AUT Journal of Electrical Engineering

AUT J. Electr. Eng., 56(3) (2024) 399-406
DOI: 10.22060/eej.2024.22943.5576

Carbon Nanoparticles-Based Sensor Analysis

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ABSTRACT: In this article, an electrochemical sensor in the form of a field effect transistor is designed and fabricated. This sensor is based on carbon nanoparticles grown between two metal electrodes. These nanoparticles have been synthesized by the electric arc discharge method in suitable environmental conditions and in different lengths from the decomposition of butane gas. Considering this set as a field effect transistor, its channel is always established and it is responsible for detecting the desired material. To check the correct functioning of the sensor, its sensitivity to different concentrations of saltwater was checked and the resistance diagram of the sensor as its electrical output was obtained in the presence of different concentrations of saltwater for different lengths of the channel.

Review History:

Received: Jan. 24, 2024
Revised: Feb. 20, 2024
Accepted: Mar. 12, 2024
Available Online: Jul. 01, 2024

Keywords:

Nanoparticles
Sensor
Electrochemical Sensor
Arc Discharge Method
FET Sensors

1- Introduction

Electrochemical sensors have been considered due to the need for low-cost measurement equipment, low detection limits, and high speed. The receptor and transducer are the basic components of these sensors. The receptor interacts with the target molecule and the transducer converts the signal generated from this interaction into an electrical signal that can be read or recorded to obtain useful analytical information[1, 2]. One of the types of electrochemical sensors are carbon-based sensors. Carbon materials have played an important role in developing electrochemical sensors due to their low cost, non-toxicity, ease of access, and high chemical stability. In nanotechnology, carbon nanoparticles have attracted much attention in the field of sensors due to their high electrical and thermal conductivity and high surface-to-volume ratio[3, 4]. In the structure of carbon-based sensors, carbon materials such as quantum dots, fluorenes, graphene sheets, graphene oxides, and single-walled and multi-walled carbon nanotubes are used[5]. Graphene, fluorene, and single-walled and multi-walled carbon nanotubes are shown in Figure 1 as the most common carbon nanoparticles[6].

By including nanoparticles on the surface of the sensor, the area as well as the electrical conductivity of the sensor can be increased. These particles provide a wider active surface for the absorption and identification of various analytes, increasing the rapid synthesis of electron transfer[5].

Graphene, as one of the most popular carbon nanoparticles, is a single-layer graphitic sheet consisting of carbon atoms with sp^2 hybridization. Carbon atoms in this two-dimensional structure are placed together in the form of hexagonal cells by strong covalent bonds[7, 8]. In this hybridization, two s orbitals and two p orbitals, i.e. p_x and p_y, are hybridized and form new molecular orbitals[9]. Graphene was theoretically discussed in 1947, until in 2004, a carbon atom-thick layer of graphite sheet was obtained[10]. One of the characteristic features of graphene is its hardness and at the same time its high tensile strength. Another important feature of graphene is the high speed of electrons when passing through it and as a result, its high electrical conductivity, which can be very useful in making sensors[11]. One-dimensional nanotubes can be formed by rolling graphene, and zero-dimensional fluorene structures can be achieved by packing graphene[12].

Carbon nanotube can also be expressed as a rolled sheet of graphene. This one-dimensional nanoparticle can form hollow cylinders with a diameter of about 0.4 nanometers to tens of nanometers[13]. Depending on the number of graphene layers in this cylindrical structure, this particle can be divided into two main categories: single-walled carbon nanotubes consisting of a rolled graphene sheet (SWCNT) and multi-walled carbon nanotubes. The wall consists of several concentrically rolled graphene sheets (MWCNT). The multi-walled carbon nanotubes themselves are divided

