reaction are injected into the conduction band of the SnO_2 which results in a decrease in the electrical resistance (or increase in conductivity). The formation of the depletion region by adsorption of oxygen and reduction of the depletion region by adsorption of reducing gas is schematically shown in Figure 1.

Experiment details

Synthesis of Tin oxide nanoparticles

$SnCl_2.2H_2O$ (10 g) was added to 30 ml of grape fruit juice taken in a beaker and the reaction mixture was stirred for 2 h at about 60°C and left overnight for digestion. The resulting pale-yellow mixture was filtered and the residue was washed with the mixture of ethanol and water (1:1) and dried at room temperature. It was then calcined in muffle-furnace at about 600°C for 4 h. A pale yellow colored SnO_2 was obtained. The scheme of preparation of SnO_2 nanoparticles are illustrated in figure 2.

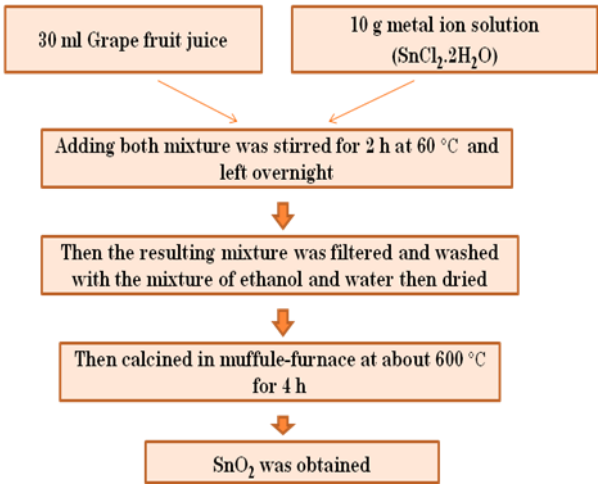

Figure 2: The scheme of preparation of SnO_2 nanoparticles using grape fruit juice

Green Synthesis of Carbon Quantum Dots

The grape fruit used as a carbon source for the synthesis of CQDs. The grapefruit was cut into small pieces and fresh juice was squeezed out. The obtained juice was centrifuged at 8000 rpm for 10 min; the above supernatant was passed through a filter paper to get pulp-free grapefruit juice. Then, 20 mL filtered grapefruit juice and 20 mL deionized water were mixed under stirring for 15 minutes. The above mixture was transferred into a Teflon-lined steel autoclave and heated at 180°C for 6 h. After the system was cooled to the room temperature, the obtained brown products were centrifuged for 15 min with a speed of 5000 rpm and then the resultant solution was dialyzed in a dialysis bag for 48 h. Finally, the products were freeze-dried for further analysis. The green synthesis of CQDs illustrated in figure 3.

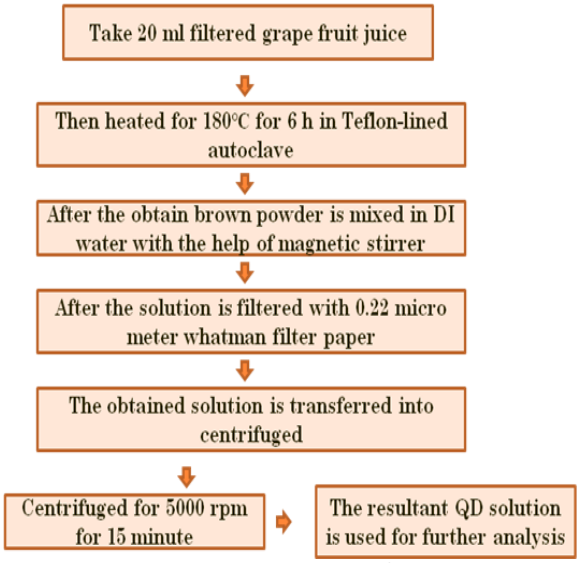

Figure 3: Green Synthesis of CQDs

Nanocomposite of Green Synthesized SnO2 and Carbon Quantum Dot

The synthesis of carbon quantum dots and SnO_2 nanoparticles NC, the carbon quantum dots (10 ml), and 2 gm of SnO_2 nanoparticles were mixed, and kept in microwave in cyclic mode for 2 min. Finally, obtained dark brown color precipitates of carbon quantum dots and SnO_2 nanoparticles nanocomposite. Further,

this nano mixture is used as active material in gas sensing application.

RESULTS & DISCUSSION

XRD analysis

The XRD patterns of as-prepared SnO_2/CQDs NC are shown in figure 4. The obtained diffraction patterns of SnO_2/CQDsNC are compared with JCPDS (Joint Committee for Powder Diffraction Studies) data card no. 46-1088. Peak intensity and close match between the observed and the diffraction peaks of as-prepared sample were consistent with those of pure tetragonal rutile structure and no diffraction peaks ofother substance could be found, which manifested that the existence of CQDs did not influence the crystalline structure of SnO_2.

