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Journal of Power Sources 171 (2007) 835-841

www.elsevier.com/locate/jpowsour

# Electrochemical separation of hydrogen from reformate using PEM fuel cell technology

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 Received 22 February 2007; received in revised form 1 June 2007; accepted 5 June 2007

Available online 19 June 2007

#### Abstract

This article is an examination of the feasibility of electrochemically separating hydrogen obtained by steam reforming a hydrocarbon or alcohol source. A potential advantage of this process is that the carbon dioxide rich exhaust stream should be able to be captured and stored thereby reducing greenhouse gas emissions. Results are presented for the performance of the anode of proton exchange membrane (PEM) electrochemical cell for the separation of hydrogen from a  $H_2$ – $CO_2$  gas mixture and from a  $H_2$ – $CO_2$ –CO gas mixture. Experiments were carried out using a single cell state-of-the-art PEM fuel cell. The anode was fed with either a  $H_2$ – $CO_2$  gas mixture or a  $H_2$ – $CO_2$ –CO gas mixture and hydrogen was evolved at the cathode. All experiments were performed at room temperature and atmospheric pressure. With the  $H_2$ – $CO_2$  gas mixture the hydrogen extraction efficiency is relatively poor. To improve the efficiency for the separation of the gas mixture containing CO, the effect of periodic pulsing on the anode potential was examined. Results show that pulsing can substantially reduce the anode potential thereby improving the overall efficiency of the separation process although the anode potential of the CO poisoned and pulsed cell still lies above that of an unpoisoned cell.

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Keywords: Hydrogen purification; Electrochemical separation; Reformate; Carbon monoxide; Periodic pulsing

## 1. Introduction

Hydrogen, as an energy carrier, has been proposed as an attractive alternative to fossil fuels. Many authors have promoted a vision of a hydrogen economy based on a combination of electricity and hydrogen. Ultimately, the energy to produce hydrogen by the splitting of water will need to come from noncarbon sources of energy, such as nuclear, solar or wind. At the present time, hydrogen produced from fossil fuels such as natural gas has a much lower cost than hydrogen produced by the electrolysis of water. Nevertheless, the depletion of global oil and gas reserves and concern over global warming caused by increasing carbon dioxide levels in the atmosphere is increasing the urgency to find a new clean and abundant source of energy for the future.

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Many agencies, such as the US Department of Energy (DoE) and the International Energy Agency (IEA), expect that during the transition to the hydrogen economy, hydrogen will be produced at distributed hydrogen production sites by reforming natural gas. Currently there are 39 hydrogen filling stations in California and 140 spread over the globe, the majority of which obtain hydrogen from natural gas. To reduce greenhouse gas emissions, an important part of this process will be the capture and storage of the carbon dioxide. At the moment, technology is lacking to do this efficiently. A recent IEA publication [1] says, "Further R&D is needed on hydrogen purification (to produce hydrogen suitable for fuel cells) and on gas separation (to separate hydrogen or  $CO_2$  from gas mixtures)".

The electrochemical separation of hydrogen from impure gas streams is not new and has been studied by a number of people, including Sedlak et al. [2], going back to the 1980s. In this scheme, hydrogen is removed from hydrogen rich but impure fuel stream by electro oxidation at the anode and evolved at the cathode as a pure hydrogen stream. The cell overvoltage for this

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process can be quite low. The results of Sedlak et al. [2] showed that hydrogen separation could be achieved at low cell voltage and with high separation efficiency. In addition, as discussed by Rohland et al. [3], the same device can be used for hydrogen compression. The separation of hydrogen/nitrogen/carbon dioxide mixtures has been studied Lee et al. [4]. In a recent paper [5], we have studied the separation of hydrogen from methane with the aim of using the existing natural gas pipeline system for the distribution of hydrogen. In this scenario, hydrogen would be injected and mixed with the natural gas at the point of production and separated at the point of use. The research that we have performed suggests that separation can be achieved quite efficiently.

This article is an examination of the feasibility of electrochemically separating hydrogen from a reformate stream that was derived from a hydrocarbon source such as natural gas or an alcohol such as methanol. A side benefit of this electrochemical process is that the essentially pure carbon dioxide exhaust stream should be able to be captured and stored thus reducing greenhouse gas emissions.

