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Development of direct methanol alkaline fuel cells using anion exchange membranes

Eileen Hao Yu, Keith Scott*

School of Chemical Engineering and Advanced Materials, University of Newcastle upon Tyne, Merz Court, Newcastle upon Tyne NE1 7RU, UK

Abstract

Research into the development of direct methanol alkaline fuel cell (DMAFC) using an anion exchange polymer electrolyte membrane is described. The commercial membrane used had a higher electric resistance, but a lower methanol diffusion coefficient than Nafion® membranes. Fuel cell tests were performed using carbon supported Pt catalyst, and the effect of temperature, methanol concentration, methanol flow rate, air pressure and Pt loading were investigated. It was found that the cell performance improved drastically with a membrane assembly electrode (MEA) which did not include the gas diffusion layer on the anode, because of lower reactant mass transfer resistance. To give suitable cathode performance, humidification of the air and a subtle balance between the air pressure and water transport is required.
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1. Introduction

The direct methanol fuel cell (DMFC), which directly converts the chemical energy of methanol into electrical energy, is one of the most promising alternative power sources for transportation, potable electronics and stationary applications [1]. Although the DMFC has a lower power density ($W\,m^{-2}$) than the hydrogen fuel cell, the high theoretical energy storage capacity of methanol ($5019\,Ah\,kg^{-1}$) has the potential to increase the energy density of the DMFC system above that of competing electrochemical power sources. The ease of use of a liquid fuel, which is more convenient to store and refuel, makes the DMFC an attractive alternative to batteries and combustion engines. Solid polymer electrolytes (SPE), mainly proton conducting polymer membranes, have been used in DMFCs. Nafion® appears the popular membrane electrolyte for DMFC applications. However, the poisoning issue of the anode due to formation of CO species during methanol oxidation and the methanol crossover limit the performance of DMFCs.

It has been shown that methanol oxidation in alkaline media is kinetically faster than that in acidic media [2–5].

In addition, the ionic current in alkali fuel cells is due to conduction of hydroxide ions and is the reverse direction to that in proton conducting systems. As such, the direction of the electro-osmotic drag is reversed, reducing the methanol permeation rate. An alternative approach for the development of direct methanol fuel cells has so been proposed.

The first fuel cells using aqueous KOH electrolyte were patented by Reid in 1902 and Noel in 1904, 63 years after Grove's acid fuel cells. It is well known that the reaction kinetics and catalytic activities of the anodic oxidation of fuel and the cathodic reduction of O_2 in alkaline media are significantly higher than those in acidic media. In NASA's Apollo missions, fuel cells using 80–85% KOH (molten KOH), operated at $250\,°C$ and close to atmospheric pressure, were used as a part of the power supply system. The first DMFC using a solid polymer membrane was conceived by Hunger [6]. It contained an anion exchange membrane which porous catalytic electrodes pressed on both sides. Studies on developing alkaline anion exchange membranes for applications in low temperature portable DMFCs [7,8] have been carried out. Ogumi et al. [9] investigated the feasibility of using OH form anion exchange membrane on fuel cells using ethylene glycol as the fuel. The results suggested the potential application of anion exchange membranes in direct alcohol fuel cells.

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* Corresponding author. Tel.: +44-191-2228771;
fax: +44-191-2225292.
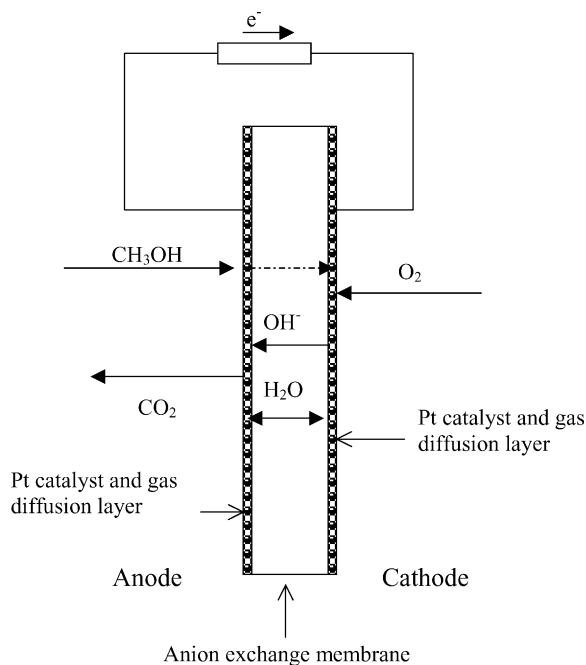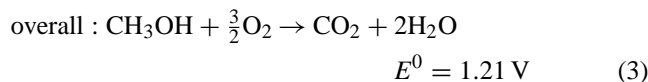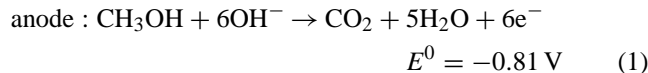*E-mail address:* k.scott@ncl.ac.uk (K. Scott).

Fig. 1. Schematic diagram of transport processes in DMAFC.

Fig. 1 shows the schematic diagram of the processes in a DMAFC. The reactions occurring on the electrodes are as follows:

$$\text{anode}: CH_3OH + 6OH^- \rightarrow CO_2 + 5H_2O + 6e^-$$
$$E^0 = -0.81\,\text{V} \qquad (1)$$

$$\text{cathode}: \tfrac{3}{2}O_2 + 3H_2O \rightarrow 6OH^- \quad E^0 = 0.402\,\text{V} \qquad (2)$$

$$\text{overall}: CH_3OH + \tfrac{3}{2}O_2 \rightarrow CO_2 + 2H_2O$$
$$E^0 = 1.21\,\text{V} \qquad (3)$$

A further potential advantage of the DMAFC is that the methanol oxidation catalyst is less structure sensitive in alkaline media than in acid media, which could lead to the use of less expensive non-precious metal catalysts, such as Ni or Ag.

