

Research Article

Electrochemical Corrosion Prevention in Oilfield Wastewater for Effective Dissolved Oxygen Removal Using a Novel Upflow Bioelectrochemical System

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Towards the corrosion issues of oilfield wastewater for water recycling, the dissolved oxygen (DO) is a subsequent corrosive factor after the air desulfurization tower for high-efficiency removal of sulfides. However, an in situ biological technology for efficient DO removal has not been well developed by using organics in oilfield wastewater. A novel upflow bioelectrocatalytic system assembled with three electrodes (cathode-anode-cathode) was designed in this study, in which waste organic matter of oil wastewater was degraded by a bioanode for electron production and dissolved oxygen was efficiently reduced by a biocathode under an assistant external voltage. The results showed that the average current was kept over 6 mA by applying a fixed voltage of 0.8 V to treat oil wastewater with DO as high as 3–5 mg/L. The bottom cathode contributed the largest to DO removal rate, reaching 67%; contribution of the middle anode and the upper cathode for DO removal was 11% and 9%, respectively. The whole DO removal rate by the bioelectrocatalytic system was up to about 90%, and the effluent DO was reduced to below 0.6 mg/L by removing 40–50% COD.

1. Introduction

At present, for equipment corrosion, pipeline blockage, filter material pollution, and other hazards caused by sulfide in the oilfield system, the air desulfurization tower with high treatment efficiency, low operating cost, and convenient operation is usually used to remove sulfides, but desulfurization produces water with high dissolved oxygen (DO) and causes complex and rapid chemical, electrochemical, and biological corrosion of the pipeline [1]. At present, the dissolved oxygen through multistage filtration and deoxygenation in the oilfield still reaches 0.5–0.8 mg/L, which is much higher than the 0.05 mg/L refill water standard requirement [2]. The trace amount of dissolved oxygen causes serious chemical corrosion on water injection pipeline equipment and casing for a long time, and it increases the corrosion rate under high oxygen concentration on the surface of steel in sediments [3]. In addition, dissolved oxygen oxidizes residual organic matter in the crude oil and forms a fine precipitation, which leads to the reduction of oil pores

and reduces the oil recovery rate. At the same time, the metal oxide of the metal tube wall also causes blockage of the formation and has a great influence on the permeability [4]. Experiments have shown that the dissolved oxygen in water oxidizes the polymer, making the polymer chain shorter and less viscous, which results in oil displacement [5]. To achieve efficient DO removal before treated wastewater is reinjected, oxygen scavengers are used as chemical agents, like sulphite and hydrazine hydrate. However, an in situ biological technology for efficient DO removal has not been well developed by using organics in oilfield wastewater.

Microbial electrochemical system (MES) refers to an electrochemical system in which an antimicrobial (anode) and/or reduction (cathode) reaction is carried out on an electrode [6]. Driven by the electrode potential, the bioanode in the microbial electrochemical system can efficiently oxidize organic matter, produce reducing electrons, and directly or indirectly carry out electron transfer and metabolic reaction with the electrode. Compared with the microbial fuel cell (MFC) using air cathode to reduce oxygen [7, 8], MES with an

external voltage can enrich extracellular electron transfer bacteria [9] and perform more efficient cathodic reduction to various reductive compounds (oxidants, organic azo bonds, nitrobenzene, etc.) [10, 11]. The bioelectrodes are a kind of compound extracellular electron transfer microorganism with a sustainable and efficient biocatalytic reaction system [12, 13]. In addition, relevant research reports have also found functional bacteria with special conditions such as salt tolerance and pressure resistance in microbial electrochemical systems. For example, Parot et al. isolated *Halomonas aquamarina* and *Roseobacter* from seawater-enriched negative biofilms and showed oxygen reduction catalytic activity [14]. It has been found that iron porphyrin adsorption mediates oxygen reduction on the surface of the electrode.

Based on the principle and structure of the microbial electrochemical system [15, 16], this study will build an efficient microbial electrochemical process to enrich and remove oxygen in accordance with the characteristics of oilfield wastewater. A three-electrode (cathode-anode-cathode) upwelling oilfield wastewater microbial electrocatalytic deaeration reactor will be adopted. Under a fixed external voltage, the brush anode is acclimated and enriched with a large number of extracellular electron-transporting bacteria using organics in oil wastewater. The downside biocathode rapidly consumes dissolved oxygen through electronic reduction, providing an anaerobic environment for the subsequent bioanode and biocathode. The bioanode facultative anaerobic microorganism can further consume residual dissolved oxygen in the water and increase oxygen removal rate. The research provides a new insight to develop an efficient biological technology to achieve simultaneous removal of residual organic matter and dissolved oxygen for oil wastewater.