At first sight, because  $CO_2$  is essentially inert, it would appear that this process should work as well as separation of hydrogen from methane [5] and or nitrogen [4]. A major issue, however, is the poisoning of the catalyst [6–8] by the small amounts of CO that are always present in the reformed gas. The mechanism of CO poisoning of a Pt catalyst is well established. As shown in the following equations, CO competes with hydrogen for the active sites on the platinum at normal anode operating potentials:

$$H_2 + 2S \leftrightarrow 2H - S \tag{1}$$

$$2H-S \leftrightarrow 2S + 2H^+ + 2e^- \tag{2}$$

$$CO + S \leftrightarrow CO - S$$
 (3)

where S represents a catalytic site on the electrode.

The work of Papageorgopoulos and de Bruijn [9] has shown that 98% of the active platinum sites are blocked by CO by hydrogen containing 1% CO<sub>2</sub> at 25 °C. The adsorbed CO can be removed by raising the anode potential to about 700 mV versus RHE. At this potential, as shown in the following equations, the CO reacts with hydroxyl species that are formed on the platinum surface to form CO<sub>2</sub>:

$$H_2O + S \leftrightarrow S - OH + H^+ + e^-$$
(4)

$$CO-S + OH - S \rightarrow 2S + CO_2 + H^+ + e^-$$
(5)

To try to overcome the CO poisoning problem, bifunctional platinum-alloy catalysts such as Pt/Ru have been developed. With these catalysts, formation of OH on the surface, as per Eq. (4), occurs at substantially lower potential. The use of a Pt/Ru catalyst still results in a substantial loss of cell potential [4]. The results of Iorio et al. [10] show that, while the cell potential is increased by about 250 mV when compared to pure platinum, there is still a loss of about 200 mV (or a loss of 30% in voltage efficiency) when compared with the use of pure hydrogen as a fuel.

Operation of the anode of either an electrochemical separation cell or a fuel cell at a potential high enough so that CO is removed by oxidation to  $CO_2$  would result in a serious loss of efficiency. Previous work [11-13] has shown that CO poisoning of the anode of a PEM fuel cell can be alleviated by periodic pulsing. In this paper we examine the efficacy of this process for alleviating CO poisoning in an electrochemical separation cell.

# 2. Experimental

#### 2.1. Cell and membrane electrode assembly

Experiments were made using a single electrochemical cell having a 25 cm<sup>2</sup> active area manufactured by ElectroChem Inc. The membrane electrode assembly (MEA) set used in these experiments had carbon supported platinum and ruthenium at both anode and cathode (loading: 1 mg Pt, 0.5 mg Ru cm<sup>-2</sup>) and used Nafion<sup>®</sup> 115 as membrane material. All of the measurements were made at room temperatures (293 K). The cathode, operating in the hydrogen evolution mode [5], was used as a reference. Distilled water was circulated through the cathode compartment at  $2.3 \text{ mL s}^{-1}$  to ensure membrane humidification and to allow the cell temperature to be controlled. In this configuration, hydrogen gas was consumed at the anode and evolved at the cathode. The operating pressure of both the anode and cathode compartments was 1 atm.

#### 2.2. Test setup

A Kikusui Model PBX 20-10 Bi-Polar Power Supply, connected in parallel with the electrochemical cell, was used to control the cell current. The power supply was programmable so that the cell could be cycled through a predetermined current profile. A Fluke Hydra data logger was used to monitor and capture cell potential and current for final analyses using a desktop computer. One channel of the data logger is directly connected to the electrochemical cell to measure the cell potential while a second channel monitors cell current from a connection on the bipolar power supply. All of the polarization curve experiments were performed by ramping the current from 0 to 10 A at a rate of  $20 \text{ mA s}^{-1}$ . When the limiting current was reached, the experiment was terminated. In the periodic pulsing experiments, the cell was run at a constant (base) current and, periodically, a variable width 10 A pulse was applied. The transient voltage data was collected using a Nicolet 310 storage oscilloscope (collection time in the millisecond range).

