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# Development of Optimum Operating Parameters for Bioelectricity Generation from Sugar Wastewater Using Response Surface Methodology

M. O. Aremu<sup>1</sup>, E. O. Oke<sup>1</sup>, A. O. Arinkoola<sup>1</sup> and K. K. Salam<sup>1\*</sup>

<sup>1</sup>Department of Chemical Engineering, P.M.B. 4000, Ladoke Akintola University of Technology (LAUTECH), Ogbomoso, Oyo State, Nigeria.

## Authors' contributions

*This work was carried out in collaboration among all authors. Author AOA conceived the modeling aspect of this work. Author MOA conducted the experiment which was used for modeling. Author EOO wrote the review, designed the experiment and generated the model while author KKS analyzed and discussed the results. Conclusion was written by all the authors. All authors read and approved the final manuscript.*

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## ABSTRACT

Two response surface methodologies involving historical data designs have been successfully developed with the aim of predicting optimum operating parameters for bioelectricity generation from sugar wastewater. The regression models evaluated the effect of waste water concentration, experimental process time and agitation speed on bioelectric current generation from microbial fuel cell. In the first historical data design, regression model was developed for the investigation of the effect of waste water concentration and experimental process time on the response (bioelectric current). Second historical data design was performed to determine the extent to which agitation speed and process time influence the response. The polynomial regression models were validated with statistical tool whose values of  $R^2$  for first and second model were 0.95 and 0.98 respectively. The optimum combination of wastewater concentration and process time for the maximum generation of bioelectric current from second model in numerical optimization of response surface methodology are 246.7 and 6 minutes with 1.94 mA electric current. Optimum conditions generated were process time of 40.14 minutes and

\*Corresponding author: E-mail: [kaykaysalam@yahoo.co.uk](mailto:kaykaysalam@yahoo.co.uk);

agitation speed of 39.7 rpm which generated bioelectric current of 1.633 mA. The optimum operating conditions developed could be used to enhance commercialization of bioelectricity generation from sugar wastewater.

*Keywords: Historical data design; sugar wastewater; bioelectricity; microbial fuel cell.*

## 1. INTRODUCTION

Waste water is any water that has been adversely affected in quality by anthropogenic influence; it comprises liquid waste discharge by domestic residences, commercial activities, industry or agriculture effluents encompass a wide range of potential contaminants in different concentrations. Wastewater discharged from various industries has been a major source of pollution in water and land environment. Effluent from sugar industries is municipal waste water that contains a broad spectrum of contaminant resulting from the mixing of water from different sources during the production processes [1].

The goal of process/environmental engineers is ensuring continuity of human existence by controlling hazards posed by the pollutant and seeking a means of making worth out of waste. Wastewater can be re-used by treating the water to produce de-ionized water which can be used for drinking and other activities. Not only treating the water but it can be used as a source of electrical energy generation using microbial fuel device (MFC) [2]. One of the greatest advantages of MFCs over conventional fuel like hydrogen and methanol fuel is that a diverse range of organic material can be used as fuel. Microbial Fuel Cell (MFC) is a biochemical-catalyzed system, which generates electricity as a by-product by oxidizing biodegradable organic matter in the presence of either fermentative bacteria or enzymes [3]. A typical MFC consists of anode and cathode separated by cation specific membrane otherwise known as cation exchange membrane (CEM) or proton exchange membrane (PEM). Microbes in the anode oxidize fuel gain energy for metabolism by transferring electron from an electron donor such as glucose or acetate to an electron acceptor and the resulting electron are transferred to cathode through the circuit and the membrane respectively.

Microbial fuel cells convert chemical energy to electrical energy through catalytic activities of microorganism [4]. Electricity has been generated in MFCs from various wastewater sources and effectiveness of the process is determined using chemical oxygen demand (COD) removal efficiency as well as coulomb efficiency [5]. Traditionally, waste water from processing plants has been treated using aerobic sequencing batch reactor and up flow anaerobic sludge batch reactor [4]. These methods require a high energy input and are thus costly, new approaches for waste water treatment which not only reduce cost but also produce useful side-products have recently received increasing attention. The microbial fuel cell (MFC) technology offers a valuable alternative to energy generation as well as waste water treatment.

