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Azo dye wastewater treatment in a bioelectrochemical-aerobic integrated system: effect of initial azo dye concentration and aerobic sludge concentration

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ABSTRACT

An integrated system that consisted of a bioelectrochemical reactor (BER) and an aerobic completely stirred tank reactor (ACSTR) was constructed to treat simulated wastewater mixed with azo dye Acid Orange 7 (AO7) and real municipal wastewater. It was found that AO7 could be efficiently decolorized in BER within 6 h while initial concentration fluctuated from 25 to 100 mg/L. With an initial AO7 concentration of 75 mg/L, BER reached a decolorization efficiency of 93.65±2.45% at 6 h and contributed 26.56±8.91% of chemical oxygen demand (COD) removal. Coulombic efficiency (CE) of current generation varied from 23.19±6.96 to 37.23±3.48% and CE for AO7 decolorization decreased from 30.45±3.19 to 14.59±1.21% at the initial AO7 concentration of 50 mg/L, which illustrated the important contribution of electrochemical reduction on AO7 decolorization. The effluent of BERs can be subsequently refined by ACSTR, the residual COD was 72±10 mg/L after 6 h treatment with an activated sludge concentration of 6000 mg/L. Further increasing the sludge concentration to 9000 mg/L, final COD concentration decreased to 62±7 mg/L. Results showed that the bioelectrochemical-aerobic integrated system effectively treated the mixed azo dye containing wastewater.

Keywords: Azo dye wastewater; Electrochemical-aerobic integrated system; Initial azo dye concentration; Coulombic efficiency; Sludge concentration

1. Introduction

Nowadays, in order to achieve economies of scale and control pollution, printing and dyeing industrial enterprises are encouraged to cluster together in some industrial parks by government policies [1]. In these parks, enterprises are forced to pretreat the dyeing wastewater on site before it is discharged into centralized wastewater treatment plants (WWTPs). Meanwhile a certain amount of municipal wastewater is discharged into WWTPs as well. These mixed wastewater (consisted of pretreated dyeing wastewater and municipal wastewater) are characterized by low biodegradability, high toxicity and large discharging amount [2]. Conventional treatment methods are unqualified for mixed wastewater treatment due to their low efficiency and time-consuming nature, and thus there is an urgent need to develop proper technologies to deal with the ubiquitous mixed dyeing wastewater [3].

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Azo dye is the most widely used synthetic dyestuff and contains one or more azo bonds (-N=N-) [4]. Unfortunately, the utilization of azo dye is inefficient during the dying process, around 10% of which are unemployed and discharged in the wastewater [5]. Special structural features provide azo dye stability in the aerobic conditions, while the azo bonds could be cleaved under anaerobic conditions and resulting in decolorization. On contrary, the decolorization products (aromatic amine) are stable under anaerobic conditions but require aerobic environment for further mineralization [6]. Therefore, it is necessary to build an integrated system that consists of both anaerobic and aerobic reactors to fulfill the strict discharging standards for azo dye containing wastewater [7,8]. Due to the slow reaction kinetics of anaerobes compared to aerobes, the anaerobic decolorization has been considered as the limit step during the entire dye wastewater treatment period. However, conventional anaerobic treatments frequently suffered from insufficient electron donors and low decolorization efficiency.

Currently, bioelectrochemical technologies are widely employed in recalcitrant wastewater treatment, such as decolorization [9], dehalogenation [10], nitro compounds and heavy metals reduction [11,12], etc., due to the directed and efficient electron utilization [13,14]. In a bioelectrochemical reactor, organic matter is oxidized by anode respiration bacteria with electrons transfer to the anode and further to the cathode for driving the decolorization [15]. Implementation of bioelectrochemical technology could led to superior contaminants transform efficiency and much lower electron donor requirement, which may benefit efficient removal of azo dye from mixed wastewater. Previous reports explored the feasibility of bioelectrochemical technologies used for azo dye decolorization [16,17], few of them considered the following aerobic treatments [18,19]. To date, comprehensive studies on the overall integrated systems for in simulated real wastewater treatment have not been revealed yet.

