

# Fabrication and Performance of Membrane Electrode Assembly Prepared by a Catalyst-Coated Membrane Method: Effect of Solvents Used in a Catalyst Ink Mixture

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The effect of solvent types used to prepare catalyst-coated membrane (CCM) electrodes on the coating ink, applied coating process, interaction of catalyst ink and Nafion membrane and, thus, on the performance and power density of the proton exchange membrane fuel cells (PEM fuel cells) was investigated. Among seven types of solvents studied, the catalyst ink prepared using the isopropanol solvent showed the highest cell performance, followed in descending order by ethanol, acetone, water–ethylene glycol, ethylene glycol dimethyl ether (EGDME), and ethylene glycol diethyl ether (EGDEE). The superior performance was due to the good attachment of catalyst layer to membrane and the good surface coverage revealed by the results from the scanning electron microscope (SEM) of the CCMs, optical transmittance of the CCMs, and solvent absorption capacity of membrane. In electrode preparation by the CCM method, the solvent–ionomer mixture in solution form was more favorable than that in colloid form, as it gave higher cell performance. Finally, the catalyst-coated membrane on the H<sup>+</sup> form Nafion demonstrated better power density than the CCM on the Na<sup>+</sup> form Nafion.

## 1. Introduction

In order to be competitive with conventional power generation systems, fuel cell system cost has to be reduced, especially for automotive applications.<sup>1</sup> The cost of proton exchange membrane fuel cells (PEM fuel cells) depends primarily on the costs of platinum and the membrane; hence, the increase of Pt-utilization for fuel cell electrodes is one of the major issues in fuel cell research activities for the development of a new electrode fabrication technique. In previous literature, there were three different electrode preparation methods, a so-called decal method, a gas diffusion layer (GDL)-based method, and a membrane-based method or catalyst-coated membrane (CCM) method.<sup>2</sup> For the decal method, the catalyst ink is brushed onto a Teflon film before being transferred to form the catalyst layer on the Na<sup>+</sup> form Nafion membrane using a hot pressing process.<sup>3</sup> In the GDL-based method, the electrode is formed by applying the catalyst ink directly onto the GDL and then hot pressing it with the membrane.<sup>4,5</sup> For the membrane-based method, the catalyst ink is directly applied onto a dry, blank membrane, followed

by a hot pressing step.<sup>6,8</sup> It has been reported that the decal method has a risk of uneven or incomplete transfer of catalyst from the transfer film to the membrane<sup>2</sup> in contrast to the membrane-based technique, which also produces a higher MEA performance than the decal transfer process since it provides a better ionic connection between the membrane and the ionomer in the catalyst layer.<sup>6,7</sup> For these reasons, we would restrict our investigations to the membrane-based method.

To form a high-performance electrode which has high catalyst utilization, as well as low contact and mass transfer resistances, the solvent used for the catalyst ink preparation plays a key role because solvent type not only affects the formation of the proton conduction network but also influences the intimate contact between the catalyst layer and the membrane due to the swelling property of the membrane during the coating process. Although various solvents had been studied to fabricate PEM fuel cell electrodes with the CCM technique, these published data could not be compared because of the differences in many parameters used in those studies.<sup>2,6,7</sup> Previously, the effects of organic solvent types on the performance of PEM fuel cell electrodes prepared by the GDL-based method<sup>4,5</sup> and the decal method<sup>3</sup> were investigated. However, the results could not be applied to the efficient method like CCM. Therefore, the aims of this research were to investigate the effects of solvents used for forming PEM fuel cell electrodes with the CCM technique on cell

