

Enhancement of Bioelectricity Generation from Treatment of Distillery Wastewater Using Microbial Fuel Cell

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Distillery wastewater is an important potential sources for electricity generation using Microbial Fuel Cell (MFC) because of it has high content of organic waste and can be easily degraded. This study investigated the effect of feed pH and buffering conditions on electricity production and treatment efficiency using distillery wastewater as a substrate in MFC. The anodic chamber was operated with diluted distillery wastewater (4000 ± 20 mg COD/L) at various pH between 5.4 and 10 while the cathode chamber was maintained at pH 7.5. The MFC peak power density of 168 mW/m^2 (580 mA/m^2) with COD, color and TDS removal efficiency of 68.2, 26.4, and 15.4%, respectively was achieved at pH 8. Cyclic voltammetry revealed that an exoelectrogenic activity of microorganism was significantly influenced with respect to pH. The effect of buffering salts in the anolyte on MFC performance was also investigated. When the system operating with borate buffer, highest power density of 194.7 mW/m^2 (624 mA/m^2) at 100Ω was achieved. The result indicated that alkaline condition (pH 8) and borate buffer was favored for obtaining maximum power generation and treatment efficiency from distillery wastewater in the MFC. © 2017 American Institute of Chemical Engineers Environ Prog, 00: 000–000, 2017

Keywords: microbial fuel cell, distillery wastewater, borate, power density, COD

INTRODUCTION

Sugar cane molasses is most important raw materials for production of alcohols and amino acids in fermentation industries. However, after use of raw materials, a large quantity (8–15 L of wastewater for every liter of alcohol production) of wastewater is discharged which creates serious environmental issues [1]. The molasses based distillery industries wastewater contains high organic matter in terms of Chemical Oxygen Demand (COD: 65,000–13,0000 mg/L), high concentration of minerals, dark brown color, and burnt sugar odor [2]. Several methods includes chemical, electrochemical, biological (aerobic and anaerobic) methods have been exploited for the treatment and disposal of molasses wastewater [3]. Conventionally, an anaerobic digestion method is used for treating such waste and generates methane gas followed by an aerobic treatment prior to disposal

[4]. An intensive conventional wastewater treatment system are required to develop an alternative technology which should be reliable, cost effective and also energy recovery.

Microbial fuel cell (MFC) is a novel bio-electrochemical system that generates bioelectricity and simultaneously removes pollutant such as COD, color, salinity etc. from wastewater which is one of the most advantageous [5–7]. The MFC consists of biotic anode and abiotic cathode chamber separated by a proton exchange membrane (PEM). The potential developed between metabolic respiration on electron donors and electron acceptors conditions in the anode and cathode generates electricity and water [8]. Earlier, the MFC was operated using pure organic matter such as glucose, acetate and lactose as a substrate to understand the fundamental of process [9,10]. Nowadays more complex substrates (dairy, domestic, starch processing, and paper recycling wastewater etc.) have been used to improve the performance and exploiting the waste into useful product [11–14]. Though, the molasses based distillery wastewater containing high organic matter that provides a great potential source for electricity generation. Additionally, the MFC used sulfide (that generated during the anaerobic process) as a fuel to generate electricity and oxidize sulfide into elemental sulfur [15].

In recent year, the MFC performance was enhanced by varying the operational and design parameters such as reactor configuration, electrode material, electrode distance, pH, microbial communities and substrate concentration etc. [16–19]. Nevertheless, the wastewater feed pH played a significant role on overall performance in dual chamber MFC, because it controls not only bacterial growth and also supports an efficient movement of protons through PEM. Generally, bacteria respond to change internal and external pH by adjusting their activity associated with many different processes, including proton translocation, amino acid degradation, adaptation to acidic or basic conditions [20]. Since, the wastewater pH in anode chamber should be identified to enhance power generation in the MFC. Mohanakrishna *et al.* [21] evaluated the MFC performance with distillery wastewater by keeping at acidic environment. Ha *et al.* [22] adjusted the distillery wastewater at pH 7 using buffer for evaluating the performance using bacteroidetes dominant thermophilic MFC. As of our knowledge none of studies have been performed at what pH enhanced performance in terms of power

generation and treatment efficiency will be obtained during treatment of distillery wastewater in the MFC.

