Deriving electricity from dye processing wastewater using single chamber microbial fuel cell with carbon brush anode and platinum nano coated air cathode

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Abstract

Single chamber air cathode microbial fuel cell (MFC) is a promising and sustainable technology to generate electricity. In the present study, the potential of air cathode MFC treating dye processing wastewater was investigated at various organic loads with interest focused on power densities, organic removal and coulombic efficiencies. The highest power density of about 515 mW/m² (6.03 W/m³) with 56% of coulombic efficiency was procured at 1.0 (g COD/L) organic load. The high potency of TCOD (total chemical oxygen demand), SCOD (soluble chemical oxygen demand) and TSS (Total Suspended Solids) removal of about 85%, 73% and 68% respectively was achieved at the organic load of 1.0 (g COD/L). The bacterial strains in anode region at the initial stage of MFC operation were reported to be responsible for potential organic removal. The bacterial strains in air cathode MFC were identified as *Paenibacillus* sp. strain JRA1 (MH27077), *Pseudomonas* sp. strain JRA2 (MH27078), *Ochrobactrum* sp. strain JRA3 (MH27079), *Sphingobacterium* sp. strain JRA4 (MH27080), *Stenotrophomonas* sp. strain JRA5 (MH27081), *Bacillus* sp. strain JRA6 (MH27082) and *Clostridium* sp. strain JRA7 (MH27083) using phylogenetic analysis. After 60 days of air cathode MFC operation, the bacterial community in biofilm samples was dominated by *Bacillus*, *Ochrobactrum* and *Pseudomonas* (20–22%). The biofilm sample collected from the carbon brush consisted of *Bacillus* (33%), *Ochrobactrum* (30%), *Pseudomonas* (28%), *Clostridium* (6%) and *Stenotrophomonas* (3%). The present study revealed the treatment efficiency of dye processing wastewater along with power generation in single chambered air cathode MFC.

Keywords Dye processing wastewater · Air cathode MFC · Polarization curve · Coulombic efficiency

Introduction

To protect environmental and human life, treatment of wastewater is more essential. Until past decades, a portion of the wastes generated from industries were drained off directly into the waterways without any proper treatment (Puspasari and Peinemann 2016). With concerns of suitable

development, removal of contaminants via biological treatment is the best alternative when compared with other methods (Chen et al. 2016). Among the biological treatment systems, bioelectrochemical based technologies are gaining attention of researchers due to biofuel generation along with wastewater treatment (Li et al. 2016). Microbial fuel cell (MFC) is one of the technology to treat the wastewater along with corresponding electricity generation (Feng et al. 2008). MFC have emerged as a promising as well as challenging technology in recent years. For MFC, the chemical energy of organic substance is converted into proton and electron by electrogens attached on the anode surface. These produced electrons and protons then move towards the cathodic region through external circuit where they bonded with oxygen to form water molecules (Yang et al. 2015). MFC is advised to be an promising sustainable technology to satisfy the energy demands particularly using wastewater as substrate, which

