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Effect of crosslinked poly(vinyl alcohol)/sulfosuccinic acid ionomer loading on PEMFC electrode performance

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ABSTRACT

In this study, crosslinked poly(vinyl alcohol)/sulfosuccinic acid (PVA/SSA) ionic polymer and Nafion® N-115 membranes are used as electrolytes to fabricate different types of membrane electrode assemblies (MEAs) for proton exchange membrane fuel cells (PEMFC). Each MEA is fabricated with various crosslinked PVA/SSA ionomer loadings (0.04–1.5 mg cm⁻²) in catalyst layer based on volumetric ratio between catalyst and ionomer. Properties of each catalyst layer are evaluated using scanning electron microscopy, cyclic voltammetry, and hydrogen pumping technique. I–V characteristics, impedance, and incubation behaviour of each cell are evaluated in fuel cell testing fixture. Incubation of cell is effective when ionomer loadings are below 0.1 mg cm⁻². At ionomer loading of 0.04 mg cm⁻², PEMFC demonstrates highest power densities than that observed with other loadings of ionomer due to higher electrochemical active surface area of the electrode. Hydrogen pumping studies reveals that the PVA/SSA ionic polymer is a potential anode material for PEMFC electrodes while ionomer loadings are below 0.1 mg cm⁻².

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Introduction

Fuel cell is an electrochemical device which converts chemical energy of fuel into electricity with higher efficiency than the conventional heat engines. In recent years, research on fuel cells has gained prominence, since it is a promising green-energy technology. The process of energy conversion occurs on electrochemically active sites in the electrodes. Hence, better choice of electrode/electrolyte materials are necessary for stable performance of membrane electrode assemblies (MEAs) of proton exchange membrane fuel cells (PEMFCs) [1–3]. To achieve better performance of the cell, ionic polymer

in the MEA plays an important role. Perfluorinated sulfonic acid (PFSA) membranes are one of the most extensively used polymer electrolyte membranes for PEMFCs. Among PFSA family of polymers, Nafion® by DuPont has gained popularity due to its high proton conductivity in fully hydrated state [4]. However, high cost of these membranes has limited their commercialization. In addition, the lower conductivity exhibited by these membranes at low humidity have necessitated the need to look at alternate polymeric systems [5]. In this scenario, non-fluorinated membranes (such as sulfonated polyether-ether ketone, poly (vinyl alcohol) blends etc.) are considered as alternatives and numerous studies have been conducted on such membranes [6,7].

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In general, two methods are used in fabrication of membrane electrode assembly for PEMFCs. First method to prepare a MEA is the catalyst coated membrane (CCM) method where catalyst ink is initially coated on Teflon substrates. Then these Teflon substrates are dried and hot pressed on to ionic polymer membrane. The second method is catalyst coated gas diffusion layer (CCG) method where catalyst ink is directly coated on the GDL, dried, and then hot pressed on to the electrolyte membrane [8,9]. In the present study, MEAs are fabricated in CCM method, since through this method, charge transfer resistance is reduced between membrane and electrodes [10]. In CCM method, electrode is essentially a thin catalyst layer pressed between the ionomer membrane and a porous, electrically conductive substrate. It is important to have higher numbers of electrochemically active sites in the electrode to acquire high electrode performance [11]. The electrochemically active sites of the catalyst layer are the locations that fuel, electrons, and protons can access and these active sites/locations can be improved/increased with optimum content of ionomer in the electrodes. Several studies have reported that ionomer loading in the catalyst layer is critical for higher performance of the cell [7,12]. Guangchun et al. studied the effect of Nafion loading on the properties of the catalyst layers. They identified that loss of electrode performance at higher ionomer loadings is due to an increasing oxygen transport resistance. They also reported that the ohmic resistance of electrode decreases with increasing ionomer loading, and further addition of ionomer (after percolation limit) increases ohmic resistance [13]. Ticianelli et al. prepared electrodes with brushing a 5 wt.% Nafion solution onto the electrodes and studied the effect of catalyst ionomer loading on the cell performance [14] and found that 3.3 wt.% of Nafion ionomer content is optimal to minimize ohmic and mass transfer losses. Uchida et al. found the optimal content of the Nafion ionomer to be 1 mg cm^{-2} at Pt loading of 0.5 mg cm^{-2} [15]. Antolini et al. have suggested an empirical equation to calculate optimum ionomer loading [16]. They reported optimum Nafion loading as 36 wt.% for all Pt loadings. Sasikumar et al. studied the correlation of platinum loading and optimum Nafion content in the electrode [17] and reported that the optimum Nafion percentage depends on Pt loading in the electrodes. Sasikarn et al. optimised process variables to fabricate MEA using design of experiment (DOE) approach [18]. They reported that for 1 mg cm^{-2} of platinum 15% (w/w) level of Nafion is optimum, and in the process of preparation, drying temperature of $80 \text{ }^\circ\text{C}$ for 0.5 h in air is the optimal condition for fabrication of MEA. In literature, ionomer content in catalyst layer is reported in weight ratio relations between catalyst and catalyst ionomer, however, best results have been reported at different ionomer loadings. The underlying concept of ionomer loading in electrode/catalyst layer is to enhance catalyst utilization in the electrode and to improve the cell performance, since loading of ionomer will impact parameters/properties such as porosity, and ionic and electrical conductivity of the electrode. Therefore it is necessary to correlate the catalyst loading and Nafion percentage in the electrode from a volumetric ratio perspective. In the present study, PEMFC electrode performance is evaluated with different ionomer loadings in the catalyst layer using volumetric relation between catalyst and ionomer.

