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Polluted Water Sensor Based on Carbon Quantum Dots/Alq3 Using Drop Casting and Spin Coating Techniques

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Abstract

Water quality sensors have recently received a lot of attention due to their impact on human health including environmental sensors especially that based on carbon quantum dots (CQDs). In this study, CQDs were prepared using the electro-chemical method, where the structural and optical properties were studied. These quantum dots were used in the environmental sensor application after mixing them with three different materials: CQDs, Alq3 polymer and CQDs and Alq3 solutions using two different methods: drop casting and spin coating, and depositing them on silicon. The sensitivity of the water pollutants was studied for each case of the prepared samples after measuring the change in resistance of the samples at a temperature of 30 °C. Through the results, it was found that the highest sensitivity of sample 3 (CQDs solution) to the carbon continuous dot was in the case of the contaminant fructose and was 99.55%, while the highest sensitivity of sample 4 (Alq3 solution) was for the one sensitive to the contaminant (mercury chloride) and was 81. As for sample 1 (CQDs/Alq3 composite films by drop casting), the highest sensitivity was in the case of detecting the contaminant lead chloride and was 80. The results showed that the best sensor was obtained using a spin-coating technique when the solution sample of CQDs+Alq3 was placed on a silicon slide in fructose and the sensitivity was 200%. This demonstrates the importance of quantum dots in measuring the sensitivity of water pollutants. The thin film thickness was measured to be 500 nm.

Article Info.

Keywords:
Biosensor, Carbon Quantum Dots, Alq3 Polymer, Electro-Chemical Method, Pollutants.

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1. Introduction

Environmental sensors have recently attracted a lot of attention, ranging from biological to environmental to agricultural uses [1-4]. Common diagnostic techniques are expensive and time-consuming [5]. Researchers have been working on creating rapid, sensitive, and accurate environmental sensors as means to early identify different forms of pollutant in order to address these problems [6]. Furthermore, environmental sectors have made substantial use of biosensors as they are used to detect the level of ionic heavy metals in wastewater due to the increased their level. Numerous environmental sensing relies on nanotechnology to achieve quick, easy, and sensitive detection, particularly for early detection of diseases, because nanomaterial have distinctive properties to use them to increase the efficiency of bio-sensing systems [7, 8]. Environmental sensors are devices with three main components: signal transducer, signal processor, and biological recognition element [9, 10]. For instance, electro-chemical environmental sensors are often used in environmental and biomedical monitoring [11-15]. Nano-sensor systems use at least one nanostructure material with notable properties, such as good thermal conductivities, electrical, optical, magnetic properties, and high stability [16, 17]. In general, quantum dots are nano-structures with unique features that have recently received much interest in environmental sensors [18-24].

The organic core's limits on chemical stability, water solubility and cytotoxicity necessitate the application of a biocompatible shell to the surface [25, 26]. Numerous biosensors created depend on carbon quantum dots (CQDs) for different diagnostic methods because of their structure and distinguishing qualities [27]. As a result, biocompatible CQDs may be substantially safer than conventional QDs when used in biosensors for disease detection. Additionally, compared to their QD counterparts, CQDs have lower manufacturing costs [27-30]. CQDs are an excellent choice for creating environmental sensors because of their physicochemical stability, conductivity, electro-activity, and excellent optical qualities, among other characteristics. In recent years, it has become more important to study the properties and synthesis of different CQD-based biosensors in order to follow up on the progress gained so far and to identify the remaining difficulties for future researches. Despite carbon's limited water solubility, CQDs have an excessive number of carboxyl groups in their structure's edges, which give them an excellent aqueous solubility [31]. There are many studies done in biosensors field using semiconductor carbon nanotubes [32] or copper phthalocyanine tetrasulfonic acid tetrasodium salt/graphene oxide [33], where these compounds give sensitivity less than the compound Alq3 and CQDs [34].

