TREATED CLINOPTILOLITE-MODIFIED GRAPHITE FELT BIOANODE MICROBIAL FUEL CELLS FOR POWER GENERATION AND DYE DECOLOURISATION

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Specially dedicated to my beloved Dad and Mom,
Reza Kardi and Maria Hadighi

and

To my adorable husband
Nima

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ABSTRACT

One important factor in microbial fuel cells (MFCs) study is the anode. In MFCs, the anode acts as the key component in the generation of bioelectricity and power. Despite the fact that there have been some improvements in the electrochemical performance of MFCs in recent years, their low power generation is still deemed a major drawback. The effects of surface modifications of the anode as biofilm carrier on the performance of MFCs were investigated. This research focused on the role of the novel fabricated anode as support material for the adhesion of bacterial consortium (NAR-2) consisted of *Citrobacter* sp. A1, *Enterobacter* sp. L17 and *Enterococcus* sp. C1 were used in MFCs reactor for the decolourisation of Acid Red 27 (AR27) and the simultaneous generation of electricity. The performance of a modified anode fabricated using surfactant-treated clinoptilolite (S-TC) with common type of carbon-based material, namely treated clinoptilolite-modified graphite felt (TC-MGF) anode was evaluated with different MFCs constructions. Prior to the MFCs experiments, the modification of anode was successfully verified using different spectroscopic and microscopic techniques such as EDX, FESEM, ATR-FTIR and BET analysis. In addition, screening of parameters for the adhesion of bacterial consortium NAR-2 onto TC-MGF anode (NAR-2-bioanode) was accomplished. The newly-developed TC-MGF bioanode was implemented in the dual-chamber (H-type) of the MFC. The performance of TC-MGF bioanode was compared to the results obtained using non-modified graphite felt (BGF) bioanode. Maximum power densities for BGF and TC-MGF bioanodes were 458.8 ± 5.0 and 940.3 ± 4.2 mWm^{-2}, respectively. In the following experimental, a small MFC reactor was fabricated with TC-MGF bioanode to compare the performance of the MFC with commonly used fuel cell membranes, Nafion (N-117 and N-115), which were examined along with the N-212 membrane in a single-chamber cubic di-air cathode (S-CCD-AC) design. The power density and columbic efficiency of N-115 membrane (1022.5 mWm^{-2} - 35.4%) were significantly higher than the values obtained for the N-117 (592 mWm^{-2} - 15.6%) and N-212 (493 mWm^{-2} - 12.3%) membranes. A novel MFC reactor with TC-MGF bioanode novel design (Conch shell) using the N-115 membrane having an air-cathode upflow (A-CU) MFC, as a combination of upflow and MFC technologies was used to compare the presence and absence of a membrane design. The A-CUMFC with membrane-less at flow rate 0.6 mL min^{-1}, anode distance of 0.5 cm and a concentration of AR27 at 900 mg L^{-1}, high decolourisation rate (98%) achieved in a 60-day operation, was 40% higher than that of the membrane-MFC. The average maximum power density obtained (1250 mWm^{-2}) using the membrane-less MFC was higher than that of the membrane-MFC (1108 mWm^{-2}) during the 80-day operation with TC-MGF bioanode.