In the past years, bio-electricity has been generated using various types of wastewater. Mohan et al. [6] subjected mixture of chemical wastewater aggregated from bulk drugs, chemical intermediates, dye and dye intermediates, pharmaceuticals, pesticides and various chemical processes for bioelectricity production and treatment of wastewater. The performance of MFC (mediatorless anode) with respect to electricity generation and substrate removal efficiency from chemical wastewater treatment was evaluated at two

different organic loading rates (OLR). It was concluded that performance and stabilization tendency with respect to power generation was found to be dependent on the applied substrate loading rate and a pH of 5.5 to sustain the activity of acidogenic bacteria along with inhibiting methanogenic bacterial activity.

In their contribution, Patil et al. [5] compared water treatment and electricity generated from three different effluents; Dairy, Sugar and Paper wastewater using inoculum *Enterobacter aerogenes* NCIM 2340 with Mediator Neutral red and Toluidine blue. The result of the investigation after 40 hours showed that sugar wastewater have the highest Power density of 16.347 mW/m<sup>2</sup>, followed by 34.274 mW/m<sup>2</sup> and 42.646 mW/m<sup>2</sup> for Dairy and Paper Wastewater using toluidine blue. Paper wastewater was more efficient in the generation of electricity compared to Sugar and Dairy wastewater and in presence of mediator more electricity was generated with toluidine blue when compared to that of neutral red.

Utilization of agro-industrial wastewater collected from cassava mills as raw material for electricity generation was analyzed in the work of Kaewkannetra et al. [7]. Mixed culture sludge was used to inoculate the bottom chamber of the MFCs while cassava mill wastewater was used in the MFCs. The investigation reported COD removal efficiencies of approximately 28% in the MFC system when compared to control experiment. It was observed that variation in cyanide concentration had no effect on the power generation capability of an MFC, thus it was able to achieve a maximum power density of 1.8 W/m<sup>2</sup>. The result of COD and coulomb efficiency in this study was compared with results of seven published experimental investigations and it was discovered that current work has superior performance when compared with the published work. Catalyst was introduced into mediator-less microbial fuel cells (CAML-MFC) for Electricity generation from food processing wastewater at the industrial estates in Zahedan, Iran. The results of this investigation indicated that catalysts and mediator-less microbial fuel cells (CAML-MFC) can be considered as a better choice for simple and complete energy conversion from wastewater [8].

Baranitharan et al. [9] investigated feasibility of bioelectricity generation from Palm Oil Mill Effluent (POME) in Microbial Fuel Cell (MFC) Using Polacrylonitrile Carbon Felt as Electrode. POME was treated using double chamber microbial fuel cell with simultaneous generation of electricity. Polyacrylonitrile carbon felt (PACF), a new electrode material was used as electrode throughout the MFC experiments. Various dilutions of raw POME were used to study the effect of initial chemical oxygen demand (COD) on MFC power generation, COD removal efficiency and coulomb efficiency in the presence of anaerobic sludge used as inoculum for all the MFC experiments. The results showed that anaerobic sludge enhanced the power production due to better utilization of substrates by various types of microorganisms present. MFC possesses great potential for the simultaneous treatment of POME and power generation using PACF as electrode because initial COD has great influence on coulomb efficiency, COD removal efficiency and power generation.

In a recent work, a model was presented for possibility of controlling temperature and pH of MFC to regulate electricity generated. MFC was inoculated with a mixed consortium while the temperature and pH was varied from 10 to 30°C and 5.0 to 7.0 respectively. Boolean logic operations was employed to logically designed for sharp thermal and pH response of the microbial anode of the MFC and the result of the work showed that bioanode could switch between electrochemically active and inactive states in response to the operation parameters resulting in the bioelectricity generation from the assembled MFC [10].

In this study, sugar waste water treatment using sequential anode-cathode MFC in which the effluent of anode chamber was used as a continuous feed for aerated cathode chamber was studied. Historical data design in Response Surface Methodology (RSM) was adopted in the design of experimental combinations of factors. This study therefore aim at the development of optimum operating parameters for bioelectricity generation from sugar waste water considering the effect of process time, waste water concentration and agitation speed on the quantity of electricity generated from sugar wastewater.