In this work, a bioelectrochemical reactor (BER) and aerobic completely stirred tank aerobic reactor (ACSTR) integrated system was developed for mixed azo dye wastewater treatment and some important influencing factors on the performance of BER-ACSTR integrated system were studied. Model azo dye (AO7) dissolved in real municipal wastewater was employed to simulate practical mixed wastewater. The influence of initial azo dye concentration on the decolorization performance of BER was investigated. To identify the contribution of electrochemical reaction in BER, different operation modes were conducted and systematic electrochemical performance was analyzed. The performance of ACSTR in further chemical oxygen demand (COD) removal was studied and the effect of activated sludge concentration was evaluated.

2. Materials and methods

2.1. Reactor configuration

BER designed for this study was made of glass with a working volume of 0.75 L [4]. Graphite fiber brushes (L 5 cm × Φ 5 cm, TOHO TENAX, Co., Ltd., Japan) were used as both anode and cathode electrodes. All graphite fiber brushes were pretreated according to the literature [20]. Titanium wires (1 mm in diameter, Baoji LiXing Titanium Group Co., Ltd., China) were stretched out of the reactors and connected with the circuit. During the experiment, each reactor was operated with an external resistance of 10 Ω and an applied voltage of 0.5 V using a DC power supply (FDPS-180, Fudan Tianxin Scientific and Educational Instruments Co., Ltd, Shanghai, China). A saturated calomel electrode (SCE) (+247 mV vs. standard hydrogen electrode) was used as reference electrode. Anodes, cathodes and reference electrodes were connected to a data acquisition system (Keithley 2700, Keithley Co. Ltd., U.S.). Electrode potentials and currents were recorded at every 10 min. Beaker flask (0.5 L of empty volume) was used as aerobic completely stirred tank reactor (ACSTR) and an aerator was placed on the bottom for air supply to the reactor. Each reactor was equipped with a magnetic rotor and placed on a six-position magnetic stirrer (84-1, Shanghai Meiyingpu Instruments Co., Ltd., China) for obtaining a homogeneous mixture.

Both BERs and ACSTRs were operated in batch mode, each cycle was consisted of 6 h reaction, 0.5 h sediment and 0.5 h medium replacement. The medium of BERs was completely replaced by fresh medium. Approximate half of the supernatant was eliminated from ACSTRs and replaced by BERs effluent. The schematic diagram of the integrated BER and ACSTR system integrated is shown in Fig. 1.

2.2. Chemicals

Model azo dye, Acid orange 7 (AO7, purity > 95%) with an azo bond, was employed to evaluate the performance of the integrated system. AO7 was purchased from Shanghai Sangon Biotech Co., Ltd., China. All other chemicals were analytical reagents.

2.3. Startup and operational conditions

The BERs were inoculated with real municipal wastewater (MW), the characteristics of MW are shown in Table 1. Sodium acetate (1000 mg/L) and azo dye (AO7, 25 mg/L) were added as electron donor and acceptor, respectively, to enhance the biofilm formation in the startup stage. When a stable current output was observed, the reactors were considered to be successfully started up. Activated sludge was acquired from Taiping municipal wastewater treatment plant (Harbin, China) and it was used in ACSTRs. Dissolved oxygen (DO) in the ACSTRs was maintained around 2–3 mg/L which was a typical level for activated sludge processes. The mediums for both BERs and ACSTRs were replaced every two days in the startup stage.

During the experiment, sodium acetate was no longer added in the influent and all the electrons donor was the organic matter in the MW. The effect of initial azo dye concentrations (AO7, 25, 50, 75 and 100 mg/L) on decolorization performance in BERs was evaluated, after that, all BERs were operated under open circuit mode to identify the contribution of electrochemical reaction. Then the initial AO7 concentration was set as 75 mg/L, and activated sludge concentrations (mixed liquor suspended solids, MLSS) in ACSTRs were set at 3000, 6000 and 9000 mg/L, respectively, to probe the influence of biomass on COD removal. Each operational condition was run in three parallels and at least three repetitions. Each condition for both BERs and ACSTRs was operated for at least ten batches to