ABSTRAK

Satu faktor penting di bawah kajian sel bahan bakar mikrob (MFCs) ialah anod. Di MFC, anod bertindak sebagai komponen utama dalam penjanaan bio-elektrik dan kuasa. Walaupun terdapat beberapa peningkatan dalam prestasi elektrokimia MFC dalam beberapa tahun kebelakangan ini, penjanaan kuasa rendah mereka masih dianggap sebagai kelemahan utama. Kesan pengubahsuaian permukaan anod sebagai pembawa biofilem terhadap prestasi MFC telah dikaji. Kajian ini menumpukan kepada peranan anod yang baru dibuat bagi melekatkan filem mikrob sebagai bahan sokongan untuk menawarkan tapak pelekat konsortium bakteria (NAR-2) yang terdiri daripada *Citrobacter* sp. A1, *Enterobacter* sp. L17 *dan Enterococcus* sp. C1 digunakan dalam reaktor MFC, dari segi penyahbauan Acid Red 27 (AR27), dan penjanaan elektrik serentak. Prestasi anod yang diubahsuai yang dibuat menggunakan klinoptilolit (S-TC) yang dirawat dengan terapi surfaktan dengan bahan yang berasaskan karbon yang biasa, iaitu anoda grafit dan klinitilolit yang diubahsuai (TC-MGF) dirawat dengan pembinaan MFC yang berbeza. Sebelum kajian MFC, pengubahsuaian anod berjaya disahkan menggunakan teknik spektroskopi dan mikroskopik yang berbeza seperti analisis EDX, FESEM, ATR-FTIR dan BET. Di samping itu, pemeriksaan parameter untuk pelekat konsortium bakteria NAR-2 ke anod TC-MGF (NAR-2-bioanode) telah dicapai. Bioanod TC-MGF yang baru dibangunkan telah dilaksanakan di ruang dobel (H-jenis) MFC. Prestasi bioanod TC-MGF dibandingkan dengan hasil yang diperoleh menggunakan bioanod grafit (BGF) yang tidak diubah suai. Keupayaan kuasa maksimum untuk bioanod BGF dan TC-MGF masing-masing adalah 458.8 ± 5.0 and 940.3 ± 4.2 mWm^{-2}. Dalam kajian seterusnya, reaktor MFC kecil dibuat dengan bioanod TC-MGF untuk membandingkan prestasi MFC dengan membran sel bahan bakar yang biasa digunakan, Nafion (N-117 dan N-115), yang dikaji bersama dengan N-212 Membran dalam reka bentuk katod di-udara kubus tunggal-ruang (S-CCD-AC). Ketumpatan kuasa dan kecekapan *columbic* (1022.5 mWm^{-2} - 35.4%) membran N-115 jauh lebih tinggi dan daripada nilai yang diperoleh untuk N-117 (592 mWm^{-2} - 15.6%) dan membran N-212 (493 mWm^{-2}- 12.3%). Reka bentuk kelompok MFC dengan reka bentuk kelompok bioanod TC-MGF (Conch shell) menggunakan membran N-115 yang mempunyai MFC aliran udara-katod (A-CU) MFC, sebagai gabungan aliran dan teknologi MFC digunakan untuk membandingkan kewujudan dan ketiadaan reka bentuk membran. A-CUMFC dengan membran yang kurang pada kadar aliran 0.6 mL min^{-1}, jarak anod 0.5 cm dan kepekatan AR27 pada 900 mg L^{-1}, kadar penyahairan yang tinggi (98%) dicapai dalam operasi 60 hari, adalah 40% lebih tinggi daripada membran-MFC. Ketumpatan kuasa maksimum purata yang diperoleh (1250 mWm^{-2}) dengan menggunakan MFC yang tidak membran adalah lebih tinggi daripada membran-MFC (1108 mWm^{-2}) dalam operasi 80 hari dengan bioanod TC-MGF.

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LIST OF ABBREVIATIONS

$A_{600\ nm}$	-	Absorbance at the wavelength of 600 nm
A1	-	*Citrobacter* sp. A1
Al^{3+}	-	Aluminum cation
AFM	-	Atomic Force Microscope
AR27	-	Acid Red 27
APHA	-	American Public Health Association
ATR-FTIR	-	Attenuated total reflection Fourier transform infrared
BET	-	Brunauer emmett and teller
BFC	-	Biological fuel cell
BS	-	British standard
C1	-	*Enterococuus* sp. C1
Ca	-	Calcium
Ca^{2+}	-	Calcium cation
CC	-	Carbon cloth
CE	-	Coulombic efficiency
CEC	-	Cation exchange capacity
CEM	-	Cation exchange membrane
CMC	-	Critical micelle concentration
COD	-	Chemical Oxygen Demand
CT	-	Contact time
CV	-	Cyclic Voltammetry
e	-	Electronic charge
EDX	-	Energy dispersive x-ray analysis
EPSs	-	Exopolysaccharides
FESEM	-	Field emission scanning electron microscopy
BGF	-	Bare Graphite felt
GC-MS	-	Gas Chromatogrphy-Mass Spectrophotometry
H^+	-	Hydrogen cation