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**Table 1. Properties of the Solvents Used in Catalyst Ink Preparation, Membrane (Swelling Ratio), and the Resultant CCM Electrodes<sup>a</sup>**

solvent	boiling point (°C)	viscosity (cP)	dielectric constant <sup>9,10</sup>	swelling ratio (%)	transmittance (%)	surface quality	power density (mW/cm <sup>-2</sup> )	performance ranking
iso-propanol	82.0	2.10	18.3	199	0.0 ± 0.002	S	442	1
ethanol	78.0	1.07	24.3	238	0.0 ± 0.002	S	352	2
acetone	56.0	0.31	20.7	154	0.121 ± 0.002	C	301	3
deionized water	100.0	0.89	80.4	39	0.266 ± 0.002	C	268	4
ethylene glycol	197.3	16.10	37.0	83	0.263 ± 0.002	C	294 <sup>b</sup>	5
EGDME	85.0	0.46	7.2	110	0.122 ± 0.002	C	213	6
EGDEE	121.4	0.70	3.8	75	0.136 ± 0.002	C and PA	200	6

<sup>a</sup> S, smooth surface; C, cracked surface; PA, poor adhesion. <sup>b</sup> Higher power density than deionized water but its performance reduced rapidly in the high current density region.

polarization curve and power density and to understand the influence of physical properties of solvents on the catalyst coating on the membrane. Eventually, among the seven solvents studied, i.e., acetone, isopropanol, ethylene glycol, ethanol, ethylene glycol dimethyl ether (EGDME), ethylene glycol diethyl ether (EGDEE), and deionized water, the most appropriate solvent would be determined.

## 2. Experimental Section

**2.1. Experimental Setup. Catalyst Coated Membrane Preparation.** The catalyst ink was prepared by mixing the commercial Pt/C (20 wt % Pt/C, E-TEK) with Nafion solution (5 wt % in aliphatic alcohol, ElectroChem. Inc.) at the ratio of 7:3 in each of the solvents. The seven solvents selected for this study were acetone, isopropanol, ethylene glycol, ethanol, ethylene glycol dimethyl ether (EGDME), ethylene glycol diethyl ether (EGDEE), and deionized water, as mentioned previously. Their physical properties such as dielectric constant, viscosity, and boiling point are shown in Table 1. The mixture of the above components was sonicated for 2 h before applying it onto the treated membrane at 0.5 mg/cm<sup>-2</sup> Pt loading to form the cathode catalyst layer of a membrane electrode assembly (MEA). The catalyst layer was applied by the painting technique under the controlled air velocity of 4.2 m/s and temperature of 44 °C. The MEA, thus, obtained was then vacuum-dried at 70 °C for 1 h in a controlled temperature vacuum drying oven. The CCMs fabricated from the catalyst ink prepared from isopropanol, ethanol, ethylene glycol, EGDME, EGDEE, and deionized water were designated as IPA-CCM, ET-CCM, EG-CCM, EGM-CCM, EGE-CCM, and DI-CCM, respectively.

Before applying the catalyst ink onto Nafion 115 membrane, the electrolyte membrane was pretreated following three main steps to remove organic material and activate the membrane. It was first cleaned in 3% H<sub>2</sub>O<sub>2</sub> solution, then soaked in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution, and followed by washing in deionized water. All treatment processes were conducted at 70 °C (±2 °C).

**Preparation of MEA.** The commercial gas diffusion electrode (GDE, LE2500E01 from E-TEK) was used as the anode for all MEAs prepared. The commercial gas diffusion layer (GDL, LT2500, E-TEK) was used as the backing of the cathode side. The commercial GDE, the prepared CCM cathode, and the GDL were inserted in place during the MEA fabrication. The cell was then hot pressed using a hydraulic press at 150 °C and 6.9 × 10<sup>5</sup> Pa for 120 s.

**2.2. Testing and Analysis. Membrane Characterization.** The pretreated membrane was characterized for solvent absorption capacity through the swelling ratio. First, the membrane was

vacuum-dried at 70 °C for 24 h, before the volume (width × length × thickness) of the membrane was measured. After that, it was soaked in each solvent for 24 h and the remaining solvent was wiped off. Then, all three dimensions were measured. The swelling ratio of the membrane was calculated by

$$\text{swelling ratio} = \frac{V_w - V_d}{V_d} \times 100\%$$

where  $V_w$  and  $V_d$  are the volumes of wet and dried membranes, respectively.

