

Design and Development of Microbial Fuel Cell Using Modified Graphite Anode

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Abstract –The microbial fuel cell (MFC) is an innovative renewable energy technology that also serves to treat wastewater through the bacteria-driven oxidation of organic substrates. The liquid anolyte contains the organic substrate to be oxidized, while the catholyte contains the substance to be reduced. In a Dual chamber MFC, the catholyte typically contains dissolved oxygen or another easily reducible compound in an aqueous solution, while in a single-chamber MFC, gaseous airborne oxygen reacts directly at the cathode. Graphite rod is widely used as an anode material in microbial fuel cells (MFCs) because of its high specific surface area, low cost, good electrical conductivity, and biocompatibility. In this paper, graphite anode samples were thermally treated with a mixed solution of 30% HNO₃ and 30% H₂O₂ with volume ratios of 1:1. The electrochemical performances of the resulting MFCs were investigated by polarization curve measurement. XRD and scanning electron microscopy were conducted to characterize the functional groups and the morphology of the Graphite rods. After modification, the number of oxygen-containing functional groups in MFC increased compared with MFC(bare anode), the start-up time of the obtained MFCs was markedly shortened, and the charge transfer resistance of the bio anode was decreased. In MFC with modified graphite anode as electrode, the maximum power density was 2.94 mW m⁻², which was 200% higher than MFC graphite rod. Increases of larger specific surface area and less impurities on the modified anodes favoured the adsorption and growth of bacteria and acceleration of electron transport between the electrode and bacteria. Thus, the electrochemical characteristics of MFCs employing these anodes were improved.

Keywords: Microbial Fuel Cell, Modified Graphite Anode, Dual chamber,

I. INTRODUCTION

Renewable energy technologies and environmentally sustainable solutions are gaining considerable momentum not only within the scientific community, but the general population as well. India has had a long involvement with anaerobic digestion and bio-energy technologies. Waste water treatment plants in the country have been established which produce renewable energy from sewage. Every year, about 55 million tones of municipal solid waste (MSW) and 38 billion liters of sewage are generated in the urban areas of India [1]. As with any fuel cell, a main objective of the MFC is to generate electrical power. However, since it is likely to be also used for the purposes of wastewater treatment, it has the added objective of effectively treating wastewater mainly through the reduction of COD, although it may also be useful for the removal of specific inorganic species such as

sulfides [2] and nitrates. [3] Given that this technology is still in its early stages of development and the addition of a biological component to a conventional fuel cell introduces a number of challenges, the research focus has been broad, covering aspects such as materials, fuel cell design and configuration, substrates for metabolism, choice of inoculant and catalyst usage. The origin of using microorganisms to generate electricity is a new concept and dates back to 1910 when Potter first discovered electricity production by *E.coli*. [4] These results were later substantiated in 1931 when Cohen demonstrated that a voltage of 35 V could be achieved from MFCs connected in series. [4] Although some studies in the 1950s and 1960s were conducted, the past 15 years have seen a significant push in the area of MFC research. This increased attention is no doubt due to the greater awareness and demand for renewable sources of energy as well as improved water treatment. Several studies have focused on delineating the activation losses occurring at the anode as well as methods by which the losses can be reduced. [5] used electrochemical impedance spectroscopy (EIS) and found that the impedance associated with the anode is the dominant loss in an MFC, even with an established biofilm. They also reported that the charge transfer resistance dropped from 2.6 to 1.5 k - cm² after 5 days of closed-circuit operation and decreased further to 0.48 k -cm² after 3 weeks of operation. This indicates that as the biofilm becomes established, biocatalytic oxidation of substrate is enhanced and thus activation losses are reduced. [6] found that a decrease in the initial suspended biomass concentration as inoculant appears to favour the growth of biofilm biomass within an MFC relative to suspended MFC biomass. Furthermore, this leads to an increase in biofilm thickness and density and consequently raises the maximum current that can be generated, although it is worth noting that the amount of charge obtained over a batch cycle was not affected. PEMFCs, MFCs also exhibit ohmic (electrolyte) resistance associated with the transport of H⁺ ions from the anode where they are liberated to the cathode. However, unlike PEMFCs which use a proton-permeable membrane as the solitary electrolyte, MFCs rely on the ions present in the wastewater such as Na⁺, Mg²⁺, K⁺, SO₃²⁻, NH₃⁺ and NO₃⁻ to provide the ionic conductivity of the anolyte. [8] Given that the wastewater solution is not concentrated; its proton conductivity is much lower than that of Nafion. Although a Nafion membrane is commonly used in MFC systems to separate the anode compartment from the cathode or cathode compartment due to its proton- permeability and hydrophobicity, its importance is not likely as critical as in a conventional PEMFC. In PEMFCs, protons liberated at the anode can transfer directly to the cathode since both electrodes are hot-pressed to the Nafion and so very closely spaced. The hydrogen source in these cells is typically gaseous and free of ionic impurities that can occupy the negatively charged sulfonic acid