H_2O - Water
H_2SO_4 - Sulphuric acid
HDTM-BR - Hexadecyltrimethylammonium bromide
HPLC - High Performance Liquid Chromatography
I - Current
K - Potassium
K^+ - Potassium cation
K_2HPO_4 - Dipotassium hydrogen phosphate
KH_2PO_4 - Potassium dihydrogen phosphate
$K_2Cr_2O_7$ - Potassium dichromate
KBr - Potassium bromide
L17 - *Enterobacter* sp. L17
MCM-41 - Mobile Composition of Matter No. 41
MFC - Microbial fuel cell
Mg - Magnesium
Mg^{+2} - Magnesium cation
NA - Nutrient agar
Na - Sodium
Na^+ - Sodium cation
NMR - Nuclear magnetic resonance
NO_3^- - Nitrate
O - Oxygen
OD - Optical Density
$(OH)^-$ - Hydroxide ion
P - Power
PBS - Phosphate buffer solution
PEM - Proton exchange membrane
Pt - Platinum
QAC - Quarternay Ammonium Cationic Surfactant
R_{int} - Internal resistance
Rpm - Revolution per minute
RP-1 - Real product-1
RP-2 - Real product-2
RT - Room temperature
S - Sulfur

Sec - Seconds
SCE - Saturated Calomel Electrode
Si - Silicon
SiO_2 - Silica
SSA - Specific surface area
S-TC - Surfactant-treated clinoptilolite
SMZ - Surfactant modified zeolite
TAA - Total Aromatic Amines
TC-MGF - Treated clinoptilolite-modified graphite felt
TNT - Total Nitrogen Test
UV-Vis - Ultraviolet Visible
XRD - X-ray diffraction
3-D - Three-dimensional

LIST OF SYMBOLS

°C - Centigrade

cm - centimetre

cfu mL^{-1} - Colony Forming Unit per millilitre

g L^{-1} - gram per litre

kΩ - Kiloohms

L - Litter

mg L^{-1} - milligram per Litre

mL - millilitre

mL min^{-1} - millilitre per minutes

mM - millimolar

M - Molar

MΩ - Megaohms

μL - Microliter

μm - Micrometre

N - Normalite

nm - nanometer

V - Voltage

ν - Volume of gas adsorbed per unit weight of anode at a pressure

ν_m - Volume of gas adsorbed for monolayer coverage

v/v - Volume per volume

w/v - Weight per volume

Ω - Ohm

θ - Critical angle of incidence of the x-ray beam on the crystal plane

λ - Wave-length

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CHAPTER 1

INTRODUCTION

1.1 Study Background

Recently, it has been observed that due to a massive increase in population and industrialisation, there has been a need for further knowledge regarding energy resources. Today, scientists are faced with the daunting task of discovering novel and innovative techniques of generating energy from alternative energy sources. To date, the most commonly used source of energy is fossil fuel, which is still able to support energy demands for the next 100 years (Daniel *et al.*, 2009). However, many reports have stated that fossil fuels, especially oil, natural gas, and coal, are being used at a very alarming rate. Additionally, it has also been observed that the use of fossil fuels has led to an increase in global warming and can cause significant variations in climatic conditions. This has resulted in an increasing demand for alternative and cleaner sources of renewable energy (He, 2007).

The demand for energy has tremendously increased over the last few years. Scientists have started focussing on waste streams, including industrial waste, agricultural waste, domestic and food processing wastes, as alternative energy resources and for a cleaner environment due to the presence of biodegradable wastes (He, 2007).

Azo dyes are one of the biggest and the most popular classes of synthetic dyes with wide applications in the paper, textile, cosmetics, food and pharmaceutical industries (Idel-aouad *et al*., 2011; Mendes *et al*., 2011; Jadhav *et al.*, 2013). It has been observed that every year azo dyes are produced approximately 7×10^5 metric tons, and approximately 5-10% of these dyes are released into the environment as waste (Rai *et al.*, 2005; Dafale *et al*., 2010). Azo dyes are very popular due to their cost-effective synthesis process, stability, and availability in many colours. Chemically, azo dyes consist of one or more azo bonds (-N=N-) associated with the aromatic structure in the molecule (Jadhav *et al.*, 2013; Jović *et al.*, 2013; Fang *et al.*, 2015).