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can produce current, also simultaneously treat wastewater, thus may minimize the operational costs of effluent treatment plant (Khan et al. 2015). In air cathode MFC's, configuration of air facing cathode materials and their efficiency was the most crucial parameter in power generation and removal of organic pollutant (Ou et al. 2016). One side of the cathode surface exposed to air has significantly enhanced the power generation, compared to aqueous cathode. Increase in internal resistance occurs due to low solution conductivity of aqueous cathode which leads to minimum power output production. Reducing space between the electrodes is the only way to overcome the higher internal resistance. Moreover, MFC produces less biomass (sludge) when compare to other technologies (Feng et al. 2008; Jayashree et al. 2016). After accounting all the positive aspects of MFC technology, in the present study single chambered air cathode MFC was employed which potentially treated the dye processing wastewater along with power generation. Many researchers have reported the electricity generation in MFC from various industrial wastewaters such as brewery (Feng et al. 2008), seafood processing (Jayashree et al. 2016), surgical cotton industry (Tamilarasan et al. 2017) and textile wastewater (Mise and Saware 2016). Around 5.5 million litres per day of wastewater are generated from various dye processing industries. The dye industry has been categorized as one among the major polluting industries in India (Mise and Saware 2016). As per the guidelines of central pollution control board (CPCB), India, the dye industry wastewater discharge limit of COD is less than 250 mg/L (Adishkumar et al. 2016). These untreated contaminated effluents when discharged into the water bodies not only affect the photosynthetic activity of the river system, but also produce harmful effects to the living organisms (Sahinkaya et al. 2017). Based on previous study by Watanabe (2008), the performance efficiency of single chamber air cathode MFC was improved gradually when compared with having different kind of diffusion layers such as platinum and cobalt tetramethoxyphenylporphyrin. Higher chemical oxygen demand (COD) and pH present in the effluent from dye industry is due to the accumulation of enormous quantity of dye ingredients and detergents. Acclimatization of the inoculum with biofilm formation in carbon brush and power generation occurs normally within 24–72 h. The presence of dyes, auxiliaries and soda in textile wastewater delay the acclimatization of the microbial consortium and also influenced the power generation (Khan et al. 2015). The presence of highly toxic material in dye wastewater cause higher resistance to microbial degradation, hence an effort was made to treat this wastewater using electrochemically active microbes. A novelistic approach was carried out using single chamber air cathode MFC with platinum coated PTFE (polytetrafluoroethylene) diffusion layers to achieve higher power and treatment efficiency of dye wastewater. The

core of present study is to analyze the treatment efficiency of dye processing effluents in single chamber air cathode MFC operated with various organic loads; and to examine the influence of four diffusion layers as cathode material for higher power production.

Materials and methods

Dye processing wastewater

Dye processing wastewater was collected from common effluent treatment plant situated at Angeripalayam, Tirupur district, Tamilnadu, India and stored at 4 °C. Table 1 summarizes the initial characteristics of dye processing effluents such as pH, total chemical oxygen demand (TCOD), soluble chemical oxygen demand (SCOD), total solids (TS), total dissolved solids (TDS), total suspended solids (TSS), chloride and alkalinity were determined as described in standard methods by American Public Health Association (Rice et al. 2017).

MFC architecture and mode of operation

A single chamber fed - batch air cathode with working volume of 260 mL was used. Anode brush were made of a core (double twisted) 15 cm long titanium wires with 4,00,000 tips of carbon fibers (PANEX 33 160K, ZOLTEK) cut to 2.5 cm in outer diameter and 5 cm long (Fig. 1a). The cathode (3.5 cm diameter, 9.6 cm² total cathode projected area) were finished by a coat of 10% Pt catalyst (0.5 mg Pt per cm²) on the 30% wet proofed carbon cloth (Sciarria et al. 2015). Moreover, air facing side of carbon cloth has been coated with carbon base layer followed by 60% PTFE four diffusion layers (Cheng et al. 2006). PTFE, a fluorinated hydrophobic chemical compound, is normally coated to the air facing side of the cathode to make a diffusion layer with adequate hydrophobicity. A PTFE layer can be very dense and disrupt the oxygen circulation, thus coated with carbon

Table 1 Initial characteristics of dye processing industry wastewater (triplicate measurements)

Parameter	Values
pH	10.7
Total solids (mg/L)	11,650 ± 170
Total dissolved solids (mg/L)	7260 ± 120
Total suspended solids (mg/L)	4050 ± 70
Chemical oxygen demand (mg/L)	2100 ± 200
Biochemical oxygen demand (mg/L)	1110 ± 150
Chloride (mg/L)	1550 ± 50
Total alkalinity (mg/L)	1725 ± 100

Fig. 1 Single chamber air cathode MFC **a** schematic representation of the device used for this experiment, **b** voltage production during acclimatization (start up) phase

black powder to increase the porosity and also providing sufficient cathode performance relative to power production (Yang et al. 2015).

