



Treatment of oil and grease in produced water by a pilot-scale constructed wetland system using biogeochemical processes



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HIGHLIGHTS

- A pilot-scale wetland treatment system was designed and constructed.
- Sequential reducing and oxidizing cells promoted specific biogeochemical processes.
- Metals and oil in simulated oilfield produced water were treated effectively.
- Biogeochemical conditions changed with mass loading of oil, affecting treatment.
- An oil–water separator decreased inflow concentrations of oil.

ARTICLE INFO

Article history:

Received 17 July 2013

Received in revised form 30 October 2013

Accepted 8 November 2013

Available online 8 December 2013

Keywords:

Constructed wetland

Produced water

Oil and grease

Metals

ABSTRACT

Constructed wetland treatment systems (CWTSs) can effectively remove many constituents that limit beneficial use of oilfield produced water. The objectives of this investigation were: (1) to assess the effect of mass loadings of oil and grease (O & G) on treatment performance in pilot-scale subsurface flow and free water surface CWTS series having sequential reducing and oxidizing cells, and (2) to evaluate effects on treatment performance of adding a pilot-scale oil–water separator. Increase in O & G mass loading from 5 to 20 mg min⁻¹ caused decreases in both dissolved oxygen concentration and sediment redox potential, which affected treatment performance. Biogeochemical pathways for removal of O & G, iron, and manganese operate under oxidizing conditions, and removal rate coefficients for these constituents decreased (0.905–0.514 d⁻¹ for O & G, 0.773–0.452 d⁻¹ for iron, and 0.970–0.518 d⁻¹ for manganese) because greater mass loading of O & G promoted reducing conditions. With increased mass loading, removal rate coefficients for nickel and zinc increased from 0.074 to 0.565 d⁻¹ and from 0.196 to 1.08 d⁻¹, respectively. Although the sequential reducing and oxidizing cells in the CWTS were very effective in treating the targeted constituents, an oil–water separator was added prior to wetland cells to enhance O & G removal at high inflow concentration (100 mg L⁻¹). The oil–water separator removed approximately 50% of the O & G, and removal extents and efficiencies approximated those observed at 50 mg L⁻¹ inflow concentration during treatment without an oil–water separator.

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1. Introduction

Oilfield produced water is defined as water brought to the surface as a result of oil production (Veil et al., 2004). Twenty-one billion barrels of produced water were generated in 2007 by onshore and offshore facilities with 87% from oil production activities (Clark and Veil, 2009). Common naturally occurring constituents of concern that limit the use of produced water include organic compounds, anions, cations, nitrogen compounds (nitrate, nitrite, and ammonia), total dissolved solids, and total suspended

solids (Veil et al., 2004; Benko and Drewes, 2008). Simulated oilfield produced water used for this experiment was based on the composition of a produced water originating from an oilfield located in sub-Saharan Africa that contains oil and grease (O & G), Fe, Mn, Ni, and Zn as constituents of concern (Horner et al., 2011a).

Strategies to manage produced water include underground injection, surface discharge, and beneficial use (Veil et al., 2004; Benko and Drewes, 2008). Beneficial use (e.g. livestock watering, irrigation, power plants, aquaculture, wildlife habitat, aquifer recharge) of produced water is attractive in regions where water demand surpasses water supply and can become an option when constituents of concern can be treated to acceptable levels (Veil

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et al., 2004). Constructed wetland treatment systems (CWTSs) are a potential treatment method for produced water. CWTSs have been utilized to treat a variety of waters including wastewater originating from farming practices, acid mine drainage, petroleum refinery effluents, flue gas desulfurization wastewater, brackish oilfield produced water, and other industrial effluents (Cronk, 1996; Barton and Karathanasis, 1998; Knight et al., 1999; Gillespie et al., 2000; Mooney and Murray-Gulde, 2008; Murray-Gulde et al., 2008). CWTSs are robust in nature, have the capacity to treat numerous constituents simultaneously, and have low associated costs (Mooney and Murray-Gulde, 2008; Rodgers and Castle, 2008). Contaminants can be targeted for removal through biogeochemical processes (transfers or transformations) by manipulating environmental conditions of the CWTS, soil type, and the types of plants (Rodgers and Castle, 2008). Flow patterns in CWTSs include subsurface flow (SSF; water level maintained below surface of the hydrosol) and free water surface (FWS; water level maintained above hydrosol). Previous research (Horner et al., 2011b) showed that pilot-scale CWTSs can effectively treat O & G at inflow concentrations up to 50 mg L^{-1} ($2.3\text{--}5.2 \text{ mg min}^{-1}$ mass loading). Because CWTSs could be exposed to greater O & G loadings, data on treatment performance at increased loads of O & G are needed. The purpose of this investigation was to evaluate a specifically designed pilot-scale CWTS for treating oilfield produced water with 100 mg L^{-1} O & G concentration (mass loading $\geq 5 \text{ mg min}^{-1}$). An oil–water separator, a passive device using gravity separation based on density differences between oil and water, was incorporated in the system. Oil collected from an oil–water separator has potential to be sold, which could help alleviate costs associated with produced water treatment. Specific objectives of this investigation were: (1) to assess effects of O & G mass loadings on treatment performance in pilot-scale SSF and FWS series, and (2) to evaluate treatment performance of a pilot-scale SSF series containing an oil–water separator.