In this research, we focus on the feasibility of applying anion exchange membrane in DMFCs, and investigate methods for preparing membrane electrode assembly (MEA). Preliminary fuel cell tests using the conventional hot-pressed MEAs with carbon supported Pt catalysts were carried out.

2. Experimental

2.1. Membrane pre-treatment

In this research, MORGANE®-ADP membranes (Solvay, S.A.) were used as the solid polymer electrolyte which was normally used for salt electrodialysis. Table 1 shows the basic properties of the MORGANE®-ADP membrane at 25 °C.

Table 1
Basic properties of the MORGANE®-ADP membrane

Membrane	MORGANE®-ADP
Material	Cross-linked fluorinated polymer
Exchange group	Quaternary ammonium
Thickness (μm) fully humidified	150–160
Resistance (in 0.6 M NaCl) ($\Omega\,cm^2$)	1.5–4.5
Resistance (in 1 M NaOH) (Ω)	0.5
Maximum operational temperature (°C)	55
Working pH	0–10

Before fabrication of the membrane electrode assembly (MEA), the membrane was soaked in 1 M NaOH for 24 h to change it from Cl^- form to OH^-. Then the membrane was rinsed and stored in Millipore water for later use.

2.2. MEAs preparation

Carbon supported Pt (60 wt.% on Vulcan XR72, Etek, USA) was used to prepare the catalyst ink. Inks were made by mixing the Pt/C catalyst, Millipore water and 5% PTFE (33%, ICI) suspension. The catalyst layer was prepared on a gas diffusion layer or non-wet-proofed Toray 90 carbon paper (Etek, USA). The gas diffusion layer (GDL) consisted of a layer of Ketjen black EC300 (Akzo Nobel, UK) carbon, with 10% PTFE suspension painted on top of the 20 wt.% Teflonised Toray 90 carbon paper. After the desired amount of Pt loading was achieved, the anode, cathode and membrane were sandwiched together and hot pressed at 120 kg cm^{-2} for 3 min at 100 °C.

2.3. Fuel cell tests

A stainless steel cell consisting of two compartments with 2 mm parallel channel flow field for methanol and air flow was employed in this research. The active cross-section area of the cell was 4.91 cm^2. The fuel used in this study was 2 kmol m^{-3} (M) methanol in 1 M NaOH at an operating temperature of 60 °C, unless otherwise specified. A peristaltic pump (Watson Marlow, UK) was used to supply methanol to the cell. A water bath (Cole Palmer, USA) and in-house-made temperature controllers were used to maintain the temperature at 60 °C. Before each test, the cell was loaded with short circuit current, which is the highest current that could be loaded from the cell by the electronic load under constant current polarisation, for few minutes to activate the cell. The open circuit voltage (OCV) and the anode open circuit potential (OCP) were recorded after stable values were reached. The anode potential was measured by a mercury/mercury oxide (MMO) reference electrode. The cathode potential was determined by subtracting the anode potential from the cell voltage. Data were taken after 24 h of cell conditioning. All potential values are referred to the reversible hydrogen electrode (RHE). A Kenwood PE-151 electronic load was used to obtain cell polarisation data.
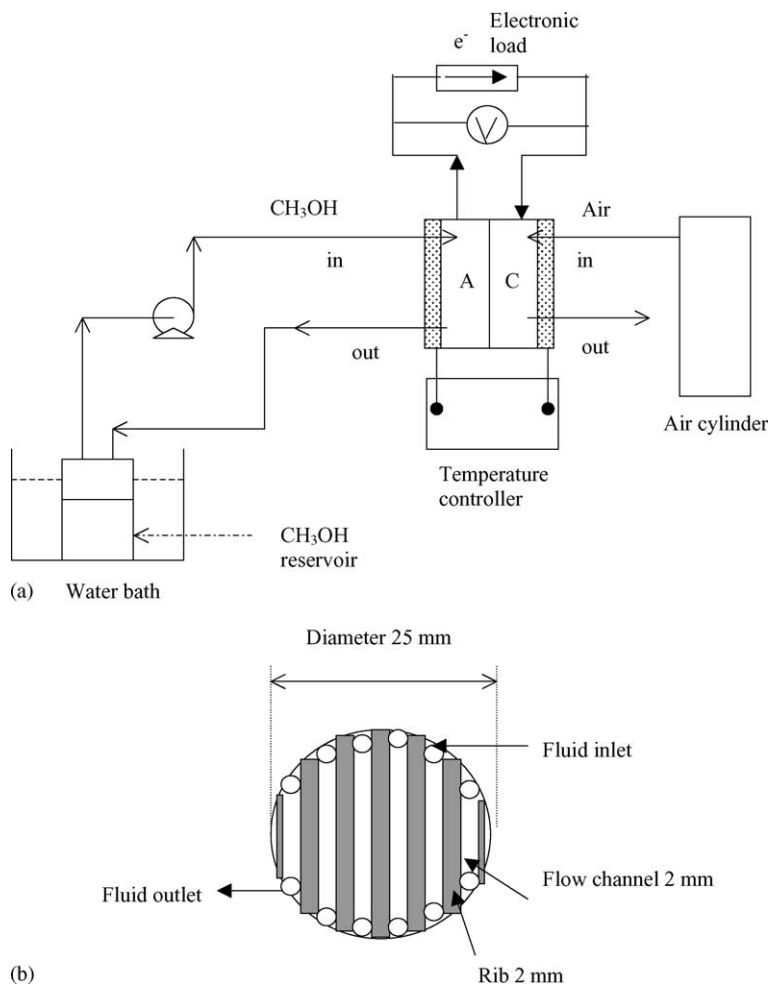
Fig. 2. (a) Flowchart of DMAFC operation process and (b) schematic diagram of the cell cross-section area and flow fields.

