Microbial Fuel Cell for the Recovery of Sludge from the Treatment of Effluents by Electrocoagulation

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Abstract. The micro-organisms present in the domestic effluents allowed the formation of an electroactive biofilm by oxidation of the organic matter on the surface of a carbon fabric anode, in a microbial fuel cell (MFC). Previous studies have demonstrated the effectiveness of the electrocoagulation process for the treatment of industrial effluents. During treatment, the metal hydroxide sludge formed adsorbs pollutants. These sludges could be recovered through their use as fuel in a microbial fuel cell. The cell was inoculated with sludge from tests on effluents taken from a wastewater treatment plant. The results showed a decrease in chemical oxygen demand by 90.27%, dissolved oxygen by 75% and turbidity by 72%. Analysis of the sludge from this treatment showed the presence of sulphite-reducing coliforms and clostridium which oxidize the organic matter contained in a substrate enriched with acetate or glucose.

The maximum power densities obtained are 41.29 mW/m^2 with acetate and 27.57 mW/m^2 with glucose respectively. The study was carried out on a microbial fuel cell with two compartments separated by an ion exchange membrane.

Keywords: Electrocoagulation · microorganism · fuel cell · biofilm

1 Introduction

Energy, like water, is one of the major contemporary issues of our time. Indeed, industrial development, population growth, agriculture and transport are, among others, sectors that contribute to the increase in energy demand and the scarcity of natural resources. The current global energy crisis has highlighted the urgency as well as the benefits of an accelerated transition to cheaper and cleaner energy sources. Microbial fuel cells (MFC) are an alternative for a double contribution: electricity production and effluent treatment.

The principle is based on the use of bacteria which convert part of the energy available in a biodegradable substrate into electricity. Indeed, microbial fuel cells are characterized

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by "the use of microorganisms as catalysts" [1]. These are deposited on the exchange surfaces, at the anodes, and produce H^+ electrons and protons, by decomposing an organic substrate, derived from wastewater, recovered from a treatment plant. If the biological mechanisms of decomposition of the substrate are complex, we nevertheless know the corresponding reduction reaction at the cathode. MFCs can be fed with a variety of simple organic molecules (sugars, proteins, etc.) or directly with the effluents to be treated. The MFCs can be completely microbial when the catalysis of the reactions at the two electrodes is done thanks to microorganisms or semi-microbial in the case where the catalysis at the cathode is carried out by mineral catalysts [2].

The anodic oxidation reactions involved depend on the type of substrate used. In the case of acetate, the reaction is such that [3]:

$$CH_3COO^- + 4H_2O \rightarrow 2HCO_3{}^- + 9H^+ + 8e^- \quad (1)$$

Electrons are transferred from the biofilm to the anode by various mechanisms such as direct contact, nanowires or mediators [4]. Electrons migrate from the anode to the cathode through an external electrical circuit, while selected ions move through a separator membrane to complete the circuit.

At the cathode, oxygen is used as an oxidant in many cases, due to its availability and high reduction potential. Indeed, an ideal electron acceptor is necessary and must be durable, without interference or toxic effect on the microbial community or any other element of the system [5].

In the case of a proton exchange membrane, once the protons have diffused through the membrane, they can react with the oxygen present, leading to the generation of water according to the following reaction [3]:

$$O_2 + 4H^+ + 4e^- \rightarrow 2H_2O \quad (2)$$

The treatment of industrial effluents by electrocoagulation generates sludge composed of aluminum hydroxide and pollutants from the treated effluent. This sludge is considered as chemical waste and its recovery makes it possible to offer an integrated treatment system. The objective of this study is therefore to evaluate the potential for electricity production from electrocoagulation sludge in the context of the implementation of a microbial fuel cell.

Thus, we are going to present the process of treatment of effluents by electrocoagulation which allowed the production of sludge. Then we will present the method of microbiological analysis as well as the process of formation of the electroactive biofilm. We will discuss the operating principle of a microbial fuel cell as well as the operating parameters to be optimized. Finally, we will present the different results obtained with a discussion focused on the power generated by the system.

2 An Overview of the Recovery of Electrocoagulation Sludge

The physic-chemical and morphological characteristics of electrocoagulation sludge depend on the type of electrode used and the nature of the treated effluent.

