J Nanostruct 13(1): 304-314, Winter 2023

RESEARCH PAPER

Detection of Carbon Monoxide Gas by Graphene Oxide/Silver and Graphene Oxide/Manganese Nanocomposites Synthesized by Ultrasonic Waves

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ARTICLE INFO

Article History:
Received 19 October 2022
Accepted 27 December 2022
Published 01 January 2023

Keywords:
Carbon monoxide
Gas sensor
Graphene oxide
Nanocomposite

ABSTRACT

Carbon monoxide is a tasteless and odorless gas produced by gas burning engines and the incomplete combustion of hydrocarbons. Carbon monoxide poisoning occurs due to the respiration of this gas. Lack of proper exhaust and ventilation of it causes poisoning and death. So inhalation of it reduces blood oxygenation and is dangerous. Therefore, timely and correct identification of this gas is important. Carbon monoxide is difficult to detect and can cause other common disorders. Graphene and its derivatives have been widely used for fabrication of gas sensors because they have two-dimensional atomic bonds that can interact by gas molecules. In this study, graphene oxide was used to detect carbon monoxide. In order to improve the response time, the sensitivity of the sensor and its selectivity were composite by silver and manganese nanoparticles. Graphene oxide was prepared by advanced Hummers method. Ultrasonic device was also used to prepare graphene oxide/silver and graphene oxide/manganese nanocomposite. The results show that the sensor prepared by graphene oxide/silver and graphene oxide/manganese nanocomposites are able to detect carbon monoxide at room temperature, with appropriate sensitivity and selectivity.

How to cite this article
Badkoobeh Hazaveh P., Nabiyouni G., Ghanbari D. Detection of Carbon Monoxide Gas by Graphene Oxide/Silver and Graphene Oxide/Manganese Nanocomposites Synthesized by Ultrasonic Waves. J Nanostruct, 2023; 13(1):304-314. DOI: 10.22052/JNS.2023.01.030

INTRODUCTION

Carbon monoxide is a colorless and odorless and highly toxic gas[1]so that is also called ''silent killer'' [2]. Carbon monoxide produce by incomplete combustion of hydrocarbons and carbon-containing substances[3]. Faulty heating appliances cause accidental carbon monoxide poisoning[4] and other source of carbon monoxide gas can be mentioned unfavorable ventilation of the heating system, fire and engine exhaust [5]. Concentrations of more than 800 ppm indoors can endanger human health[6]. The tendency of carbon monoxide to bind to blood hemoglobin is more than 240 times that of oxygen[7].Carbon binding to blood hemoglobin causes less oxygen to be carried [8], because carbon monoxide bonds to blood hemoglobin are stronger than oxygen bonds to blood, so carbon monoxide binds easily

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to blood and interferes with oxygen delivery [9]. Complications of carbon monoxide poisoning are not specific [10],because carbon monoxide can cause other common disorders, which their full effects are still unknown [11].Detection of carbon monoxide is difficult [12]. Therefore, correct and timely identification of carbon monoxide is important.

Gas sensors are known for their high sensitivity, which, they cost little to build and they are so small that they can be used in a wide range of fields [13, 14].There are different types of carbon monoxide gas sensors such as semiconductor [15], based on polymer [16], carbon nanotubes[17] graphene based and doped graphene[18]. Graphene is graphite's monolayers that arranged by carbon atoms in a honeycomb structure

[19].In 2004, Geim and his colleagues were able to make a layer of graphite which leads to the win Nobel Prize on year of 2010 [20, 21]. Graphene can produce from different ways such as chemical method[22], vapor deposition[23] and mechanical method [24]. Graphene with its derivatives is the most versatile material that has ever been discovered [25]. Graphene is given the effective surface of graphene-based materials, so, their use as a means of measuring is a hot topic[26]. In 2007 the first graphene-based gas sensor was reported [27]. Graphene has been represented as a promising gas sensor application because electron transport through graphene is highly sensitive to adsorbed molecules since it has 2D structure that makes every carbon atom a surface atom [28]. Graphene and its derivatives like graphene oxide and reduced graphene oxide have high conductivity, a special surface that gas can also be easily absorbed by the surface and easily can be improved by functional groups, so they have good gas sensing properties[29].