Fig. 2a and b show the flowchart of DMAFC experimental process and the cross-section of the cell.

The internal resistance (R_{cell}) was measured by the high frequency impedance obtained at open circuit.

3. Results and discussion

3.1. Characterisation of MORGANE®-ADP membrane

Preliminary evaluation of the methanol diffusion coefficient and electrical conductivity were investigated by previous researchers in our group. The results were compared with Nafion® membranes, and are shown in Figs. 3 and 4. It is clear from Fig. 3 that the electrical resistance of MORGANE®-ADP is much higher than Nafion®. In the case of the surface resistance, the value for MORGANE®-ADP is 16 times higher than that of Nafion®, and the resistance across the membrane of MORGANE®-ADP is about six times higher than that of Nafion®. Methanol diffusion coefficients were measured by monitoring the transfer of methanol from pure methanol solution across the membrane

into a NaOH solution. As shown in Fig. 4, the methanol diffusion coefficient for Nafion® is about 2.8 times higher than that for the ADP membrane. This low methanol diffusion coefficient indicates that the methanol crossover rate for ADP membrane will be potentially lower than Nafion®, which may improve fuel cell performance. However, the high electrical resistance could be a major obstacle limiting the ADP MEA performance. Furthermore, the ADP membrane is not stable in strong alkaline media. The suggested working pH is up to a value of pH 11. In fact, discolouring of the ADP membrane in 1 M NaOH was observed. This could affect the stability and the lifetime of a fuel cell with an ADP membrane.

3.2. Comparison between MEAs with and without gas diffusion layer

For the conventional hot-pressed electrode, the performances of cells with different anode configurations and gas diffusion layers, i.e. 20% Teflonised Toray 90 (Etek, USA) and gas diffusion layer (GDL) and non-Teflonised Toray 90 only, are compared in Fig. 5. It is clearly shown that the cell
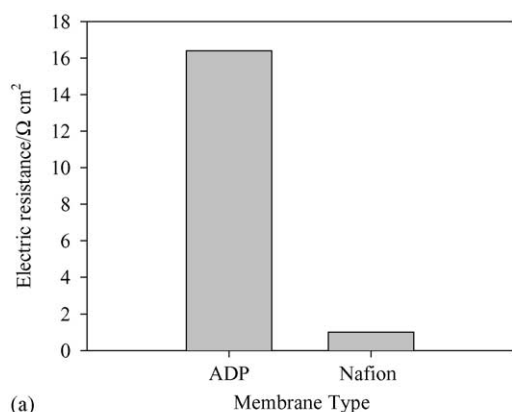
(a)
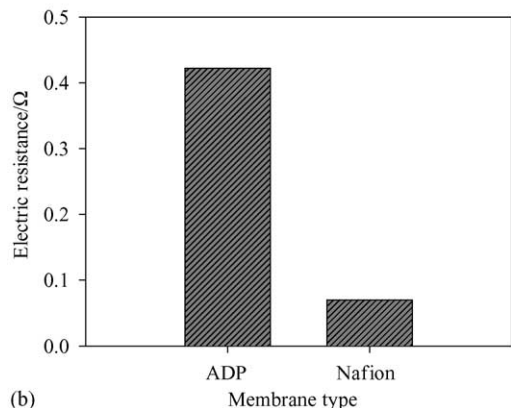
(b)

Fig. 3. Electrical resistance of ADP and Nafion® membranes. (a) Surface resistance, tested in 0.1 M NaOH and (b) resistance across the membrane. ADP: tested in 1 M NaOH, Nafion®: tested in 0.5 M H$_2$SO$_4$, all at room temperature.

performance increased dramatically without gas diffusion layer. The short circuit current densities were 69.3 mA cm^{-2} and 38.7 mA cm^{-2}, and the peak power densities were 11.7 and 5.3 mW cm^{-2}, for the MEA without and with a gas diffusion layer on the anode, respectively. As shown in Fig. 5, in the activation potential loss range 0–10 mA cm^{-2}, there is no significant difference in V–I curves for electrodes with
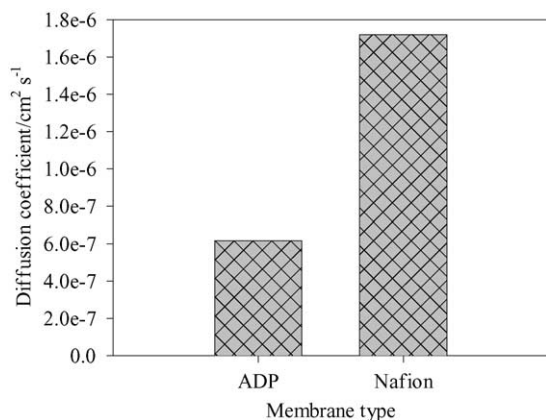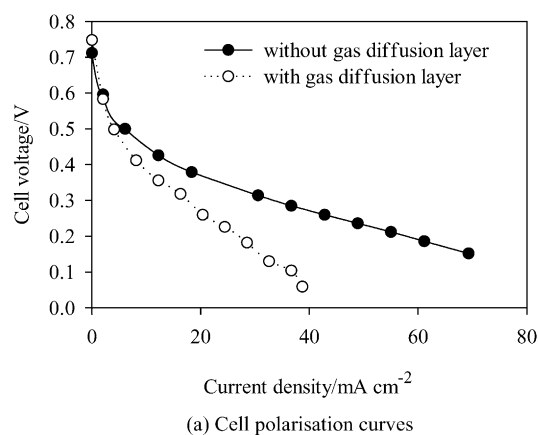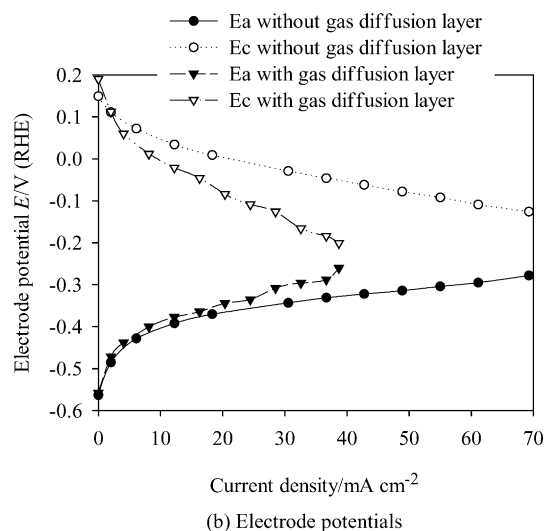