active groups on the Nafion structure. Thus, more active sites are available for proton transfer and ohmic losses are lower. Anolyte solutions in MFCs typically contain cation species at concentrations that are about 10^5 times higher than that of protons (at a typical operating anolyte pH of 7), resulting in fewer active sites for proton conduction and higher ohmic resistance. In a study on a cell in which Nafion was incorporated, [8] found that several cationic species also are transported into the catholyte solution and that these species are responsible for the majority of positive charge transfer required to maintain electro neutrality in the anolyte and catholyte solutions. Although MFCs do not necessarily require an aqueous catholyte, this study found that proton transfer from the anolyte into the catholyte must be improved to further enhance cathodic reduction of oxygen and in turn the power densities. To help minimize this effect, phosphate buffers and to a lesser extent bicarbonate buffer can be added to anolyte solutions to increase proton conductivity. [9] found that the addition of a 100 mM phosphate buffer caused a four-fold increase in the maximum power density (from 70 to 320 mW/m²) and a 25% rise in overall power density (from 70 to 98 mW/m²) obtained from a given wastewater.

Although useful for laboratory-scale experiments, the addition of phosphate buffers may not be feasible for pilot-scale and industrial-scale applications as they are non-renewable and can be expensive. Initial studies using bicarbonate buffers have shown promising results by yielding better performance than that achieved using phosphate buffers. [10] However, since very few such studies on the use of these buffers have been reported, their overall efficacy still remains questionable. organism is encountered. [11] developed a model for electron donor oxidation at a biofilm anode and reached several conclusions which are relevant to polarization losses. The conductivity of the biofilm dependence on biofilm density and thickness is a key indicator of the limitations of a particular biofilm. It also strongly influences electron donor fluxes and biomass distribution. At lower bio ($\sim 10^{-5}$ mS/cm), the anode potential is limiting (activation polarization). At high bio (10^{-3} mS/cm), mass transfer of the electron donor (acetate) to the active biofilm residing near the anode becomes limiting due to lower ohmic resistance and increased substrate utilization, to the point where the transfer of the substrate to the active portion of the biofilm near the anode becomes limiting. Marcus et al. also found that biofilm thickness should be kept to a minimum so that inert biomass does not accumulate and increase biofilm thickness and thereby inhibit transport of substrate to active biomass near the anode surface. Cathodic mass transfer losses dominate when the rate of oxidant species flux to the cathode is low. This often occurs when the oxidant is dissolved oxygen in solution due to its low solubility in water (8 mg/L at 27°C). In addition, the amount of catalyst loading may also affect mass transfer.