In addition, buffer is also another significant factor that influenced the power generation in several ways due to its chemical composition, interaction with electrodes, bacteria, and membrane. The buffer helps to reduce pH changes in the bulk solution and therefore constant pH can be maintained in the suitable range for growth of microorganism [23]. Various types of buffers with different concentration ranges have been commonly used in MFCs. Feng *et al.* [24] reported that the power output was increased with increasing the phosphate buffer concentration because of increase in solution conductivity. Although the phosphate buffer is a quite expensive and higher concentration can contribute to eutrophication conditions of water bodies when the wastewater is discharged without removal of phosphates. The borate buffer solution is another potential alternative to PBS because it is chemically stable and does not affect the biochemical reaction. Qiang *et al.* reported that appropriate concentration of borate buffer solution could greatly enhance electron recovery rate in the MFC [25]. In this study, we therefore examined the performance of dual chamber MFC in terms of power generation and treatment efficiency by varying distillery wastewater pH between 5.4 and 10. The effect of different buffering salts on power generation and COD removal efficiency were also investigated. Cyclic voltammetry analysis was also performed to investigate the effect of electrochemical activity of bacterial culture on electrode surface at different pH conditions.

MATERIALS AND METHODS

Dual Chamber MFC Construction

Dual chamber MFC was designed and fabricated using polycarbonate material (Plexiglass). It consisted of anode and cathode chamber with working volume of 250 mL of each. The proton exchange membrane (Nafion 117, DuPontTM Nafion[®]) was used as a separator between the chamber and it was pretreated using 0.5 N H₂SO₄ acid and 5% H₂O₂ solution prior to use. The plain graphite plate was used as electrode in both anode and cathode chamber. The electrodes were placed at a distance of 1 cm from either side of the membrane. The copper wire was connected to the electrodes to measure the current flow through an external circuit. Inlet and exhaust holes were made at the top of both chamber to replace electrolytes for consecutive cycles. Both anolyte and catholyte were replaced when the voltage dropped to 100 mV from its maximum Open circuit voltage (OCV).

MFC Operation

The distillery wastewater was collected nearby Trichy, India. The important characteristics of real distillery wastewater are pH: 4.7 ± 0.2, COD: 90,000 ± 2000 mg/L, TDS: 20,000 ± 200 mg/L conductivity: 33.30 ± 3 mS/cm, salinity: 19,500 ± 300 mg/L, resistivity: 29 ± 2 Ω. The distillery wastewater was stored at 4°C in the refrigerator prior to use. The anode chamber was filled with diluted distillery wastewater with COD concentration of 4000 ± 20 mg/L while cathode chamber was filled with 100 mM of potassium ferricyanide in 100 mM of phosphate buffer. The performance of MFC was firstly evaluated under anolyte pH ranges between 5.4 and 10 while the catholyte was adjusted to pH 7.5 using 0.5N H₂SO₄ and 3N NaOH. The phosphate and borate buffering salt was used to investigate the effect of buffers on power generation and treatment efficiency. No extra inoculum was added from its previous cycles. The anode chamber was continuously stirred at constant speed (300–400 rpm) using magnetic beads to maintain uniform mixing throughout the

reactor. The anode chamber was completely sealed with epoxy sealant to maintain anaerobic environment, while the cathode chamber was open to atmosphere. The MFC was operated under batch mode with ambient conditions (25°C and 1 atm) and it was repeated thrice to obtain stable performance.

Measurement and Calculations

Cell voltage (E) was measured using an autorange digital multimeter. The current (I) and power (W) was calculated using ohms law equation of $I = E/R_{ext}$, $P = E^2/R_{ext}$. where R_{ext} is the external resistance in ohm. The polarization data was obtained by connecting anode and cathode electrodes with resistors (ranging from 15,000 to 50 Ω) after steady state voltage is obtained under OCV. The current density (mA/m²) and power density (mW/m²) was calculated with respect to anode electrode area (m²). Electrolyte pH, conductivity, resistivity, TDS, and salinity was determined using a multiparameter series (CyberScan PC650, Eutech's). The TDS removal efficiency of distillery wastewater was calculated as per the standard method. Wastewater COD was measured using standard potassium dichromate titration method (closed reflux method). The wastewater treatment efficiency in terms of COD removal efficiency (η_{COD} %) was calculated as follows

$$\eta_{COD} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

where C_i and C_f represent the initial and final COD concentration (mg/L) in the anode compartment.