Graphene oxide is derivatives of graphene [30] that was first synthesized in 1859 by Benjamin so earlier than graphene was discovered [31]. Compared with graphene, there are different kind of oxygenated functional groups on the graphene oxide sheet[32] such as hydroxyl, carbonyl, epoxy, carboxylic[33]. Staudenmaier, Brodie and Hummers method are three methods for preparing graphene oxide [34, 35].These methods use potassium acids and chlorates [36]. The Hummers method uses a mixture of concentrated $KMnO_4$, $NaNO_3$, and H_2SO_4 [37].The most commonly approach to synthesis graphene oxide is Hummers method which is based on exfoliation of graphite[38, 39]. As mentioned graphene oxide has different functional groups which can interact with gas molecule [40].To improve the sensitivity and selectivity of graphene oxide to gas molecules, metals and metal oxide nanocomposites can be added to it [41]. Graphene oxide has different applications such as drug delivery [42], biomedicine and biotechnology [43], Carbocatalysts [44], gas sensors [45]. In this study, graphene oxide was prepared by Hammers method and used to identify toxic carbon monoxide gas due to its easy preparation and high sensitivity to gas molecules. In order to improve the detection, selectivity and sensitivity of graphene oxide was composited with manganese and silver metals, by ultrasonic assist.

MATERIALS AND METHODS

Hummer's method

Graphene oxide was synthesized by the Hummers method. 2 g of graphite powder was poured into beaker. 80 ml of sulfuric acid was added and stirred by magnetic stirring. Then 12 g of potassium permanganate and 10 ml of phosphoric acid were added while mixing. In this case, a green sludge mixture was obtained. Then 46 ml of ionized water was carefully added to the reaction vessel and allowed to stir for 30 minutes. After this step, 16 ml of hydrogen peroxide was added to it and a brown suspension was obtained. The suspension was placed in an ultrasonic bath for 60 minutes, and then washed it to neutralize the acidic state. The product was then dried at 50 ° C for 24 hours. The synthesized graphene oxide was then pulverized by a ball mill.

Synthesis of silver nanoparticles

For the synthesis of graphene oxide/silver nanocomposite, 0.3 g of silver nitrate was stirred with 100 ml of ionized water by a magnetic stirrer. After mixing, 1 g of lactose was added as a surfactant and continued mixing until dissolved. Then 5 ml of ammonia was added for sedimentation and stirring was completed until complete sedimentation. After this step, 2 g of graphene oxide powder was synthesized and 100 ml of ionized water was added to the mixture and stirred it to dissolve.

Finally, to form graphene oxide/silver nanocomposite, the resulting compound was subjected to 150-watt ultrasound for 21 minutes,

which was given a 3-minute break after every 3 minutes. Finally, the product was dried in an oven at temperature of 50 ° C for 24 hours.

Synthesis of manganese nanoparticles

2 g of the synthesized graphene oxide powder was mixed with 1 ml of manganese chloride solution and 200 ml of ionized water using a magnetic stirrer till the chemicals were dissolve. Then, to prepare the graphene oxide/manganese nanocomposite, the obtained compound was subjected to ultrasonic waves for 21 minutes in such a way that after every 3 minutes, the pulse was rested for 3 minutes. It was then dried in an oven at temperature of 50 ° C for 24 hours.

RESULT AND DISCUSSION

Characterization

Fig. 1a shows the X-rays diffraction of synthesized graphene oxide. The XRD pattern of our synthesized graphene oxide is in good agreement with which obtained by other research group [46]. The peak at diffraction angle of around 10 ° with the Miller index (001), is the only characteristic peak of graphene oxide. The average crystalline size is calculated to be about 12 nm. Peaks in the range of 40 ° is related to incomplete oxidation and lack of formation of graphene plates and possibly related to graphite and Miller index (100). Fig. 1b shows the X-ray diffraction pattern of graphene oxide/silver nanocomposites. The obtained pattern is consistent with the reference codes of 01-087-0717 and 00-026-1077. The peaks at 58° and 104° with Miller index of (111) and (220) are related to silver. Two diffraction peaks at 39°and 65 °(Miller of index (002) and (100)) are observed which correspond to the graphite. The crystal structure of silver nanoparticles has a cubic phase with number