In the single chamber air cathode system, the dye industry wastewater (substrate) along with the inoculum (waste activated sludge) was fed gradually into the anode chamber using peristaltic pump and maintained under anaerobic condition. BES (2-bromoethane sulfonate) was added to the anode chamber to suppress the methanogenic activity inside MFC. To acclimatize the inoculum, the MFC reactor was operated under open circuit mode for 15 days. During acclimatization phase (startup phase) the voltage production was gradually increased up to 10th day. Beyond that the voltage recorded stable condition, which indicates the adaptation of inoculum to the new environment. The startup phase power production is shown in Fig. 1b. Open circuit mode employed during acclimatization phase recorded a maximum voltage of 587 mV at the end of the phase. Concerned with

the acclimatization of the inoculum to the wastewater, normally they are able to utilize substrate for current generation within 24–72 h with carbon brush, which is well known for its high conductivity nature favoring biofilm formation. Due to the presence of dyes, auxiliaries and soda in wastewater must have contributed for longer duration of acclimatization of the microbial consortium to the wastewater. However, in the present study, the pH of the effluent (pH 10.7) was neutralized (pH 7) before being introduced into the anode chamber of air cathode MFC. Thus the pH of the dye effluent will not affect the bacterial species in the inoculum, biofilm formation ability and subsequent power production (Kim et al. 2016). During acclimatization phase, generally bacteria can uptake the substrate that is present in the wastewater for power production within 1 day. The single chamber air cathode MFC operated at various organic loads starting from 0.5, 1.0, 1.5 and 2.0 g COD/L. The pH of dye processing wastewater was maintained at 7.0 ± 0.2 before employed as substrate. The performance of MFC was analyzed over a period of 70 days at room temperature ($27 \pm 3^\circ\text{C}$). For every organic loading, the performance of the MFC reactor was evaluated based on total and soluble COD removal efficiencies, power production and suspended solids (SS) removal efficiency.

Measurements and calculations

At every organic load, digital multimeter was used to measure current and power generation in single chamber air cathode MFC based on the following equations:

$$I = E/R, \quad (1)$$

$$P = I \times E, \quad (2)$$

where I is current (A), E is voltage (V), R is external resistance (Ω), P is power (W). Moreover, power density and current density was analyzed based on cathode projected surface area. Based on the results of Min and Logan (2004) the external resistance range was maintained, who obtained effective polarization curves with external resistance ranging between 100 and 16,000 Ω during the treatment of domestic wastewater in flat plate MFC.

Coulombic efficiency (CE) is the ratio between coulombs recovered to total coulombs in substrate. Based on Eq. 3 (Logan 2008), the coulombic efficiency for fed-batch cycle was calculated

$$CE = 8I \text{ tb} / F \text{ van } \Delta\text{COD}, \quad (3)$$

where 8 is a constant, I is current (A), tb is time period (s), F is Faraday number (C/mol), V is volume of anode (L), ΔCOD is COD removal (g/L). Once the MFC reached to a

steady state condition, in every organic load the polarization curve was plotted by changing various external resistances.

Phylogenetic analysis

The water and biofilm (anode-carbon brush) samples consisting of different bacterial strains was obtained from the MFC. Bacterial DNA from the biofilm was extracted by Qiagen DNA isolation kit. The phylogenetic analysis was performed every 20 days using the water samples from MFC to analyze the difference in the bacterial community. High throughput sequencing was performed as detailed by Pugazhendi et al. (2017) with primers (515-532U 5'-GTG YCA GCMGCC GCG GTA-3' and 909-928U 5'-CCC CGY CAA TTC MTTT RAG T-3') targeting at V4–V5 region of 16S rRNA (Jamal and Pugazhendi 2018). The nucleotide sequences obtained from the high throughput sequencing were analyzed in Basic Local Alignment Search Tool Nucleotide (BLASTN) search to confirm the genus of the bacterial strains. MEGA v5 (molecular evolutionary genetic analysis) was used to depict the neighborhood relationship between the reported and identified genus of bacterial strains in the reactor study.

