Evaluation of low cost cathode materials for treatment of industrial and food processing wastewater using microbial electrolysis cells

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ABSTRACT

Microbial electrolysis cells (MECs) can be used to treat wastewater and produce hydrogen gas, but low cost cathode catalysts are needed to make this approach economical. Molybdenum disulfide (MoS2) and stainless steel (SS) were evaluated as alternative cathode catalysts to platinum (Pt) in terms of treatment efficiency and energy recovery using actual wastewaters. Two different types of wastewaters were examined, a methanol-rich industrial (IN) wastewater and a food processing (FP) wastewater. The use of the MoS2 catalyst generally resulted in better performance than the SS cathodes for both wastewaters, although the use of the Pt catalyst provided the best performance in terms of biogas production, current density, and COD removal. Overall, the wastewater composition was more of a factor than catalyst type for accomplishing overall treatment. The IN wastewater had higher biogas production rates (0.8 – 1.8 m3/m3-d), and COD removal rates (1.8 – 2.8 kg-COD/m3-d) than the FP wastewater. The overall energy recoveries were positive for the IN wastewater (3.1 – 3.8 kWh/kg-COD removed), while the FP wastewater required a net energy input of –0.7 – 1.2 kWh/kg-COD using MoS2 or Pt cathodes, and –3.1 kWh/kg-COD with SS. These results suggest that MoS2 is the most suitable alternative to Pt as a cathode catalyst for wastewater treatment using MECs, but that net energy recovery will be highly dependent on the specific wastewater.

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1. Introduction

Bioelectrochemical systems are novel processes that utilize exoelectrogenic bacteria to oxidize organic compounds and generate electrical current through the transfer of electrons to the anode. In a microbial electrochemical cell (MEC), the electrons produced by bacteria are consumed at the cathode in the reduction of H+ to H2. MEC hydrogen production requires the addition of power from an external electrical source larger than ~ 0.2 V [1–3]. MECs have achieved high hydrogen yields (3.65 mol-H2/mol-acetate) [4], and at lower applied voltages energy efficiencies of up to 400% (the energy in the hydrogen gas produced, relative to the electrical energy input) [5]. Higher applied voltages can be used to increase...
hydrogen production rates, although this reduces the energy yields \[6\]. Applied voltages >0.8 V can result in electrical energy efficiencies <100% \[7\], depending on the hydrogen gas recovery. Consequently, there is a tradeoff in setting process goals, with higher voltages chosen to increase the rate of treatment, and lower voltages used to maximize energy recovery. Typically, voltages of 1 V or less are used in MEC tests to ensure good rates and reasonable energy recoveries.

Minimizing the costs of the electrode materials, and avoiding the use of precious metals are two goals for the development of economical wastewater treatment using MECs \[8\]. One of the most critical materials to the performance of the MEC, and also one of the most expensive, is the catalyst used for hydrogen evolution from the cathode. Pt is used in many MECs, although recently several alternative materials have been proposed, including stainless steel (SS), molybdenum disulfide (MoS\(_2\)), iron, nickel oxide, nickel alloys, and tungsten carbide \[9–14\]. Stainless steel (SS) is one of the least expensive of these materials, and the use of high surface area SS electrodes can produce good rates of hydrogen evolution compared to some alternative materials \[9,15,16\]. Brushes and mesh made from SS type 304 were both shown to produce current densities comparable to those obtained with Pt and carbon cloth cathodes using acetate as a substrate for the bacteria \[17,18\]. One disadvantage of SS, however, is that it has a relatively high overpotentials, for example 0.85 V larger than Pt on carbon cloth \[13,19\]. MoS\(_2\) is a relatively inexpensive catalyst that can have lower overpotentials than SS. It can be used as a coating on the SS mesh, or as particles bound to carbon cloth. When MoS\(_2\) particles were used in a cathode in electrochemical tests (with carbon black and a Nafion binder), the hydrogen evolution reaction overpotential in perchlorate and phosphate buffer was reduced from 1.04 V to 0.105 V \[13\].