Sharma, P. et al. [6] worked on sludge from an electrocoagulation process on distillery effluents with stainless steel electrodes. These sludges were valued as an addition in

building materials. The optimal percentage of sludges that could replace cement with a marginal modification of the physicochemical properties found was 7.5.

Tezcan Un, U. et al. [7] used electrocoagulation sludge from chromium VI effluents in the production of inorganic pigments.

Sludge from effluent treatment containing TiO2-based nanoparticles has been considered as a catalyst for the activation of peroxymonosulfate (PMS) to degrade ciprofloxacin (CIP) as an emerging pollutant [8]. Treatment with titanium-based electrodes yielded sludge which was incinerated at 600 °C to produce a functional TiO2 photocatalyst. X-ray diffraction analysis revealed that the TiO2 produced under optimal electrocoagulation conditions was predominantly anatase in structure. The specific surface area of the synthesized TiO2 photocatalyst was higher than that of commercially available and widely used Degussa P-25 TiO2 [9]. The iron sludge generated by an electrocoagulation process was used for the degradation of phenol by the photo-Fenton process. Phenol degradation efficiencies were 100%, 71.3, and 51% at initial phenol concentrations of 50 mg/L, 100 mg/L, and 150 mg/L, respectively [10].

Valorisation as a dye adsorbent has also been the subject of studies [11]. Over the past ten years, we have not noted any work on the energy recovery of electrocoagulation sludge.

3 Materials and Methods

3.1 The Electrocoagulation Process

The treated effluent comes from a treatment plant. The electrocoagulation cell consists of an assembly with three electrodes (1.9 cm × 6.6 cm) immersed in the effluent with an inter-electrode distance of 2 cm. One of the electrodes is made of stainless steel serving as the cathode and the other two made of aluminum serving as the anode are connected to a galvanostat using the connection wires. A direct current of 0.66 A, i.e. a current density of 250 A/m^2, is imposed in 2 L of waste water for a period of 1 h. The experiment is repeated several times to obtain a certain amount of sludge. At the end of each experiment, three phases are observed: the lightest sludge is above, the heaviest sludge below and the treated water in the middle. Floating sludge is collected using a spatula. Then the water is decanted to recover the sludge that is at the bottom (Fig. 1).

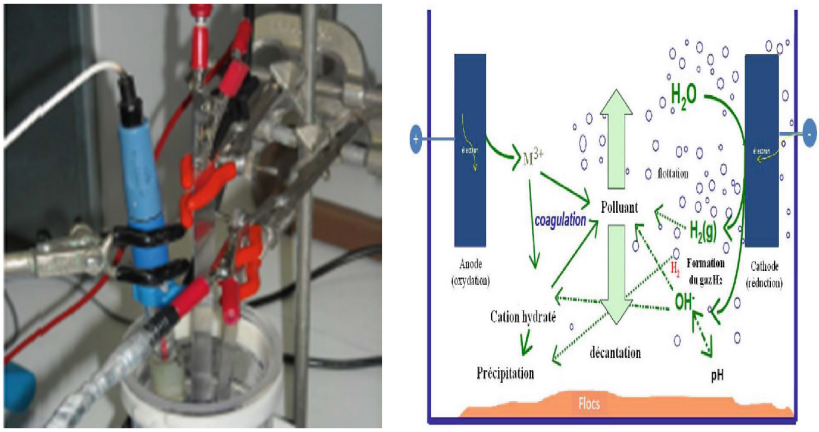

Fig. 1. Electrocoagulation cell and principle of operation

3.2 Microbiological Analysis of Sludge

The microbiological study will consist of identifying and counting the presence of microorganisms, more particularly coliforms, staphylococcus aureus and sulphite-reducing clostridium.

The identification of the microorganisms is done by preparing the EPT (Buffered Peptone Water) for the dilutions and the media where the colonies appear. The operating parameters are optimized by varying: the sample volume of 100 mL; the initial concentration from 5 to 100 mg/L; the current density from 100 to 300 A/m^2 (for an electro-active surface of 14 cm^2, the current is varied from 0.1 to 0.4 A); the initial pH of 3 to 9 (by addition of NaOH and HCl solutions at 0.1 N); the initial conductivity of 0.9 to 4.27 (by addition of a mass of NaCl ranging from 0.1 to 1 g/L); the inter-electrode distance (ied) of 0.25 to 1.5 cm.

