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Performance of TiO₂-Carbon on Ceramic Template with Sodium Hydroxide Activation as Supercapacitor Electrode Materials

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ABSTRACT

Supercapacitor has been successfully fabricated using TiO₂-Carbon with Sodium Hydroxide (NaOH) activation on ceramics template. Synthesis of TiO₂ sol was conducted by using Sol-gel method. Ceramic membrane as a growth template of TiO₂-C which is activated with NaOH was prepared as a supercapacitor electrode materials. Polyvinyl alcohol (PVA) is used as a separator and phosphoric acid (H₃PO₄) as an electrolyte. NaOH activation on the supercapacitor electrode had increased the capacitance value both at the combustion temperature of 250°C and 300°C. Based on EDX (Energy Dispersive X-Ray) reported that the greatest amount of carbon found in the activated electrode at a combustion temperature of 300°C is 60.67%, which provides the highest capacitance value that is 14 540 nF with H₃PO₄ electrolyte concentration of 0.5 M and the charging time for 30 minutes with a conductivity value of 45.4x10⁻⁵ S cm⁻¹.

Keywords: Supercapacitor, TiO₂, Sodium Hydroxide (NaOH) activation, capacitance, carbon, ceramics

INTRODUCTION

Compared to the commonly used rechargeable batteries, supercapacitors which is capable to be charged-discharged with high current, is an energy storage device which has high power density and long durability [1-3]. Supercapacitor can store energy by accumulating the opposite charge on the double layer at the electrode/electrolyte interface through electrostatic force [4].

A number of carbon materials, such as activated carbon [5-6], carbon aerogel [1, 7], lignin [8], Carbon from palm kernel shell [9] and hydroxyethylcellulose [10], have been widely used as electrode materials for supercapacitors. At present, the use of carbon as a supercapacitor electrode is more desirable because of its advantages, namely carbon has a large surface area making it possible to store charge more, the numbers are more, the price is cheap, and stable. Some research on the modification of carbon as electrodes reported to increase the capacitance of supercapacitors, including adopting the nanostructured iron oxide (Fe₃O₄)-activated carbon composite [11], the growth of platinum nanoparticles on carbon electrodes [12], Hierarchical porous carbon fiber prepared using a SiO₂ template [13]. In this study, TiO₂ synthesized using sol-gel method and then carried on ceramic coating to be used as supercapacitor electrodes. Modification of incomplete combustion of TiO₂ in a wide variety of combustion temperature is expected to increase the amount of carbon in the form of carbon-TiO₂ nanocomposite ceramics and and to expand the electrode surface made chemical activation with NaOH so as to increase the capacitance of a supercapacitor electrode.

MATERIALS AND METHODS

Equipment's and materials

Equipment's which were used are LCR-Meter (Tonghui *Electronic* TH2820-LCR), Multimeter (Heles UX-838TR), petridish, copper plate, glass (4x4 cm²), oven, *furnace, hot plate, charger* (Nokia *Handphone* 8.62 V) and other laboratory glasses equipment's

Materials are ceramics used for floor (Materina), H₃PO₄ (Merck), (CH₂CHOH)_n (Bratachem), Ti(OC₃H₇)₄ (Aldrich, 97%), CH₃CH₂(OH)CH₃ (Merck), C₄H₁₁NO₂ (Merck), and Aquades.

Preparation of electrode

Ceramics are used as floor tiles prepared to be taken inside, thinned until its thickness is 2.5 mm, with a diameter of 4 cm [14]. Titania sol preparation is done by mixing Isopropanol (CH₃CH₂(OH)CH₃), Dietanol Amin (C₄H₁₁NO₂), and Tetra Titanium isopropoxide (Ti(OC₃H₇)₄). The mixture is then stirring for 4 hours at room temperature. The coating process is done by dipping the ceramic into titania sol for 2 minutes. The coating process is performed on all sides of the ceramic [15].

Ceramics which has been coated, heated at 100 ° C for 1 hour then burned at 300° C for 2 hours. Ceramic coating that has been soaked with NaOH for 2 minutes, then dried at a temperature of 100°C. Supercapacitor electrode TiO₂-C was dried in a furnace at a temperature of 250°C and 300°C for 2 hours.

Characterization techniques

X-ray diffractograms of the samples were recorded using a GE XRD 3003TT X-ray diffractometer with monochromatic nickel filtered CuK α ($\lambda = 1.5416 \text{ \AA}$) radiation in the 2θ range of 20° to 80°. The morphology and elemental composition of the carbon were determined by scanning electron microscope (SEM) that has the attachment of an energy dispersive X-ray analyzer (EDX).