2. Materials and Methods

2.1. Bioreactor Setup and Start-Up. The experimental device was a membrane-less three-electrode (cathode-anode-cathode) upflow oilfield sewage microelectrocatalytic deoxygenation reactor with an inner diameter of 5 cm and a height of 100 cm and an effective volume of 2 L (see Figure 1). The anode was made of carbon brush electrode (carbon fiber; radius 2.5 cm; length 20 cm) and the cathode was made of nickel foam (Ni > 99.8%) (thickness 5 mm; length 20 cm; porosity 95%; specific surface area 0.9 m²/g) [17]. The anode and cathode were fixed in the middle of each cylinder accordingly through titanium wire on a plastic supporter, where the distance was around 5 cm between the neighbouring electrodes. A 0.8 V auxiliary DC voltage was applied between the anode and the cathode of the reactor to regulate the electrode potential. Wastewater is poured from the bottom inlet through the two levels of cathode and anode, where the top adopts the classic three-phase separator design. To maintain suspended communities during continuous flow operation, overflow was controlled by a lacinate downflow weir. Usually, a splitter plate is used in the middle position of the reactor, which can lead to an even flow type with good mixture through the long distance in cylinder reactors.

The reactor was started with the biochemical pool effluent from the domestic sewage treatment plant as the inoculum source, and the bioelectrode film growth and domestication were carried out with the acetate as the domesticated carbon source. The domestication of the inlet is shown in Table 1. The volume ratio of inoculated wastewater and acclimated influent water was 1:1, and it was cultured for 24 h at room temperature using the sequential batch domestication method. The inoculum and the domesticated influent mixture were replaced twice. Observed by the circuit current data collector, the steady growth of the current indicated that the bioelectrode was domesticated successfully. Continuous flow water was used for continuous and stable operation for 1 week with hydraulic retention time (HRT) of 6 h. The change of current data was monitored by an automatic data recorder in real time. The current change can reflect the working state of microbial electrocatalysis in real time [18]. The stable operation process can be replaced by the actual oilfield wastewater for deoxygenation debugging and operation, which was kept running for 2 months (HRT 6 h).

Vitamin fluid components [19] are biotin 2.0 mg, folic acid 2.0 mg, vitamin B6 10.0 mg, thiamine hydrochloride 5.0 mg, riboflavin 5.0 mg, niacin 5.0 mg, calcium pantothenate 5.0 mg, vitamin B12 0.1 mg, right aminobenzoic acid 5.0 mg, lipoic acid 5.0 mg, and deionized water 1.0 L.

Trace element liquid components [20] are C₆H₉NO₆ (nitrilotriacetic acid) 1.5 g, MgSO₄·7H₂O 3.0 g, CaCl₂ 0.1 g, MnSO₄·H₂O 0.5 g, NaCl 1.0 g, Na₂MoO₄·2H₂O 0.01 g, ZnSO₄·7H₂O 0.1 g, FeSO₄·7H₂O 0.1 g, CuSO₄·5H₂O 0.01 g, CoCl₂·6H₂O 0.1 g, H₃BO₃ 0.01 g, KAl(SO₄)₂·12H₂O 0.01 g, NiCl₂·6H₂O 24.0 mg, Na₂WO₄ 25.0 mg, and 1.0 L of deionized water.

2.2. Oilfield Wastewater. Oilfield wastewater was analyzed: COD 1000–1500 mg/L, including small molecule volatile acid 500–600 mg/L, macromolecular petroleum hydrocarbons 300–400 mg/L, ammonia nitrogen 6.57 mg/L, sulfate 565.6 mg/L, dissolved oxygen content 4–5 mg/L, conductivity 18.9 mS/cm, and pH 8.5–9.1. The treatment water samples are collected from the two-stage sampling ports for analysis, and the collection area is connected with a three-way valve to prevent oxygen entering.

2.3. Measurement and Calculation Methods

2.3.1. Main Parameter Measurement. The pH, temperature, and DO were measured using a portable integrated pH meter; the water sample ATP microbial activity was determined by an ATP instrument (GloMax 20/20 Turner Biosystems, Sunnyvale, CA, USA); a data logger (Model 2700; Keithley) was used. Instruments for real-time monitoring and recording of microbial electron transfer processes to obtain bioelectrode potential and electron transfer quantity information were used.

