



# Improvement of proton exchange membrane fuel cell performance in low-humidity conditions by adding hygroscopic agarose powder to the catalyst layer



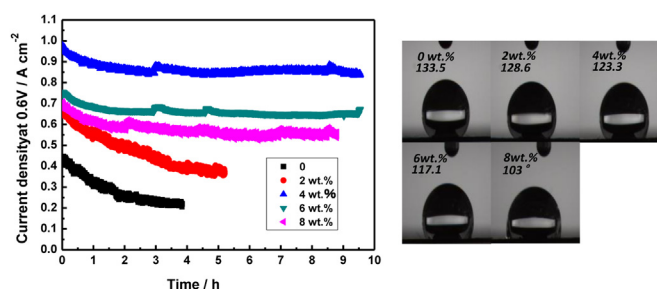
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## HIGHLIGHTS

- A self-humidifying MEA is prepared by adding agarose to the anode catalyst layer.
- The MEA shows excellent self-humidification performance at 60 °C and 20% RH.
- The current density is up to 500 mA cm<sup>-2</sup> at 0.7 V at 60 °C and 20% RH condition.
- The long term testing of 10 h reveals the good stability of the MEA at low RH.

## GRAPHICAL ABSTRACT



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## ABSTRACT

A high-performance membrane electrode assembly (MEA) with agarose added to the anodic catalyst layer (CL) was successfully prepared. The MEA exhibited excellent performance at low relative humidity (RH) in an air/hydrogen proton exchange membrane fuel cell. The effects of agarose content, RH, cell temperature, and back pressure on the low-humidity performance of this MEA were investigated. The results of water contact angles and water uptake measurements reveal that the hydrophilicity of the anode is significantly improved with the addition of agarose. With a low RH of 20% and a cell temperature of 60 °C, the optimal MEA (MEA-4), containing 4 wt.% agarose in the anode CL, achieves excellent low-humidity performance: the current density reaches 960 mA cm<sup>-2</sup> at 0.6 V and 500 mA cm<sup>-2</sup> at 0.7 V. After 10 h of continuous operation under the same conditions, the current density decreases just slightly, from 960 to 840 mA cm<sup>-2</sup>, whereas the current density of a blank MEA without added agarose degrades sharply.

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## 1. Introduction

Developing a self-humidifying membrane electrode assembly (MEA) that would make it possible for a MEA or proton exchange membrane fuel cell (PEMFC) to operate in conditions of low or no

humidity has recently become an attractive topic in the field of fuel cell research. A MEA with high-performance and self-humidifying could simplify fuel cell systems, decrease their cost, and make fuel cell water management easy—all factors that would accelerate the commercialization of PEMFCs.

A facile and effective method of preparing a self-humidifying MEA is to add hygroscopic materials into the anode catalyst layer (CL). Some researchers have reported [1–13] that the addition of hygroscopic SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, PVA, or ZnO particles into the anode CL

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could improve the wettability of MEA and thus enhance its performance under low or no humidity conditions. Jung et al. [1] prepared a self-humidifying MEA by directly dispersing hydrophilic SiO<sub>2</sub> particles into the anode CL. The MEA's wettability and performance under low-humidity conditions were remarkably improved. With 0% relative humidity (RH) at the anode and 80% RH at the cathode, an MEA with 40 wt.% SiO<sub>2</sub> in the anode CL exhibited better performance than a blank MEA: the current density at 0.6 V reached 840 mA cm<sup>-2</sup>, although they did not provide long-term discharge data. Inoue et al. [12] prepared CLs containing SiO<sub>2</sub> nanoparticles (SiO<sub>2</sub>-CLs) to improve the low-humidity performance of a PEMFC. The cell achieved the highest performance when SiO<sub>2</sub>-CLs were used for both the anode and the cathode; at 30% RH and 80 °C, the current density at 0.7 V was 2.7 times larger than that of without SiO<sub>2</sub>. Su et al. [5] fabricated a Pt-SiO<sub>2</sub>/C self-humidifying catalyst via an organic colloid method. When this catalyst was used in the anode CL, at a cell temperature of 50 °C and 28% RH for both anode and cathode, the current density at 0.6 V remained at 0.65 A cm<sup>-2</sup> without any degradation for 20 h. It was suggested that the silica on the carbon support served as a mini water reservoir, absorbing back-diffused water from the cathode layer and thereby improving the low- or non-humidification performance of the PEMFC. Liang et al. [11] attempted to prepare a self-humidifying MEA by adding polyvinyl alcohol (PVA)—an organic polymer material rather than an inorganic one—to the anode CL; the operating temperature of this MEA was up to 50 °C at an RH of 34%, with the current density at 0.6 V reaching 1000 mA cm<sup>-2</sup> and remaining at 750–780 mA cm<sup>-2</sup> after 60 h of constant voltage testing.