**Characterization of CCM and MEA.** The CCMs were characterized for surface coverage of the membrane by catalyst layer and surface appearance. The former was determined from optical transmittance analysis by a UV–vis–near infrared (NIR) recording spectrophotometer (Shimadzu, UV-3100) in the wavelength range of 400–700 nm. Measurements were made at nine different positions of each sample, and the values were averaged. The latter was physically characterized using a scanning electron microscope (SEM) (JSM 5410, JEOL). The SEM was also used to visualize the cross-sectional view of the finished MEAs and determine the thickness of the electrolyte membrane in the samples.

**Cell Polarization Test.** The performances of the MEAs were evaluated in a 5 cm<sup>2</sup> active area commercial PEM fuel cell housing (ElectroChem. Inc.) including gaskets, bipolar plates, a current collector, and clamping bolts and nuts. The test station consisted of two main systems: a gas feeding system and a control system as shown in Figure 1. The gas feeding system served to supply the fuel and oxidant gases fed to the cell, while the control system allowed the operating conditions of the single cell to be measured and adjusted. The cell testing was conducted at 80 °C cell temperature with a 90 °C humidification temperature of the reactant gases. High purity (99.99%) oxygen and ultra high-purity (99.995%) hydrogen were applied as the oxidant at the cathode and the reactant gas at the anode side, respectively, with an identical flow rate of 0.9 mL/s on both sides. Before testing the cell, the MEA was activated at 0.6 V (±0.001 V) for 24 h. The current–voltage properties of the MEA in the potential change mode were measured using a digital multimeter.

## 3. Results

**3.1. Effect of Solvent Used for CCM Fabrication.** The performances of the cells prepared using different solvents are depicted as polarization curves and power density curves in Figure 2a,b, respectively. It is shown that the performance of the cell composing the catalyst prepared from isopropanol was the best, followed by that of ethanol, acetone, water, ethylene glycol, EGDME, and EGDEE, in that order. It was noticed that all three highest performance cells were fabricated using alcohol-based solvent. The catalyst-coated-membrane electrode fabricated using ethylene glycol as the

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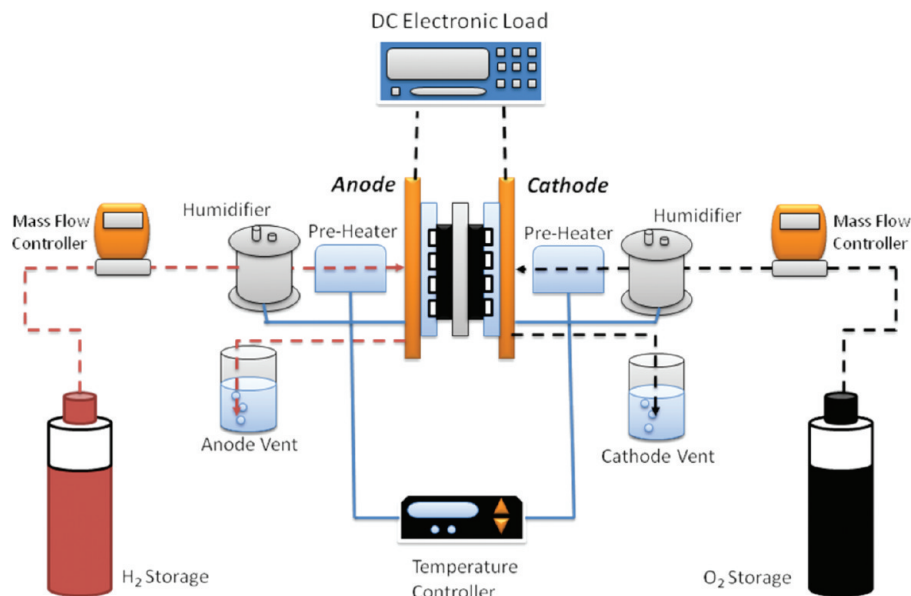


Figure 1. Schematic diagram of cell performance test setup.