COD removal rates were fitted assuming a first-order reaction with respect to wastewater COD concentration, and calculated as follows:

$$\ln \left(\frac{C_i}{C_f} \right) = -kt \quad (2)$$

where C_i is the influent COD, C_f is the effluent COD, t is time and k is the first order removal rate constant. When the kinetics are assumed to be first order with respect to concentration, this may vary with external resistance [26].

Percentage of color removal was calculated using the formula:

$$\text{Color removal (\%)} = \frac{\text{Abs}_i - \text{Abs}_f}{\text{Abs}_i} \times 100 \quad (3)$$

where Abs_i is the initial absorbance while Abs_f is the final absorbance

The Coulombic efficiency (CE) was calculated by integrating the current measured on the basis of COD consumption for a period.

$$\text{CE (\%)} = \frac{8x \int_0^t I dt}{Fv\Delta\text{COD}} \quad (4)$$

where I is the current, ΔCOD change in anolyte concentration, v is the volume of anode chamber, F is the faraday constant.

Cyclic voltammogram for each anolyte pH was recorded in a personal computers connected to Potentiostat–Galvanostat (model PGSTAT 101, Metrohm Autolab, The Netherlands) using conventional three electrode system. Voltammograms were obtained with the biofilm attached graphite electrode as

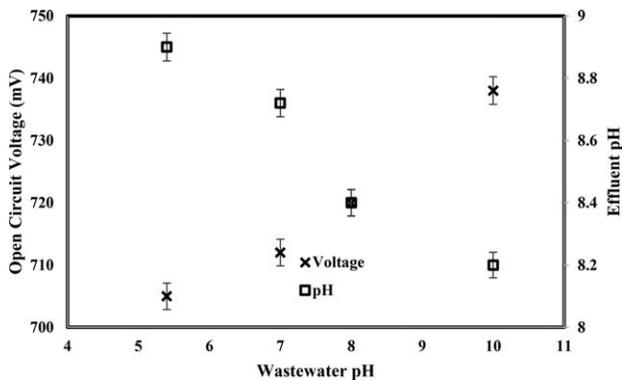


Figure 1. The effluent pH and maximum potential are presented as a function of the wastewater solution pH.

a working electrode, an Ag/AgCl as a reference electrode, and a platinum wire as a counter electrode respectively. The electrochemical cell was purged with oxygen free N_2 for 15 min before measurements. The CV was performed with the voltage ranging from -1 and $+1$ V at a scan rate of 50 mV/s. All CV experiments was conducted using wastewater as an electrolyte and it was conducted under room temperature.

RESULTS AND DISCUSSION

Effect of pH on voltage generation

The anolyte chamber was operated at different pH conditions such as acidic (pH 5.4), neutral (pH 7), and alkaline (pH 8 and 10) conditions. The biological and electrochemical reactions changed the wastewater pH during an experiment that caused variation in voltage generation in MFC as shown in Figure 1. When the system was operated at acidic and neutral pH, after 3 days reaction, the electrolyte (called effluent) pH was increased to an alkaline condition (pH 8.5 ± 0.2). Meanwhile, bacterial metabolism constantly produced weak acid compounds and maintained their intracellular pH. However, opposite trend could be observed when the feed operated at pH 10 and an effluent pH decreased to 8.7. When feed pH was fixed at 8, an effluent pH slightly increased to 8.4 which was due to transfer of protons from anode to cathode during electrochemical reactions [27]. The voltage generation was found to be dependent on electrolyte pH in the anode chamber. The system initially produced lesser potential of 320 ± 20 mV and afterwards increased to maximum value at all pH under OCV condition. The MFC under alkaline pH produced the higher voltage than acidic and neutral pH conditions. The maximum potential of 705, 712, 725, and 730 mV was generated at pH 5.4, 7, 8, and 10, respectively. The higher voltage generation observed under alkaline conditions might be due to increasing the negative potential in the anode chamber which caused higher attainable cell voltage in MFC [28]. Moreover, an inherent microorganism might be favoured for better voltage generation in alkaline pH rather than acidic and neutral pH.