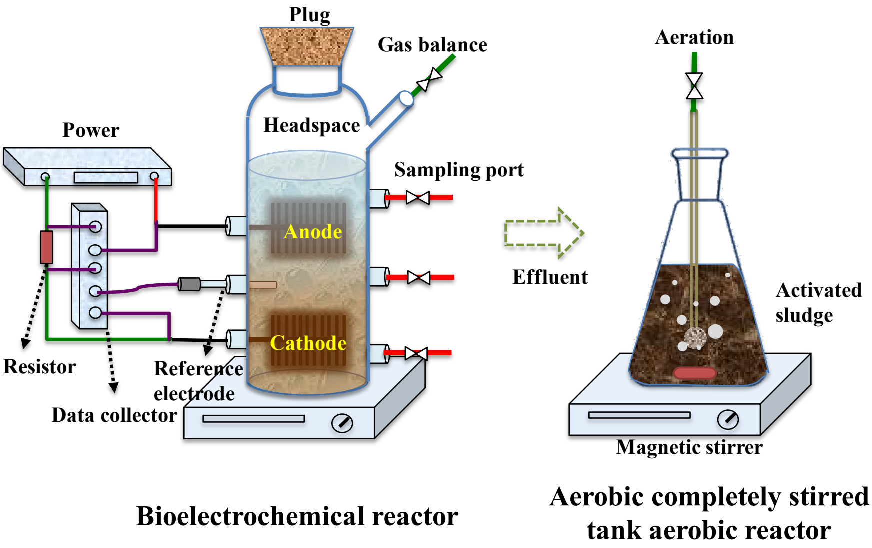

Fig. 1. The schematic representation of the diagram of BER and ACSTR integrated system.

Table 1
Composition and concentration of the municipal wastewater (MW) used in this study

Item	Value
COD, mg/L	254±31
NH_4^+-N, mg/L	46.80±7.15
TN, mg/L	52.78±9.58
TP, mg/L	5.52±0.33
VFAs, mg/L	77.83±11.01
pH	7.30±0.10

achieve stable and reliable results. All reactors were operated at an ambient temperature of 23±2°C.

2.4. Analytical methods and calculations

Liquid samples taken from BERs were immediately filtered through 0.45 μm filters (Tianjin Jinteng Experiment Equipment Co., Ltd., China). Samples taken from the ACSTRs were centrifuged at 3000 rpm for 10 min firstly for sludge deposition. The sludge pellets were returned to the corresponding reactors, and the liquid supernatant was immediately filtered through 0.45 μm filters.

AO7 concentration was quantified by a UV-Vis spectrophotometer (UV-1800, Shanghai Meipuda instrument Co., Ltd., China) at a wavelength of 484 nm. COD was determined using HACH methods. DO was measured by a portable meter (JPBJ-608, INESA Scientific Instrument Co., Ltd., China).

AO7 decolorization efficiency (DE, %) was calculated based on the difference between the AO7 concentrations of influent and effluent samples, as shown in Eq. (1).

$$DE = \frac{C_{in} - C_{eff}}{C_{in}} \times 100\% \tag{1}$$

where C_{in} and C_{eff} are the AO7 concentrations in the influent and sample, separately, mg/L.

COD removal efficiency (η, %) was calculated based on the difference between initial and sampled COD concentration, as shown in Eq. (2).

$$\eta = \frac{C_{in\text{-}COD} - C_{eff\text{-}COD}}{C_{in\text{-}COD}} \times 100\% \tag{2}$$

where $C_{in\text{-}COD}$ and $C_{eff\text{-}COD}$ are the COD concentrations in the influent and sample, separately, m/L.

Electric quantity (mAh) was calculated based on the operating time and the current of external circuit, as shown in Eq. (3).

$$Electric\ quantity = \sum I \times \mathrm{t} \tag{3}$$

where *I* is the current that calculated from the external resistance using Ohm's law, mA; *t* is the operating time, h.