One of the most extensively investigated organometallic compounds is tris (8-hydroxyquinolinato) aluminum (Alq3) with formula $Al(C_9H_6NO)_3$ because of its intriguing characteristics and possible use in optoelectronics [35]. It has traits including relative stability, ease of synthesis, and effective electronic transport that are comparable to those of inorganic semiconductors [36-38]. In organic light-emitting diodes, Alq3 is a metal chelate that is often utilized as an electron transport layer [39]. The first description of the usage of this material in OLEDs was published in 1987 [40], and since then, interest in Alq3 has increased. Fong and So looked at the hole transport characteristics of Alq3, in comparison.

The aim of this research is to manufacture an environmental sensor using carbon quantum dots and Alq3 polymer, using two preparation methods: drop casting and spin cutting. The sensor can be used to sense some water pollutants, such as lead chloride ($PbCl_2$), mercury chloride ($HgCl_2$), and fructose ($C_6H_{12}O_6$).

2. Experimental Work

Several materials were prepared in order to prepare samples for the biosensor, and they are as follow:

2.1. Carbon Quantum Dots

CQDs were prepared using the electro-chemical method, as shown in the following steps (Fig. 1). Firstly, a volume of 99.5 ml of ethanol (C_2H_5OH) was diluted with 0.5 ml of distilled water. Secondly, a mass of 0.3 g of sodium hydroxide (NaOH) was dissolved in the ethanol and thoroughly mixed for two hours using a magnetic stirrer. Thirdly, two graphite electrodes with a gap of 2.5 cm were set up inside the beaker containing 250 ml of the mixture. The circuit's current was set to 30 mA. Fourthly, the electrodes rotated every 5 min to move ions from one electrode to the other. In this case, a milky white solution (mixture) was created. Fifthly, the mixture's hue was changed to yellow-orange after being left for five days. Column chromatography was used to separate CQDs from the resultant solution, as depicted in Fig. 2. Sixthly, in another little beaker, 20 ml of diethyl ether ($C_2H_5OC_2H_5$) was mixed with 20 ml of silica gel (SiO_2). Seventhly, the prior mixture was slowly mixed with 10 ml of petroleum ether (C_7H_{16}), and then the final product was put in a chromatographic column. Eighthly, the produced filter is purified by adding CQDs. Fig. 3 illustrates how the CQDs' color changes over time. Ninthly, drop casting was used to make the films, which were subsequently heated to 50 ^{o}C. The thickness of the prepared films was measured by Tolansky's method, and the thickness of the films was approximately 600 nm.

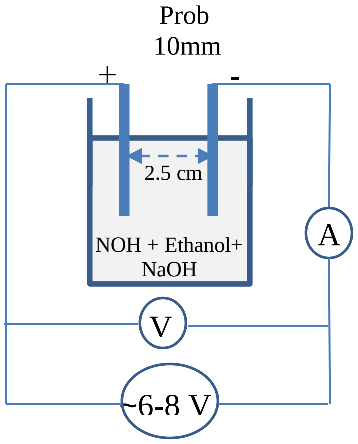

Figure 1: The electro-chemical method.

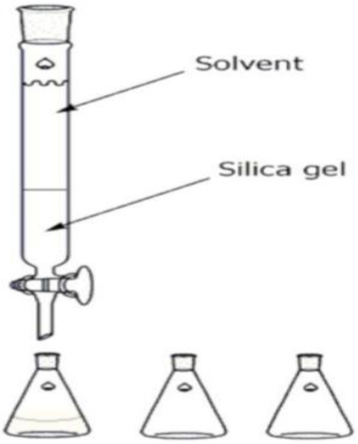

Figure 2: Column Chromatography filter.

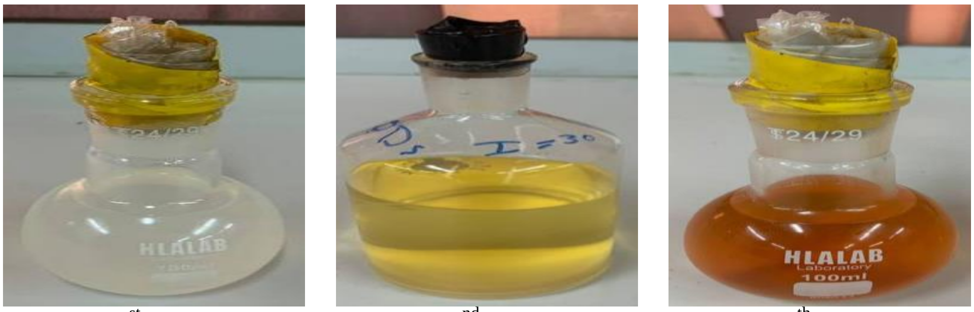
1st day 2nd day 4th day

Figure 3: The color of the CQDs solution changed with time.