All of the gases (hydrogen, a 1000-ppm CO/hydrogen mixture and carbon dioxide) used in the experiments were stored in high-pressure cylinders connected via small diameter plastic tubing to the fuel cell test station. In order to obtain the desired mixtures, the flow of each gas was controlled using a calibrated rotameters mounted on the fuel cell test station. A constant input pressure of 140 kPa, upstream of the rotameter was used with a single plastic tube connecting the stream of mixed gases to the anode compartment of the cell. The total pressure in the fuel cell was maintained at 1 atm. To simulate the stoichiometry of reformed natural gas, all of the gas mixtures consisted of 80% hydrogen and 20% carbon dioxide by volume. The total gas flow rate to the anode compartment of the electrochemical cell was varied.



Fig. 1. Polarization curve for pure hydrogen gas at the anode (293 K, Pt/Ru catalyst,  $3.25 \text{ mL s}^{-1}$  gas flow, 1 atm).

#### 3. Results and discussion

#### 3.1. Pure hydrogen

The uncorrected polarization curve for pure hydrogen gas at the anode and pure hydrogen gas combined with distilled water at the cathode using a 25 cm<sup>2</sup> MEA catalyzed with platinum/ruthenium (1 mg Pt, 0.5 mg Ru cm<sup>-2</sup>) is shown in Fig. 1. The linear nature of the initial portion of the plot indicates that *IR* losses dominate the cell overpotential in this region. By taking the slope of the polarization curve in the 0.02–0.04 A cm<sup>-2</sup> region, an estimate of 17.0 m $\Omega$  was obtained for the cell resistance. This resistance is more than a factor of two higher than the value one obtains using the conductivity value that Dupont gives [14] in their technical literature for the conductivity and thickness of Nafion<sup>©</sup> 115. The extra resistance probably arises from the resistance of the thick graphite collector plates that are used in the cell as well as from contact resistance between the plates and the MEA.

The *IR* corrected polarization curve for the cell is also included in Fig. 1. This curve shows that the maximum overpotential at the highest current density used in these experiments  $(0.4 \text{ A cm}^{-2})$  is about 20 mV. If it is assumed that this overpotential is divided equally between the anode and cathode, then the overpotential at the cathode is 10 mV or less. The use of the cathode as a dynamic hydrogen reference electrode is justified since previous work [5] has shown that the overpotentials for the hydrogen/methane and hydrogen/argon mixtures were much larger.

# 3.2. Hydrogen/carbon dioxide mixtures

Fig. 2 shows the uncorrected anode polarization curves for an 80% hydrogen/20% carbon dioxide mixture using total gas flow rates of 32.5, 62.5 and 82.5 mL min<sup>-1</sup>. Two major factors

influence the shapes of the curves. First, the maximum current, indicated by the vertical portion of the curves, is limited by the amount of hydrogen available at a given flow rate. The limiting current densities at these flow rates are 0.17, 0.28 and  $0.40 \text{ A cm}^{-2}$ , respectively. The second major factor that influences the shape of these curves is the difference in hydrogen partial pressures at the anode and cathode. While the hydrogen pressure at the cathode remains constant at 1 atm, the hydrogen pressure at the anode depends on the mixture used as well as the current density. Passage of current depletes the hydrogen in the anode chamber and thus changes the composition during the sweep. Under these conditions, the anode potential is given by the Nernst equation:

$$V = V_0 + \frac{RT}{2F} \ln \left[ \frac{P_{\text{cathode}}^{\text{H}_2}}{P_{\text{anode}}^{\text{H}_2}} \right]$$
(6)

The ohmic loss and the activation overpotential loss must also be included to determine total cell potential. The activation overpotential for hydrogen oxidation is very small and contributes very little to the total cell potential. Based on Eq. (6), the vertical portion of the lines indicates the current density at which most of the H<sub>2</sub> in the H<sub>2</sub>/CO<sub>2</sub> mixture has been consumed and the hydrogen partial pressure at the anode became small.