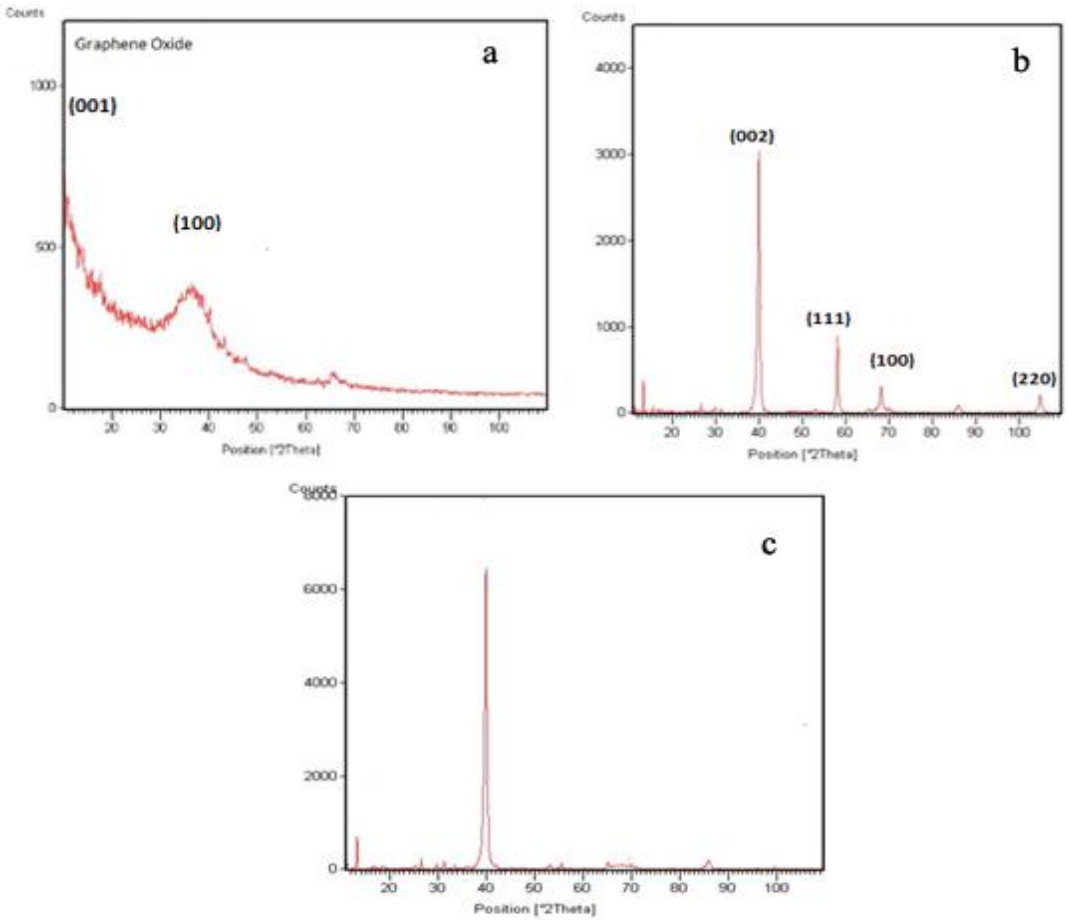

Fig. 1. X-ray diffraction of synthesized a) graphene oxide b) graphene oxide/silver nanocomposite c) graphene oxide/ manganese nanocomposite.

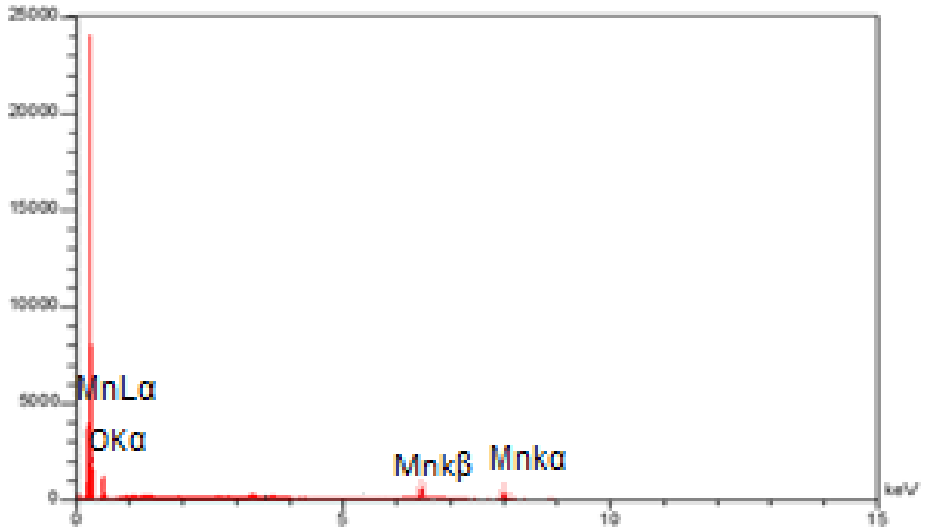

Fig. 2. EDX analysis of graphene oxide/manganese nanocomposite.

225 and space group of Fm-3m. The average size of the crystals was calculated to be about 18 nm. Fig. 1c shows the X-ray diffraction pattern of a graphene oxide/manganese nanocomposite. Due to the low amount of manganese in the sample the corresponding peaks are weak and the predominant structure is related to graphene oxide and its derivatives. EDX analysis was used to better prove the presence of manganese (Fig. 2 4), Which well proves the presence of manganese, oxygen and carbon. Peaks MnL_{α}، Mnk_{β} and Mnk_{α} indicate the presence of manganese. The average