The films that were formed using this method are nonuniform because of the inconsistent drying conditions and uneasy control. These films are thicker at the center and thinner at the edges. The drop casting method is an alternative technique for polymer semiconductors, which are expensive and have poor solubility [34].

2.2. Preparation of Alq3 Polymer

Alq3 polymer solution was prepared by dissolving 30 mg of Alq3 nanopowder in 3 ml of chloroform (99% purity) using magnetic stirring for one hour at room temperature until a clear yellow solution is obtained as shown in Fig. 4.

Figure 4: The Alq3 powder (left) and Alq3 solution (right)

2.3. Preparation of CQDs/Alq3 Composite Biosensor Samples

CQDs/Alq3 composite films were prepared by dissolving 0.5 ml of Alq3 polymer in 0.5 ml of CQDs. This solution was then stirred using an ultrasonic bath for 15 min at 60 °C. Then, two different samples were prepared using two different techniques, namely, drop casting (sample 1- deposit the solution on a silicon substrate and then the remaining of the solvent was evaporated at 60°C for 30 min) and spin-coating (sample 2- drop the solution on a silicon substrate where the speed of base of the spin is 1000 rpm and the spin time is 30 s, and the remaining solvent was evaporated at 60 °C for 30 min). In addition, two different samples were prepared for each of the basic materials manufactured for the biosensor, so the total number of samples is four samples. Two samples were prepared as follows: sample 3- is CQDs solution dropped on a silicon substrate and then the remaining of the solvent was evaporated at 60 °C for 30 min using an oven. Finally, a sample 4- drop of Alq3 polymer is dropped on a silicon substrate and then the remaining solvent is evaporated at 30 °C for 15 min.

2.4. Preparation of Contaminated Material

In this study, three water pollutants were employed; lead chloride ($PbCl_2$), mercury chloride ($HgCl_2$) and fructose ($C_6H_{12}O_6$) in different proportions (Table 1), which are considered among the most pollutants of water:

Table 1: Material and proportion affecting the person.

Material	The proportion affecting the person	Toxicity Properties
Lead Chloride ($PbCl_2$)	15 µg/l [41]	Exposure to $PbCl_2$, being soluble, leads to lead poisoning
Mercury Chloride ($HgCl_2$)	2 ppb [42, 43]	Mercury dichloride is a highly toxic compound. They can cause damage in the stomach, mouth, and throat. They can lead to acute kidney failure.
Fructose ($C_6H_{12}O_6$)	140 mg [44]	Lethal dose or concentration (LD, LC)

Each material was mixed with 20 ml of distilled water, to know the sensitivity of sample against polluting materials and measure the resistance.

2.5. Water Pollutants Configuration

The water pollutant sensor is an analytical device used to detect some toxic chemicals that affect human tissues and organs. Fig. 5 represents the configuration of biosensor; three layers as follow: Si substrate, mask (Al) and thin film (CQDs or Alq3 or CQDs+Alq3) and the mask shape.

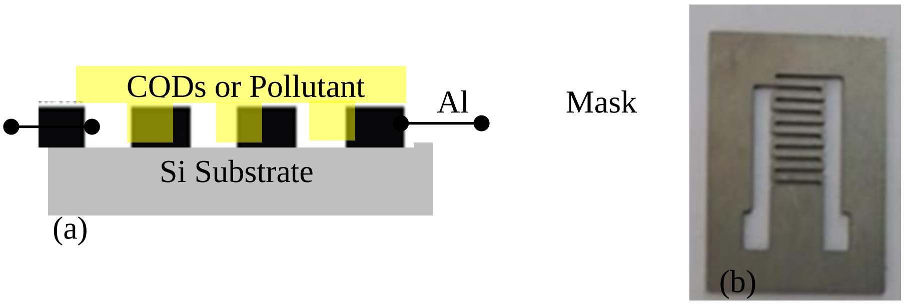

Figure 5: (a) the configuration of water pollutant sensor, (b) the Mask.