#### 3.3. Modeling hydrogen separation

To model the hydrogen separation process, a modified version of the procedure used by Zhang [6] can be used. The anode reactions consist of the hydrogen adsorption and oxidation reactions defined by the following equations:

$$H_2 + 2S \leftrightarrow 2H-S; \quad \vec{k}_{H_2,ads}, K_H$$
 (7)

$$H-S \leftrightarrow H^+ + e^- + S; \quad \vec{k}_{H_2,ox}$$
 (8)



Fig. 2. Measured anode potential for an 80% hydrogen/20% carbon dioxide mixture at various flow rates (293 K, 1 atm).

Table 1	
Model parameters	
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Parameter	Value	
$\overline{k_{\mathrm{H,ads}}}$ (A cm <sup>-2</sup> atm <sup>-1</sup> )	402	
$k_{\rm H,ox} ({\rm Acm^{-2}})$	4.0	
$K_{\rm H}$ (atm)	0.5	
$\alpha_{ m H}$	0.5	
Anode volume (cm <sup>3</sup> )	4.4	
Temperature (K)	293	
Double layer capacitance (F)	0.45	
Surface roughness	100	
Platinum mole density (mol $cm^{-2}$ )	$2.2 \times 10^{-9}$	
Cell area (cm <sup>2</sup> )	25	

Based on this model, three ordinary differential equations can be developed [5] that describe the surface coverage of hydrogen, the hydrogen partial pressure in the anode chamber and the anode potential. These equations were solved using the ordinary differential equation (ODE) solver in Scilab [15]. The kinetic parameters given by Zhang [6] (see Table 1) were used for the simulations. Fig. 3 shows the results obtained for the various total gas flow rates including the *IR* contribution. There is seen to be good agreement between the experimental (Fig. 2) and simulated (Fig. 3) results.

## 3.4. 1000-ppm CO:hydrogen/carbon dioxide mixture

To examine the effects of carbon monoxide poisoning on the anode potential needed to separate hydrogen from carbon dioxide when the electrode was poisoned with CO, experiments were conducted with a gas mixture having two components (a) 80% was a hydrogen gas that contained 1000-ppm CO and (b) 20% was a carbon dioxide gas. Anode polarization curves are shown in Figs. 4–6 for the same three gas flow rates as shown in Fig. 3. For comparison, the anode potential curves for the COfree hydrogen/CO<sub>2</sub> mixtures have been included as dashed lines.



Fig. 3. Calculated anode potential for an 80% hydrogen/20% carbon dioxide mixture at various flow rates (293 K, 1 atm).



Fig. 4. Polarization curve for an 80% hydrogen with 1000-ppm CO/20% carbon dioxide mixture compared to a 80% hydrogen/20% carbon dioxide mixture at a flow rate of  $37.5 \text{ mL min}^{-1}$  (293 K, 1 atm).

From this data, it is seen that the gas containing 1000-ppm CO poisons the anode and raises the anode potential by as much as 300 mV. From a practical point-of-view, this means that substantially more energy is required for the hydrogen separation process when the gas contains CO. When the current approaches about 5 A, spontaneous oscillations in the anode potential occur. This corresponds to self-cleaning of the anode and occurs when the anode potential rises high enough for OH to be formed at the catalyst surface. This OH then reacts rapidly with the adsorbed CO to form  $CO_2$ . When this surface cleaning takes place, the anode potential then falls. This process has been described and modeled in detail by Zhang [6].



Fig. 5. Polarization curve for an 80% hydrogen with 1000-ppm CO/20% carbon dioxide mixture compared to a 80% hydrogen/20% carbon dioxide mixture at a flow rate of  $62.5 \text{ mL min}^{-1}$  (293 K, 1 atm).



Fig. 6. Polarization curve for an 80% hydrogen with 1000-ppm CO/20% carbon dioxide mixture compared to a 80% hydrogen/20% carbon dioxide mixture at a flow rate of  $87.5 \text{ mL min}^{-1}$  (293 K, 1 atm).

These spontaneous oscillations are illustrated in more detail in Fig. 7. From these data, it is seen that the period of oscillation is between 3 and 4 s. In this experiment, the cell was held at open circuit potential to allow the surface concentration of CO to reach equilibrium (maximum CO coverage). The current was then stepped to 6.0 A. Because the surface conditions are not stabilized immediately following this current step, the period of oscillation slowly changes (decreases) with time.