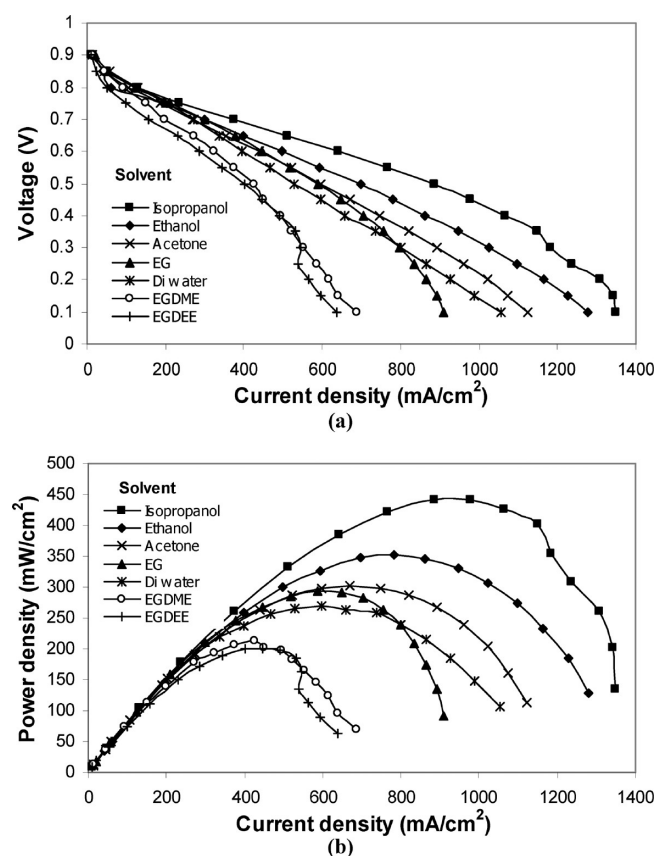


Figure 2. Effect of solvent on the (a) polarization curve and (b) power density curve of the MEAs consisting of different CCMs prepared using different solvents. Testing condition: 80 °C cell temperature, 90 °C humidifier temperature, and 1 atm.

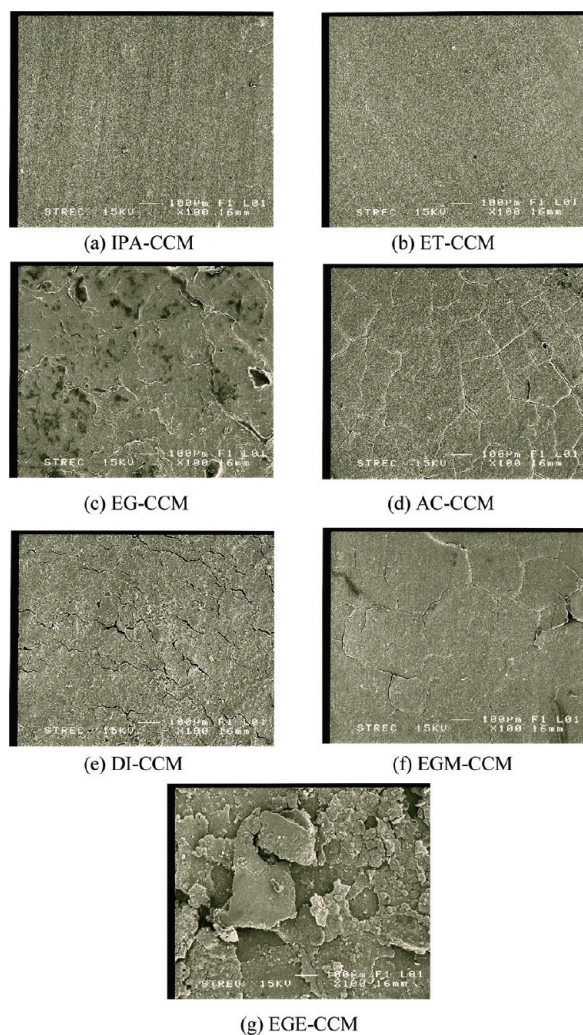
solvent showed a high performance at low and medium current density regions; however, its performance was rapidly diminished in a high current density region. Since ethylene glycol has a high boiling point, EGDME and EGDEE were selected as solvents in order to substitute ethylene glycol. Unfortunately, both EGM-CCM and EGE-CCM showed the lowest performance.