Results and discussion

Dye processing wastewater treatment efficiency

A single chamber air cathode MFC was operated under fed batch mode with varying organic load of 0.5, 1.0, 1.5 and 2.0 gCOD/L. Figure 2a shows the total COD (TCOD) concentration in influent, effluent and removal efficiencies in the individual organic load. A TCOD removal efficiency of about 62% was achieved in lower organic load of 0.5 gCOD/L. From Fig. 2a, it was noted that the TCOD removal efficiency increased while organic load increased from 0.5 to 1.0 gCOD/L. A maximum of 85% TCOD removal was achieved at the organic load of 1.0 gCOD/L. This clearly shows the utilization of substrate was effective in the organic load of 1.0 gCOD/L. Further increase of organic load beyond 1.0 gCOD/L resulted in decrease of TCOD removal with an efficiency of 74% and 58% at an organic load of 1.5 and 2.0 gCOD/L respectively. Based on the organic loading optimization results (i) at higher substrate loading (2.0 gCOD/L) TCOD removal decreased due to depletion of retention time for effective substrate degradation by the anodic microorganisms (Kim et al. 2016), (ii) since BES was added to the anode chamber along with the substrate there might be predominant role of electrogenic microorganism in the COD removal and the excess of substrate provides a niche for methanogens and methanogenesis must be anticipated whenever substrate loading is higher

Fig. 2 Effect of wastewater concentration on **a** TCOD removal, **b** SCOD removal

than the maximum electron transfer rate of the MFC (Mise and Saware 2016; Huang and Logan 2008).

The removal trend of SCOD follows the similar pattern as like that of TCOD removal. From Fig. 2b, a peak SCOD removal of about 73% was procured at 1.0 gCOD/L organic load. In the anode chamber, the electrogenic and non-electrogenic bacteria including methanogens might have contributed to maximum TCOD, SCOD removal (Tamilarasan et al. 2017). However, in this study BES was added in the MFC to suppress the activity of methanogens. This in turn increases the activity of electrogenic bacteria which plays an effective role in the removal of COD. At an organic load of 0.5 gCOD/L, 58% of SCOD removal was attained. Beyond the organic loading of 1.0 gCOD/L the substrate concentration was increased to 1.5 and 2.0 gCOD/L, a sudden decrement was recorded in SCOD removal. Based on Fig. 2b, at an organic load 1.5 and 2.0 gCOD/L, the SCOD removal was found to be 69% and 53%, respectively. In the present study, formation of thick biofilm around carbon fibre brush was noted. Similarly, work on upflow MFC, He et al. (2005) reported a strong correlation between COD removal efficiency and biofilm formation around anode. The results

of COD removal in the present study treating dye effluent was compared with various real wastewaters COD removal and enlisted in Table 2.

Power generation at different substrate concentrations

The performance of the single chamber air cathode MFC was evaluated through the polarization curve by varying external resistances (16,000–100 Ω). Figure 3a–b depicts the polarization curve of MFC. This curve was analyzed to find out the optimum power as well as current densities distributed to anode volume and cathode projected area. At acclimatization phase, bacterial biomass of the air cathode MFC was allowed to adopt the new environmental condition. During this period, organic matter (substrate) can be utilized which leads to constant power generation (Huang and Logan 2008). During acclimatization phase, the recorded open circuit voltage (OCV) was found to be quite low, this may be caused by the immobilized biomass on the surface of anode brush and suspended bacteria which shows the startup phase (Tee et al. 2016). This (OCV) was maintained for a period of 15 days, which leads to biofilm formation on the anode surface and substrate concentration to strengthen the higher power production. Power production in MFC system was actually improved by the biofilm formation on the anode surface (Bergel et al. 2005). Typical behavior of fuel cells is highly stabilized at peak resistance during the removal of external load and the stabilization decline at lower resistance (Jayashree et al. 2016). A maximum power density of 270 mW/m^2 (3.69 W/m^3) and 515 mW/m^2 (6.03 W/m^3) was observed for lower and higher resistances. The tolerant voltage and power density of about 925 mV and 515 mW/m^2 (6.03 W/m^3) was recorded at the organic load of 1.0 gCOD/L connected with external resistance (500 Ω) under closed circuit mode. Carbon brush electrode produced more power

