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Research Article

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Synthesis of Epoxy/Graphite Nanocomposite Based TiO_2 and ZnO Nanoparticles as Bipolar Plate for PEMFC

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Abstract
In this study, a polymer/graphite nanocomposite bipolar plate was prepared through bulk molding process. Nanoparticles of metal oxide such as zinc oxide (ZnO) and titanium dioxide (TiO_2) were added to composite as secondary fillers. Epoxy resin was chosen because of its low cost and easy fabrication through bulk molding. In these nanocomposites, the percentage of resin was constant while graphite and nanoparticles were variable. The conductivity and mechanical properties of these composite were studied by standard test methods. The obtained results showed that the nanocomposites prepared with a high percentage of nanoparticles have the highest flexural strength. Scanning electron microscopy (SEM) showed that fillers disperse well in the matrix.

Keywords: Bipolar Plate, Nanocomposite, Fuel Cell, Epoxy, Graphite

Introduction
Polymer electrolyte membrane fuel cell (PEMFC) is one of the most promising clean energy sources because of its attractive features such as high power density, relatively low operating temperature, convenient fuel supply, longer lifetime, and modular shape [1]. Among PEMFC components, the bipolar plate is one of the most important components, which can contribute up to 80% of the fuel cell stack total weight and 38% of the total cost of the PEMFC stack [1, 2]. To prepare bipolar plates in PEMFC several requirement must be considered; e.g., high electrical conductivity, non-porous and leak-free conduit for the reacting gases, supply of fuel and oxidant gases, supply water and product gas removal, utilization of water or air for stack cooling, good thermal conductivity to find an opportunity stack cooling, satisfactory temperature distribution between cells, construction to high tolerance in large volumes, high corrosion resistance, good mechanical stability, low weight, and low cost [3, 4]. Therefore, many research efforts are ongoing to fabricate cost-effective and lightweight bipolar plates for fuel cells [5]. The technical targets are defined by the Department of Energy (DOE) of USA, as shown in Table 1 [6].

Table 1: US DOE Technical Targets for Composite Bipolar Plates

Property	Value	Units
Weight	<0.4	$kg\ k\ W^{-1}$
Flexural strength	$>25 \times 10^6$	$N\ m^{-2}$
Electrical conductivity	>1	$S\ m^{-1}$
Thermal conductivity	$>10^4$	$J\ s^{-1}\ K^{-1}$
Gas permeability	$<2\times10^4$ and 3.04×10^5	$m\ s^{-1}$ at 353 K $N\ m^{-2}$
Corrosion resistance	$<10^{-2}$	$A\ m^{-2}$

The conventional materials for fabricating bipolar plate are based on either graphite materials or metals. Graphite is the most commonly used material for bipolar plates considering its advantages such as high electrical and thermal conductivity, excellent corrosion resistance, and lower density than those of metals. However, it has also some disadvantages such as its brittleness and difficulty in machining; thus, a graphite bipolar plate must be made large and thick, which results in a large and heavy fuel cell stack [2, 7]. For metallic bipolar plates, several metallic alloys have been proposed as possible bipolar plate materials combining excellent gas impermeability and mechanical resistance. Stainless steels, aluminum, nickel, copper, titanium, and bulk amorphous alloys are investigated in the literature for this application. However, metallic bipolar plates have poor corrosion resistance in the PEMFC environment [6, 8].

Recently, bipolar plates based on composites of polymer and conducting filler (for improving electrical conductivity) have been investigated due to their lower cost, lighter weight, and easy fabrication through compression or injection molding compared to the properties of the available materials, such as graphite and metals [5, 9]. These composite bipolar plates have been made using a thermoplastic polymer such as polypropylene, polyethylene, and poly(vinylidene fluoride) or thermosetting polymer such as phenolics, epoxies, and vinyl esters [10, 11].

In this work, Novolac epoxy, Graphite (G), and nanoparticles such as TiO_2 and ZnO were respectively chosen as the polymer matrix, the primary filler, and secondary fillers to prepare a high-performance conductive material with low percolation threshold and excellent comprehensive properties. Different types of secondary fillers were added into the graphite-polymer matrix and nanocomposites were fabricated by the bulk-molding compound process, followed by studying the properties of composite bipolar plates.

