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Effect of Microwave Treatment in Graphite Anode for Microbial Fuel Cell and Its Application in Biosensor

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Abstract

The electrode in the microbial fuel cell has a significant effect on cell performance. The treatment of the electrode is a crucial step to make the electrode surface more habitable for bacteria growth, thus, increases the power production as well as waste treatment. In the current study, two graphite electrodes were treated by a microwave. The first electrode was treated with 100W microwave energy, while the second one was treated with 600W microwave energy. There is a significant enhancement in the surface of the graphite anode after the pretreatment process. The results show an increase in the power density from 10 mW/m^2 to 15 mW/m^2 with 100w treatment and to 13.47 mW/m^2 with 600w treatment. An organic sensor was obtained for the same waste material used, where the sensitivity was weak, ranging from 100 mg/L for organic matter to 150 g /L. The sensor was used once again for each substance with better results. The sensitivity ranged from 25 g/L per liter to 150 g/L, while successful linearity has been gain. Therefore, it can conclude that the microbial fuel cell with dual chamber can be designed for a biosensor with the available and cost-effective material.

Keywords: Microbial fuel cell; Biofuel cell; microwave treatment; electrochemical, biosensor

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1- Introduction

Recently, renewable energy sources as an alternative to fossil fuels have drawn attention for many researchers. For example, bioenergy produced from microorganisms (such as microalgae [1] **,[2]** or by taking advantage of the presence of the microorganisms to generate electricity as in the microbial fuel cell. Microbial fuel cell (MFC) is an electrochemical device that uses bacteria to generate electricity by oxidizing organic matter [3]**, [4]**.

The main role of the bacteria is to make a biofilm on the surface of the anode electrode. This biofilm will act as a catalyst to degrade the organic matter [5]**.** Using the microbial fuel cell at different applications; such as wastewater treatment and energy production, showed the need for a cost-effective reactor with better performance. The electrode for the microbial fuel cell has a great effect on the cell performance. Electrode material takes the most concern of cell due to their cost or availability. The choice of the electrode depends mostly on how the bacteria grow on the electrode surface and construct the biofilm. Carbon materials are commonly used in MFC because they are inexpensive and have excellent conductivity, chemical stability, biocompatibility, and large surface area. Various carbon materials, including graphite [6] graphite foam **[7]** woven graphite, graphite felt [8] reticulated vitreous carbon (RVC) [9] carbon paper [10] have been reported. Changing the properties of the electrode can be achieved with a specific modification.

Different treatment and modification have been taken to alter the electrode surface properties especially conductivity and biocompatibility. The treatment with ammonia gas, as an example, increased the surface charge of the electrode and improved MFC performance [11].

Nanomaterials also take part in these modifications, such as the use of carbon nano-tube to decorate the anode of MFC and enhance the power generation [12].

Graphite decorated anode with Au nanoparticles produces current densities up to 20-times higher than that produced from plain graphite anodes by Shewanella oneidensis MR-1, while Pd-decorated anodes were 0.5–1.5 times higher than the original **[13]**. Wang **[14]** modified a graphite electrode with a simple method of heat treatment in an oven that also enhances the electrode performance.

The microwave technology was used in the laboratory for sample preparation and heating for different field of science. The materials which interact with microwaves to produce heat are called microwave absorbers including carbon material. The carbon-based material is good microwave absorber.

Thus, the microwave has been used for many processes, including the synthesis of different carbon materials (i.e., nanostructures, graphite, active carbons, polymers, etc.) [15]. Menéndez et al. **[15]** show that the microwave effect on carbon-based material by modifying their physical and chemical properties.

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Until now, there is no single biosensor that meets each of these necessities yet**.** The biosensors demonstrate a couple of one of a kind sorts of common signs, including change of advancement case or change in light power. Biosensors that use an electrochemical signal, for instance, present or potential is called electrochemical biosensors [14]**-[18]**.

The MFC-based biosensor is an electrochemical biosensor. In an MFC-based biosensor, microorganisms created on the anode catalyze the oxidation of regular material present in the inflow and electrons are made at the anode. An MFC meets various necessities of the perfect sensor. It can be used as an early alerting motion since it is sensitive to various portions.

Moreover, it can be low in cost, direct in operation and can screen incessantly. Various diverse biosensors require a transducer and one of the upsides of an MFC is a transducer is not required. Another favored outlook is using typically available microorganisms. No innately balanced infinitesimal living beings are required like the case in some unique sorts of biosensors [19]**, [20]**.