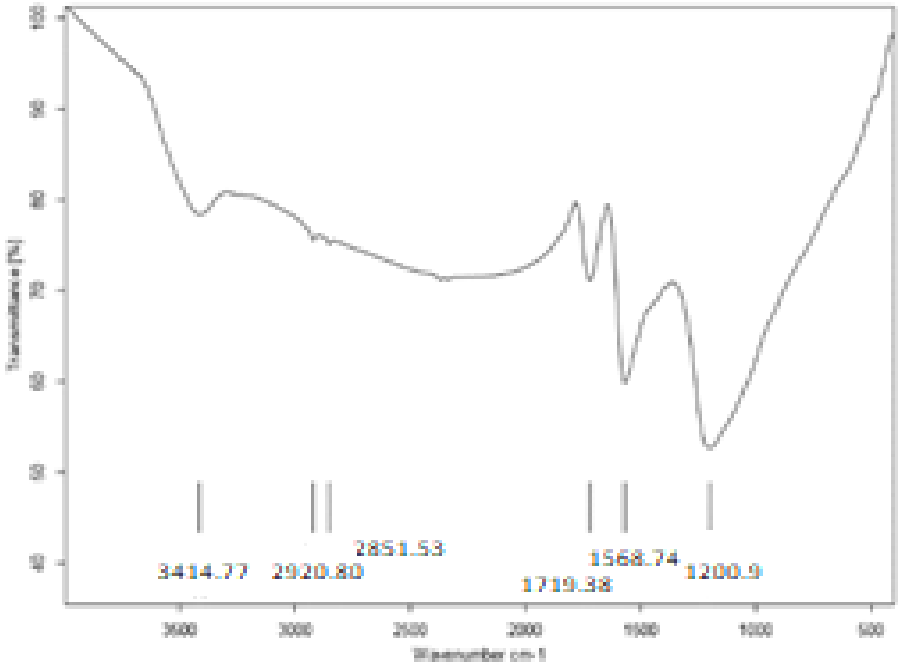

Fig. 3. FT-IR analysis of graphene oxide.

size of the crystals is about 18 nm.

Fig. 3 shows the infrared spectrum FT-IR of synthesized graphene oxide. As shown in the figure, the absorption spectrum in the range of 3414 cm^{-1} is related to the oxygen-hydrogen (O-H) bond in the hydroxyl group. The small absorption peak at