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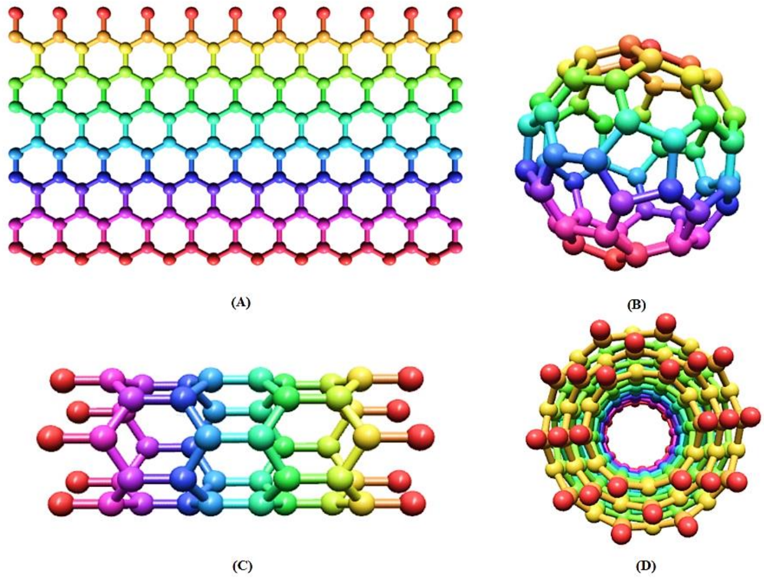

Fig. 1. (A): Graphene Sheet, (B): Fluorene, (C): Single-Walled Nanotube (SWCNT), (D): Multi-Walled Nanotube (MWCNT)

into two types. The first mode consists of rotating a graphene sheet several times (Swiss) and the second mode includes graphene sheets nested in the form of concentric cylinders. (Russian doll)[14]. Carbon nanotubes in this division can show different properties, for example, single-walled carbon nanotubes show semiconducting or metallic properties, while multi-walled carbon nanotubes generally have metallic properties[9]. The diameter of nanotubes is on the nanometer scale, while it can be synthesized in lengths of several millimeters to centimeters. For this reason, it can be said that it has a very high ratio of length to diameter, which is one of the main characteristics of these nanoparticles. For this reason, these particles are considered one-dimensional. This characteristic and the unique physical and chemical characteristics of carbon nanotubes have caused these particles to be used to improve the performance of sensors[12].

Graphene and carbon nanotubes, due to their unique structural features, which are completely composed of surface atoms, as well as their unique electrical properties, can be configured in the form of field effect transistors as a microelectronic system[15].

Fluorene is one of the common nanoparticles. In the structure of this three-dimensional nanoparticle, carbon molecules are spherical or hollow oval. In this structure, pentagonal and hexagonal faces are formed from carbon. The first example of this structure named C60 was discovered, which contained 60 carbon atoms[16, 17]. Each carbon atom in this structure bonds with three neighboring atoms due to existing hybridization. In pentagonal structures this bond is single and in hexagonal structures this bond is double[18, 19].

This article presents an electrochemical sensor fabrication method based on the synthesis of carbon nanoparticles between two metal electrodes. In the second part, the growth methods of nanoparticles are discussed. In the third part, the method of fabricating the sensor and in the fourth part of this article, the results of saltwater detection by this sensor are given.

2- Classification of nanomaterial synthesis methods in general

Nanoparticle synthesis methods can be generally classified into two classes[20]:

1-Top-down approach, 2-bottom-up approach

2- 1- Top-down approach

The top-down approach is a combination of chemical and mechanical techniques in the form of miniaturization processes. Meanwhile, ball milling is considered as one of the simplest mechanical techniques among low-cost methods for this approach. The basis of this approach is to break down bulk materials and convert them into nanoparticles. In this method, miniaturization processes such as attrition, milling, and etching can be used. This approach is mostly used to fabricate thin films and nanomaterials larger than 100 nm. Arc discharge methods, laser ablation, electrochemical oxidation, and ultrasonic methods are examples of this approach for the synthesis of nanomaterials.

2- 2- Bottom-up approach

Synthesis of nanoparticles in this method is done with less error. The basis of this approach, unlike the previous approach, is the creation of complex nanostructures in desired dimensions from atoms, molecules, and carbon nanoparticles. Citrate, carbohydrates, and small alcohols are examples of these small carbon organic precursors. In this approach, compared to the previous approach, the morphology, dimensions, and characteristics of grown nanoparticles can be controlled. Hydrothermal purification, microwave synthesis, thermal decomposition, sol-gel, and chemical vapor deposition are among the conventional methods in this approach[21-24].