Dye concentration was spectroscopically determined using the Beer-Lambert law (Absorbance $= \varepsilon \times \ell \times$ C) applied at 592 nm, where ε is the molar absorptivity, ℓ is the solution thickness and C the CV concentration. The absorbance are measured by a spectrophotometer SPECORD 250 PLUS.

Preparing for the EPT

EPT (Buffered Peptone Water) is used for dilutions. The solution is prepared by 10 g of EPT in 500 mL of distilled water and distributed in tubes (9 mL for each tube) then sterilized in an autoclave at 121 °C for 15 min.

Diluting Method

The method of successive decimal dilution is done by aseptic sampling of 1 mL of the stock solution (sludge) using a sterile graduated pipette fitted with a suction bulb. This sample is transferred into the first tube (9 mL of EPT) which will be marked 10^{-1}, the pipette must not penetrate the 9 mL of diluent.

Using a second sterile pipette of 01 ml, proceed in the same way (withdrawal from tube 10^{-1} to tube 10^{-2} … up to tube 10^{-9}), using a new sterile pipette for each sample and the sample is homogenized by aspiration and discharge three times.

Media Preparation

- The VRBL (violet read bile agar) medium, also called "violet red neutral bile lactose crystal", was prepared by introducing 20.75 g into 500 mL of distilled water. This solution is stirred until completely dissolved and then sterilized in an autoclave for 15 min at 121 °C.
- For the preparation of the TSN (Tryptone Sulfite Neomycin) medium, 20 g are introduced into 500 mL of water then boiled until completely dissolved. Then the solution is distributed in the tubes and sterilized for 15 min at 121 °C in an autoclave.
- The Baird Parker Agar Base medium is obtained by dissolving 30 g in 475 mL of distilled water, then left to soak and brought to the boil with continuous stirring. Then, the solution is sterilized in the autoclave at 121 °C for 15 min and cooled to 50 °C to finally add 25 mL of the vial of egg yolk with potassium tellurite.

For the TSN medium, 0.1 mL of each dilution is poured into a tube containing the medium solution. Afterwards these tubes will be incubated at 44 °C for 24 h. And the other two media (VRBL and Baird Parker) will be well mixed to have a homogeneous solution and the Baird Parker medium is distributed in the Petri dishes because it undergoes surface seeding while the VRBL will undergo deep culture.

Surface Culture

For this method, 0.1 mL of each dilution is deposited on the surface of the agar medium (pre-poured dish) then spread using a spreading rake which is passed over the surface of the agar while turn the box. This method is used for Baird Parker Agar Base medium and is incubated at 37 °C for 24 or 48 h.

Deep Cultivation

Unlike the previous culture, 0.1 mL of each dilution is placed then 10 to 15 mL of agar medium (40 to 45 °C) is poured into the dish and mixed evenly with the inoculum. The dishes are then cooled and incubated. This technique is used for the VRBL medium and the incubation is done at 44 °C in 24 h. In this case the colonies can develop on the surface or in depth.

The counting method

The appearance of colonies in the cultured medium attests to the presence of the bacteria sought in the sample.

These bacteria are determined by the following relationship:

$$\text{Number of bacteria} = \frac{\text{Number of colonies}}{\text{volume introduced} * \text{dilution}} * \frac{\text{M}}{\text{z}} \tag{3}$$

Volume introduced: 1 mL.

The number of colonies of a dilution represents the total number of colonies on the two dishes or tubes of this dilution divided by 2. The dilution chosen represents the least diluted of the media where the colonies have grown and for each dilution two dishes or tubes are seeded.

3.3 Biofilm Preparation

The biofilm is obtained by introducing carbon tissue 1 cm wide into a solution containing sludge from electrocoagulation stored in an oven at 44 °C for 3 to 4 days. These sludges from electrocoagulation have been enriched with shredded banana envelopes and acetate to increase the proliferation of microorganisms. The latter will be deposited on the surface of the carbon tissues thus constituting the biofilms (Fig. 2).