## **2. MATERIALS AND METHODS**

### **2.1 Material**

The materials needed for construction of MFC for generating electricity from sugar waste water are: Two plastic containers, Graphite plate (anode), Graphite plate (cathode), Salt bridge (PEM), Multimeter, Clamps, Cover, Sugar waste water and Gum.

### **2.2 Experimental Design**

The microbial fuel cell result reported by earlier publication was used for this study [11]. The input parameters are process time ranging from 0 to 300 minutes and concentration of sugar waste water varied between 50 and 100 percent. The response of the experiment was quantity of electricity generated. The current (I) and potential (V) measurements were recorded at 30 minutes interval of operation using auto-range digital Multimeter (made by Kusam, model DT-830D). Historical data design in response surface methodology (RSM) was used to analyze the interactions among the independent variables and further optimize operating parameters of bioelectricity generation from sugar waste water.

Two experimental designs were formulated namely; the first experimental historical data design (HDD) which was carried out to identify the functional relationship between process time, waste water concentration and current generated (response) during the process; then, optimize bioelectricity generation from the process. The second HDD was also performed to statistically identify the dependency of current generated from the process on process time and agitation speed. Then, the optimum factors for each HDD were investigated in numerical optimization of response surface methodology. Statistical software package embedded in Design Expert 6.08 was used for the experimental data analysis and polynomial regression model was built. Response surface and contour plots were generated to understand the interaction of different variables. The experimental design was conducted using RSM of two input parameters and one response for each model.

## **3. RESULTS AND DISCUSSION**

The results of the two models are presented and analyzed using factor interactions for each model, surface plot and ANOVA. This section analyzed the model in detail.

### **3.1 Factor interactions for First Historical Data Design**

Figs. 1 and 2 show the individual and interaction behaviors of the two different factors used for the prediction of current generated for the two historical data. Fig. 1(i-iii) showed the behavior of concentration, time and combination of both on current generated. Fig. 1(i) show that at constant concentration of 75%, the relationship between the current generated and

time was inversely proportional. There was a decrease in the value of current generated from 1.27 to 1.028 when the process time was increased from 0 to 300minutes. In the case of concentration, the relationship between current generated and concentration was proportional; because at constant process time of 150minutes, the current generated increased from 1.097 to 1.209 when the concentration was increased from 50 to 100% as presented in Fig. 1(ii).

Fig. 1(iii) show the effects of combining process time and concentration on current generated. The figure show the effect of low and high values of concentration with increase in process time from 0 to 300minutes on current generated. It was observed that at a low value of concentration (50%), the current generated decreased from 1.90 to 0.292when process time was increased from 0 to 300minutes.The current generated line was within all the designed points. At a high value of concentration (100%), the current generated increased from 0.65mA to 1.76mA when process time was increased from 0 to 300. The current generated line pass was within all the designed points. The current generated decreased from 1.90 mA to 0.65 mA at time 0 when concentration was increased from 50 to 100(%) and increased from 0.292 mA to 1.76 mA at time 300 when concentration was increased from 50 to 100%.