2.3.2. COD Removal Calculation. The COD removal efficiency is an indispensable performance indicator for water

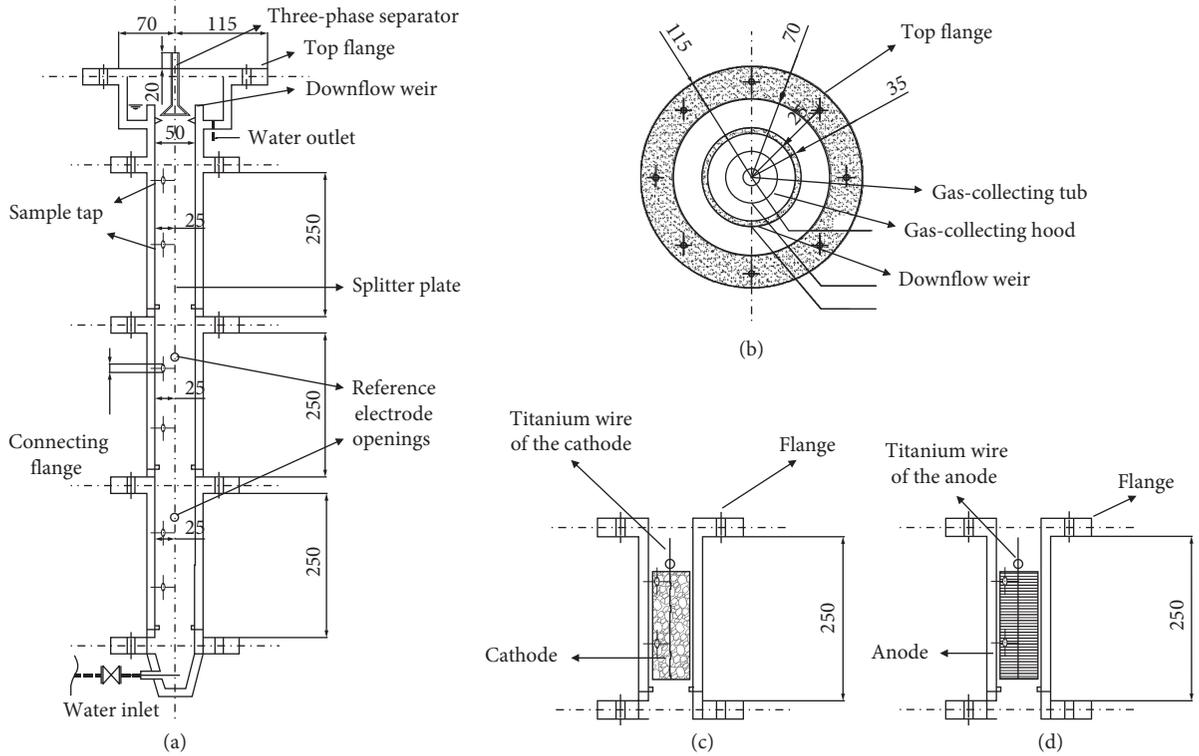


FIGURE 1: Three-electrode (cathode-anode-cathode) upflow bioelectrochemical deoxygenation reactor: (a) the reactor design; (b) the top outlet weir design; (c) the cathode design; (d) the anode design.

TABLE 1: Formula of inlet water for reactor start-up.

Substance	Acetate	Disodium hydrogen phosphate dodecahydrate	Sodium dihydrogen phosphate dihydrate	Ammonium chloride	Potassium chloride	Vitamin	Trace element
Concentration	1.50 g/L	11.55 g/L	2.77 g/L	0.31 g/L	0.13 g/L	1 mL/L	1 mL/L

treatment equipment and the most intuitive indicator. The calculation formula is as follows:

$$\text{COD}_R = \frac{\text{COD}_{\text{in}} - \text{COD}_{\text{out}}}{\text{COD}_{\text{in}}} \times 100\%, \quad (1)$$

where COD_{in} is the chemical oxygen demand of the influent, mg/L; COD_{out} is the chemical oxygen demand of the effluent, mg/L; and COD_R is the COD removal efficiency, % [21].