**Optical Transmittance of CCM.** The catalyst membrane coverage could be quantitatively determined via the optical transmittance of the CCM samples, the values of which are shown in Table 1. The lower the transmittance value, the better the coverage would be of the surface by the catalyst layer and vice versa. The values showed that the IPA-CCM and ET-CCM samples gave zero transmittance while the EG-CCM, AC-CCM, DI-CCM, EGM-CCM, and EGE-CCM samples gave higher values. These results indicated that both IPA-CCM and ET-CCM samples had good catalyst layer coverage whereas the remainders performed more poorly in this regard.

**SEM Images of MEA.** The morphology of the catalyst layer surface and the cross-section of the resultant MEAs investigated by SEM micrographs are shown in Figure 3. It can be seen that the IPA-CCM and ET-CCM had smooth surfaces without any cracks, whereas the rest of the CCMs showed rough surfaces with a few cracks. These results correspond with the optical transmittance values, where the crack-free catalyst layer showed a zero optical transmittance value while the cracked layer showed substantial transmittance.

**3.2. Effect of Charge Form of Membrane Used for CCM Fabrication.** In the decal-transfer method, the  $H^+$  form of the Nafion membrane was normally converted to the  $Na^+$  form by boiling it in NaOH solution prior to the transfer-printing step. In this research, the effect of the membrane form on the cell performance was investigated for the catalyst coated membranes prepared using isopropanol and acetone solvents. The polarization and power curves of the MEAs prepared from both  $Na^+$  and  $H^+$  type membrane are compared in Figure 4. It can be seen that the electrode fabricated on the  $H^+$  form membrane gave higher performance than that applied on the  $Na^+$  form membrane in the cases of both solvents. The drop in performance of the  $Na^+$  form MEA may be caused by the detachment of some catalyst powder from the MEA when boiling this coated  $Na^+$  MEA in  $H_2SO_4$  solution to convert the  $Na^+$  to  $H^+$  form during the final MEA preparation step. Comparing these two solvents, the performance difference between the  $Na^+$  form MEA and the  $H^+$  form MEA for the case of acetone was much higher than that of isopropanol. This result clearly demonstrates that the





**Figure 3.** SEM images of catalyst layer surface of cathode CCMs prepared using different solvents: (a) IPA-CCM, (b) ET-CCM, (c) EG-CCM, (d) AC-CCM, (e) DI-CCM, (f) EGM-CCM, and (g) EGE-CCM.

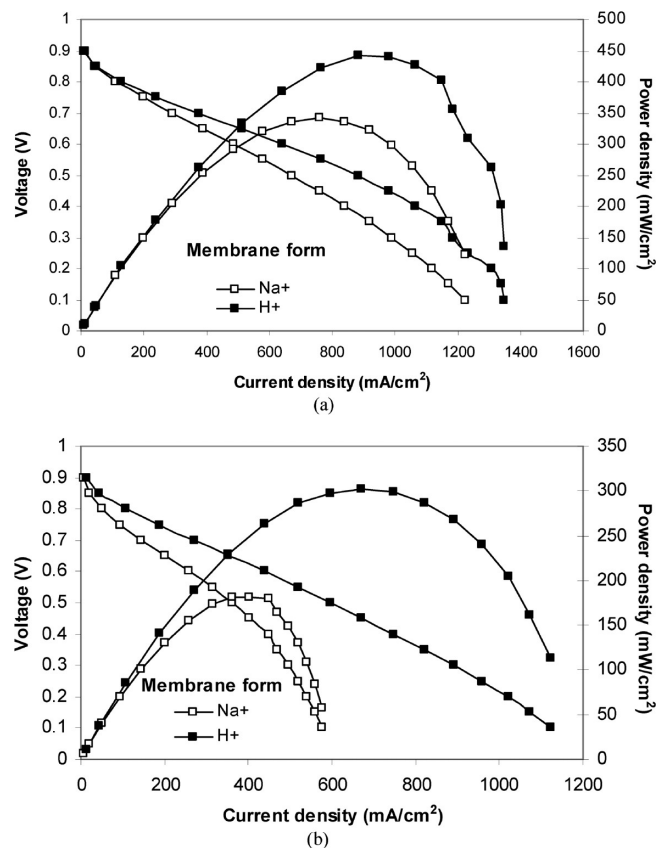
adherence between the catalyst layer and the membrane using isopropanol was much stronger than when acetone was used.

