



Sertifikat

Nomer : B/49983/UN38.3/DL.01.02/2021

Diberikan Kepada :

Nur Hidayati

Sebagai :

PEMAKALAH

dalam acara **SEMINAR NASIONAL FISIKA 2021** dengan tema :

**"New Adaptations in Physics Learning and Research to Realize 'Merdeka Belajar' Program
(Adaptasi Baru dalam Pembelajaran dan Riset Fisika untuk Mewujudkan Program Merdeka Belajar)"
yang diselenggarakan oleh Jurusan Fisika FMIPA Universitas Negeri Surabaya
pada Hari Senin, 18 Oktober 2021**



Dekan FMIPA Unesa

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Surabaya, 18 Oktober 2021

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To cite this article: N Hidayati and Munasir 2021 *J. Phys.: Conf. Ser.* **2110** 012012

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Study of Electrical and Thermal Properties of PVA-Nanosilica as A Candidate for Supercapacitor Separators

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Abstract. Separator in the supercapacitor that separates the cathode from the anode has an important role in a supercapacitor circuit. Polyvinyl Alcohol (PVA) is a substitute for polyolefin which is commonly used as a supercapacitor separator and PVA has more environmentally friendly properties. The addition of silica dioxide nanocomposites is useful for adding thermal stability and electrical insulators. The method used to make the separator is quite simple, namely by casting a PVA gel membrane with nanosilica on a glass plate and drying it at low temperature. PVA membrane with nanosilica variations different precursors are from sand by coprecipitation method and from TEOS. The properties of the two samples were characterized by scanning electron microscopy (SEM), thermo gravimetric analysis (TGA), and LCR meter. Nanosilica derived from sand and from TEOS have no much different characteristic.

1. Introduction

As the portable electronics industry develops, supercapacitors are becoming very useful components as efficient and sustainable energy storage. The supercapacitors can store electrical energy, can be easily carried and used whenever needed. Supercapacitors or double layer capacitors have a higher energy and power density than batteries [1]. The supercapacitor arrangement simply consists of an electrode (cathode and anode) with a layer of activated carbon, a separator, and an electrolyte [2].

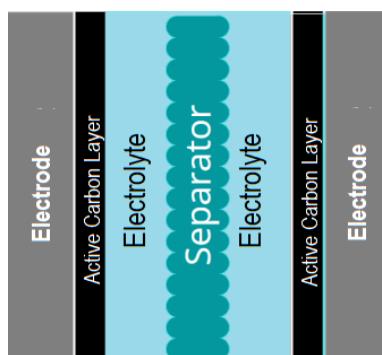


Figure 1. Supercapacitor structure

One of the components in the supercapacitor that plays an important role is the separator. The separator in the supercapacitor acts to separate the anode and cathode, so that when ion transport takes place during charging and discharging, it can avoid an internal short circuit [3]. Thickness, pore size,

chemical and thermal stability, and good ion permeability are the requirements for the separator character in the supercapacitor. At high temperatures, separators with low thermal stability can cause various safety problems. The design of a separator with a porous structure and high thermal stability is very important for a supercapacitor performance. Polyolefin-based microporous membranes such as polypropylene and polyethylene are widely used as separator materials. However, there are things that need to be considered, such as thermal stability, porosity, and poor electrochemical performance [4].

For more than three decades the sol-gel reaction has been studied extensively as a method for preparing polymer composites. Solution of TEOS and PVA with sol-gel reaction get a composite because the loading TEOS acts as a cross-linking component making the composite denser [5]. Polyvinyl Alcohol (PVA) based membranes have important characteristics, such as uniform and porous surface morphology, symmetrical interconnected porous structure throughout the thickness of the separator, and excellent electrolyte wettability, resulting in superior electrolyte adsorption/retention capacity, excellent thermal degradation, lower, and higher ionic conductivity, compared to commercial polypropylene (PP) separators [6]. PVA with the incorporation of inorganic particles such as SiO₂ can improve thermal stability and electrical insulator. In this study, nanosilica was added to the PVA-based separator in order to increase the electrical insulating properties and thermal stability of a separator. It is known that nanosilica acts as an electrical insulator with high chemical and thermal stability. Nanosilica can be synthesized from chemical precursors, namely Tetraethyl Orthosilicate (TEOS) to natural materials such as sand. Comparison of the separator characteristics of PVA with the addition of silica dioxide nanocomposite from chemical and natural precursors by polymerization method at low temperature is discussed in this article.

