

The aim of this experiment is to prepare the Nafion-SiO<sub>2</sub>-PWA composite membrane using solution phase sol-gel method with incorporation of silica and PWA in the Nafion cluster to produce Nafion-SiO<sub>2</sub>-PWA nanocomposite membrane. XRD was utilized to determine the maximum PWA loading in the silica matrix. The parameters that have been studied are: (i) annealing time and (ii) ratio of SiO<sub>2</sub>/Nafion to the physic-chemical properties of the composite membrane.

## MATERIALS AND METHOD

### Materials

Nafion solution of 5 wt% (EW 1100 Dupont), TEOS (Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>) 98%, PWA (H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub>) 96%, Dimethylformamide (DMF), Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 98%, Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>) 30% were all purchased from Aldrich. Deionized water was used as solvent in all the experiments.

### Method

#### Membrane Preparation.

Appropriately 5% wt Nafion solution was evaporated at room temperature to obtain solid Nafion. Solid Nafion was dissolved in DMF solvent to obtain 5% wt Nafion solution in DMF. PWA was also dissolved in deionized water and then mixed with TEOS at weight ratio of PWA:SiO<sub>2</sub> = 4:10. Subsequently, it was stirred in an ultrasonic bath for 30 min, and added to the Nafion-DMF solution and further stirred in an ultrasonic bath for 6 hours. The mixture was allowed to stand at room condition to release trapped air bubbles for another 24 hours without mixing. This solution was casted in a Petri dish and heated at 80°C for 2 hours to remove the solvent. In order to enhance the mechanical properties of the composite matrix, heating was continuously applied at 140°C at different periods of 2, 4, 6 and 10 hours until transparent membrane was obtained. Then, the recast composite membrane was made to detach from the Petri dish by boiling it in the de-ionized water. Finally, the membrane was cleaned by heating at 80°C in the solution of 3 wt. % H<sub>2</sub>O<sub>2</sub>, de-ionized water, 0.5M H<sub>2</sub>SO<sub>4</sub> and again in de-ionized water until the pH of the washing water becomes almost neutral. These composite membranes are designated NS10W, NS15W and NS20W, whose specifications in ratio of Nafion/TEOS/PWA are 100:10:1.1538; 100:15:1.7303 and 100:20:2.3072 (wt./wt./wt.), respectively.

## RESULTS AND DISCUSSION

### Saturated Loading of PWA Particle in the SiO<sub>2</sub> Matrix

The XRD pattern of pure silica, pure PWA and the mixture of Silica-PWA are as show in Fig 1. The amount of heteropolyacid (PWA) loaded on silica was limited