Experimental

Materials

The natural flake graphite, with a purity of 99.9% was purchased from Qingdao Guyu Graphite Company, China. The epoxy resin (EP) – a Bisphenol A based epoxy resin, with a viscosity of 6P and was obtained from US Composites. The curing temperature of the epoxy recommended by the manufacturer was 80℃. The curing agent, 4-aminophenylsulphone, was purchased from NewJersey, USA. The curing agent was a diamine type (tetrafunctional) to facilitate a rapid and dense cross- linking in the epoxy resin. Nano-metal oxides were obtained from Merck company (Darmstadt, Germany), with 25-50 nm particle size.

Preparation of the Nanocomposite Materials

The bulk molding compounds were prepared by mixing the resin, graphite, nano-metal oxide powder, and hardener in a vessel of polypropylene (PP) for 2h. In this work, materials were mixed based on the percentages shown in Tables 2 and 3. Then, the compounds were molded at ambient temperature for 5 min followed by curing them at 80℃ in an oven for 24 h.

Table 2: Percentage of Materials in Epoxy/ Graphite /TiO_2 Nanocomposite

	Sample 1 / %	Sample 2 / %	Sample 3 / %	Sample 4 / %	Sample 5 / %
Epoxy resin	77.5	77.5	77.5	77.5	77.5
Graphite	22.5	21.0	19.5	17.5	15.5
Nano-TiO_2	0	1.5	3.0	5.0	7.0

Table 3: Percentage of Materials in Epoxy/Graphite/ZnO Nanocomposite

	Sample 6 / %	Sample 7 / %	Sample 8 / %	Sample 9 / %
Epoxy resin	77.5	77.5	77.5	77.5
Graphite	21.0	19.5	17.5	15.5
Nano-ZnO	1.5	3.0	5.0	7.0

Characterization

The electrical resistance of composite bipolar plate, shown in Table 4, was measured according to ASTM C611. The flexural strength of composite bipolar plate was tested according to ASTM D790-15 with a universal testing machine (Instron WDW-E200). The specimen is 60.0 mm in length, 5.0 mm in width, and 3.0 mm in thickness. The supporting span is 40.0 mm at a constant crosshead speed of 0.5 mm min^{-1}. Table 5 gives the values of flexural strength for nanocomposites.

The density of samples was measured according to ASTM D792-13A and reported as the average of three weights per sample (Table 6). The section morphologies of the composite bipolar plate were observed on a JSM-6700F type scanning electron microscope (SEM). Figure 1 (A to G) presents the SEM of these nanocomposites.