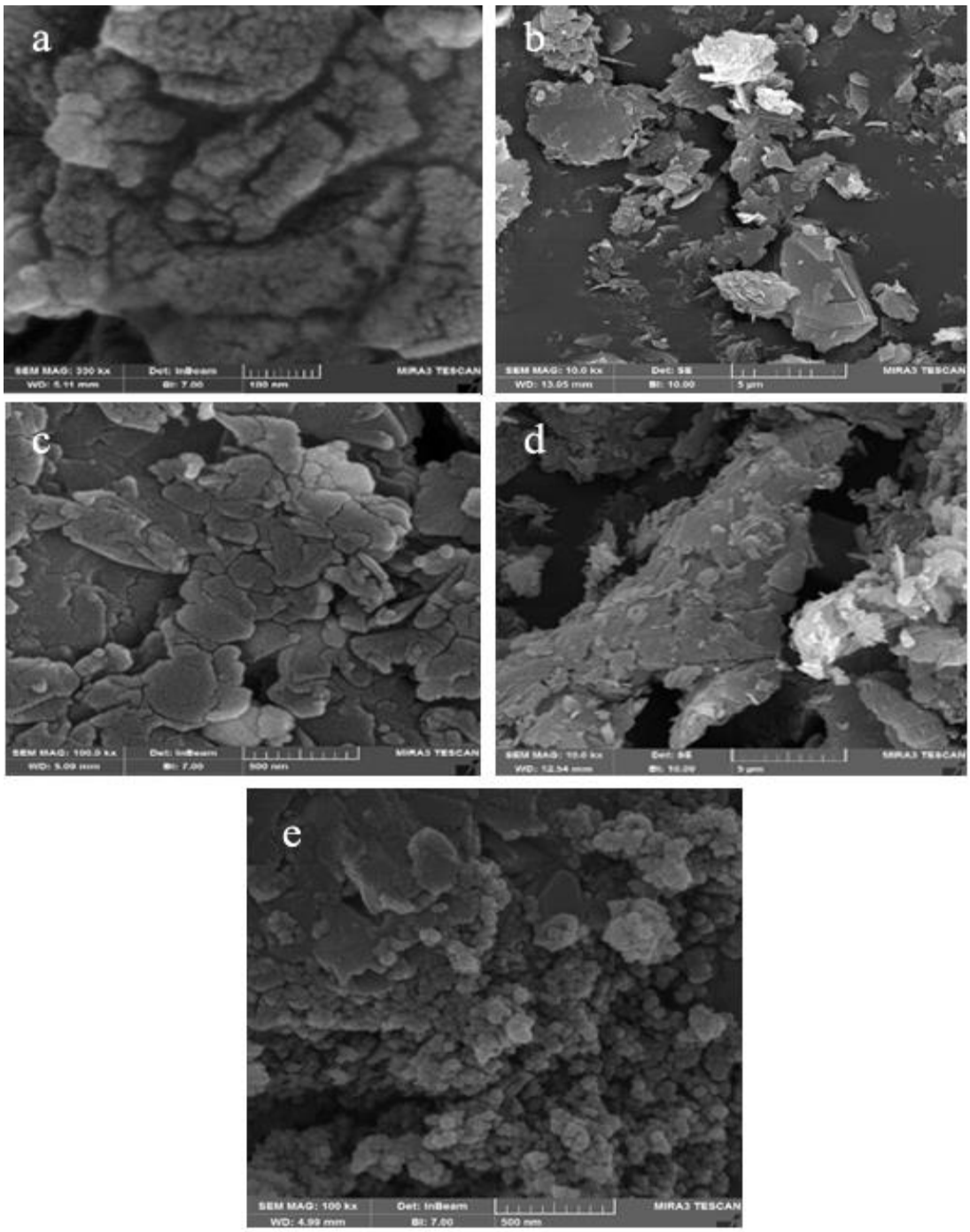

Fig. 4. SEM images of a) graphene oxide b) Graphene oxide/silver nanocomposite before applying ultrasound waves c) Graphene oxide/silver nanocomposite after applying ultrasound waves d) Graphene oxide/manganese nanocomposite before applying ultrasound waves e) Graphene oxide/manganese nanocomposite after applying ultrasound waves.

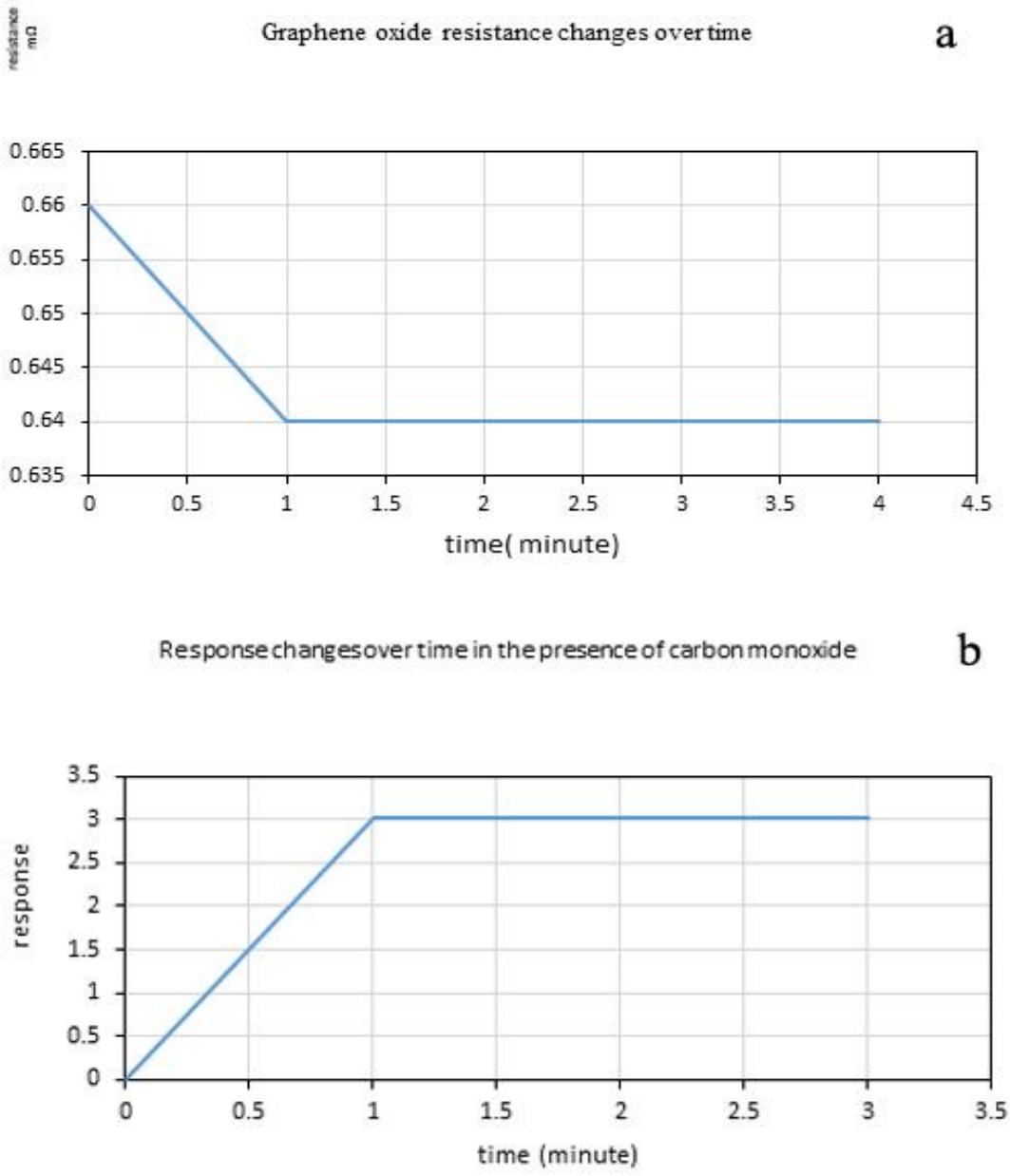

Fig. 5. a) Graphene oxide resistance changes over time in the presence of carbon monoxide b) Graphene oxide response changes over time in the presence of carbon monoxide.