#### 4. Discussion

To facilitate the discussion of solvent effect on CCM performance, the important physical properties of all seven solvents are summarized in Table 1 along with the maximum power density, the ranking of cell performance, and the coating quality. The effect of these properties is discussed in three categories: catalyst ink, coating process, and interaction of catalyst ink and Nafion membrane as follows.

**4.1. Factors Influencing Properties of Catalyst Ink.** The catalyst ink or coating solution influenced the CCM performance both during the coating and after the cell fabrication. For the painting technique used in this study, stable suspension of Pt/C catalyst powder in the ink is the key to obtain well uniform the catalyst coating on the membrane. It was observed that in the catalyst ink preparation step, the catalyst (Pt/C) powder could well be suspended in these solvents for more than 8 h without a sign of settling.

In addition, solvent can also affect the form of Nafion ionomer, solution, colloidal, and precipitate form that



**Figure 4.** Polarization and power curves of the CCMs prepared using different forms of membrane and solvents: (a) isopropanol and (b) acetone.

consequently have an impact on the CCM performance during cell operation. The physical property of solvent suggesting the form of Nafion ionomer in catalyst ink is the dielectric constant ( $\epsilon$ ). The dielectric constant value of the solvents compared to that of the Nafion ionomer in the catalyst ink indicated both the dissolution of ionomer (the formation of Nafion network structure) and the microstructure of catalyst layers. It has been known that Nafion solution forms a solution in the solvents having  $\epsilon > 10$ , a colloidal solution with  $\epsilon$  between 3 and 10, and a precipitate with  $\epsilon < 3$ .<sup>12</sup> From dielectric constant values of the solvents used, it was suggested that the Nafion ionomer in the catalyst inks prepared using isopropanol, ethanol, ethylene glycol, acetone, and deionized water were in solution form, since the dielectric constants of these solvents are higher than 10.<sup>3,4</sup> In contrast, the Nafion ionomer formed a colloid when EGDME or EGDEE ( $3 < \epsilon < 10$ ) was used. From the performance testing results, the catalyst ink in solution form produced a higher performance than that in colloidal form. The solution form of catalyst ink would assist solvent in penetrating into the membrane, creating more attachment of catalyst to the membrane compared to the colloidal form during the catalyst application step. This could be the reason why the ink prepared from water illustrated higher performance than the cells fabricated from EGDME and EGDEE-containing catalyst inks. This finding contradicts that in the GDL-based method found in the works of Shin et al.<sup>4</sup> and Fernandez et al.<sup>5</sup> However, our result is in agreement with the work of Yang et al. who investigated the decal method in electrode preparation using different solvents.<sup>3</sup> Therefore, it can be concluded that the influence of solvent type on

electrode performance was not the same for different electrode forming techniques.