2- 3- Common methods of carbon nanoparticles growth

Among the methods stated in the two general approaches in the synthesis of carbon nanoparticles, electric arc discharge, chemical vapor deposition, and laser ablation methods have been used more.

The electric arc discharge method involves applying a potential difference between two graphite electrodes as anode and cathode. This process usually occurs in the presence of inert gases such as argon. In this method, as a result of the discharge of the arc between the anode and the cathode and as a result of their temperature rising, the graphite is evaporated from the anode and deposited in the cathode in the form of carbon nanotubes and cooled[25]. The morphology of the electrodes, the energy source, and the distance between the electrodes are the determining factors in this process[26].

Another conventional method for the growth of carbon nanoparticles is the chemical vapor deposition method. This method is carried out at high temperatures and in the presence of a catalyst. In this method, carbon precursors, including hydrocarbons, are decomposed at high temperatures and after cooling will be available in the form of carbon nanoparticles. In this method, carbon nanotubes are usually obtained in bulk. Another feature of this method is the possibility of controlling the morphology of the synthesized nanotubes[19, 27].

In the laser ablation method, laser energy is used for the synthesis of nanoparticles. In this way, a laser pulse is irradiated to the solid carbon target, and by breaking and disintegrating the target, carbon nanoparticles are obtained. This process is usually done in aqueous or gaseous environment. Ease of use, no need for complex and multi-step chemical methods, and compatibility with the environment are among the positive features of this method[28, 29].

3- The growth mechanism of nanoparticles:

As it was said, among the conventional methods for the growth of nanoparticles, the electric arc discharge method can be a suitable option due to its cheapness and the possibility of controlling the growth. In this article, the growth of carbon nanoparticles with this method is presented. These nanoparticles have been synthesized in normal environmental conditions and at room temperature between two metal electrodes. After the completion of the synthesis and stability of the synthesized particles, according to the schematic shown for this system in Figure 2 the set of two electrodes and nanoparticles grown between them acts as a field effect transistor and its electrical behavior can be checked for different concentrations of the target analyte.

By applying the appropriate voltage between two electrodes and the presence of gas between them, an electric arc can occur as a type of electric discharge. This voltage is high and is proportional to the distance of the electrodes from each other. If we place a set of electrodes that can be placed at different micrometer distances from each other inside a chamber, the electric arc created by the application of voltage will break and ionize the air molecules between the two electrodes. In this work, butane gas is used between two electrodes. Therefore, butane is ionized between the electrodes and turns into carbon precursors. The generated ions are placed in the path of the strong electric field between the electrodes. This field gives force to the carbon ions and throws them towards one of the electrodes. With the deposition of the first ion on an electrode, the growth process is started, and other ions are also thrown towards the same electrode under the same field and collide strongly with the deposited ions, so that a covalent bond is established between them and the growth process continues. According to Figure 2, This phenomenon happens until a bridge of carbon ions is established between two electrodes. After the completion of the growth, in each length of the channel, the sensor output can be measured in the form of electrical resistance by a multimeter for different concentrations of the saltwater solution.

In this work, 9 samples with different concentrations of salt water have been used to measure and confirm the accuracy of the fabricated sensor. To prepare these samples, first by considering the solubility of salt in water, the most concentrated solution of salt water with a concentration of 0.12 g/ml was prepared, and then to obtain other concentrations, by increasing the amount of distilled water in each Compared to the previous test tube, diluted solutions were obtained in 8 different concentrations.

This is a bottom-up method. In this method, the synthesized carbon bridge between the electrodes is not pure and contains different types of carbon nanoparticles. As mentioned, after the growth and stability of the grown particles, this platform can be used as a FET sensor.

4- Results

4- 1- Resistance analysis of growth nanoparticles exponentially:

The relation between the electrical resistance of nanoparticles grown along different channels before injecting any material for detection as a Figure 3 is visible. This chart with $R = \alpha e^{\beta L}$ It can be expressed. In this equation, R is the electrical resistance of the sensor and L is the length of the sensor channel. Also, α and β coefficients are parameters that determine the type of grown nanoparticles. In this work, $\alpha = 0.0009$ and $\beta = 0.1306$ have been obtained for the grown carbon nanoparticles.