2.3.3. Coulomb Efficiency Calculation. Coulombic efficiency (CE) is the ratio of the actual amount of electricity (Q) generated by the anodic microbial oxidation of organic matter to the circuit and the theoretical amount of electricity generated by the substrate's own oxidation (Q_T). The electron recovery efficiency [22] is calculated as follows:

$$\text{CE} = \frac{Q}{Q_T} \times 100\%. \quad (2)$$

When acetate is used as the substrate for the reactor, the efficiency of the system can be calculated by the following formula:

The current from the positive level to circuit is calculated according to the actual current:

$$Q = It = \int i \cdot dt. \quad (3)$$

The total amount of acetate is converted into the coulomb number of electrons, that is, the theoretical amount of electricity:

$$Q_T = \frac{F \cdot b \cdot m}{M}, \quad (4)$$

where F is the Faraday constant, $96485 \text{ C} \cdot \text{mol}^{-1}$; b is the amount of electrons lost by 1 mol of substrate conversion (acetate $b = 8$); m is the mass of substrate consumed, g; M is the molar of the substrate quality, g/mol.

In the process of treating organic wastewater, due to the complexity of the composition of organic wastewater, it is impossible to determine the specific reaction substance that will react. Therefore, the chemical oxygen demand (COD) consumed by the substrate is generally used to calculate Q_T , so Q (the calculation formula of TF106) is improved as follows:

$$Q_T = F \cdot b \cdot (\text{COD}_{\text{in}} - \text{COD}_{\text{out}}) \cdot \frac{V}{M_{\text{O}_2}}, \quad (5)$$

where COD_{in} is the chemical oxygen demand of the substrate at the beginning of the reaction, mg/L; COD_{out} is the

chemical oxygen demand of the substrate after the end of the reaction, mg/L; V is the volume of the reaction solution, L; M_{O_2} is the molar mass of organic matter based on oxygen, 32 g/mol; and b is the number of electrons transferred by oxidation of 1 mol of organic matter with oxygen as the standard, $b = 4$.

3. Results and Discussion

3.1. Start and Run of Three-Electrode Biological Catalytic Deoxygenation Reactor. In order to achieve rapid start-up of the reactor, the carbon source in the anode adopts the acetate which is most suitable for the growth of extracellular electron-transporting bacteria, and its water intake concentration was about 1026.67 ± 106.93 mg/L. After the start-up, the reactor was after 6 h residence time. The COD concentration of the effluent was about 613.33 ± 119.3 mg/L, and the removal rate was $40.65 \pm 5.8\%$ (see Figure 2), indicating that the biofilm was basically formed during this stage. According to the amount of COD utilized and the amount of extracellular electron transport, the Coulombic efficiency of the growth process of the extracellular electron-transporting bacteria of the anode biofilm was calculated to be 5%. The coulomb efficiency of the positive level indicates that the acetic acid salt was passed on to the positive level through the respiration of microorganisms, and the electrons generated by oxidation are transferred to the anode through the extracellular electron transport system of the microorganism. Therefore, the Coulombic efficiency indicated that the anode had successfully domesticated the extracellular electron-transporting bacteria.

The acclimated influent water was used under the sequencing batch condition. The average current of the reactor can be stable above 6 ± 0.25 mA, and the positive potential was mainly stable at -500 ± 50 mV after successful start-up (Ag/AgCl was the reference electrode) (see Figure 3). The potential of the anode reflected the ability of the microorganism to carry out extracellular electrons transfer. At this potential, the anode had the highest microbial activity and can effectively oxidize the organic matter for extracellular electron transfer [19, 23]. At the same time, the potential also indicated that the dissolved oxygen in the medium environment near the anode had been exhausted [24].

3.2. Removal Efficiency of Dissolved Oxygen in the Oilfield Wastewater

3.2.1. Removal Contribution of Every Electrode on Dissolved Oxygen. In order to specifically study the oxygen-scavenging capacity of each electrode unit, the dissolved oxygen was segmentally monitored using a mobile dissolved oxygen meter. The content of dissolved oxygen in the water of the oilfield was about 5 ± 0.25 mg/L. After passing through the first stage of the reactor (bottom of the reactor), the dissolved oxygen dropped to about 1-2 mg/L, passing through the anode area of the reactor (in the middle of the reactor). The dissolved oxygen reached about 1 mg/L, reaching the second stage cathode (above the reactor), and the dissolved oxygen drop to 0.58 ± 0.11 mg/L.

Therefore, the dissolved oxygen concentration of the effluent of the reactor reached 0.6 mg/L under the condition that the influent dissolved oxygen concentration was close to 5 mg/L (see Figure 4). In addition, the main dissolved oxygen removal occurred at the cathode, and the dissolved oxygen removal effect on the anode was the lowest, and the overall dissolved oxygen removal rate was close to $88.35 \pm 2\%$.