2.6. Bio and Water Pollutant Sensor Testing System

In order to determine the sensitivity parameters mainly the response time and recovery time of the fabricated CQDs/Alq3 nanocomposite biosensor, a suitable setup was prepared. The cross-sectional view of a biosensor testing system is consisted of a test chamber, and images of the installed sensor are shown in Fig. 6. The device consists of a cylindrical test chamber constructed of vacuum-tight stainless steel with 24 cm in diameter and 40 cm in height, and has a removable bottom base that is O-ring sealed and vacuumed by a rotary pump. The actual volume of the chamber is 10990 cc. Electrical connections to the heater assembly and the sensor electrodes were made using spring-loaded pins through a multi-pin feed at the bottom of the chamber.

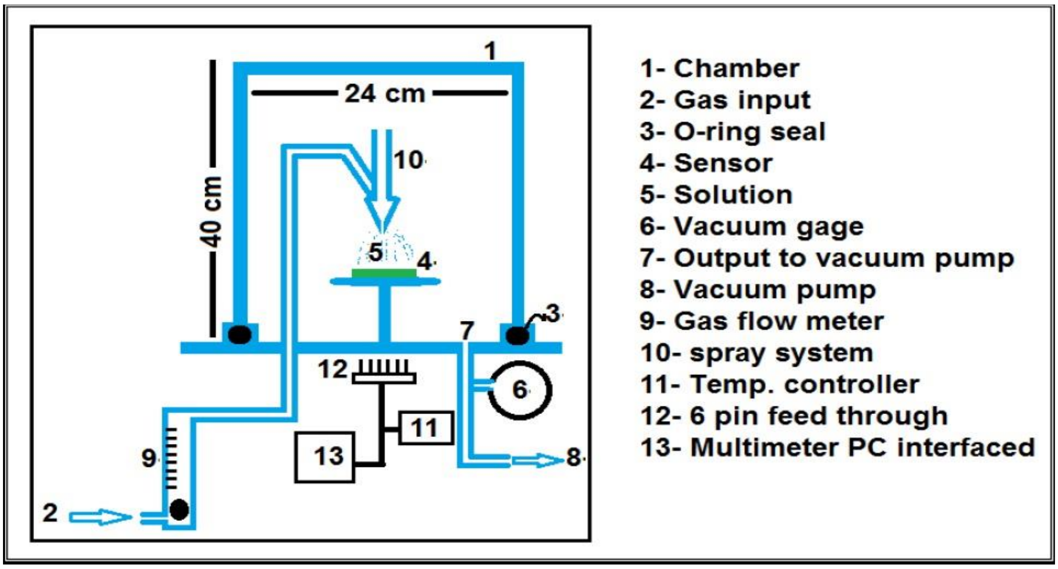

Figure 6: Diagrammatic representation of the biosensor testing system

To regulate the temperature of the sensor, heater components include a hot plate and K-type thermocouple were kept inside the chamber. The fluctuation in the resistance of the sensors, when spraying biological material (lead chloride, mercury chloride, and fructose), via a flow-meters and needle valves arrangement was recorded using a PC-interfaced digital multi meter (UNI-T UT-803) and monitored by PC. A rotary pump can be used to evacuate the chamber to a rough vacuum of 1×10^{-3} bar.

2.7. Testing Protocol for Sensors

The test setup operates according to the steps are listed below:

1- The sample was placed on the heater inside the test chamber. The pin feed through the sample firmly loaded pins, as well as the thermocouple is all electrically connected. The test container is so shut.
2- In order to reduce the atmospheric pressure in the test chamber to roughly 1×10^{-3} bar, the rotary pump was turned ON.
3- The PC-interfaced digital multi meter (DMM) measures the resistance change of the sensor based on the known concentration in test biological material ratio.
4- The needle-shaped valve of the sample of biological material is closed and returned to its initial base resistance value R_0 after each single measurement.
5- The previously mentioned measurements were carried out for other necessary concentrations of fructose, lead chloride, and mercury chloride.

