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## Optimization of the performance of a double-chamber microbial fuel cell through factorial design of experiments and response surface methodology



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#### HIGHLIGHTS

- Design of experiment was used to optimize performance of MFCs.
- The optimal levels of pH and buffer concentration of catholyte were assessed.
- The joint effect between the factors was highly significant in the responses.
- Buffer concentration affected the responses differently at different pH levels.
- Lower optimum level of buffer concentration made the process more economic.

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#### ABSTRACT

Although microbial fuel cells are potential candidates for electricity generation with concomitant treatment of wastewater, operational and technological improvements are still required to make them a cost effective process. In this paper, a full factorial design in combination with a central composite design were employed to probe the effects of pH and buffer concentration of catholyte on the performance of a two chamber microbial fuel cell. It was observed that at high level of buffer concentration (150 mM) varying the pH between 5.8 and 7.4 did not markedly influence the maximum power density and columbic efficiency. However, at a low level of buffer concentration (25 mM) reducing the pH led to higher power density. Furthermore, it was observed that higher concentrations of buffer were beneficial to power generation when pH was at its high level, but negatively impacted the cell performance at low pH levels. The highest maximum power was predicted to be 461 mW m<sup>-2</sup> (at a pH of 6.3 and a buffer concentration of 82 mM) and this was confirmed by experimental results. The findings of this paper highlight the importance of the joint effect of pH and buffer concentration, which is even more influential than their main individual effects.

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#### 1. Introduction

Although a large amount of research have been conducted on microbial fuel cell (MFC) technology [1–3], MFC's application in commercial scales is limited due to some bottlenecks in the process. Cathode and electrolyte resistances are evaluated to be the major factors limiting electricity generation by MFCs [4–6]. Buffer is commonly used to serve as the main component of

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the catholyte and believed to affect the performance of cathode, directly [7]. Buffer has different functions in the system, including establishment of an appropriate environment with suitable pH for growth of exoelectrogens as well as increasing the conductivity of the solution [8]. In addition, it plays an important role in providing sufficient protons that would be required in oxidation reduction reaction (ORR) in the cathode chamber of MFCs [9]. Besides, the characteristics of the buffer solution significantly influence oxygen reduction kinetics on a catalyst coated cathode [10]. Finally, pH of anode and cathode chambers are shifted unfavorably without using a buffer, resulting in a considerable drop in power output [11]. Therefore, it is not surprising that many studies have focused on composition and



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concentration of the cathodic solution to improve the performance of MFCs [12–14].

Increasing the power of an MFC by using a higher concentrations of buffer has been studied [6]. From a practical point of view, however, using a higher concentration of buffer is not cost effective [15], particularly when MFCs are used in wastewater treatment. In many studies buffer solutions with different pH have been applied in anode and cathode compartments to determine the effect of pH on the performance of the MFC [8,14,16]. For instance, a neutral buffer as well as an alkaline buffer (with a pH around 10) in the anode and an acidic buffer (with a pH around 2) in the cathode have been proposed [9,17].

The aim of this work is to apply a full factorial design and response surface methodology to study the effect of pH and buffer concentration on power output, columbic efficiency, COD change, and COD removal efficiency of a double-chamber MFC. It was postulated that different or even conflicting findings concerning the optimized levels of these two factors in published reports resulted from the deficiency of one-variable-at-a-time approach, which is not able to identify the possible interactions among the factors.

Optimizing the parameters involved in the cathodic chamber of an MFC can lower the cost of operation and simultaneously improve the performance of the MFC. Statistical optimization techniques can be employed to search within a wide experimental domain with a minimum number of runs and can also provide the contribution of each factor and its share on the different responses [18]. In addition, the joint effect between different variables can be evaluated, simultaneously. Finally, the effect of a specific factor can be assessed at different levels of the other factors, therefore the conclusions are reliable over the entire experimental space [19]. Factorial design (FD) and central composite design (CCD) are powerful statistical methods that have been widely used to determine the effect of several parameters on a response, simultaneously. These methods have been successfully applied to different areas such as biotechnology [20], analytical chemistry [21], nanotechnology [22,23], and petroleum industries [24]. To the best knowledge of the authors, this is the first time that such approaches are been used to study the concurrent effect of several parameters in a double-chamber MFC.