Fig. 3 Polarizations of MFC using dye wastewater as feedstock **a** power density vs current density, **b** voltage vs current density

Table 2 Comparison of COD removal with various wastewater

Type of reactor	Anode material	Type of wastewater	Maximum COD removal efficiency (%)	References
Air—cathode MFC (continuous mode)	Graphite felt	Swine wastewater	77.1	Zhuang et al. (2012)
Air—cathode MFC (fed—batch mode)	Carbon brush	Paper recycling wastewater	76	Huang and Logan (2008)
Air—cathode MFC (fed—batch mode)	Stainless steel mesh	Fish market wastewater	80	Noori et al. (2016)
Air—cathode MFC (continuous mode)	Graphite fiber brush with titanium core	Domestic wastewater	54.2	Kim et al. (2015)
Air—cathode MFC (fed—batch mode)	Graphite rod	Slaughterhouse wastewater	72	Mateo-Ramírez et al. (2017)
Air—cathode MFC (fed—batch mode)	Carbon fibre brush with titanium wire	Dye industry wastewater	85	This study

generation when compared with carbon paper electrode (Logan et al. 2007). At an organic load of 0.5 gCOD/L, voltage and power density of about 735 mV and 325 mW/m² (3.44 W/m³) was procured. When substrate loading was increased to 1.5 and 2.0 gCOD/L, a rapid decrement in voltage and power density was observed (Fig. 3a, b). At higher organic load 1.5 and 2.0 gCOD/L, voltage and power density of about 720 mV (440 mW/m²) (4.31 W/m³) and 693 mV (270 mW/m²) (3.69 W/m³) was achieved. This gradual decrement in power density at higher organic loading rate was due to inhibition caused by COD concentration. At higher organic loading, feeding nutrients was higher than consumption rate affecting the balanced nutritional requirement of microorganisms. This in turn causes substrate inhibition and less electron formation rate (Fikret and Serkan 2009).

The ohmic losses of the system relevant to electrode distance, contact of wire and substrate concentration (Kondaveeti et al. 2014). The substrate loading rate of the wastewater performs a major role of power generation in MFC (Kim et al. 2016). From Fig. 3a, b it was noted that at an organic load of 1.0 gCOD/L, the substrate utilized by the inoculum could pave the way for higher power generation. From these observations, an organic load of 1.0 gCOD/L and external resistance of 500 Ω was considered as an optimum for microbial growth for producing maximum voltage (925 mV) and power density (515 mW/m²). An external resistance of 500 Ω was found to be optimum for growth of bacteria on the anode surface and enhancement of higher power productivity.

Coulombic efficiency of the MFC

Coulombic efficiency (CE) was evaluated based on the COD concentration converted to electrical energy during fed batch cycle of single chamber air cathode MFC (Pu et al. 2014). A coulombic efficiency of 26% was achieved at an organic load of 0.5 gCOD/L (Fig. 4). At an organic load of 1.0 gCOD/L, the coulombic efficiency increased to 54%. A decrease in coulombic efficiency of about 16.7% and 13% was achieved for higher organic loading of 1.5 and 2.0 gCOD/L. The following are the reasons which can affect the coulombic efficiency: (i) competition between exo electrogenic microorganisms and other types of bacteria with respect to anode surface at high organic loading can pave the way to decrease in coulombic efficiency (Lorenzo et al. 2010), (ii) methanogenesis and/or fermentation process could affect the coulombic efficiency extremely when MFC was loaded with real wastewaters (Behera and Ghangrekar 2009), (iii) utilization of substrate due to action of bacterial oxidation aided by oxygen as an electron acceptor, which leads to decline in CEs (Lefebvre et al. 2011), (iv) application of higher external resistance leads to loss of electron

Fig. 4 Profile of the coulombic efficiency of the MFC using dye wastewater as feedstock

Fig. 5 Effect of wastewater concentration on TSS removal

due to uptake of substrates other than electricity producing reactions (Li et al. 2016).