2- Material and Methods

2.1. Electrodes

Two types of the electrode have been used in the experiment:

1- Graphite rods (5cm long and 0.5cm diameter) present in the dry cell were used as electrodes for the anode. They were removed carefully and cleaned from the substances stick on the electrode surface using sandpaper. The electrode was treated with a Bunsen burner to remove any unwanted substances on the surface. Any substances on the surface of the graphite anode may poison the bacteria and prevent the biofilm formation. The electrode was washed with distilled water before used in the MFC reactor. The electrode was attached with NICHROME wire due to its ability to resist corrosion, which will prevent any corrosion inside the cell.
2- Carbon fiber was treated with 3M HCl for 24 hours and used for the cathode.

2.2. Media

The waste used in the experiment was collected from Al-Mansour Municipalities department. The collected waste was taken and mixed with tap water and distilled water to ensure the necessary element for the bacteria and to increase the conductivity at 20 °C.

2.3. Separator

The separator used in the experiment consists of filter paper (DR. WATTS cat No. 501) placed between two supporting gaskets, the filter paper acts as a base to support the solidified solution from both sides. The solidified solution contains 5% agar by mass and 1 M NaCl solution.

The solution was mixed and heated with a microwave oven to melt the agar and the poured at both sides of the filter paper and let it solidify for a few minutes. The thickness of the separator was 4mm. This type of separator will prevent the crossover of material from the anode to cathode; also, it is much better than the long salt bridge.

2.4. Microbial Fuel Cell Assembly

A U shape microbial fuel cell was assembled from two glasses fitting, as shown in Fig. **1**.

The cathode side consists of DI water and air bubbling tube from a small compressor to supply the cell with oxygen. Three cells were made in the same way. Therefore the only difference was the treated electrode.

The cell consists of two glass tube were connected to make a U shape. Between these two parts, a salt bridge consists of 1M NaCl and 3 wt% agar as a solidifying agent.

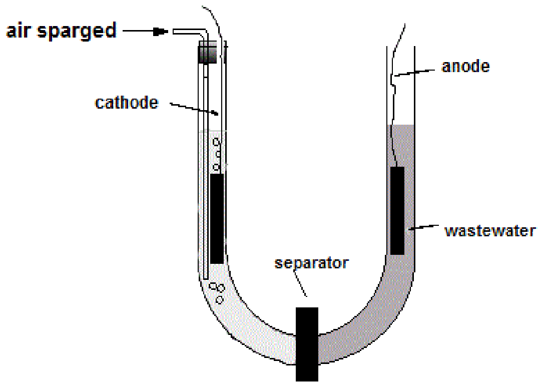

Fig. 1. Microbial fuel cell reactor

2.5. Set Up

a. Electrode Treatment Set Up

100 ml of wastewater was used in each cell and the voltage was recorded using multimedia (HoldPeak HP-90EPC Multimetro Digitalis). The two electrodes were treated with the energy of 100 W and 600 W for 10 min in conventional microwave oven equipped with vent for gas generation each was used in different cell and the third electrode was left without treatment to compare the results. In order to find the difference between the treated and non-treated electrode, a voltage-time curve and polarization curve were used the comparison purpose. This comparison approach was also used by Cheng and Logan [11] for electrodes performance with and without ammonia gas treatment. In order to obtain the polarization curve, the external resistance was changed from 10 Ω-100 kΩ, while the current was measured at a resistance (R) from the microbial fuel cell. This setup was carried out according to Lafta, 2018 [21].

b. Microbial Fuel Cell Biosensor Using Mixed Culture

1- A MFC was used with the treated anode and the modified salt bridge. The cell was left to work for 2 weeks until the voltage is stabilized and the anode is catalyzed by the bacteria.
2- The anode from the previous cell was used in the new cell where a 3kΩ of external resistor was connected
3- 3.100ml of the wastewater was poured into the new cell and 428 ml were put in Biological oxygen demand (BOD) bottle.
4- The voltage was recorded for each hour in the cell as well as the BOD device provides an hourly measure for the organic compound.

c. Mixed Culture Microbial Fuel Cell Biosensor

1- A MFC was used with the treated anode and the modified salt bridge. The cell was operated for 2 weeks until the voltage is stabilized, and the anode is catalyzed by the bacteria.
2- The anode from the previous cell was washed with distilled water and used in a cell with sucrose as its fuel. The concentration of the sucrose was 150 mg /L. The cell also connected with a 5 kΩ the external resistor.
3- Voltage and the concentration of the sources were measured each hour.