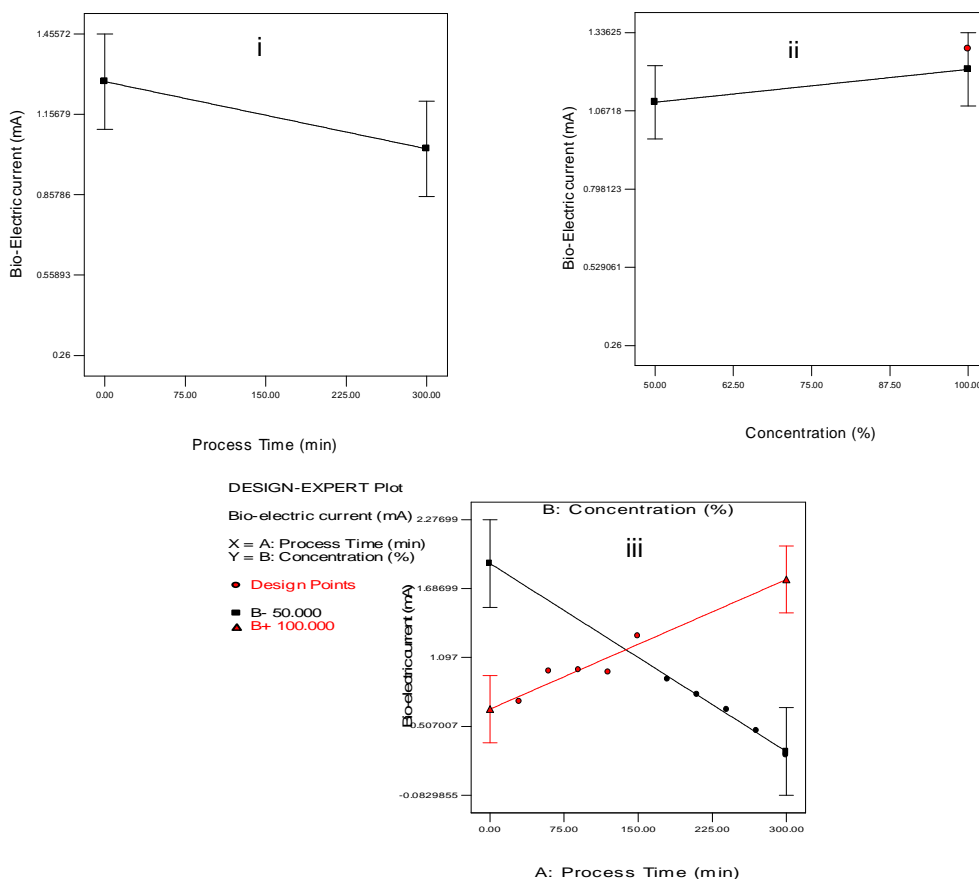


Fig. 1. Effect of factor interactions on current generated for first historical data

Fig. 2 (i-iii) show the behavior of speed, time and combination of both on bio-current generated. Fig. 2(i) show that at constant speed of 20rpm, the relationship between the current generated and time was inversely proportional. There was a decrease in the value of current generated from 1.406 to 1.032 when the process time was increased from 0 to 300minutes. In the case of speed, the relationship between current generated and speed was directly proportional because at constant process time of 150minutes, the current generated increased from 1.195 to 1.245 mA when the speed was increased from 0 to 40rpm as presented in Fig. 2(ii).

Fig. 2(iii) show the effects of combining process time and speed on the current generated. It was observed that in the absence of speed at 0rpm, the current generated increased from 0.575 to 1.815 mA when process time increased from 0 to 300. The line of current generated passed through all the designed points. At a high value of speed (40rpm), the current generated decreased from 2.238mA to 0.25mA when process time was increased from 0 to 300. The line of current generated passed within all the designed points. The current generated increased from 0.575 to 2.238 at time 0 when speed was increased from 0 to 40rpm and decreased from 1.815 to 0.25 at time 300 when speed was increased from 0 to 40rpm.

