CORROSION PREVENTION THROUGH PERFORMANCE OPTIMIZATION OF BIOLOGICAL CATHODIC PROTECTION SYSTEM

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DEDICATION

Specially dedicated to my beloved parent

(Azman Bin Ali and Che Ramlah Binti Yusof)

who gave me inspiration, encouragement, motivation and valuable support towards the success of this study

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ABSTRACT

Unprotected pipelines, whether exposed to the air, submerged in seawater or buried underground are susceptible to corrosion and this phenomenon has been identified as one of the major mechanism that affects failures in oil and gas pipelines. At present, impressed current cathodic protection (ICCP) system is one of the methods used to control the corrosion of the steel pipeline. In this study, a new system was developed based on ICCP namely biological cathodic protection (CP) system. This system used the concept of microbial fuel cells (MFCs) to generate a protective current to the cathode (pipe). Little to know on the effect of both hydraulic retention time (HRT) and electrode surface area to anode-compartment volume ratio (ESAVR) simultaneously on the voltage output particularly for biological CP system. Therefore, this study analysed the optimum value of HRT and ESAVR of the biological CP system in generating adequate protective current for the carbon steel pipe with respect to copper/copper sulfate ($Cu/CuSO_4$) reference electrode which is -850 mV. Optimization of these two parameters on the biological CP system was done using response surface methodology based on central composite design (CCD) approach. CCD predicted values and experimental results showed a strong agreement, with an R^2 value of 0.7953. The estimated optimal conditions for HRT and ESAVR of anode were at 9.21 h and 0.239 cm^2/cm^3 respectively. Consequently, the polarization study was conduted to determine the main limiting factors that occurred in the system. It was found that the main losses that occurred was due to the concentration losses whilst the maximum power point recorded was 1.1908 mW/cm^2 (Run 11). The methodology supporting the purpose of this study by providing protective current to carbon steel pipe and thus verified the ability of this system to protect the pipe from corrosion.

ABSTRAK

Saluran paip yang tidak dilindungi, sama ada terdedah kepada udara, tenggelam di dalam laut atau tertanam di dalam tanah adalah terdedah terhadap kakisan dan fenomena ini telah dikenal pasti sebagai salah satu mekanisme utama yang menyebabkan kegagalan dalam saluran paip minyak dan gas. Pada masa ini, sistem perlindungan katod arus bekasan (ICCP) adalah salah satu kaedah yang digunakan untuk mengawal kakisan pada saluran paip keluli. Dalam kajian ini, satu sistem baharu dicipta berdasarkan ICCP iaitu sistem perlindungan katod secara biologi (CP). Sistem ini menggunakan konsep sel bahan api mikrob untuk menghasilkan arus pelindung ke katod (paip). Secara amnya, pengetahuan mengenai kesan kedua-dua nilai masa pengekalan hidraulik (HRT) dan luas permukaan elektrod kepada nisbah isipadu anod (ESAVR) secara serentak pada pengeluaran tenaga terutamanya untuk sistem CP biologi adalah terhad. Oleh itu, kajian ini menganalisis nilai optimum HRT dan ESAVR dalam menghasilkan arus pelindung yang cukup bagi paip keluli karbon berdasarkan elektrod rujukan tembaga / tembaga sulfat (Cu / $CuSO_4$) iaitu -850 mV. Pengoptimuman kedua-dua parameter sistem ini dilakukan dengan menggunakan kaedah sambutan permukaan berdasarkan pendekatan reka bentuk komposit pusat (CCD). Nilai ramalan CCD dan hasil eksperimen menunjukkan kolerasi yang kuat, dengan nilai R^2 bersamaan 0.7953. Anggaran optimum bagi HRT dan ESAVR anod masing-masing pada 9.21 jam dan 0.239 cm^2/cm^3. Seterusnya, kajian polarasi dilakukan untuk menentukan factor pembatas utama yang berlaku dalam sistem. Kehilangan utama yang berlaku adalah dari kehilangan kepekatan manakala titik maksima kuasa yang direkodkan adalah 1.1908 mW/cm^2 (Ujian 11). Metodologi menyokong tujuan kajian ini untuk menyediakan arus pelindung kepada paip keluli karbon dan dengan demikian mengesahkan kemampuan sistem ini untuk melindungi paip daripada kakisan.