Since platinum is still the most widely used catalyst for oxygen reduction in an MFC, the minimization of the catalyst loading while still maintaining performance is important to reduce costs. One study revealed that only a minor drop in cathode potential by 20 mV occurred when the Pt catalyst loading was decreased from 2 mg/cm² to 0.1 mg/cm² at a current density of 1 mA/cm². [12] A loading of 0.5 mg/cm² at the same current density caused the cathodic potential to drop by only 10 mV. The second major

function of an MFC is to convert oxidizable substrates in a feed stream to fully reduced species. By doing so, the chemical oxygen demand (COD) of the effluent is reduced. COD can be from natural sources such as domestic [13,14] or industrial [15, 16] wastewater, or by addition of 12 synthetic sources such as volatile fatty acids [13, 17], sugars [18, 19], or starches. [20] The COD is an important wastewater treatment parameter because any oxidizable compounds that are discharged to a body of water will then be aerobically reduced by organisms, thus reducing the dissolved oxygen concentration in the water and consequently suffocating aquatic biota. The amount of COD removal in an MFC is a function of the complexity of the substrate, the hydraulic residence time (HRT) of the solution, and the maturity of the microbial community. Complex substrates such as sugars and starches take a longer time to fully metabolize as they are first degraded into simpler sugars, and then volatile fatty acids, before finally being oxidized to methane, hydrogen sulphide, and carbon dioxide. [21] MFCs can be operated using a wide variety of HRTs, ranging from 22 minutes [17] to several days [16] though most that are operated continuously or as fed-batch have HRTs of 2 days or less. The HRT is inversely proportional to the COD removed; the shorter the HRT, the lower the COD removal. [14, 17] For example, one study by Min and Logan varied the HRT and observed its effect on COD removal as well as power production. They found that as the HRT decreased from 4.0 hr to 1.1 hr, power production from a domestic wastewater feed increased from 43 to 72 mW/m², while COD removal increased from 42% to 79% (246 mg COD/L feed). [17] Recent research has also attempted to further treat the effluent from an MFC by looping it to the cathode chamber for cathodic reduction of nitrates and nitrites.

After oxidation of the carbonaceous compounds in the anolyte chamber, the ammonium-rich effluent is sent through an air stripper to convert the ammonium to nitrate. Finally, the discharge from the stripper is cycled through to the cathode chamber, where it is reduced to nitrogen gas. One study by Virdis *et al.* successfully ran this type of dual-treatment configuration, producing a maximum of 34.6 W/m³ while removing 100% of a 1 g COD/L as acetate feed at a coulombic efficiency of 40.8%, and 58.9% of a 586 mg/L nitrate feed at a coulombic efficiency of 72.2%. [22] These types of MFCs may play a larger role in situations where wastewater treatment is of higher priority, as aeration of stripper for conversion of ammonium requires a pump decreasing net power production. Although recent advances in MFC architecture have shown great promise, the viability of this technology on a large scale requires that these cells be stacked so that either voltage or current is increased to the point where it can be distributed to the power grid or practicably usable. As MFCs research progresses, recent efforts are beginning to focus on stacked cell architectures. An early design by Aelterman *et al.* used a 12-chambered cell split into 6 dual-chambered flat-plate cells (no serpentine flow), as shown in Figure 1 of reference [29]. In fuel cells, the catalyzed reduction of oxygen at the cathode is not limited to one simple reaction. Several steps are required, each with their own specific reaction kinetics. Determination of the molecules in the individual reactions can be difficult, especially for non-precious catalysts given that the actual sites for catalysis are not entirely understood themselves. The reduction of oxygen can either produce water or hydrogen peroxide. The water-producing

pathway is a 4-electron reaction, meaning that 4 electrons are required per molecule oxygen, while the hydrogen peroxide-producing pathway is a 2-electron reaction. [30] From an efficiency standpoint, the water-producing pathway is preferable since more electrons are consumed per oxygen molecule. Hydrogen peroxide is also reactive, potentially causing degradation of the Nafion membrane [31]. In fuel cells, the catalyzed reduction of oxygen at the cathode is not limited to one simple reaction. Several steps are required, each with their own specific reaction kinetics. Determination of the molecules in the individual reactions can be difficult, especially for non-precious catalysts given that the actual sites for catalysis are not entirely understood themselves. The reduction of oxygen can either produce water or hydrogen peroxide. The water-producing pathway is a 4-electron reaction, meaning that 4 electrons are required per molecule oxygen, while the hydrogen peroxide-producing pathway is a 2-electron reaction. [30] From an efficiency standpoint, the water-producing pathway is preferable since more electrons are consumed per oxygen molecule. Hydrogen peroxide is also reactive, potentially causing degradation of the Nafion membrane [31].