Table 4: Electrical Resistance of the Composites Bipolar Plates

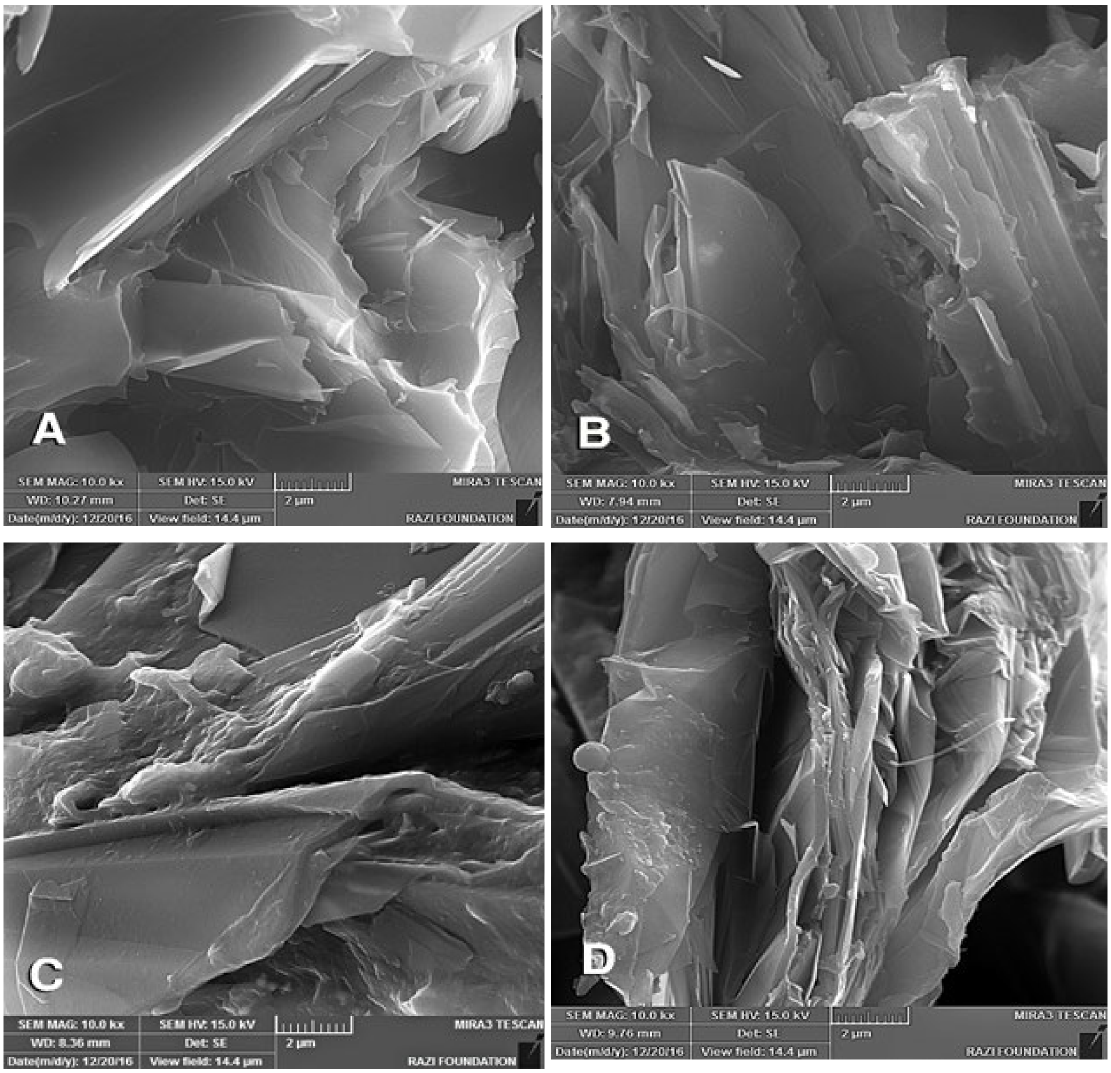
Samples number	1	2	3	4	5	6	7	8	9
Electrical Resistance / $V A^{-1} m^{-1}$	740.0	1534.3	661.3	531.2	673.0	845.3	1697.6	2104.3	1803.7

Table 5: Flexural Strength of the Composites Bipolar Plates

Samples number	1	2	3	4	5	6	7	8	9
Flexural Strength / N m^{-2}	3.61×10^7	4.60×10^7	3.75×10^7	3.52×10^7	3.21×10^7	3.85×10^7	4.07×10^7	4.63×10^7	4.83×10^7

Table 6: Density of the Composites Bipolar plates

Samples number	1	2	3	4	5	6	7	8	9
Density/ kg m^{-3}	1282	1272	1289	1297	1308	1273	1288	1296	1309