Some researchers [14–20] prepared self-humidifying MEAs by inserting a hydrophilic layer between the gas diffusion layer (GDL) and the CL. The MEAs with a thin hydrophilic layer coated on the carbon paper effectively avoided membrane dehydration and cathode flooding, enabling them to achieve high performance in low humidity. Nonetheless, although great progress has been achieved, there is still a long way to go before a self-humidifying MEA is developed that meets the requirements for practical use and commercialization.

Agarose is a natural polysaccharide polymer material, generally extracted from seaweed, with a linear polymer made up of repeating units of agarobiose. Its water uptake is 98–99%, and it is highly stable—characteristics that make it a potential hygroscopic additive in self-humidifying MEA applications. As part of our continuing endeavors to prepare self-humidifying MEAs, in the present study we added agarose to the anode CL. As expected, the MEAs exhibited excellent self-humidifying performance under low humidity at a cell temperature of up to 60 °C.

## 2. Experimental

### 2.1. Preparation of MEAs

MEAs with an active area of 5 cm<sup>2</sup> were fabricated using a direct catalyst spraying technique developed in our laboratory [21]. Before spray deposition of the catalyst layers, Nafion 212 membranes (DuPont, USA) were pretreated using a standard procedure: treatment in a 5 wt.% H<sub>2</sub>O<sub>2</sub> solution at 80 °C for 1 h, followed by treatment in deionized water at 80 °C for 1 h, then in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution at 80 °C for 1 h, and finally in deionized water again at 80 °C for 1 h. Anode catalyst inks were prepared by mixing commercial Johnson Matthey HiSPEC 4100 Pt/C catalyst (40 wt.%), agarose powder, isopropanol, and 5 wt.% Nafion solution (DuPont, USA). The cathode catalyst ink was prepared by the same method, but without agarose. The Pt loadings at the anode and cathode were 0.1 and 0.2 mg cm<sup>-2</sup>, respectively, and the dry Nafion content in the

anode and cathode CLs was adjusted to 25 wt.%. The anode CL and cathode CL were prepared by spraying the catalyst ink onto both sides of the pretreated membrane. In this work, we prepared four MEAs, with anode CL agarose contents of 2, 4, 6, and 8 wt.%, which we refer to as MEA-2, MEA-4, MEA-6, and MEA-8, respectively. For comparison, we also prepared a MEA without adding agarose, which we call MEA-0.

### 2.2. Measurement of anode catalyst layer contact angle

The sessile drop method is the most common means of measuring the contact angle of a liquid on a solid surface. Using this technique, we set a drop of water on the surface of the anode CL and measured the contact angle by fitting a tangent to the three-phase point where the liquid surface touched the solid surface. To characterize the wetting property of the CL of our MEAs, we determined the contact angle of a water drop on the CL experimentally by direct image analysis of the shape of the drop at room temperature.

### 2.3. Measurement of water uptake

The water uptake of the anode CL was measured by a method described in the literature [11], whereby the wet weight was measured after immersing the sample in hot water for 4 h, and the dry weight was measured by drying the membrane at 80 °C for 4 h. The water uptake (%) was calculated using the following equation:

$$Wup\% = \frac{m_{wet} - m_{dry}}{m_{dry}} \times 100\%$$

where  $m_{wet}$  and  $m_{dry}$  are the weights of the wet and dry samples (g), respectively.

### 2.4. Single-cell tests

A MEA was assembled using a catalyst-coated membrane and two pieces of GDL without hot pressing, and performance testing was carried out in a standard single cell with an active area of 5 cm<sup>2</sup>, using a Fuel Cell Testing System (Arbin Instruments, USA). Pure hydrogen and compressed air were fed to the anode and cathode, respectively, at flow rates of 300 ml min<sup>-1</sup> (hydrogen) and 800 ml min<sup>-1</sup> (air). Before the performance test, the cells were activated in a continuous discharge mode until stable performance was obtained; the activation was processed at a cell temperature of 70 °C and with 100% humidification of the hydrogen and air. The cell performance at various RH levels was measured at a cell temperature of 60 °C, with the back pressures of both hydrogen and air at 30 psi. The temperatures of the gas lines to the anode and the cathode were always set at 5 °C above the humidification temperatures to prevent water condensation.

### 2.5. Electrochemical measurements

Electrochemical impedance spectroscopy (EIS) was used to study the polarization resistance of the MEAs, using a Zahner IM6e electrochemistry station (Zahner, Germany). These measurements were carried out at a cell potential of 0.8 V, with amplitude of 5 mV, and in the frequency range of 0.1 Hz–1 kHz.

## 3. Results and discussion

Fig. 1 presents photographs of the contact angles of the anode CLs with and without the addition of agarose. The water contact angles of the anode CLs with 0, 2, 4, 6, and 8 wt.% agarose were 133.5, 128.6, 123.3, 117.1, and 103°, respectively, showing that the

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