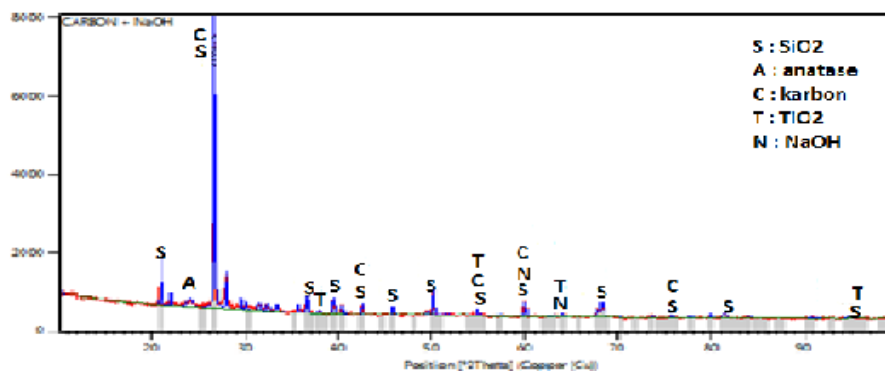
Preparation of supercapacitors

Supercapacitor electrode is made like a sandwich, wherein the polymer electrolyte is placed in the middle between the two electrodes. On both sides of electrodes placed metal plates of copper (Cu plate), then pressed with a glass plate.

RESULTS AND DISCUSSIONS

Synthesis process and structure characterization

XRD patterns of NaOH-activated ceramic at combustion temperatures of 250°C and 300°C are shown in Figure 1 (A) and (B), respectively. Ceramic electrodes were performed on NaOH activation at both the combustion temperature of 250°C and 300°C found contains of SiO₂, TiO₂, carbon, and NaOH. The diffraction pattern of TiO₂-coated electrode at the combustion temperature of 250°C and 300°C showed peaks of a TiO₂ anatase, but in very low intensity. The positions of the diffraction peaks at 25° and 48° indicating TiO₂ in the anatase phase and in good agreement with the results obtained in the previous report [16].



A.

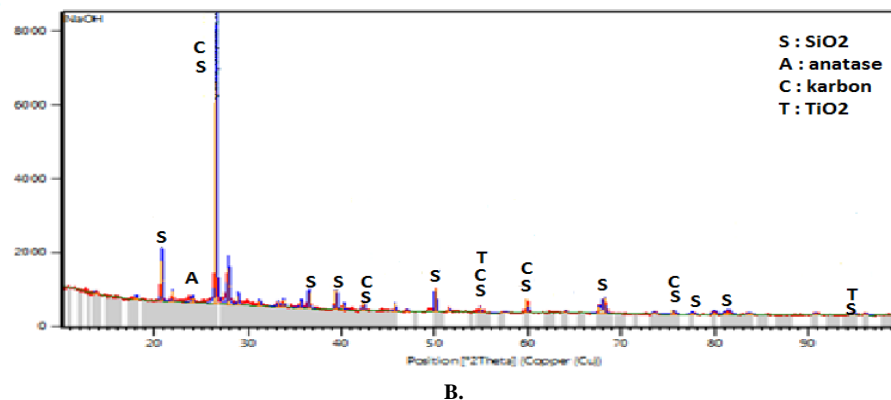


Fig. 1. XRD pattern of ceramic electrode with a combustion temperature at (A) 250°C (B) 300°C

Fig. 2 (B) shows that the ceramic electrode surface looks more smooth and homogeneous than in Fig. 2 (A). This is because of the activation process in the combustion temperature at 300°C provide a TiO₂-C structure with the active group and new functional groups which will play an important role in the charge storage processing [17].

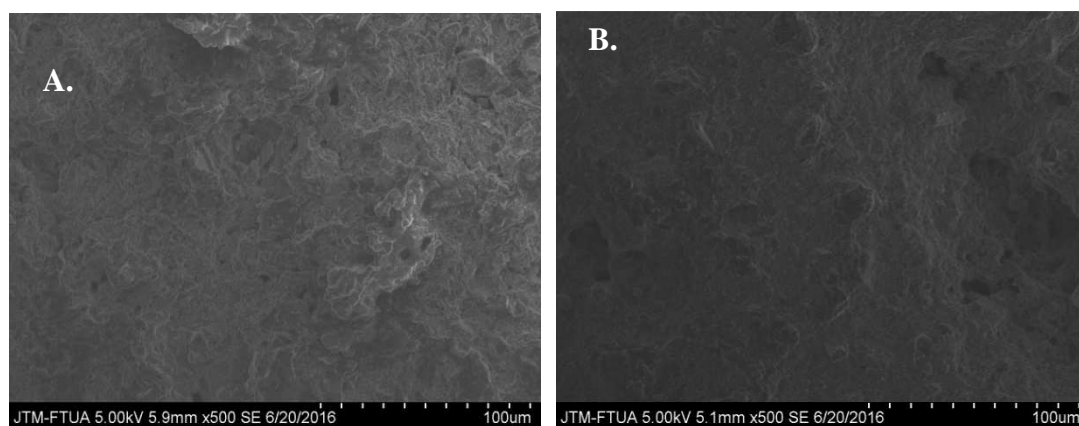


Fig. 2. SEM images of ceramic electrode are activated NaOH with a combustion temperature (A) 250°C and (B) 300°C