The coulombic efficiency (CE, %) for current generation (CE-current) and AO7 decolorization (CE-AO7) were evaluated according to Eq. (4) and Eq. (5), respectively.

$$CE\text{ - }Current = \frac{I \times t \times 3.6}{4 \times \frac{\Delta COD \times 0.75}{32} \times \frac{F}{1000}} \tag{4}$$

$$CE\text{ - }AO7 = \frac{4 \times \frac{\Delta AO7 \times 0.75}{350.32} \times \frac{F}{1000}}{I \times t \times 3.6} \tag{5}$$

where ΔCOD and ΔAO7 are the change of COD and AO7 concentrations, mg/L; 3.6 is the conversion coefficient between mAh and Coulomb, 1 mAh = 3.6 C; 4 is the number of electrons transferred per mole of COD and azo bond; 0.75 is the volume of the BER, L; 32 and 350.32 are the molar mass of COD and AO7, g/mol; F is Faraday's constant, 96485 C/mol e^-.

3. Results and discussion

3.1. Effect of azo dye concentration on the decolorization in BERs

Performances of AO7 decolorization at varying initial concentrations in BERs are shown in Figs. 2A and 2B. AO7 could be efficiently decolorized in BERs within 6 h while initial concentration fluctuated from 25 to 100 mg/L, the decolorization efficiencies (DEs) were 87.83±4.31 to 93.65±2.45%. AO7 concentration decreased to around 3 mg/L (3.52±0.60 and 3.31±1.26 mg/L) at 4 h when initial concentration of 25 and 50 mg/L and maintained stable till the end of the experiment. The corresponding DEs were 86.11±4.01 and 92.88±5.20% at 4 h. Higher initial AO7 concentration (75 and 100 mg/L) led to higher residual dyes in short period (4 h) that were 20.12±1.86 and 49.15±2.08 mg/L, DEs dropped to 73.33±1.54 and 50.60±1.69%. After other 2 h operation, AO7 concentration decreased to 4.79±1.17 and 10.34±0.94 mg/L, respectively. These results clearly indicated that the BERs efficiently decolorized AO7 in a relative short operating time compared to conventional anaerobic technologies [3]. Sun et al. employed BES to treat azo dye (Alizarin Yellow R) and observed similar decolorization efficiency with acetate as electron donor source compared to the present study [21]. It suggested that complex electron donor source (such as MW) did not obviously affect the decolorization efficiency, which implied the feasibility of applying bioelectrochemical technology in treating practical wastewater.

Initial COD concentration increased with the elevation of AO7 concentration (from 25 to 100 mg/L), as shown in Figs. 2C and 2D. COD removal presented similar trend among all initial AO7 concentrations. COD removals were around 10 mg/L in the first hour and then increased to 30–43 mg/L in the second hour. The corresponding COD removal efficiencies were 2.55±0.80–4.12±1.71% and 8.00±4.97–16.28±3.40% during the first two hours. With 6 h operating, COD removals increased to approximately 80 mg/L, residual COD concentrations were 183±10, 216±25, 250±29 and 290±25 mg/L, at initial AO7 concentration of 25, 50, 75 and 100 mg/L, respectively. AO7 was proved to be removed by azo bond cleavage under bioelectrochemical conditions and generated aromatic amines, sulfanilic acid and 1-amino-2-naphthol [22]. AO7 and the intermediate products accumulated in the anaerobic bulk [23]. Therefore, the COD removal in anaerobic stage was attributed to the oxidation of organic matters from MW. The general COD removal efficiencies were quite low and stable, which were around 23.71±5.12 to 31.45±5.54% at all initial AO7 concentrations.

3.2. Evaluation of electrochemical contribution on azo dye decolorization in BERs

AO7 decolorization in BERs was attributed to both anaerobic and electrochemical reactions. In order to evaluate the electrochemical contribution on AO7 decolorization, the applied voltage on BERs was removed and the reactors were operated under pure anaerobic condition at an initial AO7 concentration of 50 mg/L. It can be seen in Fig. 3A, DEs of BERs under open-circuit condition were obviously lower than those at closed-circuit condition. The DE differences between closed and open circuit condition were continuously increasing in the first 4 h, and gave a lead up