Polarization Behavior

The polarization behavior was studied to visualize the electron discharge phenomenon with respect to experimental (wastewater pH) variations in MFC. The polarization curve clearly showed the influence of feed pH on power density as shown in Figure 2A,B. The MFC operated at pH 8 was observed a maximum power density as compared to other pH conditions. The peak power density of 168 mW/m^2 (580 mA/m^2 ; 290 mV), 104 mW/m^2 (456 mA/m^2 ; 228 mV), 101.4 mW/m^2 (247.9 mA/m^2 ; 409 mV) and 74.6 mW/m^2 (164.6 mA/m^2 ; 453 mV) was observed for pH 8, 7, 10, and pH 5.4,

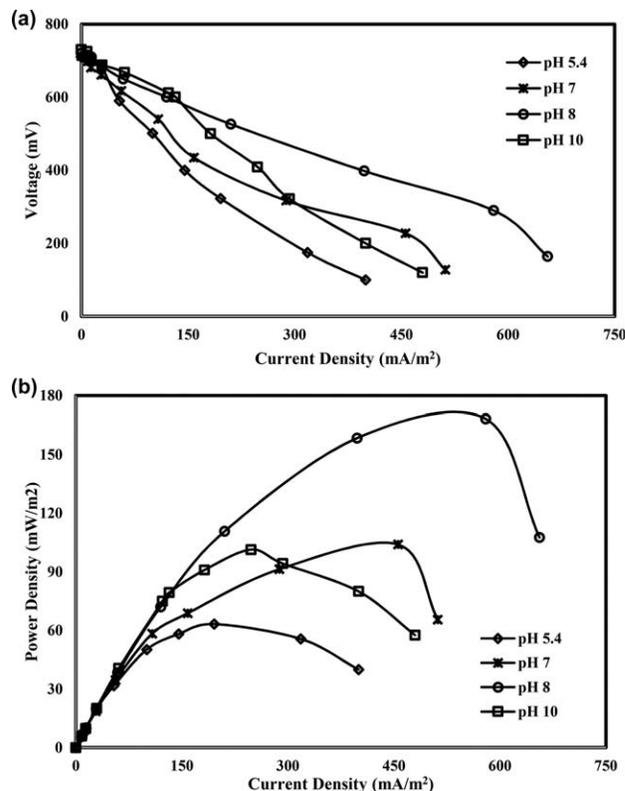


Figure 2. (a, b) Polarization and power density behavior with respect to different anolyte pHs.

respectively. In additions, to account the Coloumbic efficiency (CE), the system was connected with 1000Ω for 24 h and it found to be maximum at pH 8 than at other pHs. The Coloumbic efficiency (CE) was decreased in the order of pH 8 (13.5%) > pH 7 (9.51%) > pH 5.4 (7.05%) > pH 10 (6.32%), respectively. The maximum performance observed in alkaline conditions which might be the presence of microbial communities and its exoelectrogenic activity in the distillery wastewater. Different pH microenvironments will produce diverse biochemical pathways associated with diverse electron transfer phenomena. Under alkaline conditions, the exoelectrogenic microorganism at the anode have a better chance of outcompeting methanogenic bacteria for the degradation of organic matter in the wastewater. Decreased current output beyond pH 9 might be attributed to reduced bacterial activity as well as to slower electron discharge activity of bacteria at highly alkaline pH [28]. Moreover at pH 10, the proton gradient across anode and cathode might be poor which reduced the power generation in MFC. At low pH 5.4, the methanogenic bacterial growth would be faster than exoelectrogenic bacterial which decreased the power production in MFC [23]. The power output in this study is in good agreement with those reported by He et.al (2008) and Puig *et al.* [29,30] in which pH between 8 and 10 was also determined to be favorable power generation in dual chamber MFC with different wastewater.