The elemental composition obtained from EDX profile of the the resources indicated the presence of carbon, sodium, aluminum, silicon, calcium, and titanium. whereas silicon compounds derived from the ceramic constituent, while Ti is derived from the surface of the ceramic coated titania. Activation and increasing combustion temperatures can increase the amount of carbon in the ceramic electrode, as shown in Table 1 at 300°C, the amount of carbon can raised to 60.67%.

Table 1. EDX measurement of ceramic electrode

Atom	% mass		
	250°C	300 °C	300 °C (Nonaktivasi)
C	29.62	60.67	28.63
O	45.73	25.51	48.88
Na	7.03	3.33	0.61
Al	2.56	2.14	5.99
Si	8.59	5.91	9.78
Ca	4,10	0.33	3.68
Ti	2.36	2.11	2.44

Determination electric properties

Effect of activation with NaOH

In Fig. 3(A) shows that the combustion temperature of 250°C optimum capacitance value ceramic electrodes which are activated (5398 nF) increased 1008 times greater than the electrode before activation (5.350 nF) with H₃PO₄ electrolyte concentration of 0.5 M.

At a temperature of 300°C combustion electrodes activated with NaOH (14540 nF) increased by 148 times greater than the electrode before activation (98.12 nF) as shown in Figure 3 (B). The electrodes are activated and the combustion temperature to 300°C (14540 nF) has the optimum capacitance is almost three times greater than the electrode with the combustion temperature to 250°C (5398 nF) as shown in Fig. 3 (A) and 3 (B). This is due to the combustion temperature of 250°C most of isopropanol and diethanolamine burns to make TiO₂-C while the combustion temperature to 300°C, isopropanol and diethanolamine was burned to form TiO₂-C so as to increase the capacitance value.

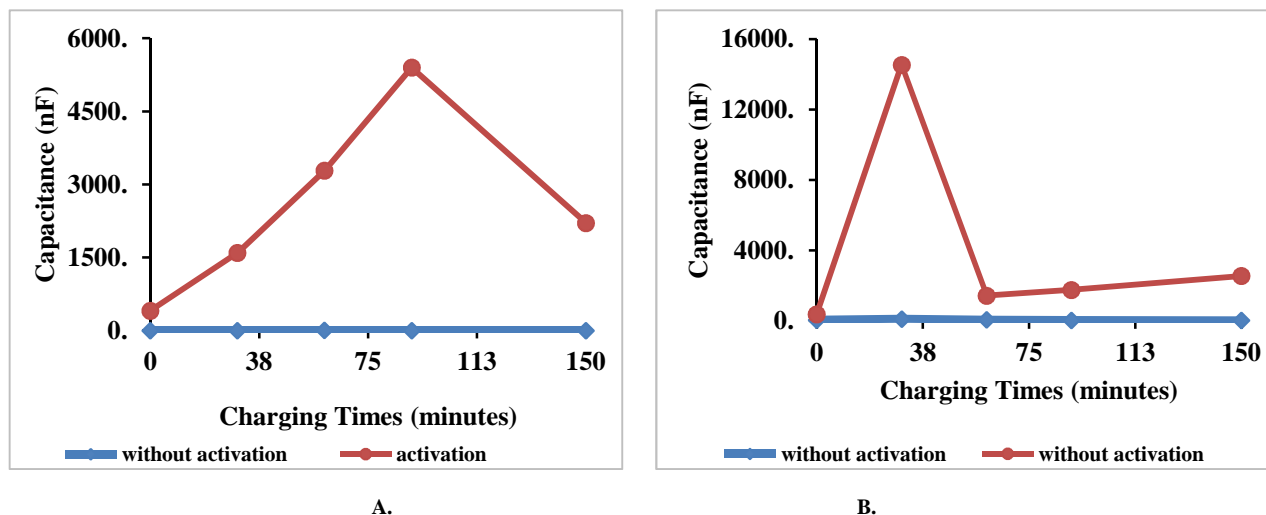


Fig. 3. Effect activation of the capacitance on the temperature of combustion (A) 250°C and (B) 300°C

Effect of H₃PO₄ electrolyte concentration

Fig. 4 shows that with increasing H₃PO₄ concentration give a greater optimum capacitance value at 0.5 M and then decrease in higher altitude toward stationary. This is because the higher the concentration of the electrolyte, more the amount of electrolyte ions, so that at the time of charging, the ions will be collected and accumulated at the interface of electrolyte and separator. A large amount of these ions will slow down the process so that the polarization discharge process of H₃PO₄ ions will be disturbed. The high concentration of H₃PO₄ also can damage the electrodes and the separators [9, 11].