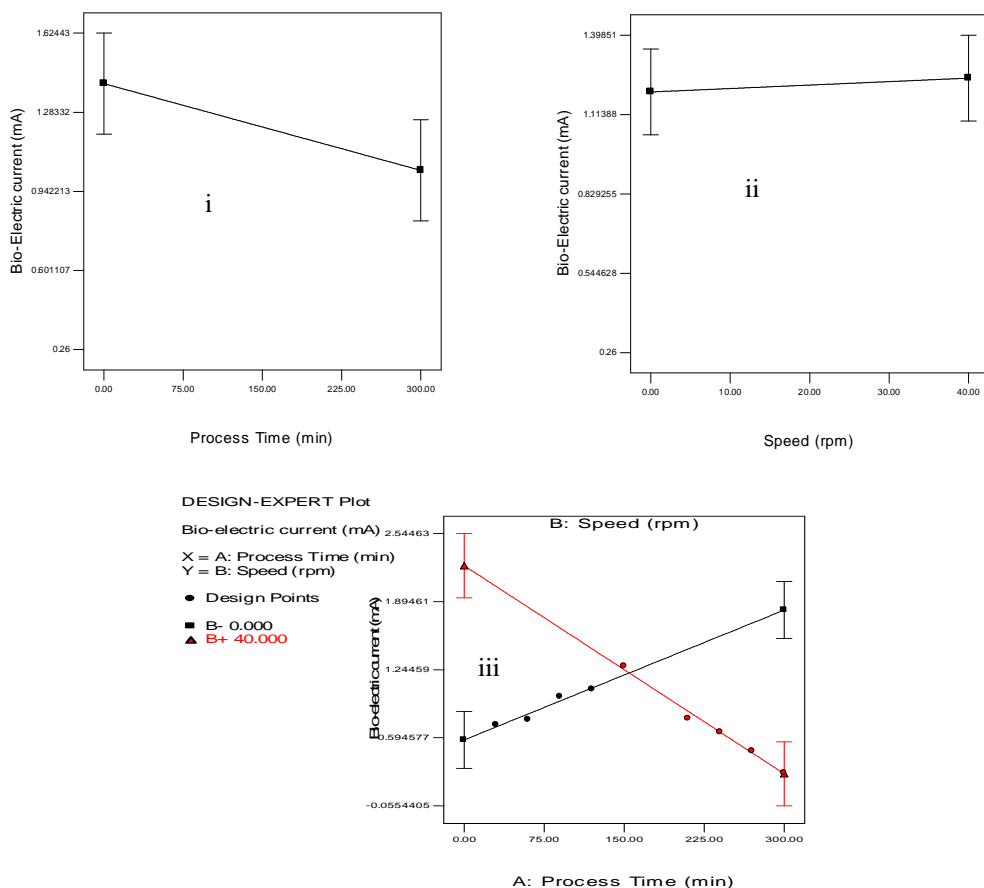
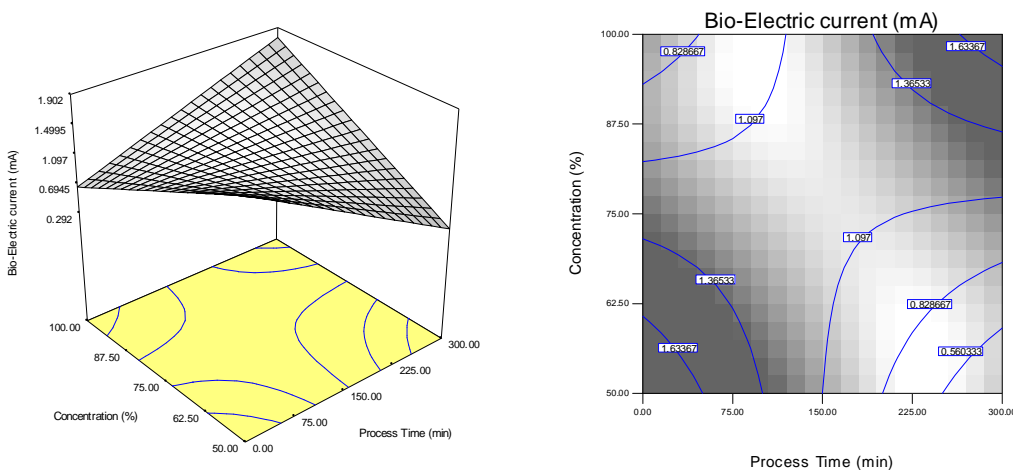


Fig. 2. Effect of factor interactions on current generated for second historical data

### 3.2 Surface Plot

The 3D response surface plot is a graphical representation of the regression equation (1). It was plotted to show the interaction of the variables. Each response surface plotted for bioelectric current generated at the different combinations of two variables at a time while maintaining the other variable constant. The convex response surfaces suggested that there are well-defined optimal variables. If the surfaces are rather symmetrical and flat near the optimum, the optimized values may not vary widely from the single variable conditions [12]. The graphic representation of response surface shown in Fig. 3 helps to visualize the effects of wastewater concentration and process time of MFC experiment on bioelectricity current generation. Fig. 3 show the effects of combining process time and concentration on current generated. It was observed that at a low value of concentration (50%), and process time 0min, the highest current generated was experienced at a value of 1.90 mA and the lowest current generated of 0.65 mA was experienced at a concentration of 100% and time 0min.