3. Results and Discussion

In a previous study, the structural and optical properties of the nanostructures were studied [34], where the results showed the formation of spherical nanostructures was down to 30 nm, and the optical results also showed an increase in the energy gap to reach 3.18 eV. In this study, silicon substrate was used to sensitize the materials, as the samples were sensitized to distilled water and their resistance was measured. The sensitivity of the samples to distilled water solutions and pollutants and their resistance were also measured. The percentage of sensitivity (S) can be determined using [45]:

$$S\% = [\,(R_w - R_{sol})\,/\,R_w] \times 100\% \qquad (1)$$

where R_w represents the resistance in water state and R_{sol} is the resistance in pollutants solution. Table 2, shows that the resistance and sensitivity of the CQDs (Sample 3) are higher in case of mixing with $C_6H_{12}O_6$ solution and lower than that of $PbCl_2$and $HgCl_2$. While, Table 3 shows that the sensitivity of the Alq3 polymer deposited on the silicon substrate (sample 4) is higher when mixed with $HgCl_2$ solution and lower than that of a $C_6H_{12}O_6$ and $PbCl_2$. Table 4 shows that the sensitivity of CQDs+Alq3 solution, which was prepared using the drop-casting technique, is higher when mixed with $PbCl_2$ solution and lower than that of $HgCl_2$ and $C_6H_{12}O_6$. Finally, the result of CQDs+Alq3 sample, which was prepared using the spin coting technique (sample 2), Table 5 shows that the sensitivity is higher when mixed with $C_6H_{12}O_6$ solution and lower than that of $PbCl_2$ and $HgCl_2$. Through the results obtained, it was found that the best bio-sensor for the lead chloride $PbCl_2$ pollutant is CQDs+Alq3 using the spin coating technique and deposition on a silicon substrate (sample 1), with a sensitivity ratio is 92.72%. While the best bio-sensor for $HgCl_2$ pollutant with Alq3 using coating technique rotation and deposition on a silicon substrate (sample 4), with a sensitivity ratio 81.60%. Finally, it was noted that the best bio-sensor for fructose $C_6H_{12}O_6$ is the CQDs+Alq3 using the spin coating technique and deposition on a silicon substrate (sample 2), with a sensitivity ratio 200%.

There are many researchers conducted carbon quantum dots in different ways and they can be compared with present study [46-52].

Table 2: Resistance and sensitivity of CQDs deposited on a silicon substrate in $PbCl_2$, $HgCl_2$ and $C_6H_{12}O_6$ (Sample 3).

Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $PbCl_2$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+$PbCl_2$)**	**S %**
19.2	3.8	15.4	80.20
Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $HgCl_2$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+ $HgCl_2$)**	**S %**
21.7	18	3.7	17.05
Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $C_6H_{12}O_6$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+ $C_6H_{12}O_6$)**	**S %**
22.7	45.3	22.6	99.55

Table 3: Resistance and sensitivity of Alq3 placed on a silicon substrate in $PbCl_2$, $HgCl_2$ and $C_6H_{12}O_6$ (Sample 4).

Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $PbCl_2$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+$PbCl_2$)**	**S %**
0.8	0.98	0.18	22.5
Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $HgCl_2$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+ $HgCl_2$)**	**S %**
0.87	0.16	0.71	81.60
Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $C_6H_{12}O_6$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+ $C_6H_{12}O_6$)**	**S %**
0.84	0.918	0.077	9.155

Table 4: Resistance and sensitivity of solution (CQDs+Alq3) by the drop-casting technique and deposited on a silicon substrate in $PbCl_2$, $HgCl_2$ and $C_6H_{12}O_6$ (Sample 1).

Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $PbCl_2$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+$PbCl_2$)**	**S %**
65	27	38	80.20
Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $HgCl_2$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+ $HgCl_2$)**	**S %**
65.7	17.4	39.3	69.31
Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $C_6H_{12}O_6$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+ $C_6H_{12}O_6$)**	**S %**
55.2	75.7	20.5	37.13

Table 5: Resistance and sensitivity of CQDs+Alq3 solution using the spin coating technique and deposited on a silicon substrate in $PbCl_2$, $HgCl_2$ and $C_6H_{12}O_6$ (sample 2).

Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $PbCl_2$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+$PbCl_2$)**	**S %**
40	85	45	92.72
Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $HgCl_2$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+ $HgCl_2$)**	**S %**
39.5	19.2	20.3	51.39
Resistance in water(kΩ) R(H_2O)	**Resistance in(kΩ) R(H_2O+ $C_6H_{12}O_6$)**	**ΔR (kΩ) = R(H_2O)-R(H_2O+ $C_6H_{12}O_6$)**	**S %**
40	120	80	200

The increasing of sensitivity when adding Alq3 with CQDs is the ability of Alq3 to act as an electron transport material as well as host material for several materials such as CQDs dopants [53]. It is known that substitution of electron withdrawing/donating functional groups in specific position in the ligand, 8-hydroxyquinoline, would change the emission property of the Al complex. In Alq3, the HOMO of 8-hyrdoxyquinoline is a highly light emitting metal complex with applications in several electronics devices. Increasing the ability of the materials to transfer electrons leads to a significant decrease in the resistance value of the manufactured sensor in water case $R(H_2O)$ that equal 40 kΩ, and thus the value of the difference between the resistance of the sensor in the case of water and in the case of pollutants increase ($\Delta R(k\Omega) = R(H_2O)-R(H_2O+C_6H_{12}O_6)$) that equal 80 kΩ. This leads to a significant increase in the sensitivity to reach 200%, especially in the case of using the spin coating technique in the deposition process. The reason is that in this technique a total homogeneity of the two solutions (Alq3 and CQDs) was obtained and it is completely distributed on the silicon substrate, in contrast to the drop casting technique. The above explanation applies to all samples, but the percentage of sensitivity varies from one case to another depending on the type of pollutant used and the method of preparation [53].

4. Conclusions

In conclusion, the electrochemical method was used to generate CQDs. This technique showed impressive results for obtaining sensors with high sensitivity, due to their nanostructure, which led to an excellent change in their electrical properties. Through the results, it was found that the sensitivity of environmental sensors increases when carbon quantum dots are mixed with Alq3, so that their sensitivity becomes 200%.

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Conflict of Interest

Authors declare that they have no conflict of interest.

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مستشعرات تعتمد على النقاط الكمومية الكربونية/ Alq3 باستخدام تقنيات الصب المتساقط والطلاء الدوراني

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الخلاصة

اكتسبت المستشعرات الحيوية مؤخرًا الكثير من الاهتمام، لا سيما في مجالات التصوير البيولوجي والكشف. نظرًا لسماتها المميزة، فإن أجهزة الاستشعار الحيوية تعتمد على النقاط الكمية الكربونية.(CQDs) في هذا البحث، تم تحضير النقاط الكمية الكربونية (CQDs)بالطريقة الكهروكيميائية. تم استخدام هذه النقط الكمية الغروانية في تطبيق المستشعر الحيوي بعد مزجها بثلاث مواد مختلفة:1- Alq3, 2-CQDsبوليمر 3- محاليل CQDs و Alq3باستخدام طريقتين مختلفتين: الصب المسقط والطلاء الدوراني، وترسب على السيليكون. تم حساب الحساسية لكل حالة من العينات المحضرة بعد قياس التغير في مقاومة العينات عند درجة حرارة 30 درجة مئوية، حيث أظهرت النتائج أن أفضل حساس كان في العينة (محلول CQDs + Alq3) بالدوران. يتم وضع تقنية الطلاء على شريحة سيليكون في الفركتوز) وكانت كمية الحساسية (200٪) وهذا يوضح أهمية النقاط الكمومية في قياس حساسية المادة.

الكلمات المفتاحية: المستشعر الحيوي، نقاط الكم الكربونية، الطريقة الكهروكيميائية، بوليمر Alq3، كبريتيد الرصاص، كبريتيد الزئبق، الفركتوز.