



Sequential solid entrapment and *in situ* electrolytic alkaline hydrolysis facilitated reagent-free bioelectrochemical treatment of particulate-rich municipal wastewater



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ABSTRACT

We introduce here a novel process for the treatment of particulate-rich wastewater. A two-stage combined treatment process, consisting of an electrolysis filter and a bioelectrochemical system (BES) configuration was designed and evaluated to remove particulate and soluble organic matter from municipal wastewater. The system was designed such that the electrolysis step was used as a filter, enabling physical removal and *in situ* alkaline hydrolysis of the entrapped particulate matter. The alkaline effluent enriched with the hydrolysed soluble compounds (soluble chemical oxygen demand, SCOD) was subsequently loaded into the BES for removal via bioanodic oxidation. The coupled system was continuously operated with a primary sedimentation tank effluent (suspended solids (SS) ~200 mg/L) for over 160 days, during which SCOD and total COD (TCOD), SS removal and current production were evaluated. With no sign of clogging the process was able to capture near 100% of the SS loaded. A high Coulombic efficiency (CE) of 93% (based on overall TCOD removed) was achieved. The results also suggest that the SCOD-laden alkaline liquor from the electrolysis step compensated for the acidification in the bioanode and a final effluent containing low COD with neutral pH was achieved. Overall, since the system can effectively entrap, *in situ* hydrolyse and oxidise organic matter without external dosing of chemicals for pH control, it has desirable features for practical application.

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1. Introduction

Waste stabilisation ponds (WSPs) are commonly used to treat municipal wastewater particularly that of remote communities (Mara et al., 1998). However, maintenance of stable effluent quality of WSPs is challenging and additional treatment processes are often required to minimise discharge of suspended solids (SS) (mostly in form of algal biomass) and dissolved organic carbon (DOC) into the environment (Ellis and Mara, 1983; Mara et al., 1992). Rock filters are one of the widely used downstream polishing technologies designed to entrap and oxidise both particulate matter and DOC

discharged from WSPs. They rely on passive diffusion of oxygen from the atmosphere to facilitate aerobic biological oxidation of the entrapped organic matter. However, excessive biomass growth and particulate matter accumulation can hinder passive aeration and could result in anaerobic conditions in the filter compromising organic matter removal and hydraulic throughput of the treatment unit. Hence, alternative technologies are desirable for efficient treatment of particulate-rich liquor such as those emanating from failed WSPs.

Recently, there has been an effort to develop new technologies to substitute rock filters (Khalfbadam et al., 2016b). Khalfbadam et al. (2016b) proposed a filter-type bioelectrochemical system (BES) termed as “BES filter” to retain and oxidise high particulate matter and soluble chemical oxygen demand (SCOD) effluent. The tubular, dual chamber BES prototype is composed of an outer

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anodic chamber that encircles an inner cathodic chamber and the two chambers are separated with a cation exchange membrane. The outer anodic chamber was designed as a filtration unit, whereby a carbon felt was installed as both a physical filter and a bioanode to entrap and bioelectrochemically oxidise the suspended organic matter from the wastewater. The study successfully demonstrated the ability of the BES filter to oxidise SCOD in wastewater with a high Coulombic efficiency (CE) of >80% (based on the SCOD removed). The study also demonstrated effective removal of organic particulate matter from municipal wastewater (83% of SS removal). However, if the CE calculation was based on the coulombs derived from the entrapped particulates, the CE would be very low (~1%). Such a low CE was most likely due to inefficient microbial hydrolysis of the entrapped particulate matter. Indeed, the system proposed by Khalfbadam et al. (2016b) suffered from the following limitations: (1) inefficient hydrolysis of entrapped particulate matter; (2) the need for active control of pH both in the cathodic and anodic chambers of BES; and (3) a huge overpotential for reduction of oxygen at BES cathode. It is imperative to overcome the above limitations should this innovative design be considered as an effluent polishing technology for WSPs.

Generally, hydrolysis is often the rate-limiting step in biological treatment processes for waste streams rich in organic solids. Various pre-treatment methods (physical, chemical, biological, and electrochemical) have been examined to improve hydrolysis of organic particulate matter. For instance, Khalfbadam et al. (2016a) effectively harnessed hydrolytic bacteria in anaerobic sludge to increase *in situ* oxidation of algal biomass in a BES. Chu et al. (2001) found that ultrasound pre-treatment (sonication at 0.33 Watt/mL, 2 h) significantly increased the SCOD content of a waste activated sludge (WAS) (from 42 to 1084 mg SCOD/L). Alternatively, drastically altering the pH of the feedstock by dosing acids or alkali could also enhance hydrolysis of organic particulate matter (Chen et al., 2007; Huang et al., 2016; Yi et al., 2013). However, these methods require either substantial retrofit of infrastructure or usage of chemicals, and hence may not be desirable for practical application. Recently, Charles et al. (2013) investigated the use of a dual chamber electrochemical cell to enhance anaerobic digestibility of a WAS. With an applied voltage of 12 V, the acidic and alkaline conditions created by the electrolytic reactions in the anodic and cathodic chambers remarkably increased the SCOD in the WAS by 31% and 34%, respectively. Since this method could effectively use electrodes to hydrolyse particulate matter without dosing external chemicals, it may be suitable for addressing the abovementioned limitations in the BES filter process of Khalfbadam et al. (2016a).