Azo dyes itself is hazardous; however, it can be more hazardous when the azo bonds are reduced to give amines which are more carcinogenic than the parent structure (Zouari-Mechichi *et al.*, 2006; Mendes *et al*., 2011; Costa *et al.*, 2012). Hence, the incomplete treatment of azo dyes leads to the formation of aromatic amines as their breakdown products, which are carcinogenic in nature (Jonstrup *et al*., 2011). Azo dyes are used extensively in the textile industry, thereby resulting in massive water pollution because the azo dyes are resistant to degradation. One such example is Acid Red 27 (AR27), a naphthylamine sulfonic azo dye which is widely available in the form of atrisodium salt. It is also commonly known as C.I. 16185, C.I. Food Red 9, Amaranth, or Azorubin S (Hong *et al*., 2007).

The physicochemical methods used for the removal of dyes from wastewater containing azo dyes include coagulation, adsorption, membrane filtration, chemical oxidation, and ozonation (Selcuk, 2005; Dos Santos *et al.*, 2007; Alaton *et al*., 2002; Forgacs *et al.*, 2004). Many reports have stated that these methods result in massive amounts of sludge and lead to secondary pollution as they use a lot of chemicals. However, ozonation method would not produce solid waste or sludge as in the physical methods, but the energy consuming has turned this method to be less practical (Robinson *et al.*, 2001; Hong *et al.*, 2007; Saratale *et al.*, 2011; Dafale *et al*., 2010). Additionally, the physical and chemical methods used for the removal of azo dyes are very expensive and time-consuming (Sarkar *et al.*, 2011; Chengalroyen and Dabbs, 2013). Microbes play very important roles in bringing about the degradation of xenobiotic compounds. Hence, bioremediation using microbes (and their enzymatic reaction) was developed as an alternative and an environmentally-friendly technique for degrading azo dyes (Saratale *et al.*, 2011; Chengalroyen and Dabbs, 2013).

Biodegradation using a mixed microbial consortium is the most common technique for azo dye degradation. The anaerobic-aerobic treatment of wastewater containing azo dyes is an effective combination method for the biodegradation of azo dyes (Van der Zee and Villaverde, 2005; Jonstrup *et al.*, 2011). The anaerobic treatment procedure removes the colour of azo dyes; however, it also results in the formation of aromatic amines as the decolourisation products (McMullan *et al.*, 2001; Murali *et al.*, 2013; Pandey *et al.*, 2007). These aromatic amines are easily degraded using aerobic processes (Jonstrup *et al.*, 2011) through hydroxylation and ring-fission of the aromatic molecules (Supaka *et al.*, 2004). Earlier, a novel NAR-2 bacterial consortium was developed consisting of the *Citrobacter* sp. A1 (a bacterial strain previously isolated and characterised from the sewage oxidation pond at Universiti Teknologi Malaysia in Johor, Malaysia), *Enterobacter* sp. L17 strain (known as *Enterobacter cloacae* L17) and the *Enterococcus* sp. C1. These were the stock cultures available in the Nanomaterial Lab at UTM. These bacterial strains were given the acronyms A1 for the *Citrobacter* sp. A1, L17 for the *Enterobacter* sp. L17 and C1 for the *Enterococcus* sp. C1 (Chan *et al.*, 2012a). In their study, Chan *et al*. (2012a) reported on the decolourisation of Acid Red 27 using the NAR-2 bacterial consortium. They observed that the C1 culture was a more dominant decolouriser of the dye during the microaerophilic condition, whereas the A1 and the L17 cultures improved the resultant biotransformation of the dye intermediates during the deamination and desulphonation processes.

Microbial fuel cells (MFCs) are novel and ground-breaking technology that can help to reduce the dependence on fossil fuels for energy production. They have been successfully used for the production of energy from biological processes involved in wastewater treatment (Logan *et al*., 2006; Zuo *et al.,* 2007; Murali *et al.,* 2013; Fang *et al.,* 2015). MFCs use microbes as the biocatalyst. The electrogenic bacteria forming biofilm on the anode surface produce electrons and protons during organic matter degradation under anaerobic conditions. The electrons are transferred to the cathode through an external circuit. Meanwhile, the protons are moved to the cathode through the electrolyte and the separator. Eventually, the electrons and protons combine with oxygen to form water molecules to complete the reaction scheme (Song *et al.,* 2015). To maintain the electro-neutrality, the cations are transported from the anode to the cathode through a cation exchange membrane (CEM) (Logan *et al*., 2006; Chen *et al.,* 2010).