Ionomer facilitates the transport of protons necessary for oxygen reduction reaction (ORR) and hydrogen oxidation reaction (HOR) within the catalyst layer and also plays a crucial role in exchanging protons between the catalyst layer and the membrane. Perfluorosulfonic acid ionomers promote platinum dissolution during fuel cell operation due to formation of the degradation product HF. Pt dissolution can be avoided by using an alternate non-fluorinated ionomer [2]. Also due to exothermic electrochemical reactions, PFSA ionomer in catalyst layer easily dehydrates. In our previous study, low cost crosslinked PVA/SSA ionic polymer (hydrocarbon ionomer) was used as an ionomer to prepare the catalyst layers [7]. We identified that the performance of PVA/SSA ionomer is as good as Nafion at the anode catalyst layer and PVA/SSA ionomer makes MEA less sensitive to relative humidity of reactant gases. In the current study, effect of crosslinked PVA/SSA ionomer loading on electrode performance is analysed. Though, performance of crosslinked PVA/SSA polymer as PEM is low [7], in the current study it has been used as an PEM to understand the effect of ionomer loading in catalyst layer on crosslinked PVA/SSA based MEA systems. To carry out this ionomer loading study, crosslinked PVA/SSA polymer is used as a polymer electrolyte membrane as well as ionomer in the catalyst layer, and five MEAs are fabricated with different PVA/SSA ionomer loadings as shown in Table 1. Performance of crosslinked PVA/SSA ionomer based catalyst layers is also evaluated using Nafion[®] N-115 membrane based MEAs and specifications of each MEA are shown in Table 1. The performance of all these MEAs is evaluated using a $\text{H}_2\text{-O}_2$ fuel cell testing fixture.

Materials and methods

Materials

Polyvinyl alcohol (87–89% hydrolysed, high molecular weight, Alfa Aesar), sulfosuccinic acid solution (70 wt.% in H_2O , Sigma–Aldrich Co.), deionized water ($18.2 \text{ M}\Omega \text{ cm}$) and platinum black (high surface area $25.0\text{--}30.4 \text{ m}^2 \text{ g}^{-1}$, Alfa Aesar) were used to prepare the catalyst layers and membranes. Nafion[®] N-115 membrane, SIGRACET[®] GDL 35 CD gas diffusion layer (SGL-GDL) were used to make the MEAs.

Membrane preparation

Aqueous 10 wt.% PVA solution was prepared by dissolving the pre-weighed amount of PVA in deionized water at $80 \text{ }^\circ\text{C}$ and stirring for 6 h. The PVA solution was mixed with SSA solution to enable 10% crosslinking of hydroxyl groups of PVA. The mixed solution was then vigorously stirred at room temperature for 24 h, to get homogeneous PVA/SSA solution. The solution was then cast in a petridish and dried at $60 \text{ }^\circ\text{C}$ for 12 h to form membranes of $200 \text{ }\mu\text{m}$ thickness. The membranes were then crosslinked at $110 \text{ }^\circ\text{C}$ for 1 h. These membranes were labelled as PVA/SSA membranes and used as electrolyte membranes for all PEMFCs. The homogeneous PVA/SSA solution was also used as the ionomer for preparation of the catalyst ink [7].

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