Preparation of materials for economical proton exchange membrane fuel cell using non-precious metal catalyst and oil waste

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1. Introduction

Nowadays, non-renewable fossil fuels cause a huge amount of pollution. Alternative energies, including nuclear, wind, solar light, and fuel cell, are considerably employed to overcome this issue. Proton exchange membrane fuel cell (PEMFC) is one of promising candidates, due to its zero emission. Although PEMFC possesses many benefits, the materials for fuel cell production are still expensive, especially commercial platinum (Pt)/Carbon catalyst. Moreover, waste oil disposal problem is continuously spoiling the environment. Therefore, these two problems are concerned to be solved in this research work.

2. Objectives

[1] To overcome the drawback of commercial PEMFC by synthesizing economical non- precious metal nanoparticles embedded in carbon particles as PEMFC catalyst. The catalyst consists of two main parts, carbon particles and metal nanoparticle. The formation of carbon particles is achieved by conversion of vegetable oil (palm oil) waste, and the metal nanoparticles are produced from non-precious metal, iron (Fe).

[2] To construct a prototype fuel cell for daily uses at KVIS for minimizing the consumed energy from fossil fuels and electricity.

3. Methodology

3.1 Preparation of FeNPs/Carbon particles

The research methodology started by collecting vegetable oil waste from KVIS canteen as carbon precursor. A direct discharge process in solution, named solution plasma process (SPP), was employed for material preparation. Due to the advantages of SPP, e. g. short processing time, operation in atmospheric temperature and pressure, and controllable material properties, this technique is versatile to be utilized as a single-step nanomaterial synthesis. In order to set up the experiment, the system which consists of a glass reactor containing vegetable oil waste and two pairs of Fe electrodes as Fe nanoparticle precursor immersed in the oil was set up. The process was simply done by connecting a bipolar-pulsed power generator (Pekuris, MPP-HV02, Japan) to the Fe electrodes under vigorous stirring. The conditions were set at 1 μs, 15 kHz, 0.5 mm, 90 min of pulse width, frequency, electrode gap, and discharge time, respectively. The Fe nanoparticles embedded carbon (FeNPs/ Carbon) particles were consequently obtained, which was then filtered and washed several times with hexane and ethanol. The washed product was dried in an oven at 100 °C for 12 hours. Subsequently, the material was heat-treated at 900 °C for 30 min under the flow of N_2 (500 sccm). The purpose of this heat treatment was to rearrange the carbon structure, and thus increase the conductivity of the carbon-particle matrix.

3.2 Characterizations

SEM and EDX (Hitachi TM3030Plus) analyses were carried out at an acceleration voltage of 15.0 kV to examine the microstructure, shape, size, and elemental composition of NiNPs/Carbon. The crystal structure of NiNPs/Carbon was examined by its XRD pattern, which was collected by a Rigaku SmartLab at 40 kV and 30 mA. The XRD analysis was carried out using a Cu Kα radiation (1.54 Å) over a scan range (2θ) of 5–90° with a scanning step of 0.01 and a scanning speed of 4°/min. The catalytic activity of NiNPs/ Carbon was electrochemically evaluated by CV. The CV measurements were carried out in a three-electrode cell (μAUTOLABIII/FRA2) using 0.1 M H_2SO_4 as an electrolyte. The electrolyte was saturated with O_2 by purging O_2 gas into the cell. A glassy carbon (GC) disk (3 mm in diameter) was used as the working electrode, a platinum electrode was used as the counter electrode, and an Ag/AgCl (saturated KCl) electrode was used as the reference electrode. The CV scans were performed in a voltage range of -0.2 - 1.4 V, at a rate of 50 mV/s.

4. Results and Discussion

4.1 Morphology and elemental analysis by SEM and EDX

FeNPs and carbon were formed by sputtering of Fe electrodes and the dissociation and recombination of carbon species from vegetable oil waste during the solution plasma discharge. SEM images of the FeNPs/ Carbon are shown in **Figure 2**. The images reveal that FeNPs were successfully embedded in the carbon matrix. FeNPs had a spherical-like morphology and were dispersed on carbon particles with a mean size of about 10 μm. The carbon particles showed a chain-like morphology attributed to their Brownian motion during the solution plasma processing.

Figure 3 shows elemental analysis results of the FeNPs/ Carbon. The EDX spectrum revealed the presence of C, Fe, O, and P. Fe was present on the carbon particles. A C peak was also detected. The O peak is attributed to the oxygen present in oil, while the P peak is attributed to phospholipid groups of the oil.

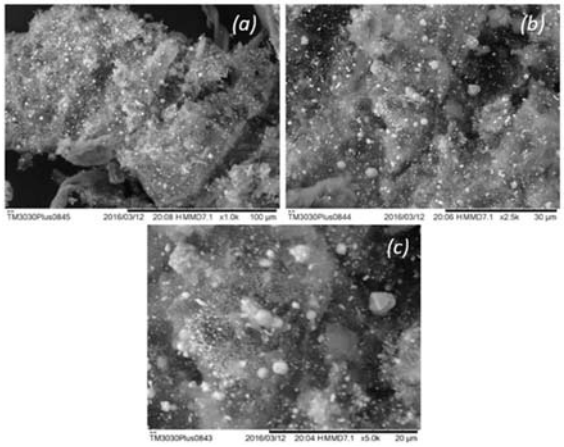

Figure 2 SEM images of FeNPs/Carbon, prepared by solution plasma in vegetable oil waste, at various magnifications: (a)×1000, (b)×2500, and (c) ×5000.