**Fig. 3. Response surface plot showing the effect of experimental process time and wastewater concentration on bioelectric generation**

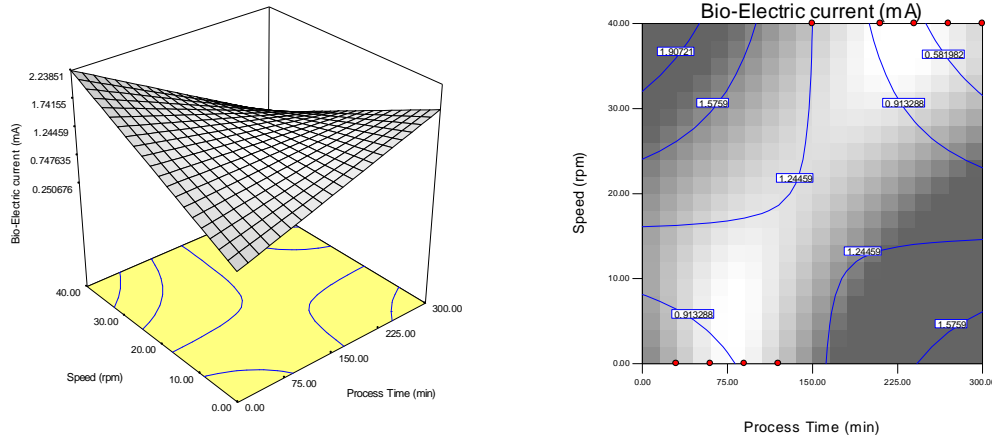
Fig. 4 showed the effects of combining process time and speed on current generated. The highest current generated of 2.238 mA was experienced when there was no speed (i.e. 0rpm) and process time of 300minutes while the lowest current generated of 0.25mA was noticed when both the speed and process time were at low values.

### 3.3 Model Equations and Interpretation

Table 1 showed the results obtained from the historical data design regarding the two sets of experimental factors: the concentration of sugar wastewater ( $X_1$ ) and experimental process time ( $X_2$ ), then experimental process time ( $Z_1$ ) and agitation speed ( $Z_2$ ) for microbial fuel cell experiment. The responses for experiment 1 and 2 are denoted by  $Y_1$  and  $Y_2$  respectively. The highest bioelectric current generated the verification experiment was 1.28 mA (as seen in run 6 of Table 1). The regression equations  $Y_1$  and  $Y_2$  demonstrated that bioelectric current generated have two empirical equations of independent variables (factors) in coded units, as shown in Equation 1 and 2:

$$Y_1 = 1.15 - 0.12X_1 + 0.057X_2 + 0.68X_1X_2 \tag{1}$$

$$Y_2 = 1.22 - 0.19Z_1 + 0.025Z_2 - 0.81Z_1Z_2 \tag{2}$$



**Fig. 4. Response surface plot showing the effect of experimental process time and agitation speed on bioelectric generation**

**Table 1. Design used for the two models**

Run	X <sub>1</sub>	X <sub>2</sub>	Y <sub>1</sub>	Z <sub>1</sub>	Z <sub>2</sub>	Y <sub>2</sub>
1	0	100	0	0	0	0
2	30	100	0.72	30	0	0.72
3	60	100	0.98	60	0	0.77
4	90	100	0.99	90	0	0.99
5	120	100	0.97	120	0	1.06
6	150	100	1.28	150	40	1.28
7	180	50	0.91	180	40	0.91
8	210	50	0.78	210	40	0.78
9	240	50	0.65	240	40	0.65
10	270	50	0.47	270	40	0.47
11	300	50	0.26	300	40	0.26

### 3.4 Analysis of Variance (ANOVA)

Table 2 showed the results of the regression models for bioelectric current generated in form of analysis of variance (ANOVA). A very low probability value [(p model>F) <0.05] indicated that the model was highly significant. The accuracy of the model was checked by the determination of correlation coefficient (R<sup>2</sup>) of 95.12%. In this case, R<sup>2</sup> value (0.9512) for equation 1 indicated that the sample variation for bioelectric current generated from the process 95.12% was attributed to the independent variables and only 4.88% of the total variation cannot be explained by the model. The adjusted determination coefficient (adj. R<sup>2</sup> = 0.9268) was also satisfactory for confirming the significance of the model.