In this study, we proposed that by operating the BES filter as an electrolytic cell, alkaline hydrolysis of the entrapped particulate matter could be driven *in situ* at the electrode (filter). This process would create an effluent with elevated SCOD and alkalinity, which could be a suitable feedstock for a subsequent bioelectrochemical (anodic) treatment. Fig. 1A depicts the concept of the proposed two-stage process. In the first stage, the particulate-rich wastewater is fed to the module, whereby the filter-electrode physically removes (filtering) the particulate organic matter from the wastewater. The filter-electrode loaded with particulate matter is then cathodically-driven to create a localised alkaline condition ($\text{pH} \geq 12$), facilitating *in situ* alkaline hydrolysis of the entrapped particulate matter. The elevated SCOD and alkalinity are expected to facilitate current production and help neutralise the acidity liberated from the anodic oxidation reaction at the subsequent stage. Overall, the sequential process is expected to yield a better quality final effluent with a higher total COD removal efficiency.

The aim of this study was to validate the proposed combination of electrolysis and bioelectrochemical anodic oxidation for effective removal of particulate and soluble organic matter from municipal

particulate-rich wastewater. Specifically, the following hypotheses were tested:

- (1) The increased cathodic pH created by the electrolysis step (first stage) can trigger alkaline hydrolysis of the particulate matter entrapped at the filter-electrode;
- (2) The alkaline effluent with elevated SCOD content can be harnessed as a more suitable feedstock for the subsequent anodic carbon oxidation step, negating the need of dosing external chemicals (e.g. NaOH) for pH control (reagent-free);
- (3) The acidity generated at the anodic reaction (captured in the counter electrolyte) of the first stage can be used to facilitate the cathodic reaction of the second stage, alleviating the overall reliance on external chemicals for pH control.

The coupled process was operated in a continuous mode with different hydraulic loadings of municipal primary sedimentation tank (PST) effluent for over 160 days, during which the performance of the system was quantified for a range of parameters such as total COD (TCOD), soluble COD (SCOD), particulate COD (PCOD) and SS removals; electrical current, Coulombic efficiencies and energy consumption.

2. Materials and methods

2.1. Configuration of the two-stage reactor process

Fig. 1 illustrates the experimental process used in this study. The process consisted of two identical, hydraulically connected dual-compartment electrochemical reactors (Fig. 1). The first reactor (R1) was operated as an electrolytic cell facilitating both entrapment and *in situ* alkaline hydrolysis of the entrapped particulate solids; the second reactor (R2) was operated as a typical BES for bioanodic oxidation of soluble organic matter. Specific configurational details of the reactor and the electrode materials have been described in Khalfbadam et al. (2016b). In brief, each reactor had a height of 1.5 m (internal diameter 0.1 m) and consisted of two concentric cylindrical stainless steel mesh columns, one with a diameter of 6 cm and the other one with a diameter of 4 cm. The two columns were served as both electrical current collector and physical support for the anode and the cathode, respectively. To prevent corrosion of the stainless steel columns, the columns were pre-treated with pickling and passivation treatments as per American Society for Testing and Materials (ASTM) standards (ASTM A380 and A967) (International Corrosion Services Ltd. Co., Perth, Australia). The two compartments were isolated by a cation exchange membrane (Ultrex CMI 7000, Membrane International Inc.), which was firmly mounted onto the outer side of the inner stainless steel mesh column and was sealed with epoxy glue to create a water-tight compartment within the module. Carbon felts (MGM-Carbon Industrial, Ltd. Co., China) were used as both the anodic and cathodic electrode materials. The carbon felts were mounted onto the outer side of the larger and inner side of the smaller stainless steel current collectors, respectively. The carbon felt at the larger stainless steel column (i.e. cathode of R1 and anode of R2) had a surface area of 0.33 m². A short distance (approximately 2 cm) was maintained between the anode and the cathode, creating a void volume where the wastewater influent was introduced at the bottom end of the unit. Since the inner column was completely enclosed, the influent wastewater was compelled to channel through the carbon felt facilitating physical removal of SS from the wastewater (Fig. 1C). In this study, it was proposed that the particulate-rich wastewater was first processed in R1 for particulate matter removal and electrolytic alkaline-hydrolysis of the entrapped particulate; the alkaline effluent created by R1 was

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