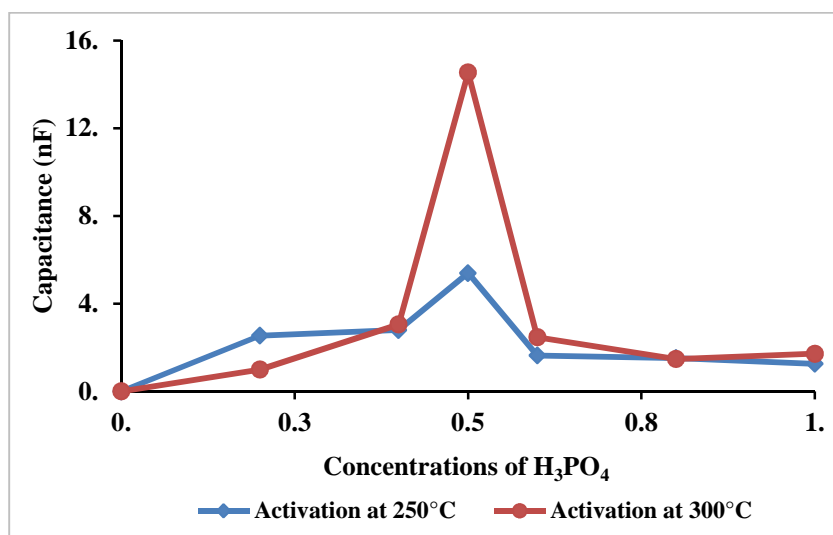


Fig. 4 Effect of H₃PO₄ electrolyte concentration against capacitance

Effect of the charging times against conductivity

Fig. 5 shows the effect of the charging time on the conductivity. At combustion temperature of 250°C the conductivity increase until the charging time of 90 minutes, and then decreased slowly. As also observed at the combustion temperature of 300°C the conductivity increase up to 30 minutes of charging time, and then decreased toward almost stationary. The greater the capacitance, makes more electrons to flow and the conductivity increase.

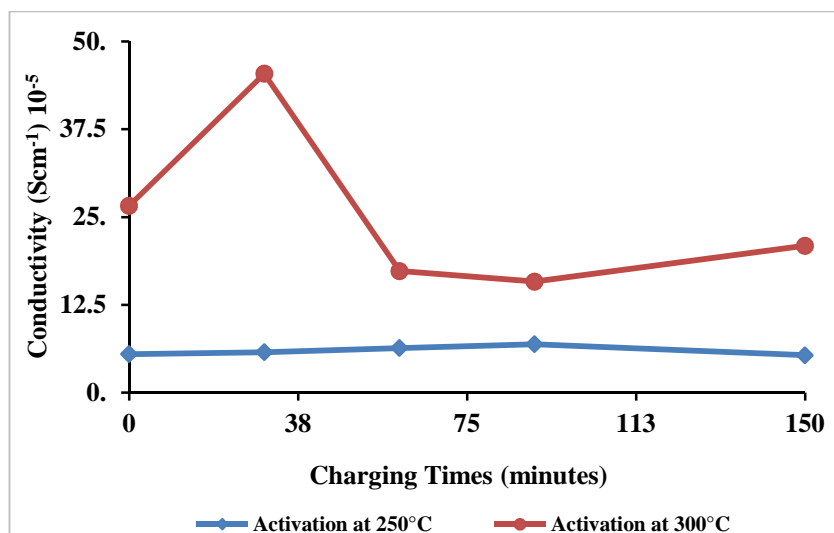


Fig. 5 Effect of the charging times against conductivity

CONCLUSION

Considering on the actual research that has been done, it can be concluded that the NaOH-activation clearly affect the performance TiO₂-C ceramic supercapacitor electrodes thus increasing the capacitance value of both combustion temperature of 250°C and 300°C. In the NaOH-activated ceramic electrode at the combustion temperature of 300°C provides an optimum capacitance value almost three times greater (14 540 nF) than one of the combustion temperature of 250° C (5398 nF) with H₃PO₄ electrolyte concentration of 0.5 M in 30 min. charging time with conductivity value of 45.4x10⁻⁵ S cm⁻¹.

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