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LIST OF ABBREVIATIONS

CCD	-	Central Composite Design
CCI	-	Central Composite Inscribed
CE	-	Coulombic Efficiency
COD	-	Chemical Oxygen Demand
CP	-	Cathodic Protection
CR	-	Corrosion Rate
ESAVR	-	Electrode Surface Area / Anode-Compartment Volume Ratio
HRT	-	Hydraulic Retention Time
ICCP	-	Impressed Current Cathodic Protection
MLSS	-	Mixed Liquor Suspended Solid
MPP		Maximum Power Point
OLR	-	Organic Loading Rates
RSM	-	Response Surface Methodology

LIST OF SYMBOLS

$C_2H_3NaO_2$	-	Sodium acetate
$CuSO_4$	-	Copper sulphate
h	-	hours
HCl	-	Hydrochloric acid
HNO_3	-	Nitric acid
KCl	-	Potasium chloride
NaCl	-	Sodium chloride
R_i	-	Internal resistance
Sb_2O_3	-	Antimony trioxide
$SnCl_2$	-	Stannous chloride
Ω	-	Ohms

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CHAPTER 1

INTRODUCTION

1.1 Research Background

According to National Association of Corrosion Engineers (NACE) International, corrosion is defined as the deterioration of a substance or its properties because of a reaction with its environment (Norsworthy, 2014). Popoola *et al.* (2013) stated that pipelines corrosion has been identified as one of the major mechanism that gives rise to failures in oil and gas pipelines. The consequences of pipelines corrosion leakages will result in the environmental impact, contamination, and shutdowns of the plant (Ngobiri *et al.*, 2015). The problem of corrosion is worth to be investigated because every year, this problem contributes to a high percentage of total costs for oil and gas producing companies worldwide (Finšgar and Jackson, 2014). In placing more emphasis, Ossai (2012) stated that, from 1980 until 2006, 50% of the European major hazards of loss arising from technical plants failures are due to ageing of plants mechanism caused by corrosion, erosion and fatigue. As the consequences, pipelines corrosion leakages can result in the environmental impact, contamination, and shutdowns of the plant. Besides that, contamination of products with residual rust leads to low quality production and finally loss of business reputation.

According to Paul *et al.* (2015), carbon steels are the most common materials used in the transportation of oil and gas from one region to another due to its cost-effectiveness and applications. However, the corrosion of carbon steel pipe still occurred due to contact with the environment. All metallic structures will tend to corrode when in contact with water, concrete, soil and moist air. Coatings in pipelines usually are not perfect and may have defects such as scratch of the coatings, pinholes, and flaws. Chemical species, groundwater, and corrosive gases may pass through the defects and reach the pipe surface. This phenomenon will cause coating disbonding and eventually pipeline corrosion at the defected area.

Therefore, to overcome this problem, all buried gas pipelines applied the concept of cathodic protection (CP) system (Ashworth, 2010; Brenna *et al.*, 2017; Kuang and Cheng, 2015). This system reduces the corrosion of the metal surface by constructing an electrochemical cell and making the surface of metal as cathode. There are two types of CP systems which are sacrificial anode CP system and impressed current cathodic protection (ICCP) system. The concept of sacrificial anode is the driving voltage is provided by the natural potential difference that occurs between the structure and a second metal in the surrounding without the need of power source. In contrast to the concept of sacrificial anode, in ICCP method, the driving voltage for protective current is supplied from a power source. The power supply is usually consist of a rectifier that converts alternating current (AC) to direct current (DC). In order to ensure adequate protection for any structure, the polarized potential of the protected structure should be measured and compared to a certain criterion. For example, the criteria for carbon steel pipe is -850mV with respect to copper/copper sulfate ($Cu/CuSO_4$) reference electrode and the cathodic protection criterion differs according to the type of metals and the soil resistivity. Despite the fact that ICCP system can be applied to a wide range of structures, it can affect other structures if it is not properly monitored (A.S. Hamdy Makhlouf and M. Aliofkhazrael, 2002). Other than that, installation of ICCP system required an external power source and have a high maintenance cost, including the mechanical maintenance of power supply and equipment (Byrne *et al.*, 2016).

Therefore, this study introduced a new approach by using the concept of microbial fuel cells (MFCs) to represent a new protection method namely biological CP system. MFCs are a sustainable energy technology which microorganisms present in the wastewater work as a catalyst to convert chemical energy into electrical (Oliveira *et al.*, 2013; Ortiz-Martínez *et al.*, 2015; Zhou *et al.*, 2011). Electricity is generated by MFCs as the microbes in the anode compartment consume substrates and thus generating electron and protons. The transfer of electrons from anode (negative terminal) to the cathode (positive terminal) through the external circuit will produce electricity. Thus, the capability of the microorganism to generate electricity can be used as a new approach for biological CP to protect the pipelines.

However, the electricity production of the system undeniably depends on the growth of microorganism. According to Al-Qadiri *et al.* (2008), the growth of microorganisms can be divided into four phases which are lag phase, exponential phase, stationary phase and death phase. During the lag phase, the microbes will adapt themselves to the growth environments. The next phase is followed by exponential growth where the microbes will grow rapidly and divide at a maximal rate possible. The next phase is the stationary phase where the growth rate of the microbes is limited due to the limiting factors such as the depletion of nutrients. Throughout this phase, the growth rate of the microbes also equals the death rate. Lastly, the population of microbes will decline in population during the death phase. Therefore, to maintain the microbial population in the wastewater, this study was constructed in continuous-mode as the microorganisms were continuously fed and removed from the system.