3- Results And Discussion

3.1. Electrode Treatment

Wastewater was added to MFC. The power output and voltage of MFC increased gradually due to the biological activity of microorganisms. Fig. **2** shows the increase of voltage with time for three cells and represents the response of the bacteria in three different cases.

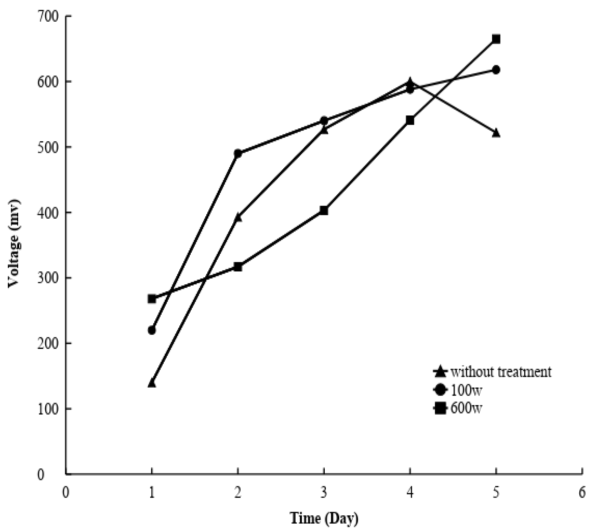

Fig. 2. Voltage generation curve from the three cells

The first one where the electrode is left untreated where the max voltage reached to 600 mV in fourth day, then, it started to decrease since the bacteria colonized not all the anode surface. As for the cell, where the anode treated with 100 W in the microwave shows a better response from the first day one up to the fourth day with keep increasing voltage above 600 mV. The reason for this increase was the anode surface that treated using the microwave. This method gave it the suitable surface property and the biocompatibility necessary **[15]** for more bacteria to inhabit the anode surface. When the anode was treated with 600 W, the initial response of the cell was the better (268 mV) on the first day. However, the increase in the voltage was slower, while it was less than the two other cells with max voltage in the fifth day (665 mV).

The polarization curve of the fuel cell was measured to investigate the amount of maximum current that can be generated with voltage. More current produced by the cell means more bacteria present on the anode surface. And that will lead to getting a thicker biofilm with higher power production. The experimental data in Fig.3 show the difference between all three cells. For the non-treated electrode at 0.00488 Ma, the voltage was the highest than all other cells with a value of 488 mV.

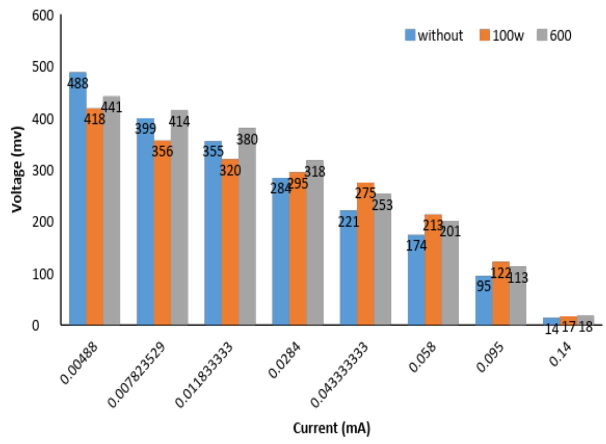

Fig. 3. Polarization curve for three cells

However, this value and the voltage of other cells decreased as higher load (resistance) applied to the cell. At current value higher than 0.0284 mA, the other treated cells by the microwave method gave a better result. The highest current was 0.14 mA, but with a different voltage of all cell. This current, the non-treated cell has the lowest voltage of 14 mV. The 100w treated anode has 17mV at 0.14 mA, and the best performance was for the 600 W treated anode with 18mv at the highest current. The figure also shows that the treated cell with microwave has a higher potential difference between the two electrodes. Since all the cells had the same cathode type, thus, the anode potential increased by the bacteria due to the microwave effect on the anode surface.

Fig. **4** shows the power density curve of the three cells that were taken when the cells reached a steady-state reading voltage by changing the external resistance.

The 0 W anode was no treatment occurs which shows the maximum power of 10 mW/m^2. For 100 W treatments, which shows a stable range of the maximum power possible of 15 mW/m^2 and for 600W, the power density was between the two other cells of 13.47 mW/m^2.