Fig. 4. Methanol diffusion coefficients of ADP and Nafion® membrane.

(a) Cell polarisation curves

(b) Electrode potentials

Fig. 5. Comaprison of cell performance between MEAs with and without GDL. With GDL: anode: Pt/C (60 wt.% Pt) 2.07 mg cm^{-2} with GDL on 20% Teflonised Toray 90 carbon paper; cathode: Pt/C (60 wt.% Pt) 2.07 mg cm^{-2} with GDL on 20% Teflonised Toray 90 carbon paper. Without GDL: anode: Pt/C (60 wt.% Pt) 2.14 mg cm^{-2} with non-Teflonised Toray 90; cathode: Pt/C (60 wt.% Pt) 2.36 mg cm^{-2} with GDL on 20% Teflonised Toray 90 carbon paper. Cell operated at 60 °C, ambient air with flow rate of 2 l min^{-1} and 2 M methanol in 1 M NaOH, 60.6 ml min^{-1}.

and without a gas diffusion layer. As the same catalyst fabrication was used, the catalytic activity was not affected by employing a gas diffusion layer. However, there is a large difference in potential with the two types of MEAs in the ohmic and mass transport potential regions. A similar difference can be observed from the anode potentials.

A large potential drop is exhibited in the higher current range for the MEA with a gas diffusion layer indicating a greater mass transfer barrier. There was no significant difference observed in the cell resistance. The cell resistances for the MEAs with only non-Teflonised carbon paper current collector and with both a gas diffusion layer and Teflonised carbon paper were both between 0.42 and 0.52 Ω. Since the gas diffusion layer does not affect the cell internal resistance significantly, the considerable difference in the cell performance with the two MEAs could be explained by the high

mass transfer resistance or diffusion resistance caused by the presence of the gas diffusion layer. Giorgi et al. [10] found that the presence of PTFE in the gas diffusion layer changed the electrode microstructure in two ways. First, the total porosity of the diffusion layer decreased; second, the catalytic surface area depended on the macro porosity of the diffusion layer. In this case, the presence of a Teflonised gas diffusion layer decreased the mass transport rate of the reactants as well as affecting the catalytic surface area. Another interesting phenomenon is that the open circuit voltage and anode potential recovered to their initial values almost instantly after removing the load for the MEA without the gas diffusion layer and Teflonised carbon paper; while for the MEA with a gas diffusion layer, it took more than approximately 2–3 min to recover. This is further evidence of an improvement of mass transport on the anode side using non-Teflonised carbon paper, without a gas diffusion layer. As a result, further fuel cell tests were performed without a gas diffusion layer at the anode side.

3.3. Fuel cell tests under various operational conditions

3.3.1. Effect of temperature

Fig. 6a and b shows the cell polarisation curves and electrode potentials obtained for tests performed at 20, 40 and 60 °C. It is clearly shown that the cell performance improved as the temperature increased. The higher temperature gave a higher open circuit voltage, which indicates that the catalyst activity is higher at higher temperatures. The electrode potentials show an interesting effect with a variation in temperature. On the cathode side, the open circuit potential and oxygen reduction polarisation increased with temperature but the differences at the three temperatures were not as significant as that for the anode. On the anode side, three almost parallel polarisation curves were observed and the curves shifted to more negative potential as the temperature increased. Furthermore, the open circuit potential also shifted to more negative values as the temperature increased. This behaviour confirms that the methanol oxidation was better at a higher temperature, which conforms with the study on methanol oxidation in alkaline media [11]. According to the cell polarisation and electrode potential curves, although the overall reaction activities were improved at higher temperature, the improvement in cell performance was mainly due to the improvement in methanol oxidation performance. This also implies that the effects of temperature on cathode oxygen reduction is not as sensitive as that for methanol oxidation in alkaline systems, which could allow the possible use of ambient air instead of preheated air in cell operation.