Reduction of suspended solids

The dye processing effluent was loaded with the suspended solids (SS) concentration of about 840 ± 120 mg/L to 3360 ± 100 mg/L with corresponding organic load of 0.5 to 2.0 gCOD/L. Figure 5 explores the influent, effluent and removal of SS concentrations. The SS removal at an organic load 0.5 gCOD/L was 55%. Maximum suspended solids reduction was 68% at the organic load of 1.0 gCOD/L. When the organic loading was increased beyond 1.0 gCOD/L, the removal of SS decreased to 64% and 49% with corresponding organic load of 1.5 and 2.0 gCOD/L. At higher organic load, removal of SS was associated to deposition of the fermentative products, which leads to reduce the pH and also

suppress the activity of anodophiles (Zhuang et al. 2012). This led to diminution in biomass that drove to higher TSS concentration in effluent. From this analysis, it was inferred that at an optimum organic load of 1.0 gCOD/L, a substantial reduction of solids in the effluent was obtained. Based on

the above results, it can be concluded that there was no waste of biomass resulting in maximum TSS removal efficiency. The overall effluent wastewater characteristics (after treatment) were summarized in Table 3.

Bacterial community analysis

The bacterial strains present in the MFC were identified as *Paenibacillus* sp. strain JRA1 (MH27077), *Pseudomonas* sp. strain JRA2 (MH27078), *Ochrobactrum* sp. strain JRA3 (MH27079), *Sphingobacterium* sp. strain JRA4 (MH27080), *Stenotrophomonas* sp. strain JRA5 (MH27081), *Bacillus* sp. strain JRA6 (MH27082) and *Clostridium* sp. strain JRA7 (MH27083) in initial stage and 20 days of MFC operation (Fig. 6). *Bacillus*, *Ochrobactrum*, *Clostridium* and *Pseudomonas* covered almost equal percent (14–16%) of

Table 3 Effluent characteristics of dye processing industry wastewater (after treatment)

Parameter	Values
pH	6.7
Total suspended solids (mg/L)	530 ± 25
Chemical oxygen demand (mg/L)	155 ± 6
Soluble chemical oxygen demand (mg/L)	173 ± 6
Biochemical oxygen demand (mg/L)	50 ± 3

Fig. 6 Phylogenetic analysis of bacterial strains in the MFC for treatment of dye processing wastewater using nucleotide sequence with neighborhood relationship

dominance compare to other strains after 40 days of operation. Previous studies were in agreement with the strains reported in the present study such *Bacillus* (Shaikh et al. 2016), *Clostridium* (Ekambaram et al. 2016) and *Pseudomonas* (Jayaprakash et al. 2016) to treat dye wastewater in MFC. *Ochrobactrum* was reported as dominant strain by Jayashree et al. (2015) in continuous upflow MFC to treat retting pond wastewater. Based on our literature survey this is the first study to report *Ochrobactrum* in MFC for the treatment of dye wastewater. *Bacillus*, *Ochrobactrum* and *Pseudomonas* dominated with 20–22% after 60 days operation of MFC. The biofilm sample collected from the carbon brush consisted of *Bacillus* (33%), *Ochrobactrum* (30%) and *Pseudomonas* (28%) as dominant strains with 28–33% occupancy. Remaining part of the biofilm was covered by *Clostridium* (6%) and *Stenotrophomonas* (3%). Thus the bacterial strains present in the reactor potentially treated the dye processing wastewater and correspondingly produced electricity.

Conclusion

The performance of single chamber air cathode MFC synergistically was influenced by varying organic load. The performance indicating parameters such as TCOD, SCOD and SS removal efficiency were found to be high at organic load 1.0 gCOD/L and their corresponding removal efficiencies were 85%, 73%, and 68% respectively. A maximum power density of about 515 mW/m² (6.03 W/m³) was recorded at 1.0 gCOD/L organic load. The bacterial community in the MFC was dominated by *Bacillus*, *Ochrobactrum* and *Pseudomonas*. The study can be concluded to scale up the process for generation of power and the treatment of dye processing wastewater.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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