In the last few years, MFCs technology has been improved significantly. There are many factors that can influence the performance of this system. These measures may include different types of electrode (Zhou *et al.*, 2011), different substrate used in the system (Pandey *et al.*, 2016; Pant *et al.*, 2010), various types of separator materials used (Kim *et al.*, 2007; Li *et al.*, 2011), as well as different MFCs design and configurations (Das and Mangwani, 2010; Oliveira *et al.*, 2013; Walter *et al.*, 2016). Different design and configurations of MFCs are subject to some important considerations such as hydraulic retention time (HRT) and electrode surface area to anode-compartment volume ratio (ESAVR) of the system.

According to Akman *et al.* (2013), hydraulic retention time (HRT) is an important parameter as it can affect the design, investment cost and energy requirement. Generally, high capital cost is required for a system with higher HRT and varying HRT also has a major effect on the power generation of MFC. Regarding the ESAVR, it has been reported that large-volume units of MFCs tend to have lower power densities and hence decreased efficiency (Walter *et al.*, 2016). A study by Ieropoulos *et al.* (2008) also proved that a smaller MFCs have a greater power density compared to large vessels because of their higher surface area to volume (SA:V) ratios.

Therefore, by utilizing the capabilities of microorganisms to generate electricity as well as the energy recovery benefits from MFCs system, biological CP offers an attractive solution in protecting the corrosion of gas pipelines. Thus, to obtain the right configurations of the system, this research investigated the optimum HRT and ESAVR by using RSM to achieve higher power density output of biological CP system. Besides, the corrosion rate of the protected pipe and reference pipe were analysed to measure the abilities of the protective current provided by the system to protect the system.

1.2 Problem Statement

One of the primary problems that resulting in failures of oil and gas pipelines is the phenomenon of corrosion at the underground steel pipeline. This problem resulted from an electrochemical reaction that takes place around the pipeline (Kuang and Cheng, 2015). CP is the most important method of all approaches to control the corrosion. CP can be achieved in two methods which are sacrificial anode CP system and ICCP system (Ashworth, 2010). However, installation of sacrificial anode has its own drawbacks such as unable to control the current and shorter lifespan. Besides that, ICCP method also has its own disadvantages. ICCP method required the installation of external power and monitoring systems and these systems are vulnerable to damage and atmospheric corrosion. This system also is prone to failure of switches, fuses and therefore it has a high maintenance cost (Byrne *et al.*, 2016).

Recently, great attention have been paid to MFCs due to their ability to produce electricity directly from the degradation of organic matters (Rahimnejad *et al.*, 2015). However, there has been no previous work describing the utilization of MFCs to generate the protective current in CP system. In this study, a new approach was developed to protect the gas pipeline namely biological CP system by utilizing the concept of MFC to generate the electricity. Still, the power generation of the MFC system fluctuated and varied depending on the configurations of the system including the HRT and ESAVR.

It was reported that high HRT of the system may not provide enough substrate for the microorganism to sustain longer while at low HRT, the biofilm may not build completely thus resulting in low power generation (Haavisto *et al.*, 2017). Besides that, recent studies have demonstrated that increasing ESAVR improved the power generation of the system (Penteado *et al.*, 2018; Walter *et al.*, 2016). Nevertheless, previous study that considers both HRT and ESAVR simultaneously is less reported particularly for biological CP system. Therefore, the right HRT and ESAVR of the system need to be studied to achieve optimal voltage output for cathodic protection method. Therefore, to study these two parameters, the experimental run was designed by using the central composite design by using Design Expert software to determine the optimal condition for high voltage output. Besides, the main limiting factors of the system also was determined by polarization study.

1.3 Research Objectives

The main objective of this study is to investigate the optimum HRT and ESAVR of the biological CP system to protect carbon steel pipe from corrosion. The sub-objectives of this study are as follows:

1. To optimize the hydraulic retention time (HRT) and electrode surface area to anode-compartment volume ratio (ESAVR) of the biological CP system by using RSM via central composite design (CCD)

2. To analyse the maximum power point and limiting factors of the system by polarization study

1.4 Research Scopes

In order to achieve the objectives of this research, the following scopes have been identified:

1. Preparing and characterizing of wastewater collected from Indah Water Gravity Thickener Pond at Taman Bukit Senang.

2. The experimental rig was fabricated and constructed with local materials based on two compartments which were anode and cathode compartment. The anode compartment was constructed with a dimension of 32.0 cm × 20.5 cm × 10.0 cm (length × width × height) and a graphite rod was used as the anode electrode. Carbon steel pipe (schedule 40 pipe dimension) was selected as the cathode electrode and salt bridge was used to connect the anode and cathode compartment. Pumps were used to feed the wastewater and substrate continuously into the experimental rig (anode compartment) through plastic tubing.

3. Determination of optimum power density output of the system via Central Composite Design (CCD) in response surface methodology (RSM) based on two independent variables (inlet flowrate of wastewater and length of graphite anode). The inlet flowrate of wastewater was varying between 2.0 – 10.0 mL/min (HRT 9.11 h -27.33 h) while the length of graphite electrode was between 6.0 cm to 15.0 cm (ESAVR of 1005.31 – 1570.80 cm^2/cm^3).

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