3.3.2. Effects of methanol flow rate

The effect of methanol flow rate on cell performance is shown in Fig. 7. Cell performance increased as the methanol flow rate increased possibly due to the improvement of mass
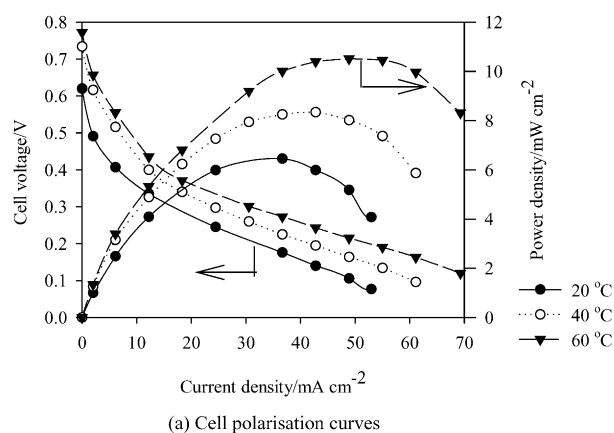
(a) Cell polarisation curves
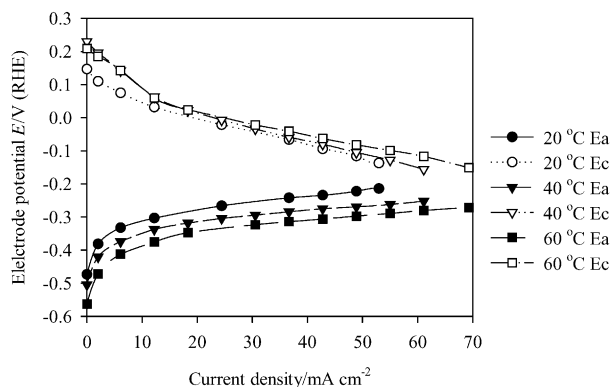
(b) Electrode potentials

Fig. 6. Temperature effects on cell performance. MEA: anode: Pt/C (60 wt.% Pt) $2.19\,\mathrm{mg\,cm^{-2}}$ on non-Teflonised Toray 90 carbon paper; cathode: Pt/C (60 wt.% Pt) $2.07\,\mathrm{mg\,cm^{-2}}$ with GDL on 20% Teflonised Toray 90 carbon paper. Cell operated at 20, 40 and 60 °C with air pressure 1 bar in 2 M methanol and 1 M NaOH, flow rate of $60.6\,\mathrm{ml\,min^{-1}}$.

transport on the anode. There was also a small effect of flow rate on the cathode reaction. The cathode performance improved with higher methanol flow rate at higher current densities, as shown in Fig. 7b. An explanation for this could lie in mass transport of methanol towards anode, which in turn, can increase the diffusion through the membrane, and affect cathode performance.

3.3.3. Effects of methanol concentration

As a high energy density in the fuel is generally desirable, a higher concentration of methanol is preferred in use. However, methanol crossover and poisoning effect are more significant at high concentration of methanol [12]. From the study of methanol oxidation, it has been shown that the reaction is not mass transfer controlled once the methanol and NaOH concentration ratio is greater than one [4]. To investigate the effect of methanol concentration in 1 M NaOH, 1, 2 and 4 M methanol were used and the cell polarisation curves are shown in Fig. 8. For 1 and 2 M methanol, cell performance was similar, although 2 M methanol was slightly better. Short circuit currents reached $70\,\mathrm{mA\,cm^{-2}}$ and peak power densities were 10–$11\,\mathrm{mW\,cm^{-2}}$.
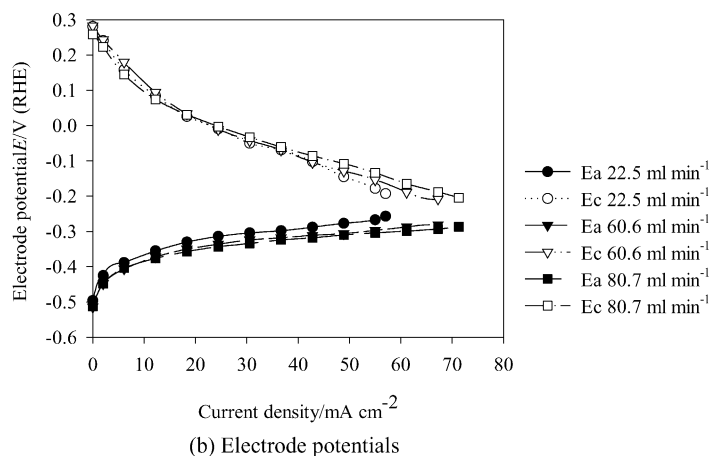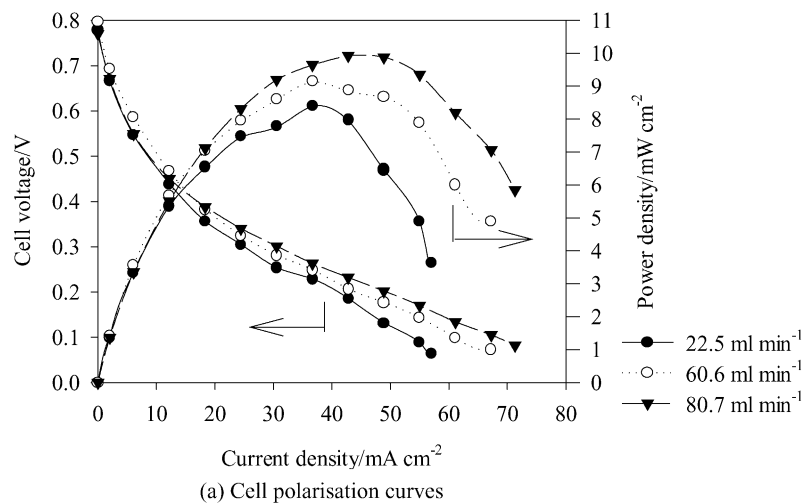
Fig. 7. Effects of methanol flow rate on cell performance. MEA: anode: Pt/C (60 wt.% Pt) 2.19 mg cm^{-2} on non-Teflonised Toray 90 carbon paper; cathode: Pt/C (60 wt.% Pt) 2.07 mg cm^{-2} with GDL on 20% wet-proofed Toray 90 carbon paper. Cell operated at 60 °C with air pressure 2 bar in 2 M methanol and 1 M NaOH with various flow rates.