2. Method

2.1 Materials

The materials needed in this study were Polyvinyl Alcohol (PVA) from Merckmillipore, Tetraethyl Orthosilicate (TEOS, 98%) from Merck, and sand (76.8 wt% SiO₂). Other supporting materials used include aquades, ethanol (C₂H₅OH, 99%), hydrochloric acid (HCl, 37%), and sodium hydroxide (NaOH).

2.2 Synthesize method

2.2.1 Synthesize of SiO₂

76.8wt% SiO₂ sand was synthesized using coprecipitation or deposition methods. This method aims to make the solute to the bottom so as to form the required precipitate. In addition, coprecipitation requires a relatively short time (\pm 12 hours) and a low working temperature [7]. In this study, previously sand was mixed with 7M NaOH to form a sodium silicate solution. Then it was allowed to stand for 24 hours while 2M HCl was titrated to form a silicate gel. The gel was washed using distilled water and then dried for 2 hours [8].

2.2.2 Nanocomposite Membrane

Nanocomposite membranes were fabricated with nanosilica from the synthesis of SiO₂ from sand and from TEOS compounds. The first membrane contains nanosilica powder that has been synthesized and then dissolved with a concentration of 1% using distilled water. Then the solution was stirred with 5wt% PVA solution for 12 hours. While the second membrane contains nanosilica from the TEOS compound, where 5wt% PVA solution is added with 5 ml of TEOS and 1 ml of HCl then stirred for 12 hours. PVA with nanosilica precursor from sand (PVA-SiO₂) and PVA with nanosilica precursor from TEOS (PVA-

TEOS) were poured on glass and dried at atmospheric pressure and room temperature for 2-3 days, respectively. After that the membrane can be peeled off slowly and dried at 60°C for 3 hours.

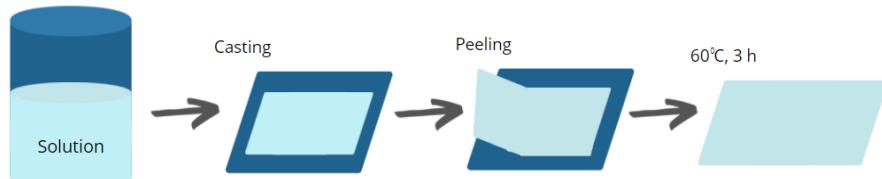


Figure 2. Membrane preparation process

2.3 Characterization.

The nanocomposite membranes were characterized using a Scanning Electron Microscope (SEM) (FEI Inspect-S50 type) to determine the transverse surface topography. Thermal stability testing was carried out using Thermo Gravimetric Analysis (TGA) (TA Instruments, DSC Q20, Waters LLC), in which the nanocomposite membrane was heated from ambient temperature to 600°C at a rate of 10°C/min with nitrogen gas. The conductivity and resistance values are obtained from the LCR meter (Inductance, Capacitance & Resistance) with two probes.

3. Results and Discussion

3.1 Nanocomposite membrane characterization results

3.1.1. Scanning electron microscope (SEM)

Figure 3 shows the scan results of the two samples using SEM on the cross-surface. The technique used to analyze is a free software image at a magnification of 5000 times. The thickness of the two samples obtained is 18-60 μm . The microstructure of the PVA-TEOS sample looks like a sheet with more structured pores than the PVA-SiO₂ sample. The Si-O-Si bond polycondensation process is formed after the hydrogel turns into a membrane. This is because the hydrogen bonding ability between the -OH groups of PVA-TEOS is stronger than that of PVA-SiO₂ [10]. PVA hydroxyl can react with the hydroxyl of the silanol group on the surface of the silica particles formed in the ^{5000X} sol-gel process. This reaction lowers the surface energy of the silica particles and thus prevents aggregation. Meanwhile, the reaction between silica particles from sand and PVA could not be observed clearly.