The effectiveness of these various cathode catalysts for effective wastewater treatment has not been well investigated. There are many studies on hydrogen production with simple organic substrates, such as acetic acid, butyric acid, and lactic acid, with Pt catalysts \[4,20–22\]. However, there are fewer tests using complex source of organic matter such as effluents from bioprocesses as cellulose fermentation or anaerobic digestion, or different types of wastewaters (domestic, winery, potato, dairy and piggery) \[5,23–28\]. SS has only been tested using a single wastewater \[25\], and MoS\(_2\) has not previously been used with actual wastewaters. The organic and inorganic components of the wastewater can affect not only catalyst efficiency but also catalyst longevity, as they can irreversibly adsorb on the catalyst and lead to its poisoning \[29,30\]. The purpose of this study was to explore more practical applications of MEC systems by using low-cost catalysts (SS and MoS\(_2\)) and two actual wastewaters as feedstocks. These wastewaters were chosen to be much different in composition: the food processing wastewater contained high concentrations of complex carbohydrates, while the industrial wastewater (from a specialty chemicals manufacturing facility) had a lower concentration of biodegradable organic matter and a high concentration of methanol. Tests using these wastewaters were also conducted using MECs with cathodes containing a Pt catalyst in order to better understand the performance of these materials relative to Pt. The effectiveness of these catalysts for effective wastewater treatment was evaluated in terms of current densities, time for treatment, COD removal, and energy recovery.

2. Materials and methods

2.1. Wastewater

Industrial wastewater samples were collected from a chemical manufacturing facility in Kentucky. The sample was collected from a neutralization pit, representative of a blend of multiple different waste streams, prior to the aerobic treatment process currently being used at this site. Food processing wastewater samples were collected from the discharge pipe of a food processing plant. Samples were placed on ice, shipped overnight to the laboratory, and stored at 4 °C. Wastewater samples were fully characterized upon arrival (Table 1). Wastewater served both as microbial inoculum and substrate in all experiments.

2.2. Reactor construction and operation

Twelve single-chamber MECs (six for each wastewater sample) consisted of Lexan cubes drilled to contain a cylindrical chamber 4 cm long by 3 cm in diameter (empty volume = 28 mL). An anaerobic culture tube was glued to the top of the reactor to collect hydrogen gas (1.6 cm inner diameter and 6 cm length; 12 mL capacity). Anodes were heat-treated graphite fiber brushes (PANEX 33 160K, Gordon Brush, 2.5 cm diameter and 2.5 cm long) \[31\].

Table 1 – Wastewater characteristics.