Although several earlier reports have observed that microbes are able to produce fuels such as methane, ethane, and even hydrogen, there are very few reports on electrogenic microorganisms with respect to their use in microbial fuel cells (MFCs). Some electrogenic bacteria reported earlier include *Aeromonas, Clostridium, Citrobacter, Geobacter, Klebsiella, Pseudomonas, Rhodoferax* and *Shewanella* (He, 2007; Humudat *et al.,* 2015).

In recent years, the azo dye decolourisation technique using MFC has become very interesting for biodegradation purposes. This system helps in a more continuous and flexible decolourisation process that can be used on an industrial scale. Many earlier reports have described the process of developing a mixed microbial consortium for degrading azo dyes by immobilising the culture on some appropriate support materials (Tony *et al*., 2009). Bacterial immobilisation on the support matrix becomes a useful technique used in bioreactor studies (Hrenović *et al*., 2005).

The main principle of the MFC revolves around the total number of electric charges which pass through the complete circuit every second. Hence, the electron transfer passage route should be as simple as possible, the distance between both electrodes has to be optimised to improve the mass transfer occurring between the electrodes (Cheng *et al.,* 2006b). Furthermore, the external resistance effect contributes greatly to the change in current density, which highlights the maximum probable power density. An earlier study reported that to produce a maximum power output when the internal resictance is equal to the external resistance of the MFC systems (Logan *et al.,* 2006).

Additionally, other studies have also noted that for MFCs, the proton exchange membranes (PEMs) play a vital role in separating the anodic and cathodic chambers.

The permeable membranes allow a smooth migration of the protons, produced at the anode, to the cathode. The different pore sizes of the PEMs are able to offer better stability, while the membranes themselves contribute the least internal resistance (Hou *et al.,* 2011a; Leong *et al.,* 2013).

When designing the MFC reactors, the performance of the anodes need to be taken into consideration. Several researchers have tested a wide variety of materials, and many configurations have also been developed in the past few years for improving the performance of MFCs (Liu and Logan, 2004). In recent years, many studies have focused on the use of carbon electrodes and carbon papers for developing MFC electrodes as they are cost-effective, non-corrosive and perfectly biocompatible (Wei *et al.,* 2011). On the other hand, these carbon materials have very low electrocatalytic movements for the anode microbial response and therefore, adjustment on the carbon electrode considered as main method for improving their activity (Park *et al.,* 2013).

In order to improve the efficiency of MFCs, investigation on modification of conventional carbon-based materials, such as graphite felt, carbon paper, and carbon cloth for the fabrication of the anode were conducted (Wang *et al.,* 2013a; Wang *et al.,* 2013b; Cheng and Logan 2007). Modification of anode may lead to a more efficient MFC; with specific properties of anodes, including biocompatibility, electronic conductivity, surface wettability and surface area were investigated (Park *et al.,* 2013; Chen *et al.,* 2013; Wei *et al.,* 2011). The anode property is strongly linked to the electrochemical reaction that affects the performance of the cell adhesion (Ginsburg and Karamanev 2007; Wu *et al*., 2015).

Additionally, changes to the surface of the anode are known to enhance the bacterial adhesion and, at the same time, to improve the electron transfer between the microbes and the surface of the electrode. Many researchers have implemented a variety of physical and chemical modification processes, such as ammonia treatment, acid oxidation, and electrochemical oxidation, for improving the transfer of electrons (Du *et al.,* 2007; Wei *et al.,* 2011). An earlier study indicated that enhancing the electron transfer through an anode biofilm would result in an improvement of power output in the MFCs (Xie *et al*., 2012; Fiset and Puig, 2015).