**Table 2. Analysis of variance (ANOVA) for the first model**

Source	Sum of squares	DF	Mean square	F value	Prob> F
Model	0.73	3	0.24	38.96	0.0002
$X_1$	0.012	1	0.012	2	0.2075
$X_2$	7.51E-03	1	7.51E-03	1.2	0.3155
$X_1, X_2$	0.37	1	0.37	59.06	0.0003

The probability (p) values were used as a tool to check the significance of each of the coefficients in Table 2. A large magnitude of the *F* value and smaller p-value denoted greater significance of the corresponding coefficient [13]. The results in Table 2 show that the model is significant based on high value of *F*, followed by independent variable ( $X_2$ ) has a significant effect, as it has a positive coefficient in equation 1, according to which an increase in its concentration led to an increase in the response of the experiment. It was observed, from Table 2, that *F* value for interaction was high and p value was, however, low which show high significance of interaction for the model.

Table 3 showed the results of the regression model for the response in the form of analysis of variance (ANOVA). The ANOVA of the second regression model demonstrates that the model is very significant, as was evident from the *F*-test with a low probability value [(p model>*F*)<0.05], which indicated that the model was significant. The coefficient of determination ( $R^2$ ) was 0.9849, indicating that 98.49% of the variability in the response could be explained by the model. The adjusted correlation coefficient (adj. $R^2$ =97.58) of 0.9758 was very close to correlation coefficient which confirmed agreement between the predicted and actual values. It was noticed from Table 3 that variables  $Z_1$  and  $Z_2$  are not significant to the model based on low *F*-value and high p value obtained in Table 3 but the combining effect of the two factors was significant to the model development. Above observation gave an indication on how variables can be introduced in model development because despite that the individual variables having low *F*-value, the effect of combination of both variables proved crucial to the development of the model equation.

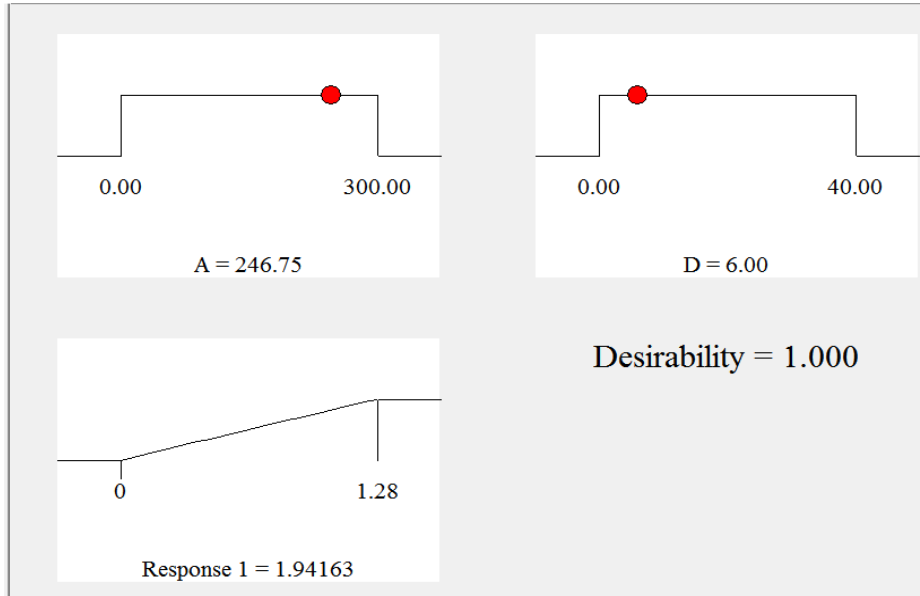
**Table 3. Analysis of variance (ANOVA) for the second model**