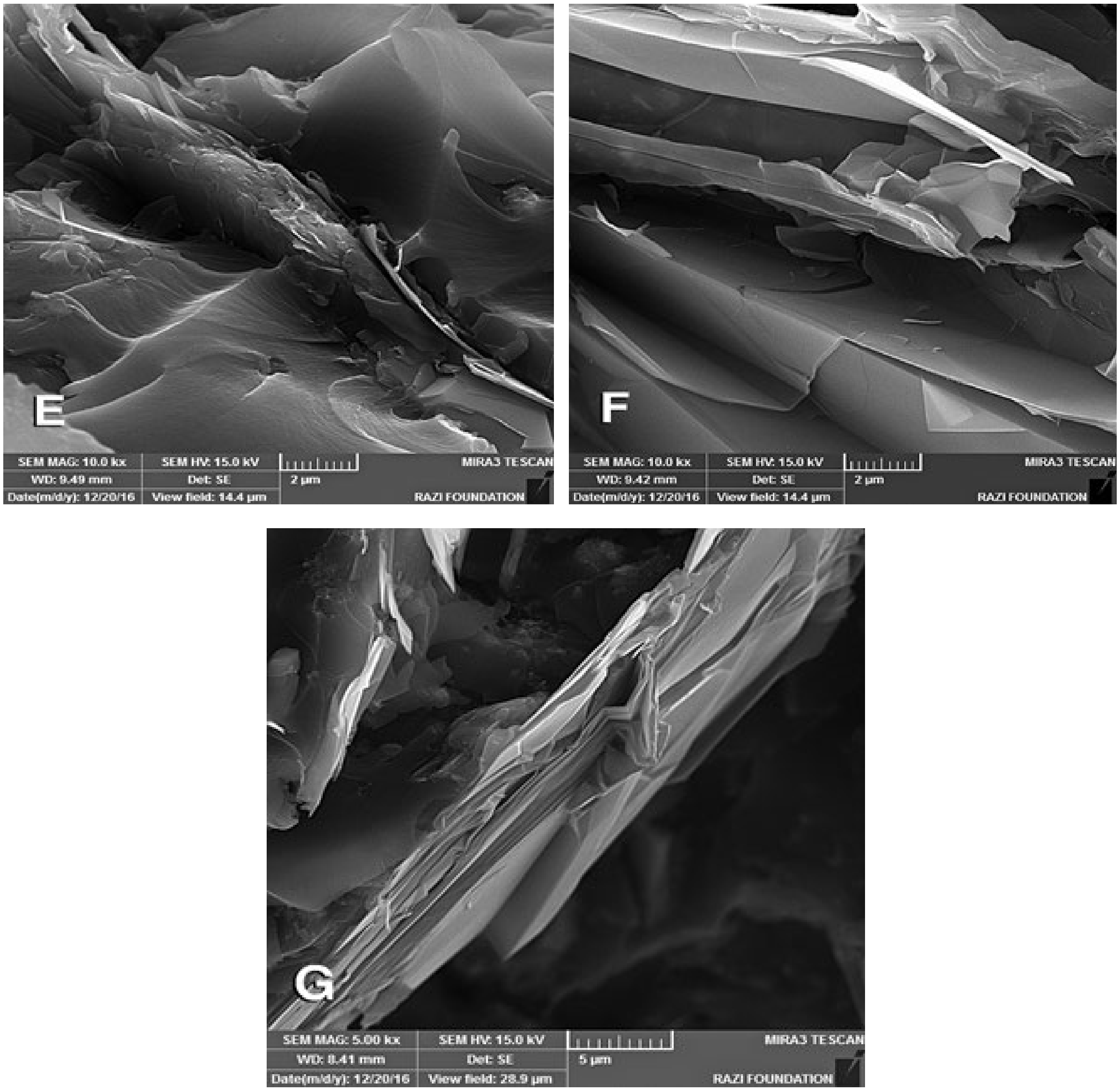


Figure 1: SEM of Nanocomposites: (A) Epoxy/Gr; (B, C) Epoxy/Gr/TiO_2; and (D, E, F, G Epoxy/Gr/ZnO)

Results and Discussion

In this study, the rate of resin and hardener was constant while contents of graphite and nanoparticles were varied. As shown in Figure 2, increase in the nano-metal oxide content results in a decrease in the conductivity resistance in TiO_2 composites and an increase in ZnO composites. This trend continues until 5% of nano-metal oxides and changes at 7% of nano-metal oxides. By increasing the nano-TiO_2 content, it was observed that graphite particles make better contact with their conductive neighbors because there is a lot of amount electrons around TiO_2. On the other hand, electrical resistance and electrical conductivity indicate a decrease and increase, respectively. The electrical resistance of composite of Epoxy/Graphite has been determined as 740 ($VA^{-1}m^{-1}$). However, this test has shown the addition of TiO_2 to Epoxy/Graphite composite leads to a decrease in the electrical resistance to 531.2 ($VA^{-1}m^{-1}$).