The ATP activity was used to directly measure the microbial activity in the solution. The results showed that the microbial activity in the reactor was similar at 1.79 mg/L when the water was oilfield wastewater; according to the number of ATP contained in a single cell (1.75×10^{-10} nmol ATP/cell), the result was converted into the number of cells and the number of cells in the reactor solution was about 7.13×10^8 . Compared with the number of microbial cells (10^{11}) when using acetate as a substrate, the difference was relatively large, and the difference was close to two orders of magnitude, as shown in Figure 5. Therefore, the activity of microorganisms was higher when acetate was used as a substrate, and the microbial activity of the anode and cathode was rapidly decreased when it was replaced with oilfield sewage.

3.2.2. Removal Efficiency of Dissolved Oxygen. Through the operation of a single oxygen-scavenging reactor, it was found that the dissolved oxygen concentration in the single reactor was less than 1 mg/L under the condition of dissolved oxygen concentration of 5 ± 0.25 mg/L, which was far below the 0.02 mg/L effluent compliance requirement. However, the actual oilfield wastewater had certain impact on the activity of the bioelectrode. Therefore, for the deoxidation experiment of the oilfield wastewater as the carbon source mode, the two reactors were connected to form a series process of two sets of bioelectrochemical deaerator. The influent water of the reactor was selected separately, and the positions of 1, 3, and 4 and the water outlet were dissolved oxygen-monitoring sites. The main results are shown in Figure 6.

After 40 days of continuous operation, the dissolved oxygen concentrations at positions 1, 3, and 4 decreased to 0.04–0.16 mg/L, 0.03–0.13 mg/L, and 0.01–0.08 mg/L, respectively, indicating that the first cathode (No. 1) was still the main area for deoxidation, while the late position 4 indicated that the second cathode can effectively ensure a residual DO removal. After the first stage of the process, the DO concentration obtained at position 3 was very close to the minimal level, so the second stage process was essential for the effluent dissolved oxygen to reach the standard.

As shown in Figure 7, the reactor was switched from the acetate medium to the actual oilfield wastewater in the first 5 days, and the oxygen removal capacity was affected. After the adaptation, the oxygen removal capacity of the reactor was gradually increased, the dissolved oxygen concentration in the influent water was maintained at the level of 5 ± 0.25 mg/L, the dissolved oxygen concentration of the effluent gradually decreased with the operation of the reactor, and finally reached 0.02 ± 0.02 mg/L of effluent (day 54), where the dissolved oxygen removal rate was almost 100%.

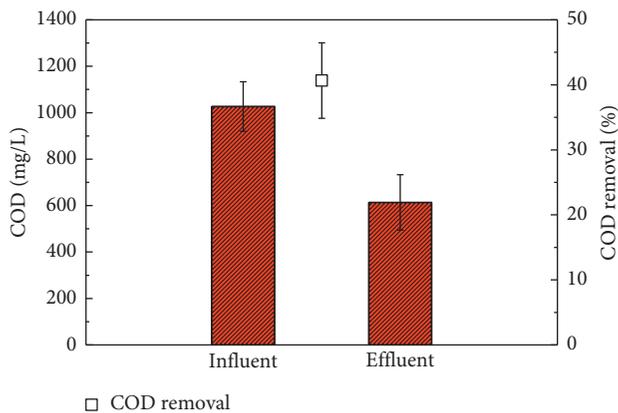


FIGURE 2: COD of the influent and effluent and COD removal rate of the effluent.

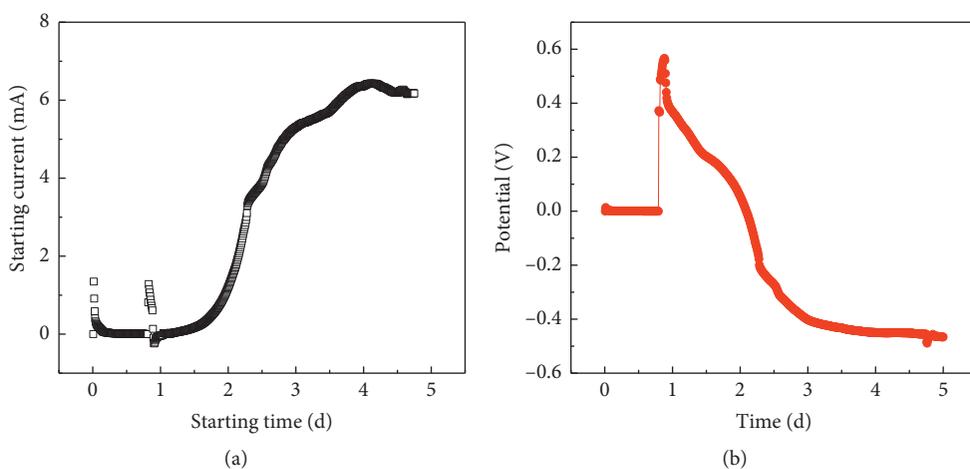


FIGURE 3: Starting current and anode potential versus time.