about 2900 cm^{-1} is related to the carbon-hydrogen (C-H) bond. The peak at 1528 cm^{-1} corresponds to the adsorption of the carbon-oxygen (C=O) double bond to the carbonyl group. The peak at 1200$cm^{-1}$is due to the dual bond of carbon to carbon (C=C).

Fig. 4a shows a scanning electron microscope (SEM) image of synthesized graphene oxide nanoparticles. The pictures were taken by MIRA3 TESCAN device. These images show nanoparticles at a scale of 100 nm with a magnification of 330,000 times and a working distance of 511 mm, 5 µm with 10,000 times magnification and a working distance of 5.09 mm. As shown in the picture, the graphene oxide plates are uniform but separated and broken by ultrasound waves while the carbon quantum dots are well formed. Fig. 4b shows SEM

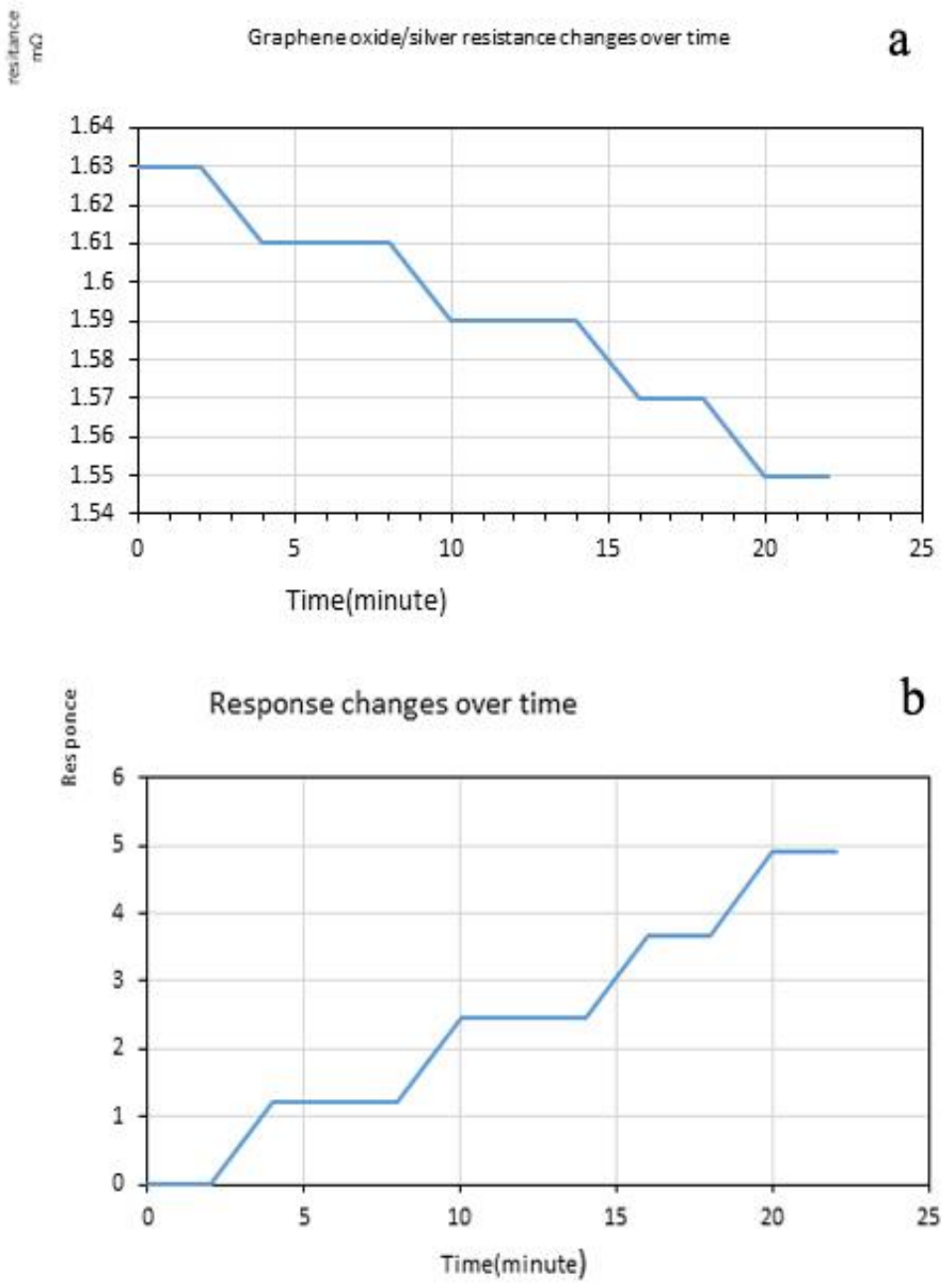

Fig.6. a) The rate of change in resistance of graphene oxide/silver nanocomposites in the presence of carbon monoxide b) Graphene oxide/silver nanocomposite response changes over time in the presence of carbon monoxide.