Cyclic Voltammetry at Different pH

Cyclic voltammetry (CV) also helps to elucidate the electrochemical reaction occurring on the electrode surface and measures redox activities of the components in electrolyte. Cyclic voltammogram permits direct electrochemical detection of redox signals and senses the potential difference across the interface. The intensity of flow of e^- against the potential difference generated between cell and surrounding medium is called current (I). At a scan rate of 50 mV/s, CV

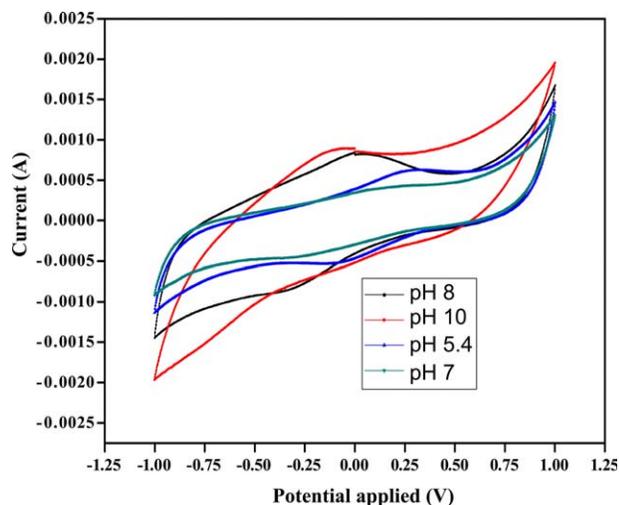


Figure 3. Cyclic voltammety profiles generated during the system operated at acidic, neutral and alkaline pH environments. [Color figure can be viewed at wileyonlinelibrary.com]

observed a significant variation in the electrochemical behavior with respect to different pH environment as shown in Figure 3. In comparison, a higher current was generated in alkaline than acidic and neutral pH respectively. During forward scan, the maximum current of 1.1, 1.3, 1.6, and 1.9 mA at 1 V was observed at pH 5.4, 7, 8, and 10, respectively. Higher current generation of applied potential compared to the corresponding alkaline pH suggesting higher electrochemical activity during forward scan. The current peak was appeared on the voltammogram when the components were oxidized or reduced during potential sweep. If one of the peaks disappeared, the component could be regarded as permanently oxidized or reduced [31]. A redox peaks appeared during forward and reverse scan at all pHs. In acidic pH, a clear redox peak at 0.25 V versus Ag/AgCl (0.62 mA) and -0.04 V (0.49 mA) was observed during forward and reverse scan. A poorly defined redox peak was observed and it showed at 0.08 V (0.37 mA) and -0.29 V (-0.457 mA) was achieved under neutral pH. During forward scan, a peak at voltage of 0.03 V (0.8 mA) and -0.1 V (0.89 mA) was observed at pH 8 and 10, respectively. But poorly defined redox peak was observed at -0.288 and -0.57 V during reverse scan for pH 8 and 10, respectively. The oxidation and reduction peaks appeared in voltammograms at pH which mainly corresponds to NADH/NAD^+ ($E^0: -0.32$ V). Samsudeen *et al.* [32] reported that the oxidation and reduction peaks observed during scanning due to the electrochemical activities of isolated culture in electrode may be involved in extracellular electron transfer. This result indicated that the alkaline condition (pH 8) would be favored the electricity generation from distillery wastewater in MFC.

Effect of Buffers on Power Production

Buffering salt also significantly affected the power production in MFC. The phosphate and borate buffers with 50 mM concentration were used in the anode chamber to investigate their effects on power generation. The pH of the anolyte was fluctuating from its feed pH 8 during the reaction. It was observed that, pH was slightly decreased to 7.8 while using both phosphate and borate buffers in the anolyte during operation. Under OCV conditions, the voltage changes were observed when the anolyte contained buffers as compared with nonbuffered control. The MFC produced a maximum voltage of 745 mV in borate buffer followed by 730 mV in phosphate buffer and lowest of 715 mV in nonbuffered control.