In their study, Park *et al.* (2013) conducted a magnetite/multiwall carbon nanotube (MWCNT) for coating anode, and it was observed that the reception of electrons by the anodic electrode was significantly enhanced by the use of *E. coli* biofilm in powered MFCs. Reports have stated that the anodic component and the arrangement can greatly affect the electron transmission, bacterial supplement, and substrate oxidation (Logan *et al.*, 2006; Lanas *et al.*, 2014). Recent studies have also revealed that positively-charged modifications and the use of natural surfaces, such as sand and quartz, improve the adhesion of the negatively-charged microbial culture due to the electrostatic force of attraction between the cells and the surface. Cheng and Logan (2007) studied the ammonium treatment and their results indicated that the positive charges on the carbon surfaces were greatly enhanced due to the ammonia gas which decreased the process and improved the power produced in the MFC with a microbial consortium. This approach proved to be effective in allowing the bacterial nanowire to adhere to the carbon cloth electrodes. In addition, Wu *et al.* (2015) observed that the use of different varieties of zeolite, namely NaX and Mobil Catalytic Material Number 41 (MCM-41), as a coating for the electrodes improves the performance of the MFC. This is due to an increase in the surface area in the range of 6.1-11 $m^2 g^{-1}$, which favours the formation of a thick biofilm.

1.2 Problem Statement

Over the years, it has become very important to find novel and cleaner energy sources. Nowadays, many industries around the world contribute to water and environmental pollution. Many reports have stated that the microbial fuel cell (MFC) system is a very good technique for energy production, while several researchers have suggested that the MFC is capable of bringing about the treatment of azo dyes from wastewater and simultaneously producing electricity (Li *et al.*, 2010; Hou *et al.*, 2011a). Despite the fact taht MFC being a novel technology for wastewater treatment and energy production, there are also some major drawbacks which limit the actual use of this technique, for example, the unsustainability of biocatalysts present on the anode, the configuration of the MFC, which limits the power output mainly during the scale-up, and the impractical batch-operation type of MFC systems as the microbes would easily wash out from the system. Therefore, the anode compartment needs a stable biofilm to be formed on a suitable and large anode surface. Furthermore, the characteristics of the anode need to be adapted to the biofilm and the used application in the MFC system.

Thus, the power output from MFCs must be improved by optimising the reactor configuration and operating conditions, while deploying proper anode materials with biocompatibility and large specific surface area. Nevertheless, no researche has demonstrated the use of the treated clinoptilolite-modified graphite felt (TC-MGF) anode as a support material for the adhesion of the NAR-2 bacterial consortium and the effect of TC-MGF on MFC performance. It is therefore important to fully understand the properties of this modified anode and its effect on the performance of the MFC system for simultaneously Acid Red 27 (AR27) azo dye decolourisation and electricity generation.

1.3 Objectives of this study

In view of the current understanding and limited research to date, this study was conducted to determine the effect of a modified anode involved in the immobilisation and performance of a developed MFC on dye degradation and electricity generation. Hence, the following objectives were established to achieve the aim of the research:

1. To charachterise treated clinoptilolite with surfactant hexadecyltrimethylammonium bromide (HDTMA-Br) followed by graphite felt anode modification using treated clinoptilolite (TC-MGF).
2. To determine the optimum conditions for the immobilisation of NAR-2 consortium onto TC-MGF anode using conventional method and charachterise the TC-MGF bioanoe.
3. To evaluate the effectiveness of TC-MGF bioanode for the generation of electricity from Acid Red 27 (AR27) dye in a dual-chamber (H-type)

MFC and its impact on the biodegraded products in comparison to graphite felt (BGF) bioanode.

4. To charachterise and evaluate the performances of the different Nafion membranes for the decolourisation of the AR27 dye and the generation of electric power using a single cube-chamber di-air cathode MFC with TC-MGF anode.
5. To assess the generation of electric power and decolourisation efficiency in a continuous upflow single column air cathode MFC system by a TC-MGF spiral design along membrane-less and membrane operations followed by analysis of the resultant degraded products.

1.4 Scope of the Study

This study has focused primarily on investigating the improvement of anode using modification technique to improve bacterial adhesion, that would then be incorporated in the different types of the MFCs system. Hence, the performance of the MFCs were evaluated at their optimised conditions.