The research objectives of this thesis project are three-fold. Firstly, the operation of a dual chamber MFC is monitored under different effluents using a platinum catalyst. Secondly, the raw graphite rod performance compared with modified graphite rod with different water effluents, different loading levels and compared to the optimum performance obtained with the platinum catalyst. During MFC runs, electrochemical parameters such as current and power outputs are continually monitored on-line, while wastewater parameters such as COD removal are regularly measured off-line. Specific research objectives are outlined as follows: Construction of Dual chamber microbial fuel cell and observation or characterization of catalyst when coating on air cathode. To improve surface area of electrode (graphite rod). To Characterize of the structure of the selected anode electrode by scanning electron microscopy (SEM), and crystal composition by x-ray diffraction (XRD). Assessment of the performance of the MFC to reduce the COD level of the feed stream and accounting for the fate of COD fed to the system on the basis of a COD balance. To generate Electricity and Observation of power density in both cases of microbial fuel cell.

II. MATERIALS AND METHODOLOGY

Materials – MFC Construction

The MFC was re-designed so that the graphite plate anode was replaced with two plain carbon cloth electrodes and a non-conductive plate placed between them to electrically and physically separate the two cells. The cathodes were also replaced with single-sided membrane-electrode assemblies

(MEA). Each MEA was comprised of two layers: carbon paper impregnated with hydrophobic Pt/c (5% weight) to reduce water loss, a carbon/PTFE micro porous layer to improve oxygen diffusion. The connections of each cell to the external circuit were made directly at the graphite rod anode and at All liquid-containing parts of the cell were sealed using rubber gaskets. The anodes of both cells were separated by a non-conductive plate. The MFC stack as a whole was constructed as a plate-and-frame design fastened together by threaded Nylon screws. The overall nominal flat-plane area of each anode and cathode was both 80 cm² and the overall anolyte chamber volume in each cell was 50 ml.

III. EXPERIMENTAL SETUP

Fig.1.MFC SET UP AND CONSTRUCTION

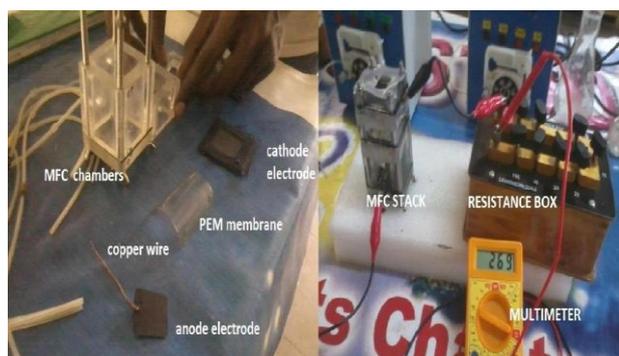
The MFCs, with 50ml volumes for the anode and 30 ml cathode chambers, were gas sterilized for 12 hrs using ethylene and assembled in a sterile container. Anode and cathodes were placed into the MFCs and a proton exchange membrane (Nafion 117, 5cm x 5cm²) was inserted between them MFC assembly's main goal is to avoid leakage in the cell and assemble all cells in an identical manner. The first step is to place the anode over the center of the opening on the MFC flange. After this, an inert plastic mesh is placed over the anode. In subsequent steps, the anode and cathode will be pushed together to minimize the distance between them and thus the internal resistance. The approximate distance between anode and cathode is 5 mm. This mesh serves as a spacer that will prevent a short circuit in the cell. On top of the mesh is placed a closed cell sponge silicon gasket. This material was chosen because it is soft and compressible and thus will form a good seal without damaging the cathode, for its thermal properties which allow it to be autoclaved, and finally because its closed cell structure makes it gas-tight and will not allow oxygen diffusion into the cell. The cathode electrode is placed on top of the gasket, followed by the copper wire current collector. The copper wire is bent into a pleated shape in order to increase the contact area and ensure good contact even in the event of a small shift in either cathode or current collector position. A second gasket is placed over the current collector, and finally the front cover.