Also, according to Figure 3, it can be seen that as the

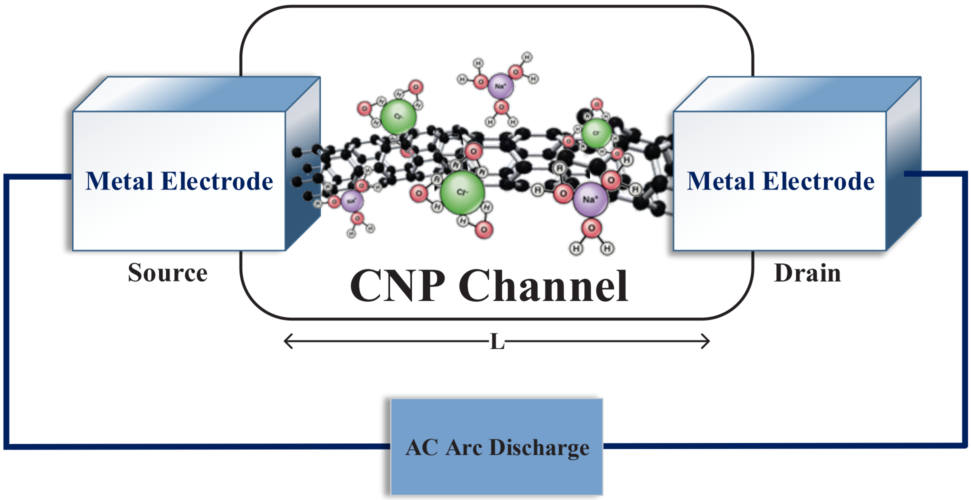

Fig. 2. The schematic related to the fabricated sensor as a field effect transistor including carbon nanoparticles grown between two electrodes as a transistor channel

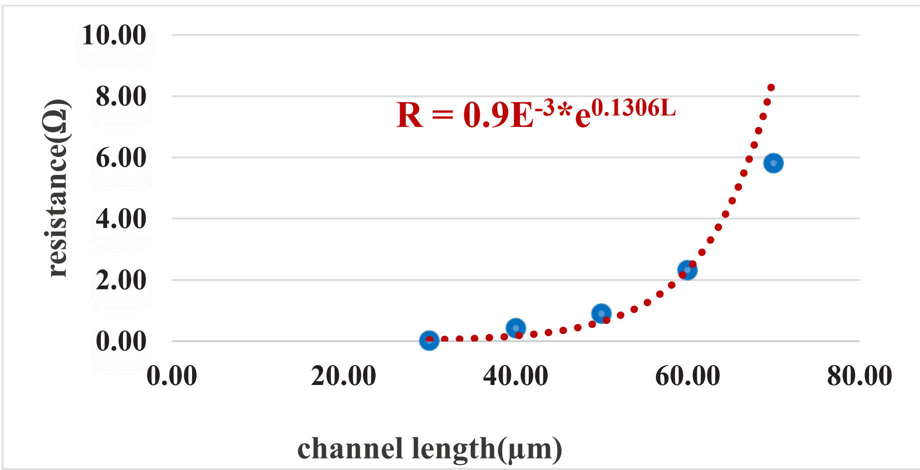

Fig. 3. Exponential relation between channel lengths and resistance of grown nanoparticles