image of graphene oxide/silver nanocomposite before ultrasound waves is applied. This image is taken at a scale of 5 μms with a magnification of 10000 times and a working distance of 13.05 mm. As can be seen from the picture, the graphene oxide sheets are not separated and are lumps. Fig. 4c show the SEM images, specification with 500 nm, 100000x magnification and 5.09mm working distance and 200nmwith 200,000x magnification of 1.04μm working distance. After the application of ultrasound waves, it is clear that the graphene plates are broken and uniform, and the carbon

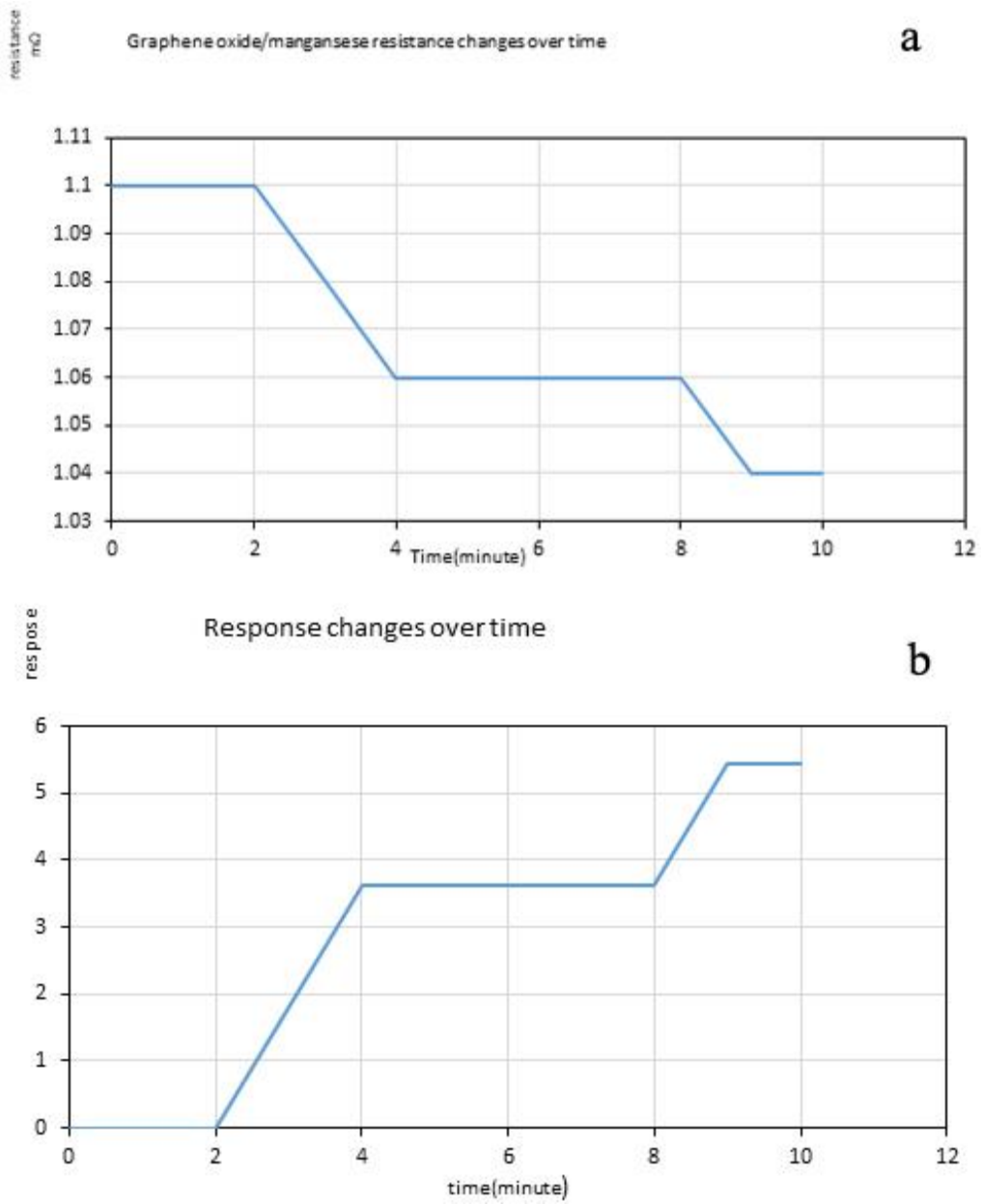

Fig. 7. a) Graphene oxide/manganese resistance changes over time in the presence of carbon monoxide b) Graphene oxide/manganese nanocomposite response changes over time in the presence of carbon monoxide.