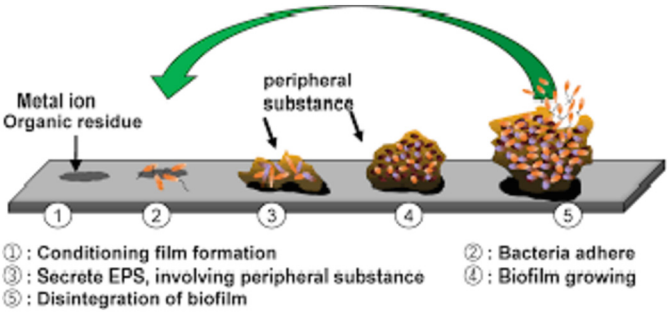

Fig. 2. Biofilm formation

3.4 Design of the MFC

The cell designed in this study is entirely microbial. The sludge from electrocoagulation is introduced into the two compartments of the cell. The previously prepared biofilm is immersed in each compartment then these electrodes are connected by a connecting wire to establish a potential difference between the anode and the cathode. A voltmeter and an ammeter were used to measure voltage and current respectively.

Indeed, in the anode compartment 150 mL of sludge are introduced and 75 mL in the cathode to a height of 8.2 cm. To increase the conductivity of the compartment considered as the cathode, NaCl was added. The addition of the substrate (acetate or glucose) in the anode allows the multiplication of microorganisms.

The measurement of current and voltage was done within one day. To maintain the multiplication of microorganisms, the addition of 2 g of substrate was done as soon as there was a decrease in the amount of energy (Fig. 3).

Fig. 3. Microbial fuel cell

3.5 MFC Characterization

The Power

To better visualize the optimal operating point for the use of the battery, the voltage-intensity product is calculated in order to obtain the power output.

$$P = U * I \tag{4}$$

U: the voltage of the battery, I: the intensity of the battery,

The power can be expressed per unit of projected area of electrode (anode or cathode), which gives a surface density of power expressed in mW/m^2.

$$d = 1 - \frac{P}{S} \tag{5}$$

S: surface of the electrode.

Polarization Curves

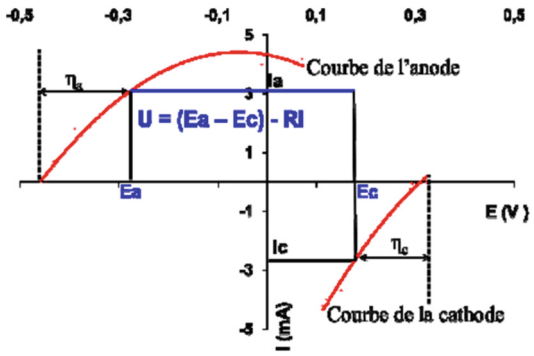

Fig. 4. Polarization curves of the electrodes of MFC

The polarization curves at the anode and at the cathode make it possible to correlate the functioning of the PCM to the kinetics of the electrodes. The current flowing through the MFC is plotted at low scanning speed as a function of the potential of each electrode measured with respect to a reference electrode (Fig. 4). The shape of the polarization curve varies according to the electrode materials and the inter-electrode distance.

For each resistor when the system reaches a pseudo-stationary state (state which is generally established after a few minutes depending on the system and the value of the resistor used), the potential and current values are recorded. This can be done using a potentiostat or simply by charging the battery through external electrical resistors of different values.

Faradic Yield

The faradic yield (R) of a PCM depends on the microorganisms responsible for the oxidation, the nature of the organic carbon serving as fuel and all the alternative reactions that consume this organic carbon [12]. Processes such as aerobic and anaerobic respiration, biomass growth or the formation of reaction intermediates from fermentation can reduce the faradic yield [13].