The scope of this research was to study the modification effect of the anode surface on the performance of the MFC for simultaneous azo dye decolourisation of the Acid Red 27 (AR27) and electricity generation, using bacterial consortium NAR-2. An initial attempt was made using a dual-chamber MFC utilising modified anode, namely treated clinoptilolite-modified graphite felt (TC-MGF). This study introduced a modification process into the MFC design using an inert, inorganic and robust material known as surfactant-treated clinoptilolite (S-TC). The TC-MGF anode was then further improved by optimisation studies in a single-chamber air cathode MFC system with the evaluation of the physical properties, the decolourisation and power output performance of the variable Nafion membranes (N-115, N-117and N-212) in the MFC. Furthermore, in order to improve the MFC system for the biodegradation process and power generation, further modifications were made to the MFC design using a spiral anode with membrane-less operation with respect to the chemical analysis in a continuous upflow single column air cathode MFC reactor.

1.5 Significance of this Study

Earlier studies focused on the treatment of azo dyes using MFCs system for power generation. However, this study used a modified anode for immobilising the bacterial consortium, thereby improving the decolourisation and degradation of the model azo dye AR27, and also the resultant electricity production. The main idea behind this study was based on the ionic or hydrophobic interactions which take place between microbes and the modified anodes, based on the actual design. The anode was modified to be positively charged, so that it would then attract the negatively charged bacteria cell wall and the azo dye molecules. Hence, interaction between the dye molecules, nutrients and bacteria present would result in a desired decolourisation efficacy, especially if the system is continuous in operation. Therefore, the modified clinoptilolite anodes were installed to form an MFC system and were immobilised using the bacterial consortium, NAR-2. This system was used to carry out dye decolourisation and production of electricity. This was an initial attempt to construct an MFC system to generate electricity by decolourising the AR27 mono azo dye using the NAR-2 bacterial consortium. In several earlier studies, it was noted that when the bacteria were immobilised, they were able to be more resistant to the shock loads of the dye compared to the suspended bacterial culture (Ab.llah, 2012). The use of an upflow continuous MFC system along TC-MGF spiral anode (Conch Shell) with membrane-less design, as reported in this study, was demonstrated to be very significant and noteworthy as it brought more effective degradation of the azo dye within improved power generation and therefore, can be applied in the future investigation for the treatment of real wastewater containing different types of azo dyes for the generation of bioelectricity.

1.6 Thesis Organisation

The entire thesis consists of six chapters. Chapter 1 presents a concise introduction to the role of the MFC system in the degradation of recalcitrant chemical pollutants and also in the production of electric power using an active biofilm that is formed on a modified anode. Moreover, the chapter also exploits the use of the modified anode as a site for the adhesion of bacterial colonies. Furthermore, the chapter covers the problem statement, and the objectives, scope and significance of the study.

In Chapter 2, the basics of the modified anodes and the assessment of their role in the bacterial and anode interactions will be explained. The chapter also discusses the various anodic procedures and the MFC design, and also hypothesises about the reaction format mechanism. Furthermore, the chapter also includes earlier researches into the performance of the MFC system on the degradation of pollutants along with the subsequent power generation. Thereafter, the chapter presents the research framework, depending on the current understanding of the available studies.

In Chapter 3, an in-depth analysis of the methods used, along with the research methodology which would be applied in the study, is presented. Furthermore, some laboratory experiments were conducted to determine the performance of the MFC based on the electricity calculations. Also, the chapter discusses the characterisation studies which were carried out using the modified anodes with the help of various microscopic and spectroscopic techniques along with optimisation (conventional method) studies. All the results of these experiments are presented and discussed in further detail in Chapters 4 and 5.

Chapter 4 focuses on the fabrication and the behaviour of the modified anodes in immobilising the bacterial consortium and performance on MFC. In Chapter 5, the optimisation parameters and the evaluation of the physical properties, the decolourisation and power output performance of the variable Nafion membranes in the MFC are clarified. The optimised parameters were then developed into continuous MFC systems with/without a membrane for carrying out biodegradation and electricity production. One of these systems was subsequently further explored for lower degradation sensitivity.

Lastly, Chapter 6 revolves around the conclusion of the whole study and highlights the major contributions of this work. Furthermore, the chapter also presents some recommendations for future studies.

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