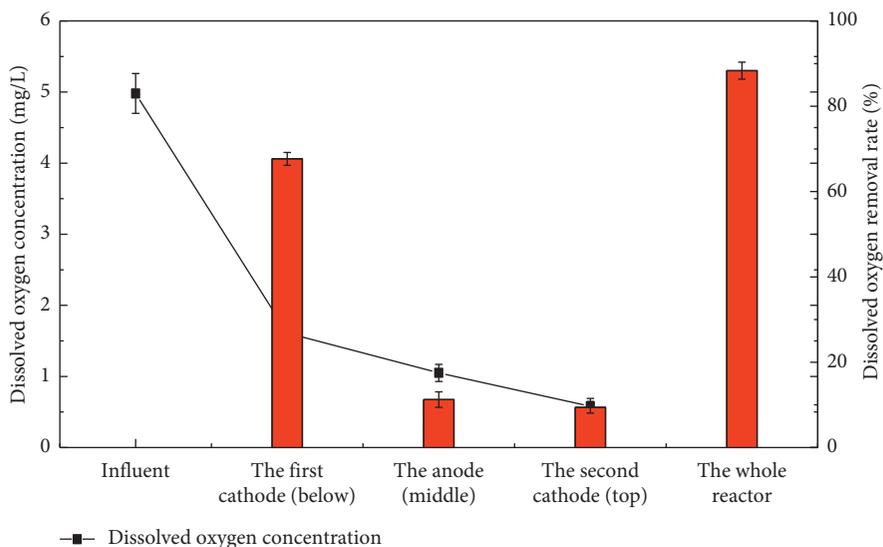


FIGURE 4: Dissolved oxygen concentration changes and removal rates at different effluent locations.

The DO removal methods for oil reinjection water are mainly divided into physical removal and chemical removal in common. The physical methods mainly include thermal

deaeration and vacuum deaeration. The thermal deaeration energy consumption is large and the operation is difficult, which cannot meet the requirements of deaeration in the

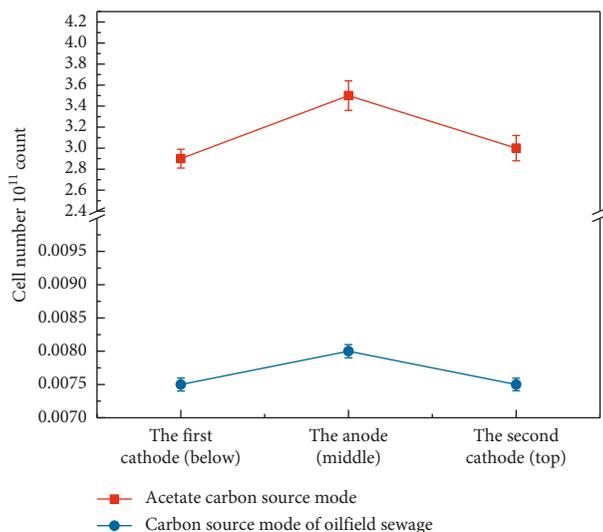


FIGURE 5: Microbial activity (number of cells).