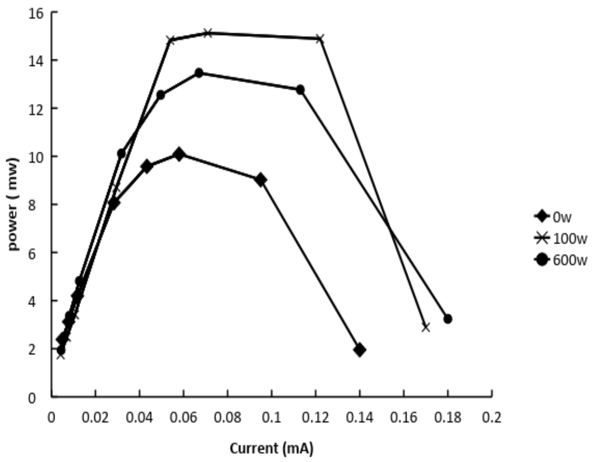

Fig. 4. Power density curve of the three cells (with 0 W, 100 W, 600 W)

3.2. Mixed Cutler Biosensor For Same Wastewater

The BOD was measured for the same wastewater used in the biosensor and the relation was drawn as shown in Fig. **5**.

The voltage from the cell was 60 mV simultaneously the BOD value was 143 mg/L. The voltage continues to increase and the bacteria digest the organic material.

The increase of the voltage has a linear relation with the BOD value. At the first three hours, the voltage was changing in linear behavior with the BOD value.

After 3 hours the line deviated from its linearity, and after 4 hours the value of voltage was constant because the equilibrium state has been reached. Despite the voltage after 4 hours was reached the equilibrium, the BOD value continued to increase.

This means that the response voltage of the cell cannot sense the organic compound concentration, thus, the sensor has reached its limit.

At the first four hours, the measurement started to get narrow until the voltage stabilized for the cell, but, not for the BOD value.

This sign indicates the variety of the bio-sensing and it is correct for the BOD base test because the bacteria outranged their linear growth.

Therefore, the sensing range was from 143 mg/L to 175 mg/L by using the growth procedure.

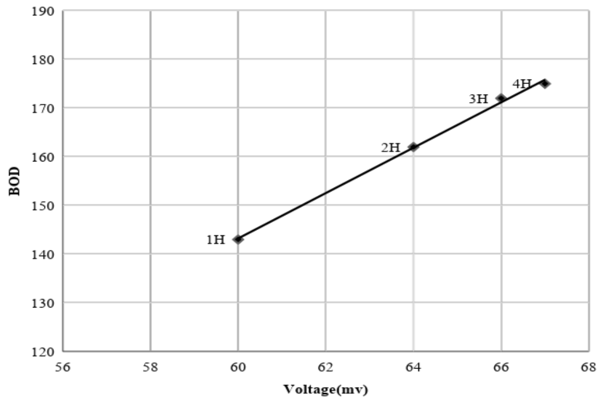

Fig. 5. the Response of biological oxygen demand (BOD) with voltage

Comparing the results with similar work done by Sumaraj and Ghangrekar **[22]** with the use of COD analysis instead of BOD was studied. Their result was a linear relation with R^2 equal to 0.954, while the present results have R^2 (0.9977). Moreover, the BOD test was used to measure the biodegradable organic matter concentration. Indeed, it gives a realistic amount of more organic matter that can be guessed by COD measurement.

In addition, the microbes in the MFC degrade biodegradable organic matter. Therefore, in measuring concentration they are in the same category While the COD measure organic and inorganic matter, as well as, the microbes in the MFC cannot degrade the inorganic matter.

3.3. Mixed Culture Biosensor for Sucrose

For the cell that contains 150 g/L of sucrose, dropping the voltage was observed for the catalyzed anode, as shown in **Fig. 6**. The external resistance used was 5 kΩ, the voltage at the beginning was recorded 125 mV. As the cell continues to operate the canalized layer of bacteria, the sucrose and the voltage was decreased with time as the sucrose decreasing. The decrease in voltage with time is linear in 4 hours. This period shows that the range of sucrose measure occurred in the first four hours only.

After four hours, the voltage was 51 mV and after another hour, it was the same. It indicates that equilibrium is reached at the bacterial culture in the anode surface.

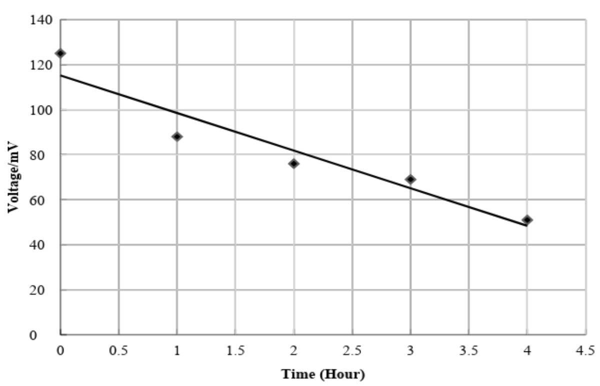

Fig. 6. Voltage with the resistance (5kΩ) for the sucrose cell

Each hour, when the voltage measured the consternation of the sucrose also measure using spectroscopy technique as shown in Table **1**.