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<tr>
<th>Parameters</th>
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<th>Food processing</th>
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<td>pH</td>
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<td>6.35 ± 0.25</td>
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<tr>
<td>Conductivity (mS/cm)</td>
<td>2.04 ± 0.02</td>
<td>2.53 ± 0.04</td>
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<td>TCOD (kg/m(^3))</td>
<td>4.07 ± 0.18</td>
<td>8.10 ± 0.62</td>
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<tr>
<td>SCOD (kg/m(^3))</td>
<td>3.81 ± 0.16</td>
<td>1.83 ± 0.20</td>
</tr>
<tr>
<td>BOD (kg/m(^3))</td>
<td>0.8</td>
<td>2.00 – 5.00</td>
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<td>TS (kg/m(^3))</td>
<td>1.34 ± 0.09</td>
<td>4.76 ± 0.10</td>
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<tr>
<td>TSS (kg/m(^3))</td>
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<td>2.43 ± 0.09</td>
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<tr>
<td>Inorganic compounds</td>
<td></td>
<td></td>
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<tr>
<td>Phosphorus (mg/L)</td>
<td>8.9</td>
<td>57.3</td>
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<tr>
<td>Sulfate (mg/L)</td>
<td>55.5</td>
<td>686 ± 25</td>
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<tr>
<td>Nitrate (mg/L)</td>
<td>&lt;5</td>
<td>5.7 ± 0.1</td>
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<tr>
<td>Nitrogen Ammonia (mg/L)</td>
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<td>9.5 ± 0.9</td>
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<tr>
<td>Organic compounds</td>
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<tr>
<td>Total carbohydrates (mg-COD/L)</td>
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<td>Soluble carbohydrates (mg-COD/L)</td>
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<td>Propanol</td>
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<td>1.5 ± 0.2</td>
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<td>Butanol</td>
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<td>Volatile fatty acids (mg/L)</td>
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<tr>
<td>Acetate</td>
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<td>116.3 ± 7.5</td>
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<td>Propionate</td>
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<tr>
<td>Butyrate</td>
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<td>29.5 ± 8.7</td>
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</table>
Three different MEC types of cathode catalysts were examined (in duplicate reactors): SS, MoS2, and Pt. All cathodes had a total projected surface area of 12 cm², with 7 cm² exposed to the solution. SS cathodes were flat sheets of type 304 stainless steel (Trinity Brand Industries, Inc.) that were sanded smooth with silicon carbide sand paper, ultrasonically washed in deionized water, rinsed with acetone, and then rinsed in deionized water. They were dried overnight (>12 h) before being used. MoS2 cathodes were prepared as previously described [13] by mixing MoS2 powder (Aldrich, 99%) with carbon black (Cabot, VULCAN XC-72R, >99%) at a mass loading ratio of 33.3%, and with 50 µL/cm² of a 2:1 volume solution of Nafton polymer and iso-propanol. This mixture was applied to the solution side of a carbon cloth (E-TEK, B-1/B/30WP, 30% by weight PTFE Wet-Proofed). Pt/C catalyst was constructed by applying a mixture 10/90 of platinum powder and carbon black (E-TEK, C1-10, 10 wt.% Pt on Vulcan XC-72) onto carbon cloth (Type B-1B, E-TEK, 3.8 cm diameter) cathodes.

The microbial communities on the anodes were first enriched in single chamber MFCs using Pt/C cathodes, and then transferred into MECS [1]. The MFCs were operated for two months in order to enrich current generating biofilm on the anodes to the specific wastewater. After this period steady exoelectrogenic activity was demonstrated by reproducible cycles of voltage production over several cycles. The reactors were then converted to operate as MECS by replacing the cathodes with new cathodes, and by covering the cathode with a solid plate to exclude air.

A voltage of 0.7 V was applied to the MECS with an external power supply (model 3646A; Circuit specialists, Inc.). When the complete gas production cycle ended, as indicated by zero gas production rate for 1 h or more, reactors were drained, refilled with fresh substrate, and flushed with ultra high-purity nitrogen gas (99.998%) for 15 min. All the tests were run in fed batch mode with duplicate reactors at 30 °C in a constant temperature room.

2.3. Measurements and chemical analyses

Voltage was measured across an external resistor ($R_{ex} = 10 \, \Omega$) every 20 min using a multimeter (2700; Keithley, United States) connected to a personal computer. Current generation was calculated using $I = \frac{E}{R}$, where $I$ (A) is the current, $E$ (V) the voltage, and $R$ (Ω) the external resistance.

During each fed-batch cycle the volume of gas produced was recorded using a respirometer (AER-200; Challenge Environmental), and the evolved gas was collected in air-tight gas bags (0.2 L capacity; Cali-5-Bond, Calibrated Instruments, Inc.). Gas from the gas bag and the reactor headspace was sampled using a gas-tight syringe (200 µL injection volume) and analyzed using gas chromatography (Models 310 & 8610B; SRI Instruments, Torrance, CA) [7].