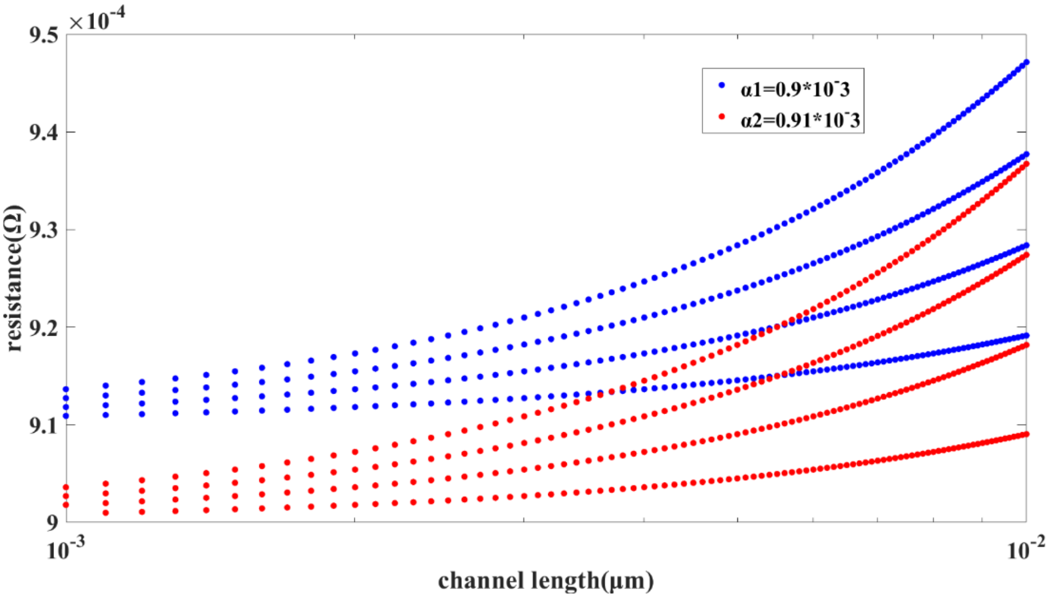

Fig. 4. The effect of α coefficient on sensor behavior

length of the channel increases, the resistance of the sensor exponentially increases. Therefore, the length of the channel can be considered as a parameter that determines the type of nanoparticles grown in terms of electrical conductivity. If the resistance of the grown nanoparticles is less than 10E-2 Ω, the channel is considered conductive. For the resistance of nanoparticles in the range of 10-2 to 10E+5Ω, the channel is semi-conductive, and if the electrical resistance of nanoparticles is more than 10E+5Ω, the channel will be insulated. Therefore, the length of the channel should be adjusted so that the nanoparticles grown in this length of the channel show their conductivity properties and as a result, they can show better performance as a sensor channel.

According to Figure 4, It can be seen that by changing in α, the electrical resistance of the sensor changes linearly as an output parameter.

4- 2- Resistance analysis of fabricated sensor:

To investigate the behavior of the sensor made by grown carbon nanoparticles, saltwater solution in several different concentrations (from low concentration to high concentration) was injected into the channel of this sensor, and the resulting electrical behavior in these concentrations was investigated. This behavior is measured as electrical resistance between two electrodes and is shown in Figure 5. Also, the relationship between the length of different channels and the behavior of the sensor in different concentrations has been investigated. This sensor is fabricated along 30-70 micrometer channels and concentrations of 0.06 to 0.12 g/ml are injected into them.

According to the results shown in Figure 5, it can be said that the length of the transistor channel made as a sensor is one of the important and influencing factors on the electrical behavior of the sensor. In examining the effect of channel length in different concentrations of saltwater, it can be concluded that in low concentrations, the effect of channel length on the electrical resistance of the sensor is significant. This is because the electrical resistance of the sensor in high concentrations does not change much with the change of channel length and they are somewhat close to each other. As a result, it can be said that with increasing solution concentration, the effect of channel length decreases.

In addition, if only the change of saltwater concentration in all lengths is examined in general, it can be seen that increasing the concentration of saltwater solution causes a decrease in the resistance of the sensor. This result is true for all channel lengths. This results from salt dissolving in water and creating Na^+ and Cl^- ions. These ions cause the desired solution to act as a conductor. By increasing the concentration of the saltwater solution, the number of ions in the solution increases and as a result, the conductivity of the sensor channel increases. So according to the relation V=IR, the resistance of the sensor decreases.