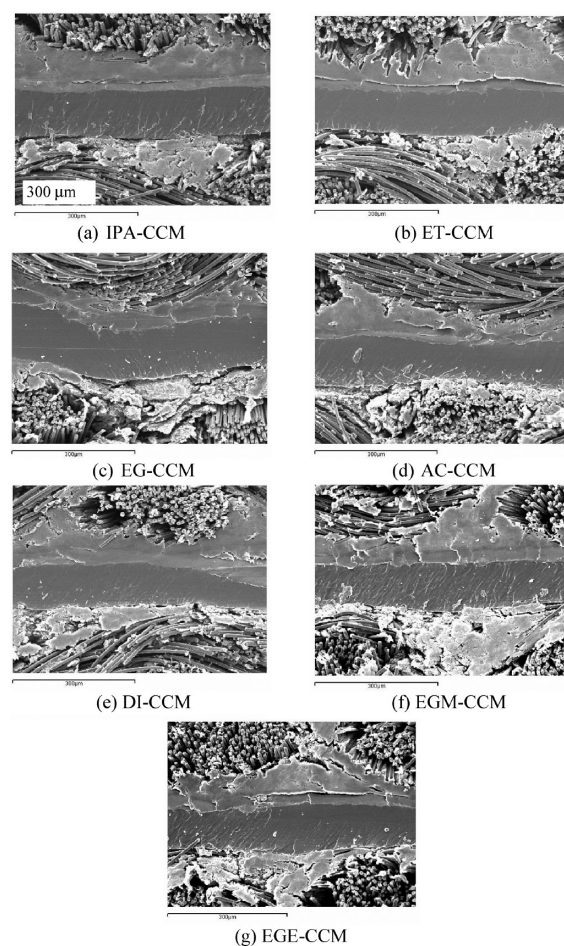
**4.2. Factors Influencing Catalyst Coating Process.** The solvent properties affected the two steps in the coating process, ink applying step, and drying step. The smoothness of the coating surface greatly depends on the boiling point and viscosity of the solvents, as reflected by evaporation rate of solvents and paintability of the catalyst ink mixtures, respectively, and these factors ultimately affect the catalyst layer coverage. For the ink applying step, control of ink flow on membrane is a critical factor to achieve a uniform catalyst layer. Catalyst ink prepared from a too low viscosity solvent could flow freely on the membrane, whereas the solvent with very high viscosity caused a poor distribution of catalyst ink on the membrane. Moreover, not only the viscosity but also the boiling point of solvent can affect the ink flowability and surface coating quality. From the SEM images of the catalyst layer surface on the membrane shown in Figure 3, it can be seen that isopropanol and ethanol having viscosity more than 1 cP provided smooth catalyst layer surfaces because they contained a correct paint viscosity and suitable evaporation rate. Therefore, catalyst layers were formed stably during the catalyst application step. On the other hand, for the solvents having viscosity lower than 1.0 cP, i.e., EGDME and EGDEE, the catalyst ink was thinner and flowed freely on the membrane surface resulting in poor surface coverage. For the catalyst ink prepared using acetone as a solvent, the flow of ink could be retarded by the fast evaporation rate of acetone due to its low boiling point and this property led to cracking of the catalyst layer during the catalyst application step. For the case of ethylene glycol, the slow evaporation rate due to its high boiling point could cause the ink to flow although its viscosity is high. The catalyst ink prepared using deionized water showed a tendency of ink agglomeration on the membrane after it had been applied for many layers. This might be attributed to the alcohol-base Nafion ionomer solution constituent in catalyst ink. It should be also mentioned that the cracks appearing on the CCM was not caused by the drying process since all cracks on the CCMs were taken place during the ink applying step.

For the EG-CCM, because of the high boiling point of ethylene glycol solvent, some of the solvent could not be removed in the drying step and remained in the membrane, resulting in rough surface of the CCM. This can be verified by the membrane thickness and the increase of CCM weight as shown in Table 2. In determining the thickness of the membrane in the MEAs prepared, SEM images of the MEA cross section were taken and the results are shown in Figure 5. It could be seen that the thickness of the membrane for EG-CCM ( $125 \pm 1 \mu\text{m}$ ) was greater than the others, which averaged at  $107 \mu\text{m}$  ( $\pm 5 \mu\text{m}$ ). This phenomenon was supported by the increased weight of the EG-CCM, approximately 156% greater than the expected value, which was not found in the other CCMs.