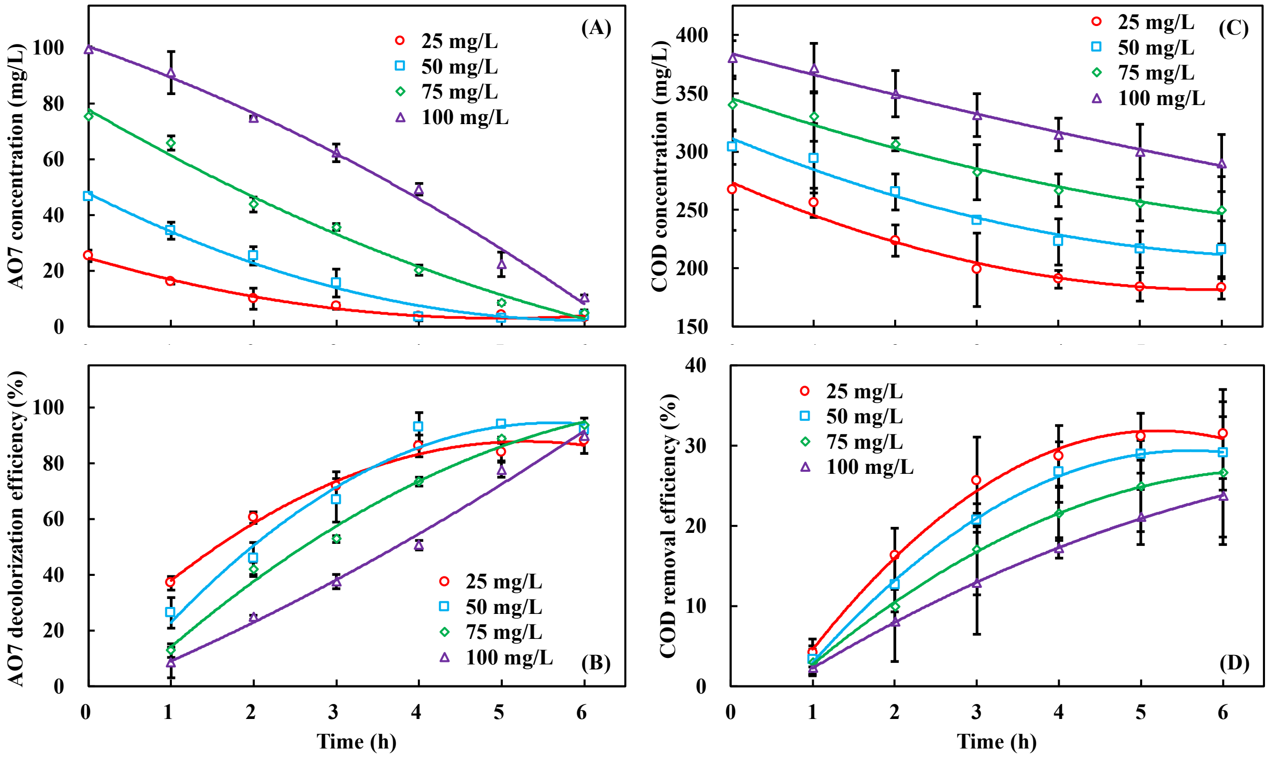

Fig. 2. AO7 concentration (A), decolorization efficiency (B), COD concentration (C) and COD removal efficiency (D) of continuous stirred bioelectrochemical reactors.

to 53%. After 4 h, DEs of closed circuit increased slightly and the DEs of open circuit reactor sustainably raised and narrowed their difference. This phenomenon emphasized the significant contributions of electrochemical reaction on AO7 decolorization.

In conventional anaerobic decolorization, organic matter was degraded to produce electrons to provide necessary reductive environment. The decolorization rate directly depended on the redox conditions of the system. In the BERs, cathode served as a continuous electron donor and the cathode potential was flexible to accelerate the decolorization but not the methanogenesis by adjusting the applied voltage. As a result, the decolorization performance of BERs was superior than conventional anaerobic treatment methods by efficient utilization of electron donors in the carbon sources limited wastewater.