It can be defined by the following relationship:

$$R(\%) = \frac{Q}{Q_t} * 100 \tag{6}$$

where: Q is the total amount of electricity transferred to the anode

$$Q = \int_0^t idt \tag{7}$$

Q_t is the amount of electricity available in the substrate.

$$Q_t = \frac{nFCV}{M_i} \tag{8}$$

$$R = \frac{M_i \int_0^t idt}{nFCV} \tag{9}$$

In the case of effluent treatment, the available organic matter is measured in terms of chemical oxygen demand (COD) and the faradic yield is calculated from the COD removed:

$$R = \frac{M_i \int_0^t idt}{nFV\Delta(DCO)} \tag{10}$$

With Mi: molar mass of the substrate (g/mol).
n: number of electrons exchanged during the reaction (mol e-/mol).
F: Faraday's constant (C/mol e-).
V: volume of effluent involved (L).
Δ(COD): variation in COD entering and leaving the reactor (g/L).

In the case of effluent treatment, Mi = 32 g/mol of O2, and therefore Δ(COD):) is expressed in g equivalent O_2/L [2].

4 Results and Discussion

4.1 Effluent Treatment by Electrocoagulation

Treatment by electrocoagulation of wastewater allowed a COD reduction from 1193 to 116 mg/L, i.e. a rate of 90.27%. As for the turbidity, it varies from 130 to 37 NTU leading to a reduction in the MES, but also a reduction in parameters such as the aluminum content. The pH went from 7.92 to 9.45 (Fig. 5).

Indeed, the decrease in turbidity is because the complex formed during the process adsorbs the suspended solids which separate from the effluent by flocculation or settling depending on the size of the flocs. This decrease is accompanied by a reduction COD and dissolved oxygen. The evolution of the pH is due to the reaction at the cathode which releases hydroxides in solution [14] (Table 1).

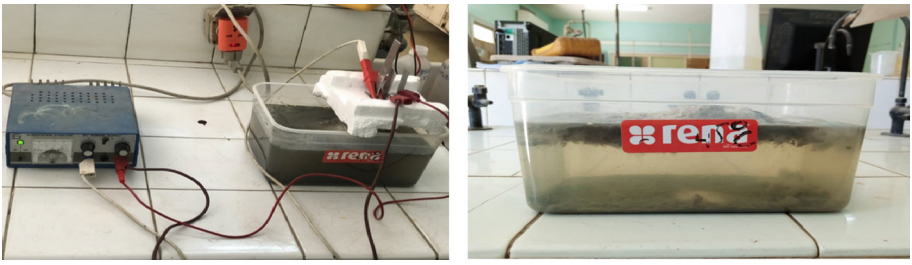

Fig. 5. Electrocoagulation reactor before and after treatment

Table 1. Physico-chemical parameters of wastewater before and after electrocoagulation

Settings	before treatment	after treatment
Conductivité (mS/cm)	2,081	1,493
DCO (mg/L)	1193	116
Teneur en aluminium (mg/L)	1	0,05
Température (°C)	25	21
Oxygène dissout (mg/L)	6,9	1,7
pH	7,92	9,45
Turbidité (NTU)	130	37

4.2 Sludge Analysis

This part of the study consists in determining the micro-organisms present in the sludge. Three species are targeted, namely coliforms, sulphite-reducing clostridium, and staphylococcus aureus. These species have shown their effectiveness for the implementation of microbial stack in previous studies [15] (Table 2).

Table 2. Micro-organisms counted in the sludge.

N(UFC/mL)	Sample		
	Sludge from electrocoagulation	Sludge + sodium acetate (MFC 1)	Sludge + glucose (MFC 2)
coliforms	92000	150	19
sulphite-reducing clostridium	10000	1000	13000
staphylococcus aureus	0	1	2

The microbiological analysis of sludge from electrocoagulation shows the presence of sulphite-reducing coliforms and clostridium and the absence of staphylococcus aureus (92000 CFU/mL for coliforms and 10000 CFU/mL for sulphite-reducing clostridium). These coliforms are more present in sludge than in wastewater (78500 CFU/mL). This can be explained by the fact that the organic matter is concentrated in the sludge compared to the wastewater, thus making its degradation easier and faster.

The sludge after addition of substrate (acetate or glucose) revealed the presence of the three species. In fact, the sludge with acetate contains 150 coliforms, 1000 sulphite-reducing clostridium and 1 staphylococcus aureus, while those with glucose reveal 19 coliforms, 13000 sulphite-reducing clostridium and 2 staphylococcus aureus.