Concentrations of solvents, alcohols, and organic acids (acetone, methanol, ethanol, propanol, butanol, acetate, propionate, and butyrate) were measured by gas chromatography (Varian Star 3400) with injector and flame ionization detector temperatures of 250 °C. Total and soluble COD (TCOD and SCOD) were measured using standard methods (HACH method 8000, HACH Company, Loveland, CO). Probes were used to measure pH (Mettler Toledo Seven Multi; Model: pH; S/N: 290843) and conductivity (Mettler Toledo Seven Multi; Model: Cond.; S/N: 291048).

2.4. Calculations

The volumetric production rates, $Q$ ($m^3/d·m^3$ of reactor), of hydrogen, methane and carbon dioxide were calculated from the measured specific gas produced normalized to the reactor volume and batch time. Treatment efficiency was evaluated in terms of TCOD removal, based on the TCOD concentrations and the beginning and end of each fed-batch cycle, and TCOD removal rate, $r$ (kg-COD/d·m³ of reactor).

Gas yields ($Y$) of hydrogen and methane were expressed on the basis of the TCOD removal (m³/kg-COD) and calculated as:

$$Y = \frac{V_{\text{gas}}}{V_i(COD_i - COD_f)}$$

(1)

where $V_{\text{gas}}$ is the total volume of a specific gas, hydrogen or methane, produced, $V_i$ the reactor volume, and $COD_i$ and $COD_f$ the COD initial and final concentrations of the wastewater in the batch test. COD removal was calculated as the average from two to five fed-batch tests.

Energy yield in the MEC was calculated as biogas energy production per unit of reactor volume (kWh/m³) normalized to COD removal (kg-COD/m³). Energy recovered as gas was determined by multiplying the moles of hydrogen and methane produced by their lower heat of combustion (−286 and −891 kJ/mol respectively) [32]. The MEC energy consumption ($W_{ap}$) was calculated by multiplying the current ($I$) in the circuit by the recorded voltage applied by the power source ($E_{ap}$) taken over time intervals $\Delta t = 20$ min for $n$ intervals and converted in (kWh):

$$W_{ap} = \sum_{i=1}^{n} I E_{ap} \Delta t$$

(2)

The net energy recovery (kWh/kg-COD) was calculated by subtracting the supplemental energy required to operate the MEC electrodes with a power supply from the value of energy produced as gas during each batch-cycle.

3. Results

3.1. Current generation

When acclimated anodes were used in the MECS, all reactors immediately produced current, with good reproducibility obtained among the duplicate reactors, with differences primarily based on type of wastewater and cathode catalyst (Figs. 1 and 2). Higher power densities were produced from the industrial (IN) wastewater than the food processing (FP) wastewater. MECS with Pt cathodes produced the highest current densities, with a maximum current density of 2.1 A/m² for the IN wastewater. Lower current densities were produced with the SS (1.5 A/m²) and MoS2 (1.2 A/m²).

There were larger difference in maximum current densities between Pt and the other two catalysts using the FP wastewater. With Pt cathodes, the maximum current was 2.4 A/m², compared to lower maximum current densities of 1.0 A/m² for both MoS2 and SS (Figs. 1 and 2). The shape of the
Current density profiles were also much different, with two separate plateaus observed with the Pt catalyst compared to one single plateau with the MoS2 and SS catalysts. The development of these two plateaus likely indicated more rapid initial oxidation of easily degraded substrates such as VFAs (Table 1), followed slower and more rate-limited degradation of complex organic matter.

The length of a fed batch cycle also was quite different with the two different wastewaters, requiring \( \approx 32 \) h (Pt, MoS2) to \( \approx 46 \) h (SS) for the IN wastewater, compared to much longer times of \( \approx 150 \) h for all three catalysts for the FP wastewater. These much longer treatment times for the FP wastewater are consistent with the reported BOD5 values by the plant of 2.0–5.0 kg/m3 for this wastewater, compared to BOD5 of 0.80 kg/m3 reported for the IN wastewater.