According to Tables 1 and 2, while at low concentrations of saltwater solution, the highest change in resistance along the length of the 60-micrometer channel is 14.2% and the lowest change in resistance along the length of the 70-micrometer channel is 4.3%. Saltwater, 31.6% has been observed as the greatest change in resistance along the 60-micrometer channel

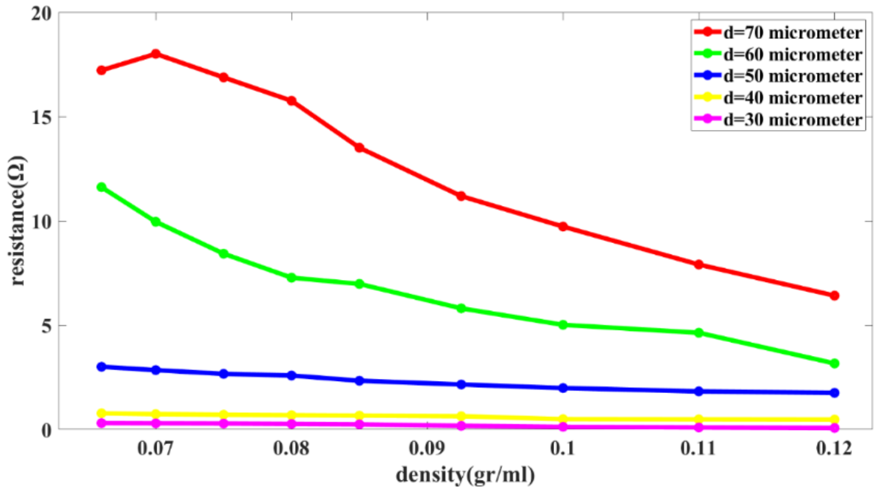

Fig. 5. Changes in the electrical resistance of the fabricated sensor as a result of changes in the density of the saltwater solution injected into the channel

Table 1. Sensitivity comparison of the fabricated sensor for different channel lengths- Low concentrations

Channel length (μm)	R_1 (Ω)	R_2 (Ω)	Percentage of changes
30	0.31	0.3	3.2%
40	0.78	0.74	5.1%
50	3.01	2.85	5.3%
60	11.62	9.96	14.2%
70	18.01	17.22	4.3%

Table 2. Sensitivity comparison of the fabricated sensor for different channel lengths- High concentrations

Channel length (μm)	R_1 (Ω)	R_2 (Ω)	Percentage of changes
30	0.1	0.098	2%
40	0.49	0.48	2%
50	1.83	1.76	3.8%
60	4.64	3.17	31.6%
70	7.91	6.42	18.8%

and 18.8% change in the resistance of the sensor along the 70-micrometer channel. Therefore, it can be concluded that the fabricated sensor performs better in the longest channel length and high concentrations.

Also, in all saltwater concentrations, it can be seen that the highest percentage of resistance changes occurs in the 60-micrometer channel. Ac Arc Discharge device includes RLC circuits. It seems that along the 60-micrometer channel, the frequency is adapted to the frequency of the RLC circuit and the resonance phenomenon has occurred. For this reason, the growth trend in this length is stronger and has shown more resistance changes compared to other lengths of the channel.

5- Conclusion

In this article, a sensor based on carbon nanoparticles was designed and these nanoparticles were synthesized by the electric arc discharge method. This sensor is a field effect transistor and includes two electrodes as source and drain and nanoparticles grown between the two electrodes as a permanent channel. Carbon nanoparticles have been synthesized as the permanent channel of this transistor in lengths of 30 to 70 micrometers. By measuring the resistance of the channel in different lengths, the exponential relation between the length of the channel and its resistance can be obtained. It was also observed that the length of the channel is used as a parameter to determine the electrical conductivity of the synthesized nanoparticles. To check the performance of this sensor, for each length of the synthesized channel, saltwater with concentrations of 0.06 to 0.12 was injected and the change in electrical resistance of the sensor was observed. In this way, at high concentrations, for every 0.01 change in saltwater concentration, an 18.8% change in sensor resistance was observed in the longest channel length and a 2% change in sensor resistance in the shortest channel length. At low concentrations, for every 0.005 change in saltwater concentration, a 3.2% change in sensor resistance was observed in the shortest channel length and a 4.3% change in resistance in the longest channel length.

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HOW TO CITE THIS ARTICLE

G. Taghizadeh Afshari, M. T. Ahmadi, A. Fathi. Carbon Nanoparticles-Based Sensor Analysis AUT J Electr Eng, 56(3) (2024) 399-406.

DOI: 10.22060/eej.2024.22943.5576