A decrease in cell performance can be observed using 4 M methanol at higher current densities. The cell peak power performance was decreased by 20% compared to that with lower methanol concentrations; the short circuit current density was 60 mA cm^{-2} and peak power density was 8.5 mW cm^{-2}. Although the anode polarisation curves were similar for the three methanol concentrations in the activation polarisation region, 2 M methanol showed slightly better performance and 4 M showed the lowest performance. A slight decrease in the cathode performance was observed as the methanol concentration increased. Probably this is evidence that cathode performance was affected due to greater methanol crossover with higher methanol concentration. Similar tests were carried out with a lower Pt loading at the anode and they are discussed in the Section 3.3.4.

### 3.3.4. Effects of Pt loading on anode

MEAs with anode Pt loadings of 2.1 and 1.4 mg cm^{-2} were tested. Not surprisingly, the cell performance was better with higher Pt loading. As shown in Fig. 9, a larger activation

potential drop was observed for Pt loading of 1.4 mg cm^{-2} comparing to Pt loading of 2.1 mg cm^{-2}. Two near parallel lines in the *V–I* curves indicate similar features for the ohmic and mass transfer resistance regions for the two electrodes. The larger activation potential drop for the lower Pt loading is due to lower active area of the catalyst.

To illustrate better the effects of Pt loading at the anode, cell test for various methanol concentrations were obtained and the results are shown in Fig. 10. As with the data for a Pt loading of 1.4 mg cm^{-2}, the 4 M methanol solution showed the lowest performance and was significantly lower than that with 1 M methanol, which gave the better performance with lower Pt loading. Also the cathode performances showed similar features as for higher Pt loading (2.1 mg cm^{-2}), discussed in the previous Section 3.3.3. Overall, with a lower Pt loading which has a lower catalyst active area, it is more easily poisoned when exposed to higher concentration of methanol. Hence, to operate the cell with higher methanol concentrations, higher Pt loadings are preferred.
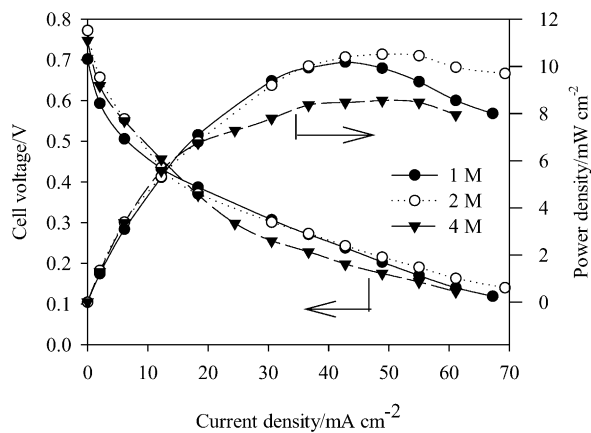
(a) Cell polarisation curves
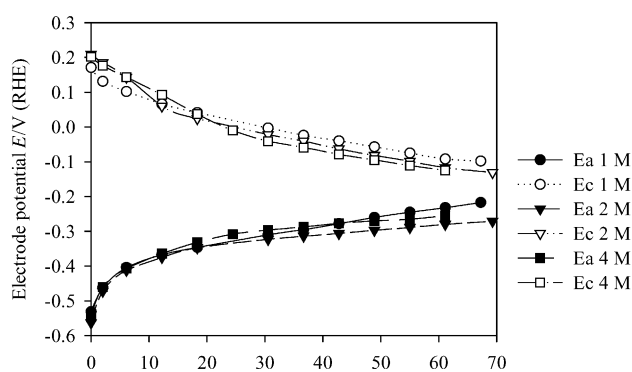
(b) Electrode potentials

Fig. 8. Effects of methanol concentration on cell performance. MEA: anode: Pt/C (60 wt.% Pt) 2.14 mg cm^{-2} on non-Teflonised Toray 90 carbon paper; cathode: Pt/C (60 wt.% Pt) 2.36 mg cm^{-2} with GDL on 20% Teflonised Toray 90 carbon paper. Cell operated at 60 °C with air pressure 1 bar in 1 M NaOH with methanol concentrations of 1 M, 2 M and 4 M, flow rate 60.6 ml min^{-1}.
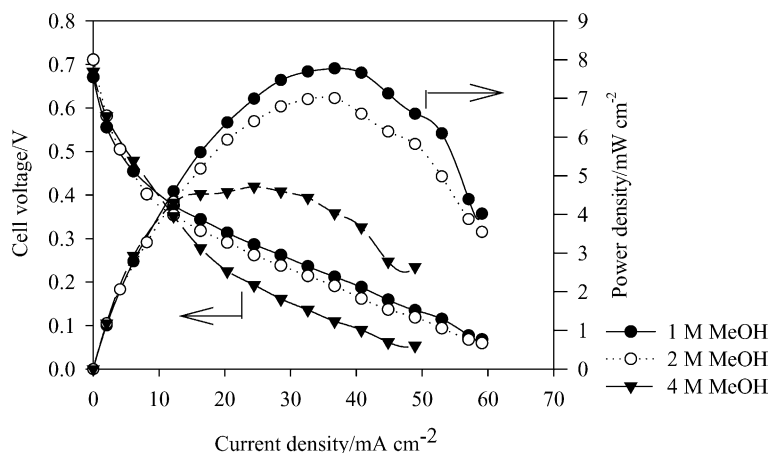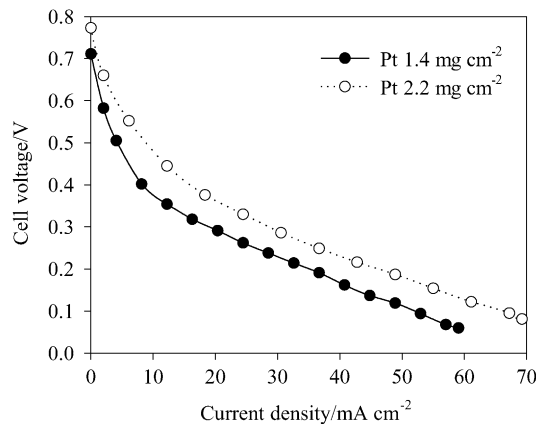