# 3.5. Effect of periodic pulsing on the anode potential of CO poisoned electrodes

We also investigated the effect of pulsing on the anode potential of a CO poisoned cell. Previous work has shown [11] that



Fig. 7. Spontaneous oscillations in anode potential (80% hydrogen with 1000-ppm CO/20% carbon dioxide; current density,  $0.24 \, A \, cm^{-2}$ , 293 K, 1 atm, 62.5 mL s<sup>-1</sup>).



Fig. 8. The effect of periodic pulsing on the anode potential (80% hydrogen with 1000-ppm CO/20% carbon dioxide; 293 K, current density  $0.2 \text{ A/cm}^2$ , 10 A, 0.5 s pulse every 10 s,  $62.5 \text{ mL s}^{-1}$ ).

the application of short duration, high-current pulses to a PEM fuel cell can alleviate the effects of anode poisoning and significantly improve stack output. The procedure was suggested earlier [12] and has been shown to be effective for PEM fuel cells using reformate [11], methanol [16] or formic acid [17] as fuels. During the pulsing process, the anode potential is momentarily raised high enough for OH to be formed at the surface, which removes the CO that is blocking the surface through reaction to form  $CO_2$ . Because some time is required for the electrode to be reblocked by CO, this process allows the



Fig. 9. The effect of periodic pulsing on the anode potential of a CO poisoned cell (an 80% hydrogen with 1000-ppm CO/20% carbon dioxide mixture is compared to both a 80% hydrogen/20% carbon dioxide mixture and to an 80% hydrogen with 1000-ppm CO/20% carbon dioxide, 293 K, with 10 A, 0.5 s pulse every 10 s). The current density was increased in increments of 0.04 A cm<sup>-2</sup> every 300 s and then held constant until the next increment occurred.

anode to operate at a substantially lower potential. Fig. 8 provides a detailed example of this process. In this experiment, the cell was operated at a constant (base) current of 5 A but every 10 s a 10 A, 0.5 s pulse was applied. Fig. 8 shows that, during the pulse, the anode potential is momentarily raised above 0.6 V, which is sufficient to strip off some of the CO that has accumulated at the anode. This allows the anode potential to immediately fall back to a much smaller potential. Repoisoning subsequently raises the anode potential until the next pulse is applied.

A comparison of the anode potentials for a CO poisoned cell both with and without pulsing and a non-poisoned cell is shown in Fig. 9. In this experiment, the current was stepped in  $1 \text{ A} (0.04 \text{ A} \text{ cm}^{-2})$  increments and held constant for a period of 300 s. A 10 A, 0.5 s duration pulse was applied every 10 s to generate the middle curve. The data shows that cell pulsing can substantially reduce the anode potential. Note, however, that the anode potential of the CO poisoned and pulsed cell still lies above that of an unpoisoned cell. In this experiment, the pulsing process has not been optimized and improved conditions could probably be achieved by adjusting the pulse height and/or width and, possibly even shape. Fig. 10 shows results for two different pulse durations, namely 0.2 and 0.8 s. As before a 10 A pulse was applied every 10 s. It is seen that anode potential is substantially lower when a 0.8 s pulse is used.

#### 3.6. Separation efficiency

The efficiency of the electrochemical separation of hydrogen has been discussed by Sedlak et al. [2] and Lee et al. [4]. Both groups define the potential efficiency,  $\eta_{V,\text{thermal}}$ , in terms of the thermal energy contained in the hydrogen (equivalent to



Fig. 10. The effect of pulse width on the anode potential of a CO poisoned cell (80% hydrogen with 1000-ppm CO/20% carbon dioxide, 293 K, 10 A pulse every 10 s). The current density was increased in increments of  $0.04 \text{ A cm}^{-2}$  every 300 s and then held constant until the next increment occurred. Pulse width data for 0.2 and 0.8 s are shown as thick and thin lines, respectively.