2. Materials and methods

2.1. Pilot-scale constructed wetland treatment system

A pilot-scale CWTS, which was designed and constructed in a climate-controlled greenhouse located in Clemson, SC (USA), was used to assess treatment performance for oilfield produced water. The pilot-scale system was designed based on biogeochemical pathways to decrease aqueous concentrations of targeted constituents of concern (O & G, Fe, Mn, Ni, Zn) in simulated produced water. Use of simulated produced water reduces transportation costs associated with using actual produced water and allows precise control of water composition. Simulated produced water was formulated by addition of Shell Rotella T[®] motor oil and high purity salts (FeCl_3 , $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, and ZnCl_2 ; Fisher Scientific Inc., Fair Lawn, NJ) to a 3785-L polypropylene carboy retention basin containing municipal water. Additional salts (NaHCO_3 , $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, KNO_3 , and CaCO_3) were added to simulate hardness, pH, and ionic composition of the produced water. The simulated produced water was formulated based on composition of a specific oilfield produced water studied by Horner et al. (2011a). The simulated produced water flowed from the retention basin to the first cell of each of three treatment series via Fluid Metering Inc. (FMI[®]) piston pumps operating at a flow rate that maintained the targeted hydraulic retention time (HRT).

The CWTS was designed for sequential reducing and oxidizing cells, with the mass loading of oil and grease to the CWTS providing organics to promote reducing conditions. Three series were constructed (2 SSF = series A and B, and 1 FWS = series C), each consisting of four wetland cells (Supplementary Figs. S1, S2). Series

A and B were identical except that series A included an oil–water separator, described in Section 2.3. Each cell was contained within a 378-L Rubbermaid[®] utility tank, 123-cm long by 64-cm wide by 61-cm deep. Hydrosol in the SSF cells, which utilized vertical flow, was composed of 20 cm of pea gravel (5–10 mm in diameter) overlain by 40 cm of medium-sized, granitic gravel (20–30 mm diameter). SSF cells were planted with *Phragmites australis* (common reed), a macrophyte native to Sub-Saharan Africa and found throughout temperate and tropical regions. Cells in the FWS series were filled to a depth of 36 cm with hydrosol composed of coarse, well-sorted, quartz sand obtained from 18-Mile Creek near Clemson, SC. The first cell in the FWS series was planted with *Schoenoplectus californicus* (bulrush). The second, third, and fourth cells of the FWS series were planted with *Typha latifolia* (cattail).

2.2. O & G mass loading and treatment performance

The effect of O & G mass loading on treatment performance of CWTS series B and C was investigated by changing O & G inflow concentration and HRT. O & G was loaded at 50 mg L^{-1} targeted inflow concentration at a rate providing a nominal 4 d HRT (24 h for each wetland cell) from August–October 2010, 100 mg L^{-1} O & G with a 4 d HRT from October–November 2010 and February–March 2011, and 100 mg L^{-1} O & G with a 2 d HRT (12 h for each wetland cell) from March–April 2011. Nominal O & G mass loadings were 5 mg min^{-1} at 50 mg L^{-1} inflow concentration and 4 d HRT, 10 mg min^{-1} at 100 mg L^{-1} inflow concentration and 4 d HRT, and 20 mg min^{-1} at 100 mg L^{-1} inflow concentration and 2 d HRT.

To determine effects of the O & G loadings on hydrosol conditions, oxidation–reduction (redox) potential was measured using platinum-tipped redox probes in the hydrosol of each cell. Probes remained *in situ* for the duration of the experiments. Redox measurements were made with an Accumet[®] calomel reference electrode using a Fluke[®] 77 voltage meter (Faulkner et al., 1989). Hydrosol redox potential was measured in September 2010, October 2010, November 2010, March 2011, and April 2011.

General water chemistry parameters (alkalinity, hardness, pH, and dissolved oxygen) were measured in aqueous samples collected in 50 mL centrifuge tubes from series inflow and from outflow of each cell. Dissolved oxygen concentration and pH were measured using direct instrumentation, and alkalinity and hardness were measured in accordance with standard methods (APHA, 2005). Temperature and conductivity of outflow water from the cells also were measured.

To determine the ability of each series to decrease concentrations of O & G, aqueous samples for measurement of O & G concentration were collected in 1L glass jars with Teflon[®] lined lids from the series inflow and from cell outflows. EPA method 1664 (USEPA, 1999), a gravimetric method involving n-hexane extraction, was used for O & G analyses.

Aqueous samples for measurement of Fe, Mn, Ni, and Zn concentrations were collected in 50-mL centrifuge tubes from series inflow and from outflow of each wetland cell to determine the ability of each series to decrease concentrations of metals. Metal analyses were performed according to EPA method 200.7 (USEPA, 1994) using inductively coupled plasma-atomic emission spectrometry (ICP-AES; SPECTROFLAME-EOP, Spectro Analytical Instruments, Kleve, Germany). Due to instrument interferences from O & G concentrations, samples containing $>50 \text{ mg L}^{-1}$ O & G concentration were diluted before analysis using ICP-AES.

O & G concentrations were measured in motor oil standards (i.e. known mass of motor oil in a known volume of water). For every tenth sample analyzed, O & G concentration in a matrix spike was measured by collecting duplicate samples, adding 20 mg motor oil to one of the samples, and analyzing both samples. Percent

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