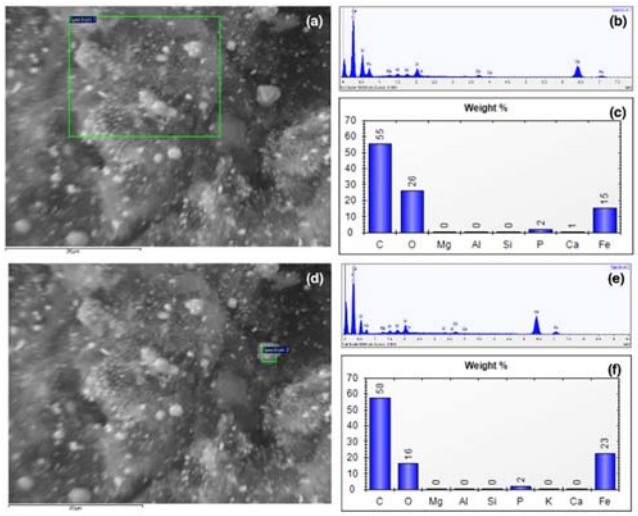

Figure 3 Result of (a and d) selected area EDX analysis, (b and c) EDX spectra for wide area of FeNPs/Carbon, (e and f) EDX spectra for specific are of FeNPs/Carbon.

4.2 Crystal structure observation by XRD

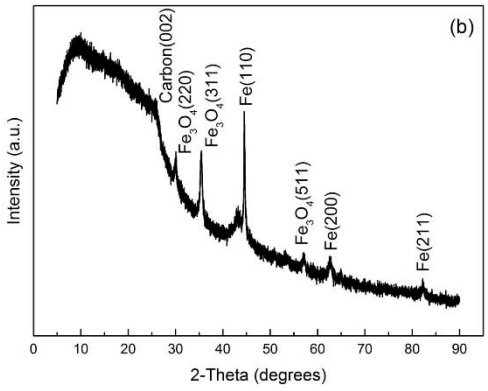

Figure 4 XRD pattern of FeNPs/Carbon.

There are two forms of Fe, including Fe-oxide and Fe nanoparticles. Seven XRD peaks were observed at 26.01, 30.05, 35.40, 44.53, 56.97, 62.72, and 82.21 degree, corresponding to carbon (002), Fe-oxide (220), Fe-oxide (311), Fe-oxide (511), Fe (110), Fe (200), and Fe (211). The XRD pattern confirmed that the FeNPs obtained by the solution plasma method had high crystallinity and an fcc structure. The C (002) peak was broad, indicating amorphous nature of the carbon particles. Moreover, the intensity of the FeNP diffraction peaks was very high, which indicates the high yield of FeNPs. The crystallite size of FeNPs (Fe (110) diffraction plane) was found to be 72 nm, as calculated by the Scherrer equation.

4.3 Catalytic activity analysis by CV

The cyclic voltammogram, as shown in **Figure 5**, indicates significant electrocatalytic activity conducted by FeNPs/ Carbon towards both the oxidation and reduction reactions.

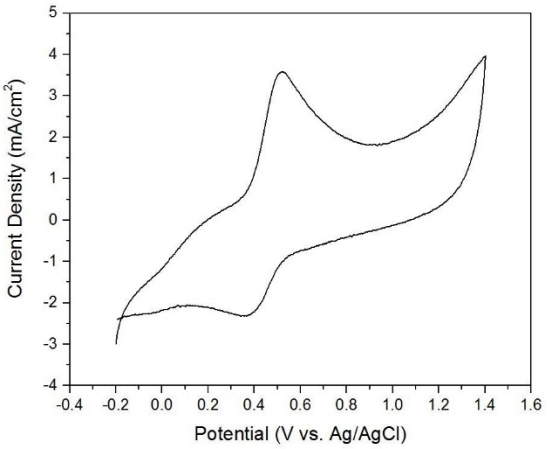

Figure 5 Cyclic voltammogram of FeNPs/Carbon in 0.1 M H_2SO_4 solution under saturated O_2 condition.

5. Conclusions and Applications

FeNPs/Carbon were successfully synthesized by solution plasma processing using vegetable oil waste as carbon precursor. FeNPs were formed by sputtering of the Fe electrodes and carbon particles were obtained simultaneously from conversion of vegetable oil waste during the solution plasma discharge. The SEM and XRD results revealed that the spherical FeNPs were embedded in the carbon-particle matrix and had a crystallite size of about 72 nm. The CV results indicated significant electrocatalytic activities. The FeNPs/Carbon produced in this study is expected to be a promising catalyst. The solution plasma method used in this study can also be used to synthesize other related materials.

6. Acknowledgements

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