quantum dots are well formed and at the nanoscale. Fig. 4d shows the SEM image of a graphene oxide/manganese nanocomposite before applying ultrasonic waves with a specification of 5 µm with a magnification of 10000 and a working distance of 12.54 mm. As can be seen from the picture, the graphene sheets and plates are not well formed and separated, nor they are uniform. Fig. 4e show SEM images at 500nm with 100,000x magnification and 4.59mm work distance and 100nm with 330,000x magnification and 5.09 mm working distance. After the application of ultrasound waves, as it is known, graphene plates are broken and uniform and are on the nanometer

scale. Also, carbon quantum dots are well formed.

Carbon monoxide gas detection

Gas measurement occurs due to surface oxygen and oxygenated functional groups. Carbon monoxide from the surface of graphene oxide and its composites binds with oxygen to form carbon dioxide.

$$CO_{gas} + O_{ads} \rightarrow CO_2$$

Loss of oxygen from the surface of graphene oxide changes the concentration of electric charge carriers and can be detected as an electrical signal. That is, because the electronegativity of oxygen is high, it absorbs surface electrons and changes the number of electrons. And after the presence of carbon monoxide and its bonding with surface oxygen and the formation of carbon dioxide electron that has absorbed oxygen, it returns to the sample. Therefore, it increases the number of electric charge carriers, i.e. electrons, and causes a high electrical conductivity and a decrease in electrical resistance. Thus, the resistance of the samples is expected to decrease in the presence of carbon monoxide. Measurements were taken at room temperature in the presence of carbon monoxide. With the arrival of carbon monoxide after at least one minute, changes in electrical resistance have been observed. The resistance of the sensor prepared from graphene oxide in the presence of carbon monoxide gas began to decrease after one minute and it reached saturation and the resistance remained constant Fig. 5a. The sensitivity of graphene oxide synthesized over time to carbon monoxide is shown Fig. 5b, which shows that the response of the sample increases at first, but after one minute the sample is saturated and its sensitivity remains constant. Response is also calculated through the following equation [47].

$$S = (R_{CO} - R_{air})/R_{air} * 100$$

R_{co} and R_{air} are resistance in the presence of CO and air.

Fig. 6a shows the rate of change in electrical resistance of graphene oxide / silver nanocomposites in the presence of carbon monoxide. As shown in the diagram, after 2 minutes the electrical resistance of graphene oxide/silver nanocomposite begins to decrease and after 20 minutes it reaches saturation and remains constant. Fig. 6b shows the sample response in the presence of carbon monoxide. The sample response in the presence of carbon monoxide gas begins to increase after 2 minutes and this increase lasts up to 20 minutes.

Fig. 7a shows the rate of change in resistance of graphene oxide/manganese nanocomposites over time. As this diagram shows, the resistance of this sample decreases over time after 2 minutes and reaches to its minimum and saturates after 10 minutes. The response of the sample also increases over time. After 2 minutes, the sample responds to carbon monoxide. After 10 minutes, when it reaches to saturation, the sample response remains constant and no further changes over timeoccurs (Fig. 7b). Due to the catalytic properties of manganese and the presence of nanopores and the uniformity of carbon quantum dots, the adsorption and interaction of gas molecules with the surface sample increasesand the number of electrons increases which leads to increase the conductivity and decrease the sample resistance.

CONCLUSION

Due to the unique properties of graphene oxide such as surface to volume ratio and the presence of oxygenated functional groups and oxygen voids had a good interaction with carbon monoxide gas. This interaction changes the density of charge carriers, i.e. increasing the number of free electrons. Thus the electrical resistance decreases and the sensor conductivity increases.

The results obtained from the detection of carbon monoxide by graphene oxide/silver nanocomposites show that its electrical resistance to graphene oxide responds faster than the detection of carbon monoxide. This is due to the conductivity of silver and its catalytic properties. In other words, the presence of silver nanoparticles in the sample catalysisthe charge transfer process, which causes the gas to be detected in less time.

The results obtained from the detection of carbon monoxide by graphene oxide/manganese nanocomposite also show that its response time is faster than that of graphene oxide. Existence of uniform surface and quantum carbon points of adsorption and interaction of gas molecules with the sample surface is increased as well. The presence of manganese also facilitates the identification of target gas and increases the conductivity and decrease the sample resistance.

Therefore, the addition of silver and manganese to graphene oxide improves its detection, selectivity and sensitivity to carbon monoxide.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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