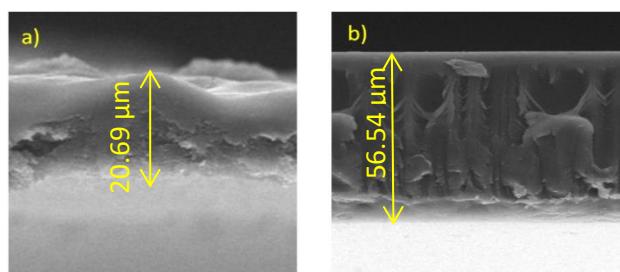


Figure 3. SEM on transverse surface a) PVA-SiO₂ b) PVA-TEOS

3.1.2. Thermo gravimetric analysis (TGA)

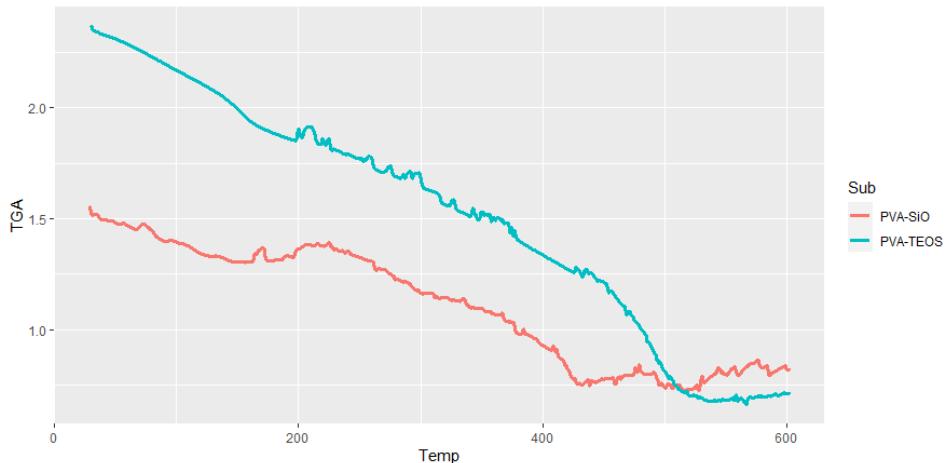


Figure 4. TGA results of the two samples

Based on the comparison of the two samples, the thermal and degradation behaviour of PVA-SiO₂ and PVA-TEOS can be known. The first decrease in the weight of the membrane between the ambient temperature to 200°C, which corresponds to the physical molecules of air and acid that occur. Most of the water molecules are in the direct state to the polymer chain via hydrogen but not in the free molecular state [9]. The first stage of weight loss in the PVA-SiO₂ sample experienced a loss of 10%, while the PVA-TEOS sample lost 19%. The second stage of PVA-TEOS membrane decomposition starts from 470-515°C, along the main PVA circuit. Meanwhile, the second stage of PVA-SiO₂ membrane weight reduction occurred at around 400-425°C. The third stage of weight loss occurs up to 600°C. PVA-SiO₂ membrane decomposes at high temperature which is more stable than PVA-TEOS. Nanosilica granules can increase thermal degradation which acts as a PVA thermal barrier [10]. The final weight residue remaining in PVA-SiO₂ is slightly more than that in PVA-TEOS.

3.1.3. Conductance and Resistance.

The membrane used as a separator in a supercapacitor has a low electrical conductivity and a high resistance value [11]. The resistance value is obtained using an LCR meter, while the conductivity value is calculated using Equation 1.

$$\text{Conductivity} = \frac{L}{R A} \quad (1)$$

Table 1. Conductivity and resistance

Separators	Conductivity Value (S/m)	Resistance Value (Ω)
PVA-TEOS	1.044×10^{-7}	5.65×10^6
PVA-SiO ₂	1.012×10^{-7}	6.31×10^6

Low conductivity values can prevent short circuits in supercapacitors. The standard value of conductivity in a polymer membrane is 10^{-7} - 10^{-3} S/m [12]. In both membranes, the conductivity and resistance values are almost equal. The PVA-SiO² membrane obtained a slightly larger resistance value than the PVA-TEOS membrane, namely 6.31×10^6 . Meanwhile, the conductivity value of the membrane after being calculated using Equation 1 obtained the same result of 10^{-7} S/m. The high resistance value

is due to the small concentration of PVA used in the membrane. The less PVA in the membrane, the more water it contains. So the resulting resistance value is getting bigger.