The microbial population of coliforms and sulfito-reducing Clostridium underwent a considerable decrease in the first pile compared to the sludge from electrocoagulation. This decrease is due to the unfavorable environmental conditions caused by the addition of acetate.

The same trend is noted for the coliform species at the level of the second MFC, on the other hand the microbial population of sulphite-reducing clostridium increases. This is consistent with the microbiological results obtained by Xin et al. (2019) as part of the study on the overview of microbial community profiles associated with the production of electrical energy in MFCs [16]. It is therefore noted that the glucose used is more favorable to the growth of clostridia than acetate. The staphylococcus aureus which was absent in the sludge appear in stacks 1 and 2 in small quantities (1 for the first MFC and 2 for the second MFC). Acetate and glucose are favorable to the growth of staphylococcus aureus but glucose remains more effective.

The microorganisms are represented in colonies as shown in the following Fig. 6.

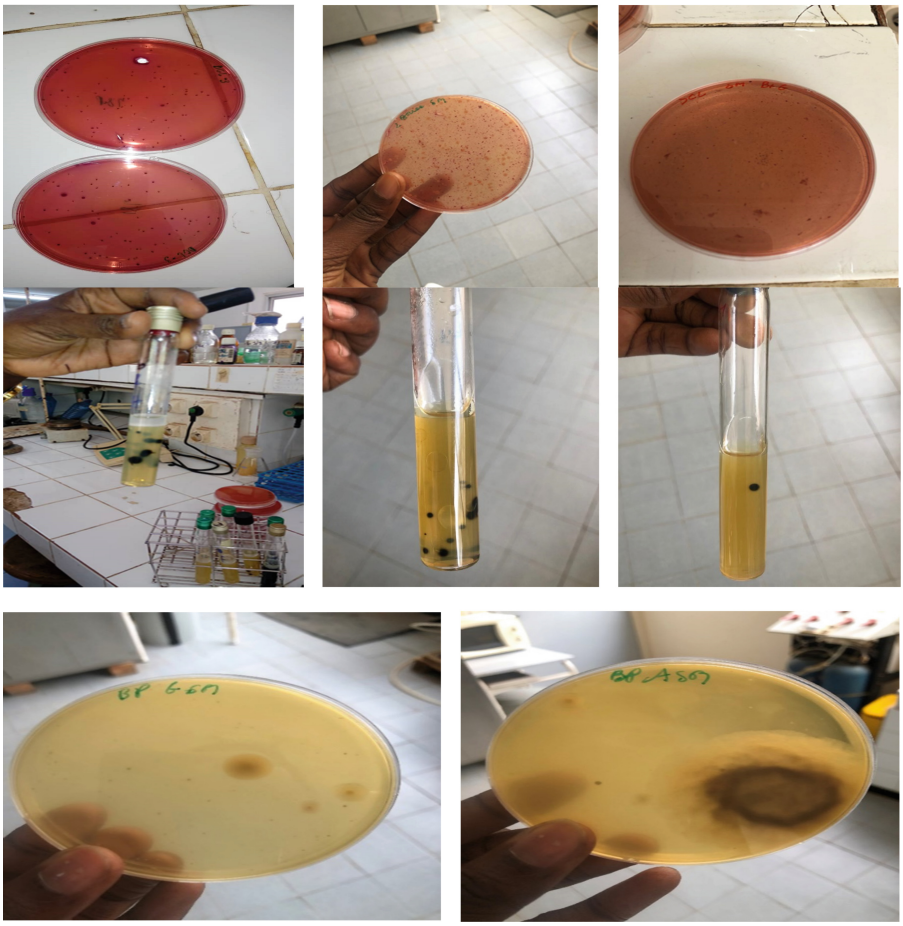

Fig. 6. Micro-organisms present on the surface (A: coliforms, B: sulfito-reducing clostridium)

4.3 MFC Settings

4.3.1 Evolution of Current as a Function of Time

To assess the efficiency of the battery designed previously with acetate or glucose as substrates, the evolution of the intensity of the current as a function of time was studied (Fig. 7).