Fig. 9. Effect of anode Pt loading on cell performance, Pt 2.2 mg cm^{-2}: anode: Pt/C (60 wt.% Pt) 2.2 mg cm^{-2} on non-Teflonised Toray 90 carbon paper; Pt 1.4 mg cm^{-2}: anode: Pt/C (60 wt.% Pt) 1.4 mg cm^{-2} on Toray 90; cathode: Pt/C (60 wt.% Pt) 2.07 mg cm^{-2} with GDL on 20% Teflonised Toray 90 carbon paper. Cell operated at 60 °C, air pressure 1 bar and 2 M methanol in 1 M NaOH, flow rate 60.6 ml min^{-1}.

3.3.5. Effects of cathode air pressure

In general, higher cathode pressure provides higher oxygen partial pressure and can reduce methanol crossover from the anode [13,14]. To examine the effect of air pressure on cell performance, air at 0 bar at a flow rate of 2 l min^{-1}, 1 and 2 bar, were applied at the cathode. As shown in Figs. 5–10, the cell polarisation curves at 60 °C, there is virtually no difference in cell performance with air at 1 and 2 bar. Also there is, as might be expected no effect of air pressure on anode performance (Figs. 5-10b). The highest open circuit voltage was obtained for an air pressure of 2 bar and the lowest for ambient air, which is a result of the effect of pressure on cathode open circuit potential. As a consequence, the cell performances increased as the air pressure increased, which indicates that oxygen partial pressure plays a role on the performance of the cathode. Similar cathode behaviour
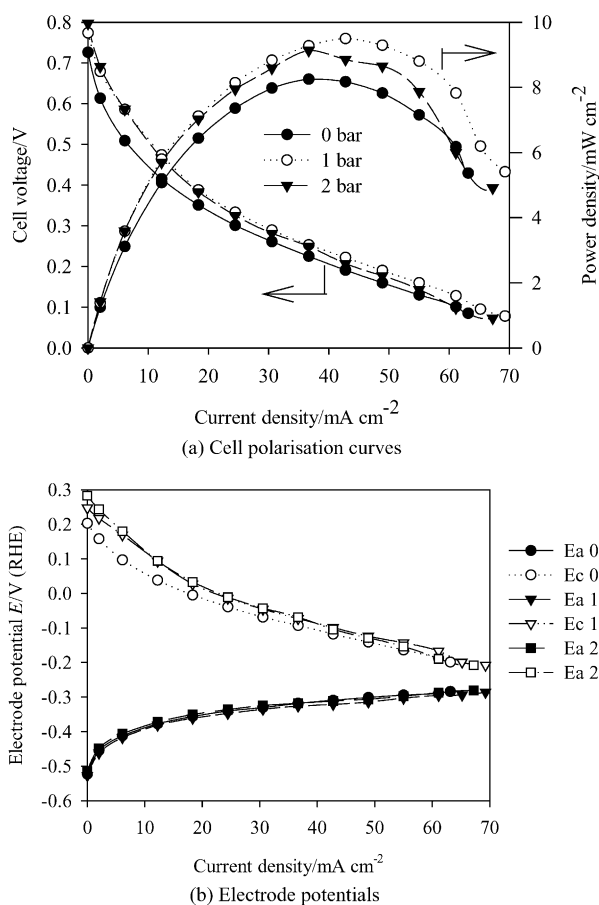

Fig. 10. Effects of methanol concentration on cell performance. MEA: anode: Pt/C (60 wt.% Pt) 1.42 mg cm^{-2} on non-Teflonised Toray 90 carbon paper; cathode: Pt/C (60 wt.% Pt) 2.05 mg cm^{-2} with GDL on 20% Teflonised Toray 90 carbon paper. Cell operated at 60 °C with air pressure 1 bar in 1 M NaOH with methanol concentrations of 1, 2 and 4 M, flow rate 60.6 ml min^{-1}.

(a) Cell polarisation curves

(b) Electrode potentials

Fig. 11. Effects of air pressure on cell performance. MEA: anode: Pt/C (60 wt.% Pt) 2.19 mg cm^{-2} on non-Teflonised Toray 90 carbon paper; cathode: Pt/C (60 wt.% Pt) 2.07 mg cm^{-2} with GDL on 20% Teflonised Toray 90 carbon paper. Cell operated at 60 °C with various air pressures: 0 bar, 1 bar and 2 bar in 1 M NaOH with methanol concentrations of 2 M, flow rate 60.6 ml min^{-1}.
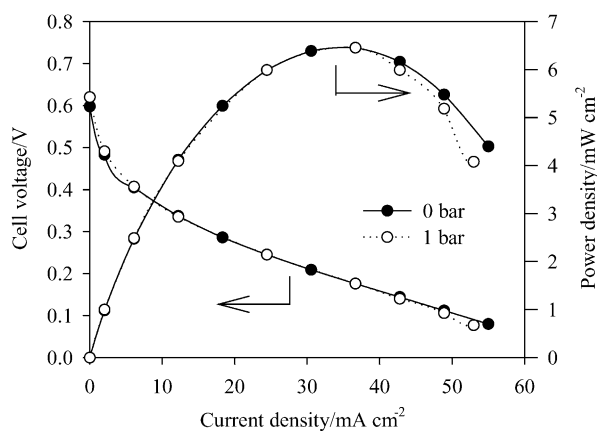