In bioelectrochemical systems, electricigens in anode biofilm oxidized organic matter produce electrons that can be utilized to implement azo bond cleavage in cathode. The electrode potentials (especially the anode potential) were important indexes to assess the electrochemical performance. In this research work, anode potentials gradually decreased from positive value to negative in a short period (less than half an hour) and further decreased to approximate –480 mV (Fig. 4A). As it can be seen from Fig. 3A, most of the AO7 was removed within 3 h and after that AO7 concentration in the BERs was quite low and steady. It was consistent with the current trend that current sharply increased to the maximum (2.54±0.10 mA) at 2.89±1.23 h (Fig. 4B), and rapidly declined an order of magnitude in the following hours. The lag of potential plateau compared to the current peak could be explained as follows: in the first stage, fresh medium supplied adequate electron donors (organic matter in MW) and electron acceptors (AO7), high electron consumption rate in the cathode resulted in a gradual electron accumulation in the anode and presented slow potential decrease. Then the electron consumption decreased due to the reducing AO7 concentration and electrons well accumulated on the anode further decreased the anode potential.

In order to quantitatively analyze the electrochemical contribution in AO7 decolorization of BERs, electric quantity and columbic efficiencies were calculated in Fig. 3B. The accumulated electric quantity gradually increased from 9.23±2.96 to 67.23±10.15 mAh along with the experimental operation, which revealed the active electrochemical reaction in the BERs. The coulombic efficiency of the current generation (CE-Current) was used to probe the ratio of the electrons from organic matters oxidation that would be presented as current flowing across the BERs. CE-Current reached 37.23±3.48% in the first hour and dropped to 23.19±6.96% in the second hour, indicating that a notable portion of electrons were converted to apparent current and this was proved by the current peak emerging in short time (Fig. 4B). After that, CE-Current smoothly increased till the end of the experiment (30.30±3.65%) and due to low AO7 concentration, reduced electron consumption in decolorization and other side reaction occurred. The CE-Current of BERs was comparable to previous reports [20,24], and the main reason for the decrease of CE was the microbes colonizing the anode [25]. Other reactions, such as fermentation and possible methanogenesis, competed for the utilization of available electron donors

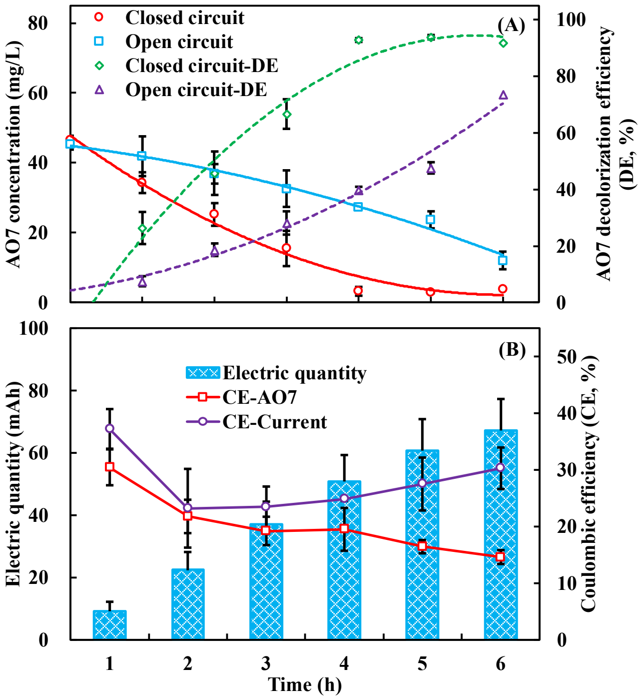

Fig. 3. AO7 concentration and decolorization efficiency (A); electric quantity, AO7 and current coulombic efficiencies (CE-AO7 and CE-current) (B) of continuous stirred bioelectrochemical reactors (BERs) operated at closed and open circuit.