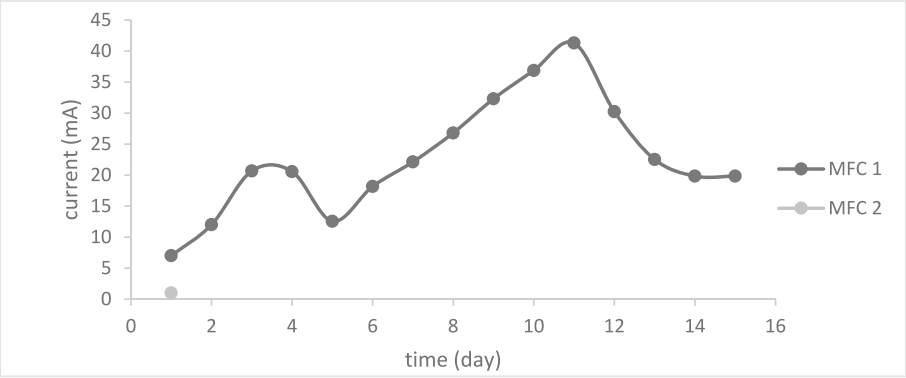

Fig. 7. Variation of current as a function of time

The acetate based MFC (MFC 1) has a current intensity that evolves sinusoidally from 0.033 to 0.102 mA and reaches its maximum on the eleventh day for a value of 0.102 mA while for the MFC with glucose (MFC 2), the values vary between 0.042 and 0.095 mA and its maximum is reached on the sixth day with an intensity of 0.095 mA.

This variation confirms the oxidation of organic matter by bacteria followed by saturation. The addition of acetate or glucose constitutes a contribution of substrate which increases the productivity of the micro-organisms.

4.3.2 Evolution of Voltage as a Function of Time

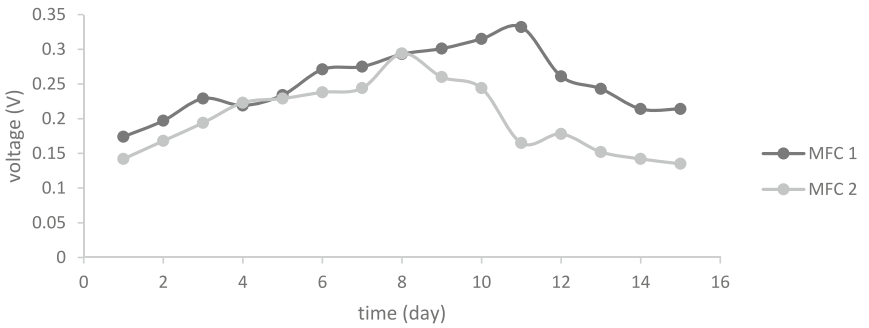

Fig. 8. Variation of voltage as a function of time

This monitoring makes it possible to characterize the speed of transfer of electrons from the biofilm to the anode. For MFC 1, the voltage varies between 0.174 and 0.332 mV and reaches its maximum on the eleventh day while that of MFC 2 varies from 0.135 to 0.294 mV and its maximum is reached on the eighth day. It is noted that the bacteria are more electroactive in the presence of acetate than in the presence of glucose. Also, the life of the MFC1 is longer. Indeed, acetate is more conducive to the development of electrophilic microorganisms than glucose [17] (Fig. 8).

4.3.3 Study of Power Density

The sludge from electrocoagulation treatment is used as inoculum for the design of the MFC (Fig. 9).

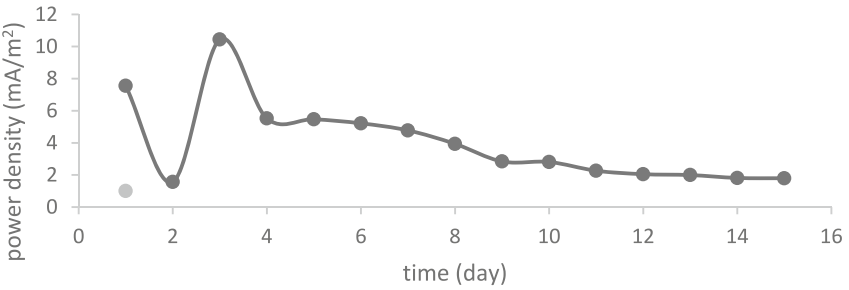

Fig. 9. Variation of power density as a function of time for MFC without substrate

The power density of the MFC varies between 1.6 and 10.4 mW/m^2. From the third day, the power density reaches its maximum (10.4 mW/m^2) then a gradual decrease in the following days was observed.