### 3.2. MEC treatment efficiency

TCOD removals in MECs using the IN wastewater ranged from 85 to 89%. The high removals indicated the components of this wastewater were highly biodegradable (Table 1). COD removal rates depended on the catalyst, with 2.8 kg-COD/m3-d with Pt cathodes, compared to 2.3 kg-COD/m3-d with MoS2 cathodes and 1.8 kg-COD/m3-d for the SS cathodes (Fig. 3a). Effluent CODs of the IN wastewater followed the same trend as removal rate results, with the lowest effluent CODs for the tests using the Pt catalyst (0.45 kg-COD/m3) and the highest effluent CODs for the SS catalyst (0.61 kg-COD/m3) (Fig. 3b).

The removal rates for the FP wastewater were much lower than those of the IN wastewater, but they showed the same general trend with catalyst type, with the highest rate obtained for the Pt catalyst (0.9 kg-COD/m3-d) and the lowest one with the SS catalyst (0.66 kg-COD/m3-d) (Fig. 3a). COD removals decreased from 67% with the Pt catalyst, to 49% with SS. The effluent COD followed the same trend, with the lowest effluent COD obtained for the Pt catalyst, and the highest for the SS (Fig. 3b). These lower removal rates with the FP wastewater compared to the IN wastewater are likely due to the high percentage of insoluble organics (nearly 80%) in the FP wastewater (Table 1).

### 3.3. Biogas production

Substantially more biogas was recovered in MECs with the Pt catalyst cathode than reactors with the other two catalysts, and much more biogas was recovered using the IN wastewater than from the FP wastewater (Fig. 4). Most of the gas recovered using the IN wastewater was methane. MECs with the Pt catalyst had the highest average gas flow rate of 1.8 m3/m3-d, and the produced gas contained 55 ± 4% methane and...
32 ± 4% hydrogen (balance was CO₂) (Fig. 4). The reactors with the MoS₂ and SS had lower biogas production rates (1.2 m³/m³-d, MoS₂; 0.8 m³/m³-d, SS), consistent with lower measured current densities than those obtained with Pt (Fig. 1). However, they also both produced high concentrations of methane (62 ± 3%, MoS₂, 70 ± 2%, SS) indicating the catalyst was not a factor in the resulting gas composition (Fig. 4).

Biogas production with the FP wastewater with a Pt catalyst was 76% lower (0.41 ± 0.02 m³/m³-d) than that produced using the IN wastewater (Fig. 4). In contrast to the IN wastewater results, most of the gas recovered from this wastewater was H₂ (86%). The MoS₂ and SS cathodes had similar but lower gas production rates of 0.17 ± 0.03 m³/m³-d (MoS₂) and 0.12 ± 0.02 m³/m³-d (SS) (Fig. 2).

3.4. Energy yields and coulombic efficiency

The net energy yields, which are used to express energy produced relative to the electrical energy applied, generally reflect the trends observed with gas production. For the IN wastewater, tests with the different catalysts all produced positive net energy recoveries, with the highest net energy recovery using of 3.8 ± 0.2 kWh/kg-COD with Pt, and the lowest with SS (3.1 ± 0.2 kWh/kg-COD) (Fig. 5). For the FP wastewater, all tests resulted in negative net energy recoveries, with similar values for the Pt (−1.2 ± 0.2 kWh/kg-COD) and MoS₂ catalyst (−0.7 ± 0.1 kWh/kg-COD), compared to −3.1 ± 0.1 kWh/kg-COD with the SS catalysts (Fig. 5). Since tests with the FP wastewater had similar current densities in MECs with the SS and MoS₂ catalysts, these results show that producing that current with the MoS₂ catalysts required much less energy input than that required with the SS cathodes.

The MECs with the IN wastewater all had much lower coulombic efficiencies of 7–12%, than the FP wastewater which were in the range of 26–35% (Table 2). This shows that the catalyst type was less important for electron recovery in these systems than the intrinsic properties of the wastewater.