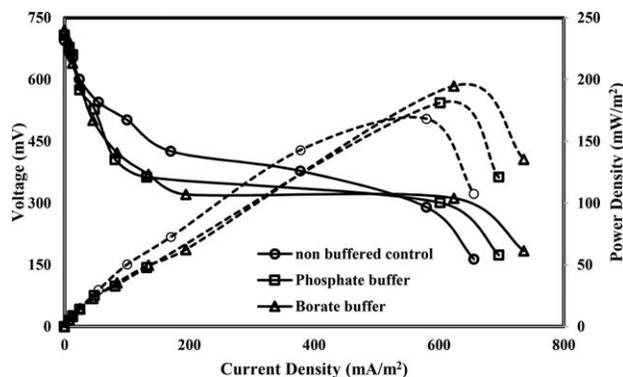


Figure 4. Voltage and power density versus current density with respect to types of buffers used in the anolyte.

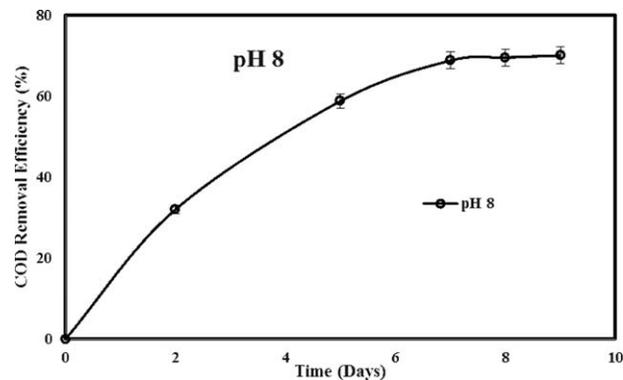


Figure 5. The COD removal efficiency with respect to time for optimum pH 8.

Polarization curve showed the buffers in the anolyte influenced the performance of MFC as shown in Figure 4. The current and power generation using borate buffer was higher in comparison with phosphate buffer used in the anode chamber. The MFC with borate buffer produced a peak power density of 194.7 mW/m^2 with corresponding current density of 624 mA/m^2 at 100Ω . The power density was reduced by 15.8% for phosphate buffer (181.2 mW/m^2) and 21.8% for nonbuffered control (168 mW/m^2 ; 580 mA/m^2) as compared to borate buffer. In additions, the CE was calculated for each buffer and it was found to be maximum of 17.2% in the MFC with borate buffer. The CE had the following values for other buffers: 15.4% (phosphate buffer) and 13.5% for nonbuffered control respectively. The results indicated that the types of buffers substantially affected the performance of MFC. When the buffering salt added in the system, the solution conductivity is increased as compared to nonbuffered control. It means that a high solution conductivity resulting from addition of buffer could reduce Ohmic resistance and internal resistance that favored electricity generation [33]. Liu *et al.* [34] reported that when the system is not limited by bacterial kinetics, the solution conductivity affected the internal resistance, which caused decrease in Ohmic voltage loss in the cell and thus the power generation was limited in the absence of buffers.

Wastewater Treatment Efficiency

The carbon fraction of wastewater functioned as an electron donor in the metabolic process resulting in the substrate degradation in concurrence with power generation and these was primarily function of pH in the anolyte. The COD

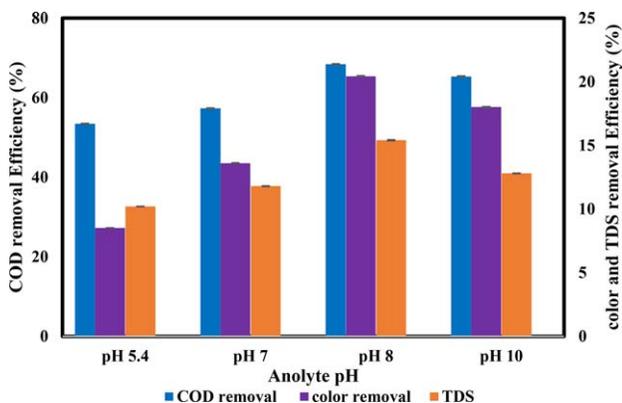


Figure 6. The COD, color, and TDS removal efficiency with respect to wastewater feed pHs in the anodic chamber. [Color figure can be viewed at wileyonlinelibrary.com]