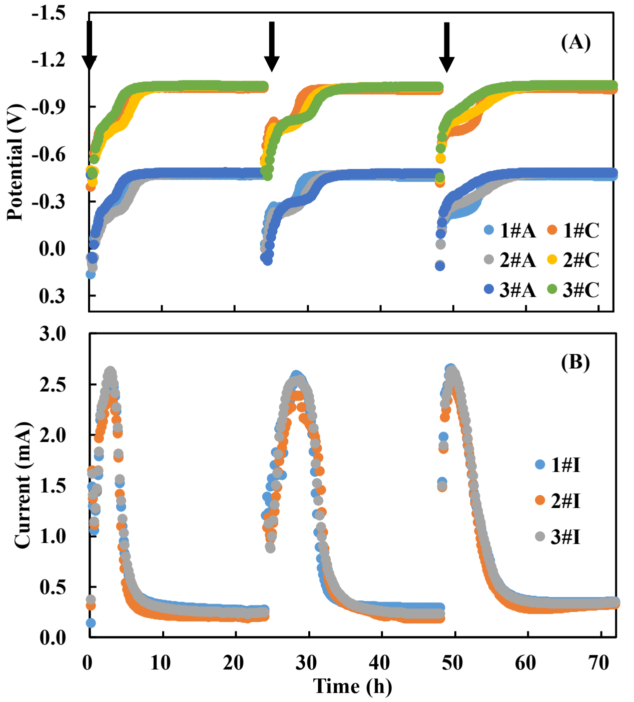

Fig. 4. (A): Anode (#A) and cathode (#C) potentials, bold arrows indicated medium replacement; (B): current of three bioelectrochemical reactors with initial AO7 concentration of 50 mg/L.

(COD) and further decreased the CE for current generation, especially in the single chamber configuration that continuously introducing microbes into the reactor by feeding with MW. Besides, oxygen invasion would be a potential electron sink that competed with the electrode at the anode and even with AO7 at the cathode as electron acceptor, resulting in a reduced CE-current.

The coulombic efficiency of the AO7 reduction (CE-AO7) at the cathode was evaluated as the ratio of the electron that would be obtained as a result of the azo bond cleavage [22]. The CE-AO7 was decreased from 30.45±3.19 to 14.59±1.21% which was due to AO7 concentration varying, implying that the electrochemical reaction was controlled by electron acceptor concentration. These findings substantiated that AO7 decolorization was a result of both electrochemical reaction and biological anaerobic reactions in the BERs. Electrochemical reaction brought noteworthy contribution and did enhance the AO7 decolorization in BERs.

3.3. BER coupled with ACSTR

The colorless reduction products of AO7 accumulated during the anaerobic condition [23]. In order to further stabilize the decolorization and remove COD from BERs effluent, aerobic completely stirred tank reactors (ACSTRs) were employed to subsequently oxidize these reduction products. It can be incurred from Fig. 5A that the COD concentration decreased in ACSTRs under all initial AO7 concentrations, with activated sludge concentration around 6000 mg/L. After a 6 h treatment, final COD concentration was deceased to 59±9, 70±19, 72±10 and 99±12 mg/L, respectively, as the initial AO7 concentrations fluctuated from 25 to 100 mg/L. The corresponding COD removal efficiencies increased to 67.52±4.69, 67.59±4.07, 71.01±8.59 and 65.87±6.37% (Fig. 5B), respectively. When initial AO7 was lower than 75 mg/L, the final effluent COD met the limit (COD ≤ 80 mg/L) of "Discharge standards of water pollutants for dyeing and finishing of textile industry (GB 4287-2012, China)".

Activated sludge concentration was adjusted to 3000 and 9000 mg/L from 6000 mg/L to evaluate the influence of biomass on the COD removal (Fig. 5C) while initial AO7 concertation was fixed at 75 mg/L. Higher sludge concentration resulted in superior COD removal effect at same DO level (2–3 mg/L), final COD concentration decreased from 99±19 to 62±7 mg/L when sludge concentration increased from 3000 to 9000 mg/L, meanwhile, the COD removal efficiency increased from 61.82±6.23 to 75.81±6.39% (Fig. 5D). In the case of initial AO7 concentration of 75 mg/L and activated sludge of 9000 mg/L, total COD removal efficiency over 80%. A previous work that employed an integrated system (up-flow bio catalyzed electrolysis-aerobic bio contact oxidation reactor) presented an effluent COD of 175±25 mg/L which was inefficient for comparison with the latest discharging standards [19]. A bioelectrochemical system integrated with bio-contact oxidation reactor was built to treat AO7 containing wastewater, the decolorization efficiency (94.62±0.63%) and COD removal efficiency (89.12±0.32%) were similar with the present