IV. EXPERIMENTAL RESULTS

Analysis of Anode Electrode

(A) SEM analysis of Graphite anode and Modified graphite anode

Scanning electron micrographs of graphite rods. Modifying with increasing concentrations of HNO₃ resulted in more rougher surface on graphite rods owing to more carbon loss, therefore provided more space for bacterial attachment. A marked change of morphology on graphite anode was caused by microbe adhering to them and colony formation, likely because oxygen-containing functional groups on the modified graphite rod surfaces significantly increased their biocompatibility.



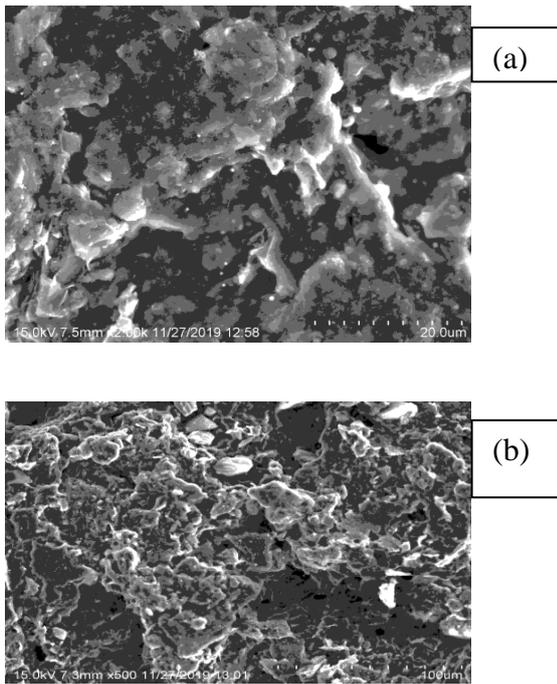


Fig.2. (a)&(b)SEM analysis of graphite anode and modified graphite anode

(B) XRD analysis of graphite anode and modified graphite anode

To determine the chemical composition of the chemical scale, elemental analysis was performed with XRD). Quantitative analysis of XRD data could not be performed due to a limitation of number of XRD images. XRD analysis of both the electrodes showed the difference of roughness as can be seen in the figures the raw graphite rod had more rough and blunt peaks and while the modified rod has slim and sharp peaks.