a 1.484 V potential). The potential efficiency then becomes:

$$\eta_{\rm V, thermal} = 1 - \frac{V_{\rm A}}{1.484} \tag{9}$$

The total cell or separation efficiency,  $\eta_T$ , is the product of the potential and current efficiencies. Inefficiency in current collection arises mainly because of cross-over of the hydrogen from the cathode to the anode. The current efficiency,  $\eta_I$ , is given by the equation:

$$\eta_{\rm I} = 1 - \frac{I_{\rm D}}{I_{\rm T}} \tag{10}$$

where  $I_{\rm D}$  is the current equivalent of the hydrogen flux that is diffusing back and  $I_{\rm T}$  is the total current. The rate of hydrogen diffusion has not been measured in this work, however, current efficiencies are usually quite high. The separation efficiencies in Fig. 11 are based on a current efficiency of unity. A plot of the separation efficiencies defined in this way is shown in Fig. 11 together with a plot of percent hydrogen separation using the experimental anode polarization data for an 80% hydrogen/carbon dioxide mixture at a flow rate of  $87.5 \,\mathrm{mL}\,\mathrm{min}^{-1}$ . From these data, it is seen that, while the separation efficiency is quite high (greater than 80% with about 80% hydrogen extraction) for the extraction of hydrogen from a hydrogen/CO<sub>2</sub> mixture, the separation efficiency is relatively poor when the hydrogen contains CO. For both the hydrogen/CO2 gas mixture and the pure hydrogen, when the hydrogen recovery increases beyond 70% there is a dramatic decrease in the separation efficiency. In this work, we have only examined hydrogen separation using a single electrochemical cell. Hydrogen recovery could probably be increased through the use of one or more additional cells in series with some energy penalty. A detailed analysis is going to be needed to see if the electrochemical separation process can compete with other separation technologies such as pressure swing adsorption and membrane separation.



Fig. 11. Separation efficiency and hydrogen recovery for various gas mixtures at a flow rate of  $87.5 \text{ mL min}^{-1}$ . Solid line: 80% hydrogen with 1000-ppm CO/20% carbon dioxide; dashed line: 80% hydrogen/20% carbon dioxide mixture.



Fig. 12. The effect of pulsing on separation efficiency for various gas mixtures. Circles: no pulsing with 80% hydrogen/20% carbon dioxide mixture; triangles: pulsing with 80% hydrogen with 1000-ppm CO/20% carbon dioxide; squares: no pulsing with 80% hydrogen with 1000-ppm CO/20% carbon dioxide; diamonds: hydrogen recovery.

The effect of pulsing on the potential efficiency is shown in Fig. 12. Data for pure hydrogen have also been shown for comparison. The data shows that, while pulsing substantially improves the separation efficiency, it is still lower that that for a mixture of pure hydrogen with carbon dioxide.

# 4. Conclusions

In this article, the feasibility of electrochemically separating hydrogen from a reformate stream that was derived from a hydrocarbon source such as natural gas or from an alcohol such as methanol has been examined. Experiments have been carried out to examine the separation of both pure hydrogen and impure hydrogen containing 1000 ppm carbon monoxide from carbon dioxide. With pure hydrogen, it has been found that the efficiency is quite high (greater than 80% with about 80% hydrogen extraction) for the extraction of hydrogen from a hydrogen/CO<sub>2</sub> mixture. When the hydrogen contains CO, however, the efficiency is relatively poor. Experiments have shown that hydrogen containing 1000-ppm CO severely poisons the anode and raises the anode potential by as much as 300 mV. From a practical point-of-view, this means that substantially more energy is required for the hydrogen separation process when the gas contains CO.

In an effort to improve the efficiency for the separation of hydrogen containing CO, the effect of periodic pulsing on the anode potential has been examined. A comparison of the anode potentials for a CO poisoned cell both with and without pulsing shows that cell pulsing can substantially reduce the anode potential although the anode potential of the CO poisoned and pulsed cell still lies above that of an unpoisoned cell. In our experiments, the pulsing process has not been optimized and improved conditions may be achievable by adjusting the pulse height, width and/or shape

# Acknowledgement

The authors gratefully acknowledge funding from the Canadian government's Natural Sciences and Engineering Research Council.

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