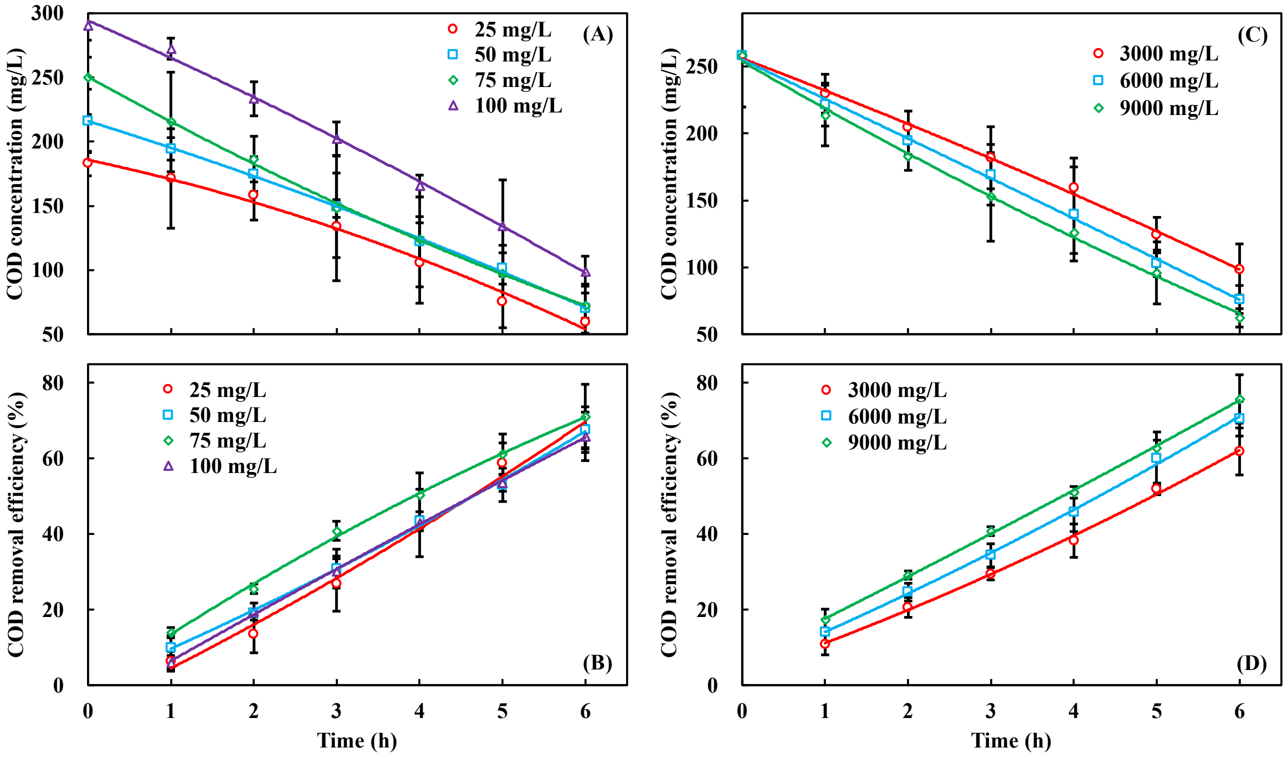

Fig. 5. COD concentration (A) and COD removal efficiency (B) of aerobic completely stirred tank reactor (ACSTRs) at varying initial AO7 concentrations; COD concentration (C) and COD removal efficiency (D) of aerobic completely stirred tank reactor (ACSTRs) at varying sludge concentrations.

study [26]. However, considering the complex real municipal wastewater, the present work would be a practical feasibility to promote bioelectrochemical technology for future applications.

4. Conclusion

An integrated system that consisted of bioelectrochemical reactor (BER) and aerobic completely stirred tank reactor (ACSTR) was proved to be an efficient solution for mixed azo dye wastewater treatment. The real municipal wastewater could be utilized as the electron donor to implement azo dye cleavage in the BER. The bioelectrochemical reduction enhanced the decolorization performance of BER. The subsequent ACSTR was efficient to decrease the total COD of the azo dye wastewater. These findings comprehensively revealed the feasibility and superiority of integrated system and illuminate the practical potential of this technology in mixed wastewater treatment.

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