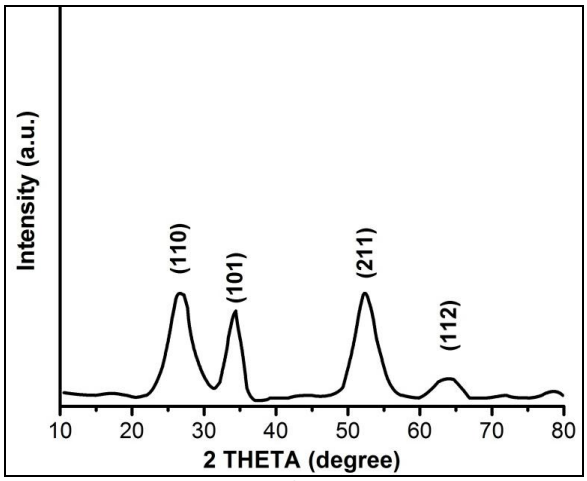

Figure 4: The XRD patterns of as-prepared SnO2/CQDs NC

The X-Ray diffraction was done for SnO_2/CQDs NC using X–rays with wavelength of 1.54 Å that is shown in Figure 4. The peaks were observed at 27.3°, 34.2°, 52.5°, and 64.6°, which corresponds to planes (110), (101), (211), and (112) respectively.

Gas-Sensing properties

Temperature dependent response of SnO_2/CQDs NC

The gas-sensing responses of SnO_2/CQDs NC to 1000 ppm carbon monoxide at different operating temperatures are shown in Figure 5. The SnO_2/CQDs NC shows highest performance towards at carbon monoxide 275ºC.

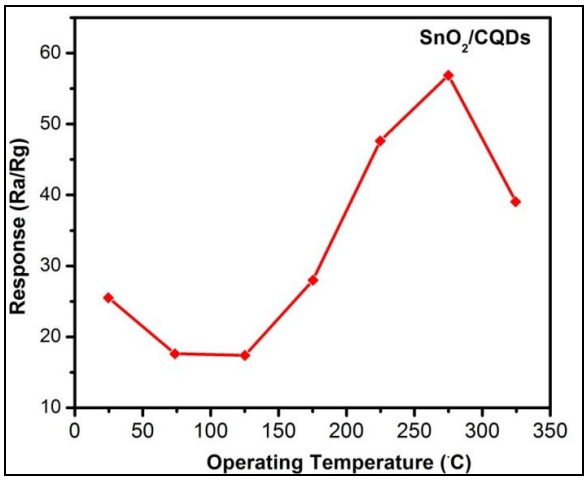

Figure 5: The gas-sensing responses of SnO_2/CQDs NC 1000 ppm CO at different operating temperatures

Figure 6 shows the transient response of the SnO_2/CQDs NC nanosensor at an operating temperature of 250°C towards 500 ppm of CO gas. Initially the sensor shows a stable resistance of 600 kΩ which instantaneously drops to 110 kΩ in about 9 sec (response time) as shown in the Figure 4. When the CO gas is expelled from the chamber the resistance starts increasing again and regains the initial value in about 11 sec (recovery time).

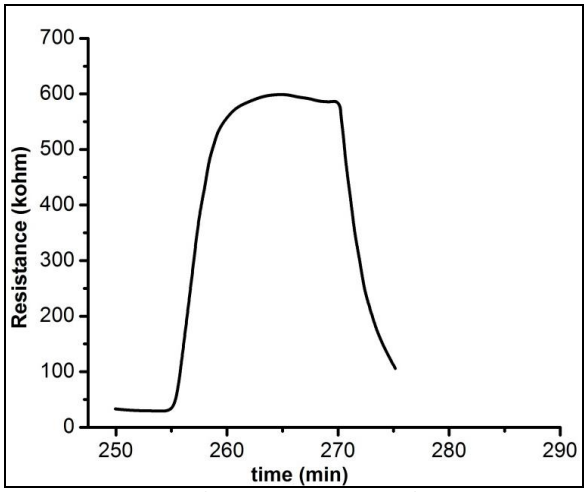

Figure 6: Stability of transient response of SnO_2/CQDs NC nanosensor

CONCLUSION

SnO_2/carbon quantum dots (SnO_2/CQDs) NC were prepared via hydrothermal method. The addition of

CQDs had a great effect on the gas sensing responses and the gas-sensing selectivity of the SnO_2/CQDs nano-composites. The SnO_2/CQDs NC shows highest performance towards at carbon monoxide 275ºC. Nanostructured-SnO_2/CQDs have been integrated as high sensitive material. Carbon monoxide gas detection tests show better responses in term of sensitivity.

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Conflict of Interest: None

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