This development confirms the presence of electroactive microorganisms in the electrocoagulation sludge without adding substrate. Electron transfer is optimum on the third day of operation. Beyond that, there is a saturation which results in a decrease in density.

Based on the observation that the life of the battery is three days, the addition of substrate was made according to this frequency (Fig. 10).

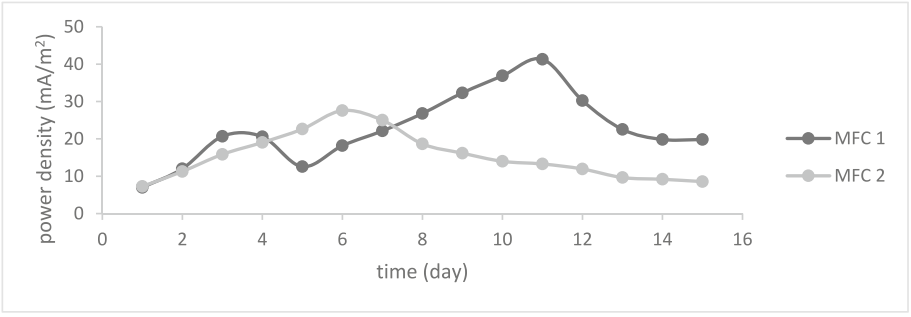

Fig. 10. Variation of power density as a function of time for MFC with substrate

The power density in the MFC 1 varies between 7 and 41 mW/m^2. From the first three days, a gradual increase in power density was observed from 7 to 20 mW/m^2 with the introduction of 2 g of acetate at the anode on the first day. After observing a decrease in power density from the fifth day, 2 g of acetate are added to the same compartment at three-day intervals. On the eleventh day, the density reaches its maximum (41 mW/m^2) and decreases in the following days regardless of the addition of acetate.

The increase in power density is due to a multiplication of microorganisms after addition of acetate while its decrease is caused by a drop in the microbial population. The latter can be explained by the fact that the microorganisms have reached their threshold, which is why they can no longer consume the substrate. Then the energy produced decreases more and more because the electro-active micro-organisms are no longer productive.

In MFC 2 an increase in power density is observed until the sixth day (27 mW/m^2) then a decrease until the end of the experiment with the gradual addition of glucose at 3-day intervals. This result confirms the greater sensitivity of bacteria to acetate.

5 Conclusion

The objective of this study was to show a possible adaptation to climate change through the oxidation of microorganisms present in an environment by bacteria, to produce electricity. Similarly, it was a question of showing the possibility of using sludge from electrocoagulation as fuel in the design of a microbial fuel cell.

These aluminum hydroxide sludges are considered chemical waste because they adsorb the pollutants present in the effluent.

The microbiological study of sludge has shown the presence of certain species such as coliforms and sulphite-reducing clostridium described in the literature as being electrophilic in wastewater.

MFCs based on these sludges have shown their efficiency for energy production. Their performance has been improved by using acetate as a substrate. MFCs therefore appear to be an effective method for the recovery of sludge from electrocoagulation.

The generated power strongly depends on the quality of the biofilm. The formation of the biofilm is conditioned by the electrode materials and the type of effluent which was used for the formation of the sludge. Limits to the development of the MFC could therefore be associated with its lifetime in the case of continuous operation. It is therefore necessary to optimize the operating parameters in order to create an environment favorable to the development of electroactive microorganisms and to the maintenance of electroactivity without the addition of chemical substrates. Parameters such as the nature of the biofilm formed, the mechanisms of electron transfer within the bacterium and from the biofilm to the anode, the electrode materials, and the configuration of the MFC. Can be optimized to improve the power supplied and the lifetime of the MFC.

In perspective, an experimental plan based on a 7-factor HADAMARD matrix will be rolled out to optimize the operation.

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