4. Discussion

The type of catalyst created clear differences in wastewater treatment effectiveness and MEC performance for a given
wastewater, but the composition of the specific wastewater was more critical to overall performance than catalyst type. In general, the Pt catalyst produced better results than the MoS2 or SS cathodes in terms of current generation, COD removal rate and extent, biogas production, and energy recovery for a specific wastewater. Higher current densities were expected with the Pt catalyst due to the lower overpotentials that can be obtained with this catalyst, which result in improved energy recoveries. In contrast with previous studies using different catalysts, where COD removals, biogas production, and performance was similar with acetate as a fuel [9,13], in this study the Pt catalyst outperformed the others both for lower COD in the process final effluent and for the volume of gas produced. However, the complex nature of the wastewaters examined here resulted in differences in gas production rates and composition with the different catalysts, likely as a result between competition for the easily degraded and soluble organics between exoelectrogenic and other microorganisms.

The differences in performance were much larger based on the type of wastewater than the catalyst type. Surprisingly, the MECs had better performance with the IN wastewater than the FP wastewater. It was expected that the food-based nature of the FP wastewater would be more readily degraded than the IN wastewater, as the IN wastewater contained a high concentration of methanol (1.5 kg/m3). Previous studies have shown that methanol was not a good substrate for electricity generation in a MFC, based on the production of very low voltages (~10 mV) [33]. The high rates of methane production and the high methane content of the gas produced here using the IN wastewater suggests that there was direct methane gas production from methanol, by the exothermic reaction 4CH3OH → 3CH4 + H2O + HCO3− + H+ [34]. This direct route of methane production could also help to explain the shorter batch times and lower current densities with the IN wastewater than those obtained with the FP wastewater. In addition, the coulombic efficiencies were much lower for the IN wastewater than the FP wastewater, indicating that a much larger percentage of the organic matter removed was degraded in non-anode dependent processes.

From the perspective of wastewater treatment, the generation of gas or current is less important than the overall energy input and extent of treatment. There was a net energy recovery using the IN wastewater, but not the FP wastewater. A typical activated sludge plant requires from –0.7 to –2.0 kWh/kg-COD for aeration [35]. Here, we obtained between 3.1 and 3.8 kWh/kg-COD, thus accomplishing both energy production and treatment. However, with the FP wastewater, there was a net energy requirement of –1.2 (Pt), –0.7 (MoS2) and –3.1 kWh/kg-COD (SS) for the different catalysts. On the basis of energy demand, this suggests that the SS catalyst is the least suitable for use in these reactors. Thus, if Pt catalysts are to be avoided, MoS2 provided a good compromise in a balance of performance and energy recovery.

5. Conclusions

These results showed that MoS2 and SS catalysts could be used in MECs to accomplish wastewater treatment, although overall rates and the extent of treatment were reduced compared to tests with Pt catalysts. The wastewater characteristics were much more important to process performance and energy requirements for wastewater treatment. IN wastewater treatment was accomplished in less time and it produced net energy, whereas the FP wastewater treatment was much slower and it required a net energy input. The SS cathode required a higher net energy input than that typically required for treatment using activated sludge, suggesting that MoS2 is the best candidate for the cathode catalyst in MECs used for wastewater treatment.

Acknowledgments

The authors thank Air Products and Chemicals, Inc. for providing wastewater samples. This research was supported by Award KUS-I1-003-13 from the King Abdullah University of Science and Technology (KAUST).

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<tr>
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<th>IN-Mo</th>
<th>IN-SS</th>
<th>FP-Pt</th>
<th>FP-Mo</th>
<th>FP-SS</th>
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I N T E R N A T I O N A L J O U R N A L O F H Y D R O G E N E N E R G Y 3 8 ( 2 0 1 3 ) 1 8 5 9 – 1 8 6 5