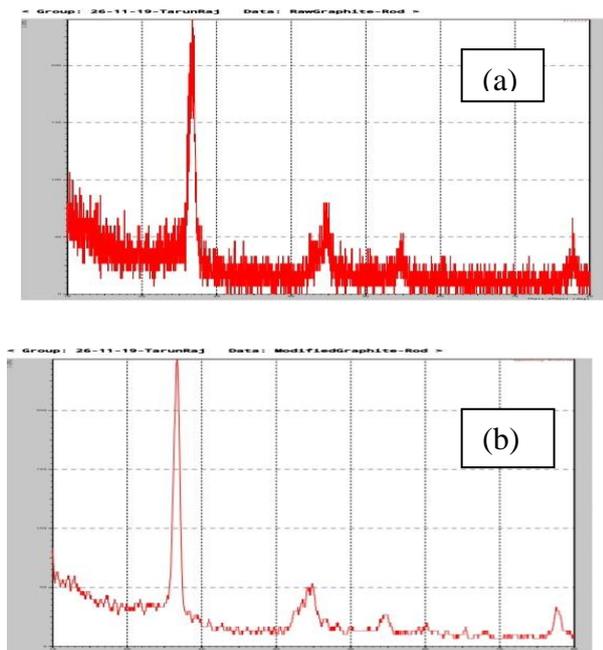


Fig.3.(a)&(b)XRD analysis of graphite anode and modified

graphite anode

Dual chamber MFC using graphite anode and modified graphite anode

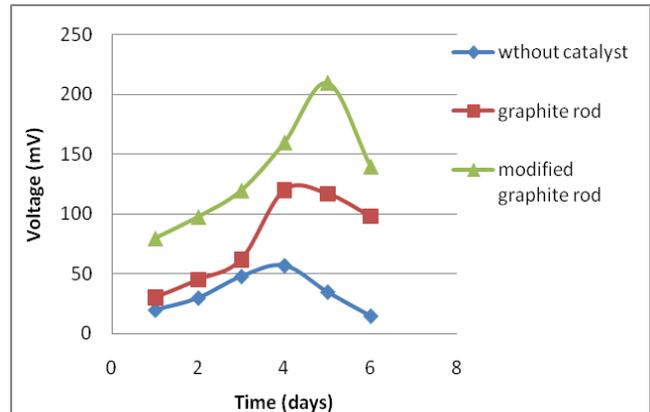


Fig.4.Dual Chamber MFC voltage and days plot for Graphite anode and modified graphite anode

The currents, voltages and power generation showed a gradual increase for few days, and then declined. This variation was due to the availability of less oxidizable substrates in the waste samples. The peak currents, voltages and power generation were observed, in all cases, on the 4th day of the preliminary experiments, as can be clearly observed in figures above. After the 5th day, the peak values started decreasing continuously. The currents, voltages and power generation from the waste substrate samples with modified graphite has higher voltage generation than raw graphite rod , in all cases, especially under anaerobic condition of the anode chambers of the MFCs.

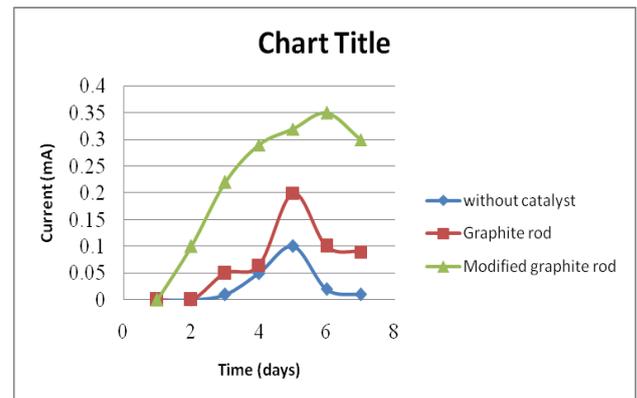


Fig.5.Dual Chamber MFC Current and days plot for Graphite anode and modified graphite anode

The figure show maximum current was obtained at 0.35mA by using modified graphite anode and it is gradually reducing until some stable current around 0.15mA. The current is significantly larger than that of MFC with GA. The result considered is that the prepared MGA offer a suitable nanostructure environment for bacterial growth and a better electron transfer from bacterial to electrode compared to GA resulting in the production of higher current by the catalytic activity of bacteria.

V. CONCLUSION

In the present work graphite rod anodes were oxidized using

HNO₃/ H₂O₂ and also the effects of parameters of both anodes used in MFC were investigated. Based on the present investigation and from the available scientific information derived from the review of relevant literature, following conclusions were drawn.

- i. The modification of graphite anode was most efficiently done.
- ii. From the results it was concluded that the maximum power density produced from using modified graphite anode is 3 times higher than raw anode.
- iii. The surface characterization of different anode used have shown different variations and was concluded that increase of oxygen containing functional groups on the modified anodes favored the adsorption and growth of bacteria and acceleration of electron transport.
- iv. The COD removal from waste water after the treatment with modified anode was more than the raw graphite anode.
- v. The surface area of the electrode and the electrochemical parameters of MFC using the anode was improved.

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