Source	Sum of squares	DF	Mean square	F value	Prob>F
Model	0.747944	3	0.249315	108.6044	<0.0001
$Z_1$	0.020902	1	0.020902	9.105024	0.0295
$Z_2$	0.001103	1	0.001103	0.480525	0.5191
$Z_1, Z_2$	0.389395	1	0.389395	169.6249	<0.0001

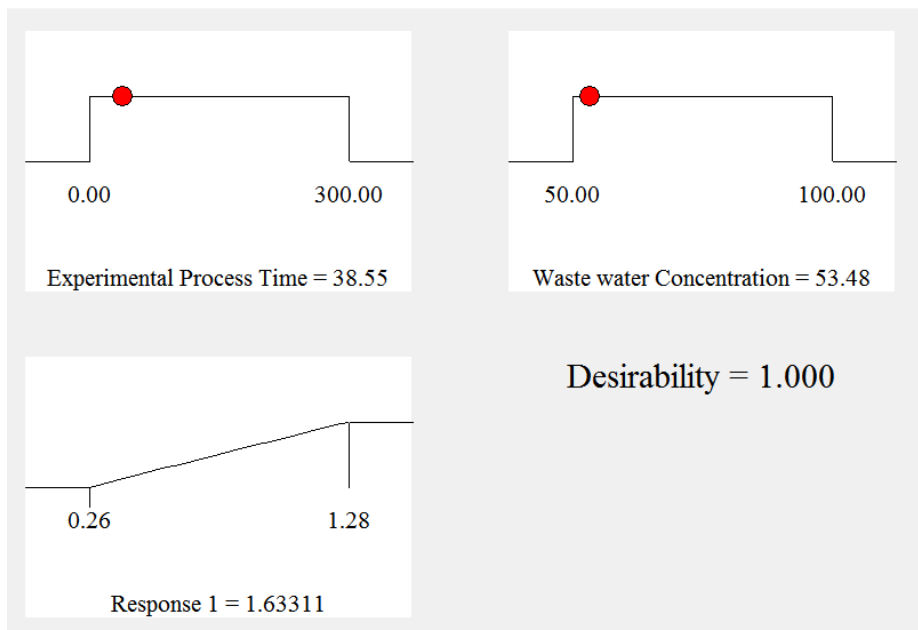
### 3.5 Optimization of Bioelectric Current Production

The main aim of this study was to find the optimum process parameters that favour generation of bioelectric current. Therefore, the function of desirability was applied using Design-Expert software version 6.0.8. In the optimization analysis of numerical optimization in response surface methodology, the target criteria (response  $Y_1$ ) was set as maximum values for the two factors of experimental process time ( $X_1$ ) and ( $X_2$ ). Fig. 5 shows the ramp of numerical optimization in response surface methodology for agitation speed and process time with bioelectric current generation of 1.941mA. Thus, the optimum operating conditions for bioelectric current generation, as shown in Fig. 5, were process time of 246.7 minutes and agitation speed 6 rpm at constant wastewater concentration. However, the optimum

bioelectric current generation of 1.633mA could be achieved when wastewater concentration and experimental process time were set to 38.4% and 53.48 minutes at constant agitation speed as shown in Fig. 6.



**Fig. 5. Ramp of numerical optimization in response surface methodology for wastewater concentration and process time of bioelectric current 1.941mA**



**Fig. 6. Ramp of numerical optimization in response surface methodology for wastewater concentration and process time of bioelectric current 1.633mA**

Then, the highest value for bioelectric current generation in Table 1 and Table 3 were compared with optimum bioelectric generation obtained in Figs. 5 and 6; it was noticed that bioelectricity generation from MFC is enhanced with more than 34% at the predicted optimum operating conditions.

#### 4. CONCLUSION

HDD was successfully used to investigate the effects of sugar wastewater concentration, agitation speed and experimental process time on the bioelectric current generation. The optimum bioelectricity generation conditions were obtained using 38.4% wastewater concentration, process time of 53.4 minutes resulting to 1.63mA; while agitation speed of 6rpm with process time of 246.7 minutes resulting to 1.941 mA of bioelectric current. Therefore, sugar wastewater showed promising substrate for bioelectricity generation in microbial fuel cell.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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