Fig. 12. Effects of air pressure on cell performance at 20 °C. MEA: anode: Pt/C (60 wt.% Pt) 2.19 mg cm^{-2} on non-Teflonised Toray 90 carbon paper; cathode: Pt/C (60 wt.% Pt) 2.07 mg cm^{-2} with GDL on 20% Teflonised Toray 90 carbon paper. Cell operated at 20 °C with various air pressures: 0 bar and 1 bar in 1 M NaOH with methanol concentrations of 2 M, flow rate 60.6 ml min^{-1}.

was also observed at 40 °C. However, the differences in the cathode and cell performance at 0, 1 and 2 bar decreased as the temperature decreased. In fact, there was no significant difference in cell performance between pressurised and ambient air at 20 °C, as show in Figs. 5–11.

3.3.6. Cell tests with humidified and dry air

One difference between the acidic DMFC and DMAFC is that in the DMFC, water is the product of oxygen reduction on the cathode; while in DMAFC, water is a reactant for oxygen reduction. Flooding is a common phenomenon in DMFC but has not been observed in DMAFC using ADP membranes. Although water does diffuse through the membrane from anode to cathode, the reverse electro osmotic drag and the low water permeation of anion exchange membrane could limit the water transport. To evaluate the impact of
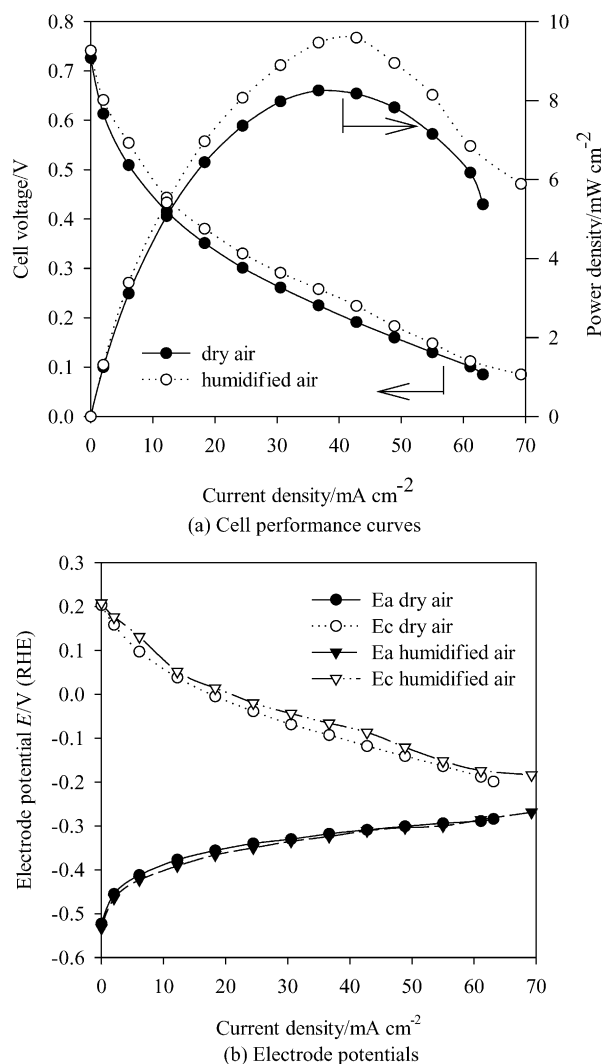

(a) Cell performance curves

(b) Electrode potentials

Fig. 13. Comparison between dry air and humidified air; MEA: anode: Pt/C (60 wt.% Pt) 2.19 mg cm^{-2} on non-Teflonised Toray 90 carbon paper; cathode: Pt/C (60 wt.% Pt) 2.07 mg cm^{-2} with GDL on 20% wet-proofed Toray 90 carbon paper. Cell operated at 60 °C, ambient air with flow rate of 2 l min^{-1} in 1 M NaOH, 2 M methanol with flow rate of 60.6 ml min^{-1}.

water on the cathode performance, humidified air was used. Humidification of air was accomplished by sparging the air stream through heated water-filled glass column maintained at 60 °C for an hour. The air was at ambient pressure and with the flow rate of 2 l min^{-1}. It is clearly shown in Figs. 12 and 13 that the cell and cathode performance were slightly improved by using humidified air. This suggests that water is an important factor influencing the cell performance, especially the cathode performance. Moreover, the cell internal resistance was decreased from 0.5 Ω, for dry air, to 0.45 Ω, for humidified air. It suggests that the cell resistance could be reduced by increasing the water content of the MEA and further improvement on cathode performance is possible by improving the humidification.

4. Conclusions

Demonstration of the DMAFC using MORGANE®-ADP anion exchange membranes was carried out conclusions drawn as follows.

- The MORGANE®-ADP membrane has a higher resistance compared to Nafion® membranes, which could be the main hindrance in improving fuel cell performance. However, the methanol diffusion coefficient for the MORGANE®-ADP membrane is lower than that for the Nafion® indicating a lower methanol crossover.
- The cell performance could be improved dramatically by eliminating the gas diffusion layer (GDL) and using non-Teflonised carbon paper as the current collector.
- The cell performance improved with increasing temperatures, mainly due to the improvement in the anode methanol oxidation.
- Air pressure of 1 bar seems the optimum cathode air pressure for good cell performance. Moreover, the cathode performance can also be enhanced by improving the humidification of the air.

- Methanol crossover and a "poisoning" of the anode increased as the methanol concentration increased. When using higher methanol concentrations, a higher Pt loading in the anode catalyst is preferred.

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