#### 2. Materials and methods

#### 2.1. MFC setup

Two-chamber MFCs were made from an acrylic cylinder with the outside diameter of 100 mm that bored through to form a 70-mm-inside diameter compartment. The total volume of each compartment was 150 ml with an approximate working volume of 135 ml. In order to fill, drain, aerate the cathode, and sparge the anode chamber with nitrogen three openings were considered on the top of each compartment. The unused holes of the anode chamber were firmly sealed to prevent oxygen diffusion to the anodic chamber. The anodic and cathodic sections were separated via a 36 cm<sup>2</sup> cation exchange membrane (Ultrex CMI7000, Membranes Inter-national Inc., USA).

The electrodes were made of carbon felts (Panex35, Zoltek, USA). The diameters of anode and cathode were 3 cm and 2 cm, respectively. Prior to use, the electrodes were immersed in acetone for 24 h and then in distilled water overnight. For the cathodes, a commercial Pt catalyst (10 wt% Pt/C, E-TEK) was mixed with 2% PTFE suspension and used as a chemical binder (7  $\mu$ l of binder per mg of Pt/C catalyst) to form a paste with final content of 0.5 mg cm<sup>-1</sup> [25]. The paste was applied to the entire surface of cathode electrode and dried at 350 °C in a furnace for 30 min. Each electrode was firmly connected to a stainless steel wire (1.5 mm in

diameter) using carbon fiber (Panex35 Continuous Tow, 50 K, Zoltek, USA). The exposed surface of the wire was isolated from the solution using epoxy and heat shrink tubings.

#### 2.2. Inoculation and operating conditions

The anodic chamber of the cells were initially fed with 350 mg sodium acetate, 1.75 ml metal salt solution [26], 60 ml wastewater, 5 ml sedimented sludge, and 65 ml phosphate buffer solution (50 mM: K<sub>2</sub>HPO<sub>4</sub>, 5.4 g L<sup>-1</sup> and KH<sub>2</sub>PO<sub>4</sub>, 2.6 g L<sup>-1</sup>; pH 7). After stabilization of the cells the amount of inoculum was gradually reduced to zero level during few cycles. The procedure was kept identical for all experiments. The waste source (i.e., sedimented sludge and wastewater) was collected from a dairy wastewater treatment unit. The collected wastes were stored in plastic bottles at 4 °C before use. The anolyte was replaced when the power dropped below 50 mW m<sup>-2</sup>. Nitrogen gas (>99%) was sparged continuously for 20 min after each inoculation to maintain anaerobic conditions in the anode chamber. The catholyte solution was phosphate buffered with different buffer concentrations and pH levels according to the experimental designs (see Table 1). The chemicals were purchased from Merck Company. The experiments were conducted in a temperature controlled chamber at 30 ± 1 °C.

#### 2.3. Data acquisition and measurements

A computer program was developed in the Lab VIEW environment (version 10, National Instrument) to continuously measure and record cell voltage and current. A data acquisition card (ADC10016, TNM Electronics) was used as an analog-digital converter to provide an interface between the cell electrodes and the computer. The OCV of a cell was measured by leaving the circuit in open mode until reaching a plateau in voltage. Polarization curves were obtained only after adoption of the MFCs to the applied experimental conditions according to each run (at least after three complete operating cycles) by varying the external resistance systematically in the range of 50,000–100  $\Omega$ . The data was taken only when quasi steady-state conditions were observed (about 20 min after changing the external load) [27]. Power and current were normalized by the projected surface area of the cathode. COD measurements were performed using the closed reflux method [28]. Columbic efficiency was determined as follows [29]:

$$\% CE = \frac{8 \int_0^{t_b} I \, dt}{F \, \nu_{Anode} \Delta COD} \tag{1}$$

where CE is columbic efficiency, *F* is Faraday constant,  $v_{Anode}$  is the working volume of the anode compartment,  $\Delta$ COD is the change in COD over time  $t_b$ , and *I* is the current.

Table 1	
Level of independent variables in FFD and CCI	).

Design	Variable	Factor	Applied levels				
			Star- Low	_ (Low)	Center	+ (High)	Star- High
FFD	$X_1$	pH of buffer	-	5.80	6.60	7.40	-
	<i>X</i> <sub>2</sub>	Buffer concentration (mM)	-	25.0	87.5	150.0	-
CCD	$X_1$	pH of buffer	5.55	-	6.60	-	7.73
	<i>X</i> <sub>2</sub>	Buffer concentration (mM)	2.0	-	87.5	-	176.0

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