Table 1. Microbial fuel cell biosensor voltage and sucrose concentration

Time(h)	Voltage (mV)	Absorbency (A)	Concentration (g/L)	Current (mA/cm^2)
0	125	0.107	150	19.44012
1	88	0.082	118.6893	13.68585
2	76	0.067	98.2683	11.8196
3	69	0.032	50.6193	10.73095
4	51	0.025	41.0895	7.931571
5	51	0.011	22.0299	7.931571

The sucrose concentration and the voltage were presented in **Fig. 7**. A curve fitting for the data has been performing using Microsoft Excel 2013. The value of R^2 is close to 0.9, which indicate the accuracy of the biosensor.

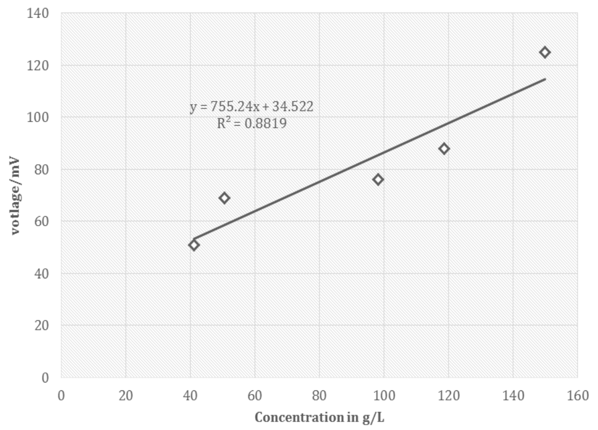

Fig. 7. The sucrose concentration and the voltage response

4- Conclusions

Measurement and calculations of the performance of the fuel cell with the microwave treatment show great promise for higher power density. The results show the effect of the microwave on the graphite material and increasing the performance of the cell and the response voltage.

The microwave reaches the micropore of the graphite and makes the necessary surface modification on the electrode for bacteria to inhabit the electrode with increase the area of biofilm formation and thus increase the power density. The modification that carried out on the cell in the present research increased the sensitivity and the accuracy of the biosensor. The sensing range of the waste was from 140 mg/l to 170 mg/l nearly the same range in the previous studies but with more accuracy. Moreover, a successful high range of sucrose sensing has been achieved.

Nomenclature

mA	current
MFC	microbial fuel cell.
P	Power in Watt (W)
R	resistance in ohm
V	voltage

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تأثير معالجة الموجات الدقيقة في أنود الجرافيت لخلية الوقود الميكروبية وتطبيقاته في المستشعرات الحيوية

الخلاصة

القطب في خلية الوقود الميكروبية له تأثير كبير على أداء الخلية الكلي. يعتبر علاج القطب عملية مهمة لجعل سطح القطب أكثر ملائمة للبكتيريا وبالتالي زيادة إنتاج الطاقة وكذلك معالجة النفايات. تم معالجة اثنين من قطب الجرافيت مع الميكروويف وتم علاج واحد منهم مع طاقة الميكروويف 100واط والآخر تعامل مع 600واط طاقة الميكروويف. تم تحسين سطح الأنود الجرافيت عند التعامل مع أفران الميكروويف. زادت كثافة الطاقة من 10 ميجاواط / م ^{2} إلى 15 ميجاواط / م ^{2} عند معالجة 100 واط وإلى 13.47 ميجاواط / م^2 وعند معالجة 600 واط. وتم الحصول على مستشعر عضوي لنفس مواد النفايات المستخدمة ، حيث كانت الحساسية ضعيفة ، تتراوح من 100 ملغم / لتر للمواد العضوية إلى 150 جم / لتر . المستشعر تم استخدامه مرة أخرى لكل مادة وكانت النتيجة أفضل . تراوحت مدة الحساسية من 25 جم / لتر لكل لتر إلى 150 جم / لتر, بينما كان الخطي الناجح هو الكسب. لذالك ممكن الاستنتاج بان الخلية الحيوية مع الغرفة المزدوجة ممكن ان تصميم كمتحسس حيوي بمواد رخيصة ومتوفرة.

الكلمات الدالة : خلية الوقود الحيوي, معالجة بالموجات الدقيقة ؛ الكهروكيميائية ، المستشعرات الحيوية