**4.3. Factors Influencing Interaction of Catalyst Ink and Nafion Membrane.** *Swelling Ratio of Membrane.* When the catalyst inks were applied to the membrane surface, the membrane absorbed each solvent at a different capacity, which in turn influenced the surface coverage of each respective catalyst ink. This property depends on the nanostructure of the Nafion membrane, as well as the polarity of the solvents which could be determined by the swelling ratio of the membrane. It has been known that Nafion membrane is

**Table 2. Catalyst Coating Weight and Membrane Thickness of MEAs after Immersing in Different Solvents**

samples	catalyst coating weight (g)	membrane thickness ( $\mu\text{m}$ )
IP-CCM	0.0215	109
ET-CCM	0.0202	108
EG-CCM	0.0481	125
AC-CCM	0.0210	103
DI-CCM	0.0215	100
EGM-CCM	0.0203	107
EGE-CCM	0.0207	112



**Figure 5.** SEM images of cross sectional MEA consisting of a commercial-GDL anode and different CCMs prepared using different solvents at cathode: (a) IPA-CCM, (b) ET-CCM, (c) EG-CCM, (d) AC-CCM, (e) DI-CCM, (f) EGM-CCM, and (g) EGE-CCM.

composed of two incompatible groups of a hydrophilic sulfonate vinyl ether side chain terminated with sulfonic acid group ( $-\text{SO}_3\text{F}-$ ) and a hydrophobic perfluorocarbon backbone ( $-(\text{CF}_2-\text{CF}_2)_m-(\text{CF}_2\text{CF}(\text{OCF}_2\text{CF}(\text{CF}_3)\text{OCF}_2-\text{CF}_2)_n-$ ).<sup>11</sup> The hydrophilic side attracts polar solvent, while the hydrophobic favors neutral or nonpolar solvent. Here, water molecules strongly adhere to the hydrophilic chain and do not have a chance to travel to the hydrophobic backbone, reflected by the low swelling ratio shown in Table 1. On the other hand, isopropanol, ethanol, and acetone are less polar than water and consist of hydrophilic and hydrophobic sites which were absorbed by the sulfonated group and perfluorocarbon backbone, respectively. Hence, the swelling ratios of

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the membrane in these solvents were much higher than in water. Unlike the above solvents, the ethylene glycol type of solvents (i.e., EG, EGDME, and EGDEE) contain a majority of nonpolar and some weak polar groups; thus, their absorption capacities fell between that of water and the alcohol solvents. Therefore, with better absorption capacity or higher swelling ratio of membrane to the alcohol solvents, the catalyst inks would adhere more strongly to the membrane and cover more of its surface.

Based on the above discussion and physical properties compared in Table 1, it is clearly seen that isopropanol and ethanol catalyst ink giving the highest cell performance provided smooth coating surface and higher swelling ratio (high interaction with the membrane) and had similar physical properties (viscosity, dielectric constant, and boiling point). The acetone coating ink giving the third highest performance had the same magnitude of dielectric constant and swelling ratio as isopropanol but contained low viscosity and boiling point. On the other hand, lower performance CCM prepared from deionized water, ethylene glycol, ethylene glycol dimethyl ether, and ethylene glycol diethyl ether had low swelling ratio, either too low or too high dielectric constant, a cracked surface, and poor adhesion on the membrane, resulting from their low viscosity and high boiling point.

## 5. Conclusion

The effect of solvent types used in the preparation of catalyst ink on the performance of the MEA prepared by the CCM technique was investigated. The important physical properties of the solvent used in catalyst ink preparation were dielectric constant, swelling ratio, viscosity, and boiling point. The viscosity and boiling point of the solvents affected flowability of the catalyst ink. The solvents that had optimum viscosity and evaporation rate can provide a uniform distribution of catalyst layer over the membrane, thus enhancing Pt utilization. High solvent absorption capacity of the membrane can enhance a good attachment between catalyst layer and membrane resulting in good proton transport and lowering ionic resistance. Additionally, in the CCM method, the MEAs prepared from the solvents forming a solution with Nafion ionomer gave higher performance than those forming a colloid with the Nafion ionomer. Among the seven solvents studied, the most suitable solvent for preparing the catalyst ink mixture for PEM fuel cell was isopropanol.

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