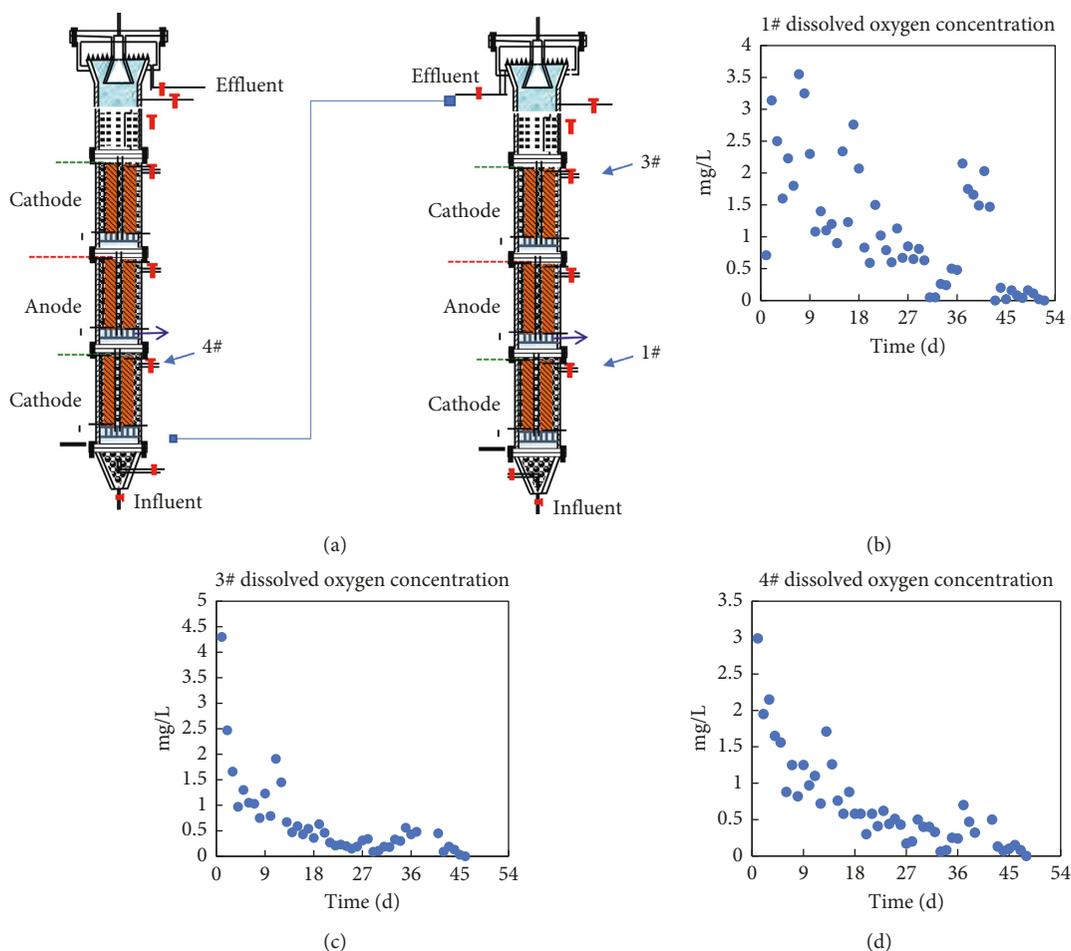


FIGURE 6: Reactor oxygenation and dissolved oxygen concentration at different effluent locations.

oilfield. The equipment of vacuum deaeration needs to be installed on-site and is too bulky to be regulated easily. Physical methods are seldom to be used at established sewage treatment stations. Chemical deaeration is mainly by adding oxygen scavengers, such as sodium sulfite (Na_2SO_3)

and hydrazine hydrate ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$). The latter is expensive and highly toxic, while Na_2SO_3 is less efficient and converted to sulfate in the pipeline system. The current study showed that an in situ biological technology for efficient DO removal can be developed by using organics in oilfield wastewater.

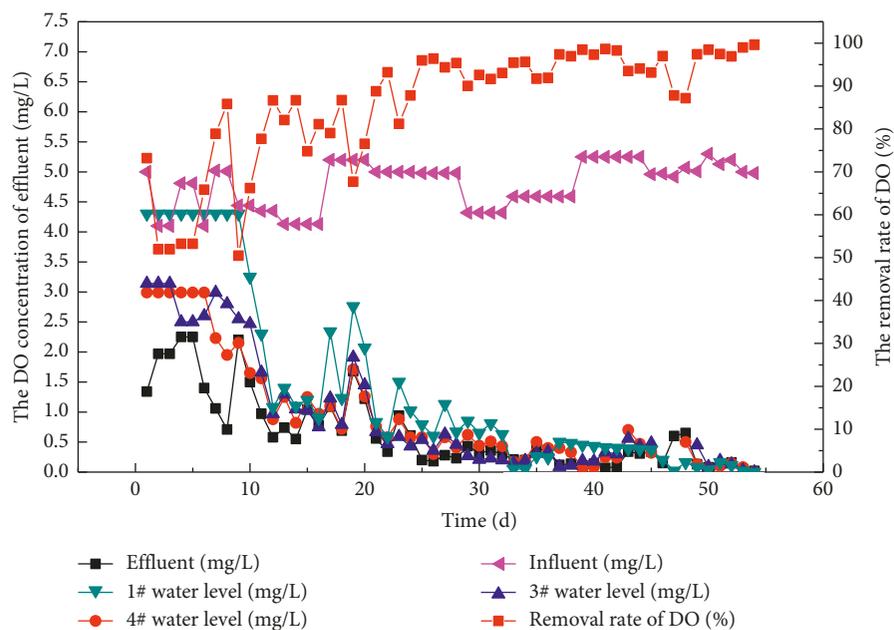


FIGURE 7: Curve of dissolved oxygen concentration and dissolved oxygen removal efficiency with time in different water outlets.