removal is a complex consequence of diverse microbial metabolisms mainly as exoelectrogenesis, fermentation, and methanogenesis [28]. Figure 5 showed that the COD removal efficiency as a function of time for optimum pH 8. The COD Removal efficiency increased over period of time and after 7th day, it achieved almost stable conditions. The COD removal rates showed good agreement with an assumption of first-order degradation kinetics and the rate constant was found to be -0.171 day^{-1} from $\ln(C_A/C_{Ai})$ versus time. The COD removal efficiency was found to be dependent on its feed pH in the anolyte as shown in Figure 6. A maximum COD removal efficiency of 68.4% at pH 8 than other pHs in the anode chamber. At pH 5.4, 7, and 10 in the anodic chamber, the maximum COD removal efficiency of 53.4, 57.3, and 65.3% respectively were observed. In comparison, the MFC operated with borate buffer achieved highest COD removal efficiency (74.8%) than the phosphate buffer (70.2%) and nonbuffered control (68.4%). Raghavulu *et al.* reported that the efficient COD removal efficiency was observed between pH 6 and 8 [35]. Higher alkalinity (pH 10) would negatively affect the growth and metabolism of exoelectrogens, thus further deteriorating their electrochemical activity.

Due to presence of melanoidins compound, the distillery wastewater is characteristically dark-brown in color. This has C—C double bonds in the structure which is responsible for the color. Its molecular weight ranges between 500 and 40,000 which are hard to remove by conventional (biochemical) method. The MFC was observed a significant color reduction at different pH conditions and types of buffers in the anolyte as shown in Figure 5. The color removal in the distillery wastewater was calculated by measuring the absorbance at a wavelength of 475 nm in the UV spectrophotometer [36]. When the system operated at pH 8, color removal efficiency of 26.42% was observed in the distillery wastewater. Though the color removal efficiency was increased when the buffers was added to wastewater. The MFC operated with borate suffer showed highest color removal efficiency of 29.5% in comparison with phosphate buffer (30.42%) and nonbuffered control. The color removal observed in the wastewater might be attributed to possibility of biologically catalyzed electrochemical oxidation during the MFC operation [37]. The color removal increased while using the buffer might be due to maintaining constant pH 8 in the anolyte which was favored to electrochemical oxidation reaction at this environment.

The distillery wastewater contains high concentration of salt contains 933.1 ± 10 ppm in the COD of 4000 mg/L. During the MFC operation at different wastewater feed pH, a reduction in TDS was observed as shown in Figure 5. The

maximum TDS removal efficiency of 10.2, 11.8, 15.4, and 12.8% was observed at pH 5.4, 7, 8, and 10, respectively. The ionic species are transferred during the MFC operation in proportion to the current generated by the bacteria during the utilization of organic matter from the distillery wastewater. Mohan *et al.* [37] reported that the salt is removed in dual chamber MFC operated with diverse catholyte. At different pH, the variation in the current generation might be influenced the TDS removal efficiency in the wastewater.

CONCLUSION

This study investigated the effect of feed pH of the distillery wastewater and buffers on the overall performance of the MFC. The experimental results demonstrated that anolyte of MFC at pH 8 showed the better power production. At pH 8, the system achieved a maximum power density of 168 mW/m^2 , which was due to the presence of microbial communities and its exoelectrogenic activity. When the MFC operated with buffer at pH 8, the performance of MFC was improved due to increase in the conductivity of the wastewater and also maintain the constant environment that is suitable for the bacterial growth and its exoelectrogenic activity. Although the borate buffer enhanced the power generation in the MFC as compared to the other buffers. The COD, color and TDS removal efficiency was also significantly varied with respect to different feed pH and a maximum of 68.4%, 26.4 and 15.4%, respectively was achieved at pH 8. The COD removal efficiency was also increased while using the buffer at optimal pH as compared with nonbuffered control. Finally, the results concluded that the optimum pH 8 and borate buffer enhanced the power generation and treatment efficiency from distillery wastewater in the MFC.

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