3.2. Discussion of PVA-SiO₂ as a supercapacitor separator candidate

Based on the results of the characterization in previous studies, it was shown that the PVA-SiO₂ membrane was very potential as a membrane in supercapacitors. The electrical and thermal properties of PVA can be improved by dispersing small amounts of SiO₂ in the polymer matrix. The interfacial interaction between PVA chains and SiO₂ nanoparticles is the most decisive factor governing the properties of PVA-SiO₂ nanocomposites. Silanol and siloxane groups on the surface of the silica produce the hydrophilic properties of the nanoparticles [13]. In addition to the PVA-SiO₂ hydrogel composite molding method, the PVA-SiO₂ nanofiber composite made by electrospinning exhibits electrical and thermal properties that can replace commercial PP separators. This is indicated by the results of the thermal shrinkage at a certain temperature variation, where the membrane mass shrinks as shown in Figure 5.

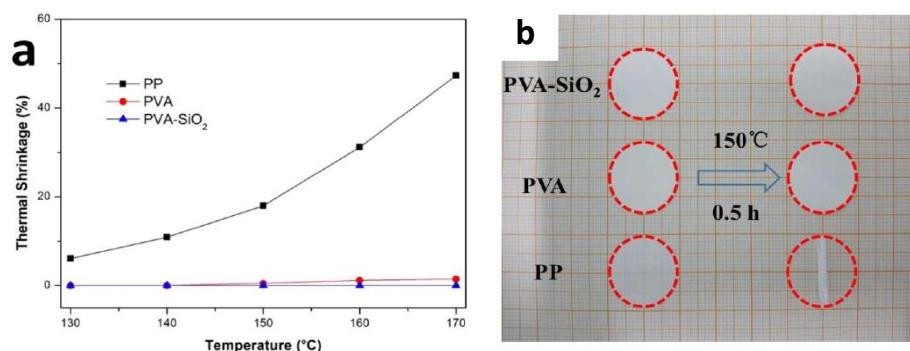


Figure 5. a) Thermal shrinkage results and b) Results after and before the membrane is heated [14]

At a temperature of 130-170°C PP membrane experienced a drastic thermal shrinkage, while PVA with PVA-SiO₂ showed almost no shrinkage. This can also be observed in Figure 5b, where PP shrinks into rolls after being heated at 150 °C for 0.5 hours (30 minutes).

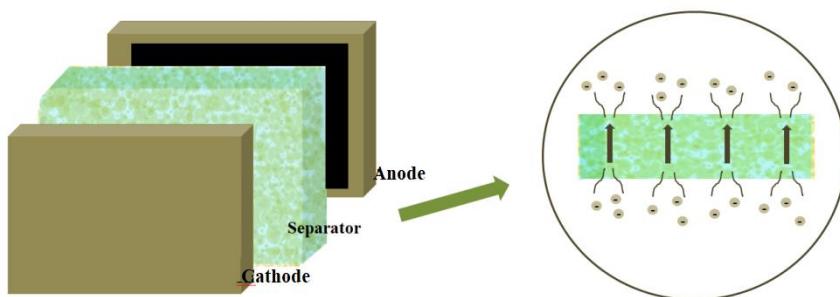


Figure 6. Schematic of the supercapacitor

The cartilage-like and porous structure of PVA makes it an ideal structure for a separator. The large concentration of silica content in the PVA hydrogel membrane can also affect the ionic mobility and the volume of the membrane cavity which affects the charge density in order to develop its electrical conductivity properties [15]. High ionic conductivity is essential for good cycling, depending on the

pore structure, porosity and absorption ability of the electrolyte. Highly porous separators are beneficial for the absorption of large amounts of electrolyte and thus lead to higher ionic conductivity [16].

4. Conclusion

In this article, PVA membrane with 5% concentration has been made with the addition of nanosilica from different precursors as a separator. The resulting membrane thickness ranges from 18-60 μm . Nanosilica derived from sand and derived from TEOS have almost similar characteristics. It can be seen from the result of TGA which decrease the weight of the membrane between the ambient temperature to 200°C. The resistance and conductivity which are not much different. In the PVA-TEOS membrane, the resistance and conductance were $5.65 \times 10^6 \Omega$ and $1.044 \times 10^{-7} \text{ S/m}$, while in the PVA-SiO₂ were $6.31 \times 10^6 \Omega$ and $1.012 \times 10^{-7} \text{ S/m}$. Based on the test results of the two membranes, it can be concluded that the membrane is suitable to be used as a supercapacitor separator in terms of thermal and electrical properties.

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