Through the construction of a novel three-electrode (cathode-anode-cathode) upwelling oilfield wastewater microbial electrocatalytic deaeration reactor, the residual organic matter in the oilfield wastewater is further oxidized into carbon dioxide by anode-respiring microorganisms (ARB), and the dissociative electrons are transferred to the cathode through the effect of extracellular electron transfer, which are used for the oxygen donor; therefore, the oxygen is reduced to water, so as to achieve the synchronous removal of oxygen and residual organic matter in the oilfield wastewater.

The biocathode is the key to achieving rapid oxygen removal. Cathode is one of the main factors restricting bioelectrocatalysis. Cathode can use oxygen as an electron acceptor and electron transport medium. From the perspective of oxygen reduction kinetics, the oxygen reduction rate directly affects the oxygen-scavenging efficiency of the reactor. By adding various catalysts to the cathode to increase the reduction rate of oxygen, the catalyst or electron conductor used has Pt, transition metal elements, ferricyanide, etc., but there are problems such as high cost, poor stability, and easy catalyst contamination [25]. The biocathode utilizes an enzyme having a specific function in the microorganism as a catalyst to reduce operating cost, improve reactor performance, and utilize microorganism metabolism to remove various contaminants in the water [26]. The biocathode can directly take oxygen as an electron acceptor. The microorganism directly transfers electrons to oxygen for oxygen reduction; in addition, oxygen can be indirectly used as an electron acceptor, that is, the microorganism utilizes a metal oxide or a high-valent iron salt (such as dioxide). Microorganisms use the reduction of metal oxides or high-valent iron to achieve the transfer of electrons to oxygen [27].

The main factors affecting the bioelectrocatalytic deoxygenation efficiency of oxygen as electron acceptor include reactor configuration, reactor volume, and operating

conditions [28]. Different configurations of bioelectrocatalytic reactors will affect the transport channels (i.e., membrane area) of protons inside the reactor, affecting the distance of proton transfer and the transfer of protons; different reactor volumes will have a certain effect on oxygen removal. As the reactor volume increases, some dead zones and short-flow conditions occur in the reactor; meanwhile, large size of the reactor also increases the distance of proton transfer, thereby limiting the oxygen removal efficiency.

4. Conclusions

- (1) Using the specified formula to enter the water, the three-electrode upflow reactor was successfully started within 5 days, the average current was stable above 6 ± 0.25 mA, and the anode potential was mainly stabilized at about -500 ± 50 mV. At this potential, the anode had the highest microbial activity. The anode can effectively oxidize organic matter and carry out extracellular electron transfer.
- (2) Under the acetate carbon source mode, the downside cathode contributed the most to the dissolved oxygen removal rate, and the dissolved oxygen concentration of the effluent at the influent dissolved oxygen concentration was close to 5 ± 0.25 mg/L. It reached 0.58 ± 0.11 mg/L and the removal rate was about $88.35 \pm 2\%$.
- (3) Two three-electrode upflow reactors were connected to treat oilfield wastewater and achieved a stable bioelectrochemical deoxygenation process. The dissolved oxygen concentration of the influent was maintained 5 ± 0.25 mg/L, and the final dissolved oxygen concentration can reach 0.02 ± 0.02 mg/L. All DO was almost removed, which meets the requirements of the refilling water standard.

Data Availability

All data were generated during the in-site measurement. Raw data sharing is not applicable to this article as data were analyzed using EXCEL or ORIGIN in this study. The data (figure) used to support the findings of this study are included within the supplementary information file.

Conflicts of Interest

The author declares there are no conflicts of interest.

Acknowledgments

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Supplementary Materials

The membrane-less three-electrode (cathode-anode-cathode) upflow reactor for oilfield sewage microelectrocatalytic deoxygenation. The reactor was assembled with an inner diameter of 5 cm and a height of 100 cm and an effective volume of 2 L. The anode was made of the carbon brush electrode (carbon fiber: radius 2.5 cm; length 20 cm) and the cathode was made of nickel foam (Ni > 99.8%). (*Supplementary Materials*)

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