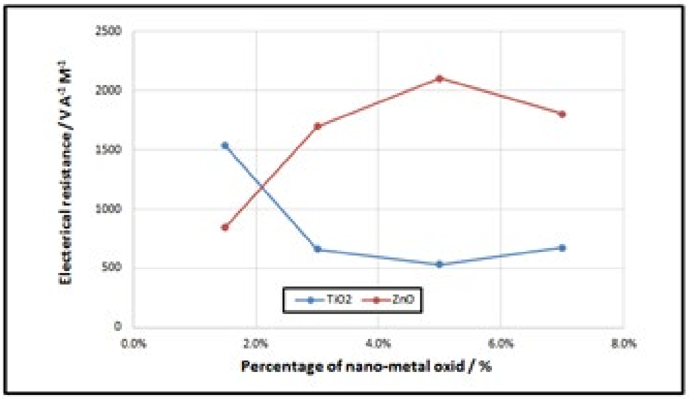


Figure 2: Comparison of Electrical Resistance

Figure 3 illustrates that the flexural strength changed with an increase in the percentage of nano-metal oxides. Flexural strength in Epoxy/Gr composite had the lowest value because the percentage of graphite is higher in this composite compared to other composites. Among other composites, bipolar plates containing 7% of nano-ZnO had the highest flexural strength while the one containing 7% of TiO_2 had the lowest flexural strength. According to Knibbs equation (Eq. 1), the flexural strength of these composite is related to their porosity and the particle size of fillers [12]:

$$\sigma = kD^{1/2} e^{bP} \qquad (1)$$

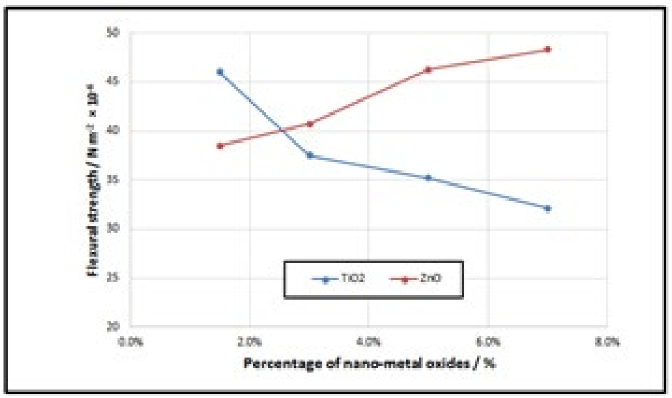


Figure 3: Comparison of Flexural Strength

Where k and b are empirical constants; D and P are the maximum diameter of grains and porosity, respectively; and σ is a flexural strength. Equation 1 shows the relationship between flexural strength and porosity at the same D.

From Figure 1 it can be seen that graphite and nanoparticles dispersed well in the polymer matrix. Also, there is a continuous matrix without any pores, leading to the enhanced flexural strength and electrical conductivity. Figure 3 shows that flexural strength of nanocomposite Epoxy/Gr/ZnO increases with the addition of ZnO, while it decreases with the addition of TiO_2. This result is attributed to the shape of nano-metal oxide particles, which could fill pores of composite better. Since nano-ZnO can make the texture less porous compared to TiO_2 nanoparticles, it shows a higher flexural strength.

As shown in Figure 4, the density of the composites bipolar plates increases with the high content of nano-metal oxides, because the space between composite is filled by nanomaterial and less air is trapped. The figure 4 also shows that density of both nanocomposites is the same and they do not have much difference.

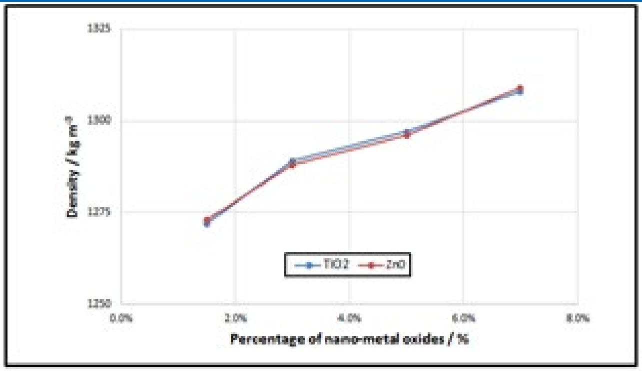


Figure 4: Comparison of Density

Conclusion

The flexural strength of composite plate increased with decreasing graphite and increasing nano-ZnO contribution. The results showed that electrical conductivity could increase with the addition of nano-TiO_2. Also, flexural strength was attributed to the morphology and density of nanocomposites. The higher density the higher flexural strength would be. The addition of nanoparticles to epoxy/graphite composites provides stronger composites that are used as bipolar plates in PEMFC. The data on mechanical properties indicate that these composite plates can be suitable for PEMFC.

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