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Preparation of SiC Fibers from Indonesian Natural Resource Prepared by Electrospinning Method

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Abstract. SiC fibers were prepared via electrospinning method. The starting materials were polyvinyl alcohol, sucrose, and SiO2. The SiO2was extracted from natural pyrophyllite. In this work, we report the effect of polyvinyl alcohol and applied voltage on structural SiC fibers. The samples were then characterized using XRF, FTIR, and SEM-EDX. Based on the results of data analysis, it was shown that the optimum composition of polyvinyl alcohol in the SiC fibers was of 13.75% (w/v). It was also found that the SiC fibers had a smooth surface with a uniform diameter. Furthermore, the optimum applied voltage was set up at about 24 kV.

Keyword. Electrospinning, poly, polyvinyl alcohol, SiC Fiber, and sucrose.

1. Introduction

Recently, silicon carbide (SiC) fibers become essential materials due to their potential applications in many areas such as aerospace, aviation, and nuclear energy. In general, such claim is related to the particular characteristics of SiC fibers, for example, high tensile strength, high elastic modulus, high-temperature resistivity, excellent corrosion and wear resistance [1–4]

Generally, various preparation methods in fabricating SiC fibers could be conducted by using pyrolysis and electrospinning [5–7]. Interestingly, the electro spinning method becomes effective and efficient method because it can be performed with easy way, inexpensive, simple experimental setup, and easy to control the fibers [8]. SiC fibers generally prepared using commercial Si powders or polycarbo methylsilaneas Si elemental source [6, 9]. On the other hand, in Indonesia, Si is available abundantly in nature almost in all area. Practically, natural Si can be obtained from SiO2containing several impurities such as TiO2, CaO, and Al2O3. Such contaminants could be trough out by leaching process to captureSiO2particles with high purity.

In this work, SiC fibers were prepared using SiO2particles as the main precursor extracted from natural Indonesian pyrophyllite. The extractedSiO2particles were then modified to form SiC nanoparticles using the sonochemical method. The SiC fibers were obtained using electrospinning method. Furthermore, in this paper, we report the effect of polyvinyl alcohol and applied voltage on structural SiC fibers.

2. Materials and methods

The SiO2 used in this study was obtained from local pyrophyllite ore. Based on the XRF characterization, the chemical composition of the pyrophyllite was Si (64.6 %), Al (16.0 %), Fe (9.0 %), Ti (7.2 %), and other elements. The pyrophyllite was ground using a mortar and a pestle. 20 grams of the ground sample were then leached using 5 M NaOH solution for 3 h and at a boiling temperature. After leaching process, the solution was diluted and filtered to separate undissolved materials. The reaction expected during this process was like in the Equation 1.

$$SiO_2 + NaOH \rightarrow Na_2SiO_3 + H_2O \quad (1)$$

Furthermore, HCl was added dropwise to the obtained solution until the SiO_2 gel was formed. Finally, theSiO_2 gel was rinsed using distilled water and dried to produce white-colored SiO_2powder.The reaction expected during the sintering process using magnesium was like the Equation 2.

$$Na_2SiO_3 + HCl \rightarrow SiO_2 + NaOH + H_2O \quad (2)$$

The SiC nanoparticles were synthesized using a sonochemical method. 3 g SiO_2 and 9 g sucrose were added to a 240 mL distilled water and ethanol mixture. Next, the mixture was stirred and continued by sonication process for one hour at 70 °C. The result was then put into a furnace for the following preheating process for 1 h at 180 °C. After that, the process continued by annealing at 950 °C in argon condition until SiC nanoparticles were produced.

To produce SiC nao fibers, 1 g SiC particles were mixed with PVA solution with concentration variation of 12.5 %, 13.75 %, and 15 % *w/v*. Eachof the solutionswas then stirred and sonicated for 1 h to disperse the SiC nanoparticles. The sonicated solution was then put into a syringe with 24 gauge needle. In this electrospinning process, the parameters were maintainedat18 kV, 20 kV, 22 kV, and 24 kV.

The percentages of elements, size, and morphology of the samples were identified through x-ray fluorescence (XRF, PANalytical) and scanning electron microscopy and energy-dispersive x-ray spectroscopy (SEM-EDX, FEI INSPECT-S50) characterizations.

3. Results and discussion

The prepared SiO2 was then extracted to form SiC nanoparticles. The EDX test results showed that the SiC nanoparticles had a high purity with the Si, C, and O contents (Table 1). Figure 1 shows the FTIR test result on the SiC nanoparticles. The sample showed the existence of a Si-C functional group at the wavenumber of 798.5 cm^{-1}, a Si-O-C functional group at the wavenumbers of 999.13 cm^{-1}, and a Si-O functional group at 1200 cm^{-1}. Such findings are in good agreement with the database of experimental results of the researchers, in which the Si-C functional group formed at a wave number of around 754 cm^{-1} until 840 cm^{-1}[6,10–13].

Table 1. Element content of SiC nanoparticles

No.	Element	Content of element (%)
1.	Si	50.94
2.	C	44.05
3.	O	5.01

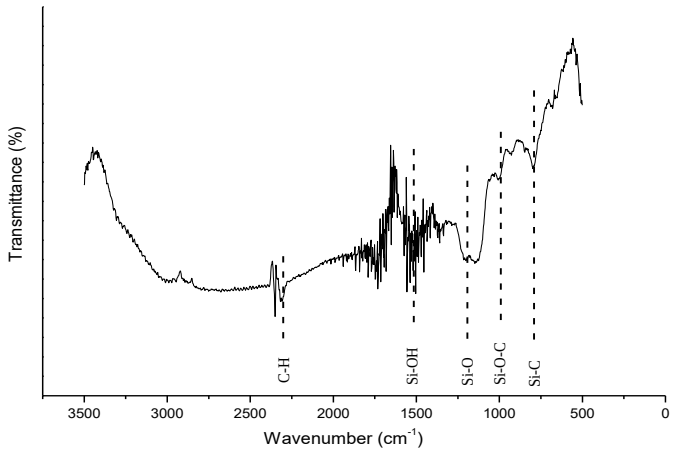

Figure 1. Result of FTIR Characterization of SiC Sample

The SEM results of SiC nanofibers with PVA concentrations of 12.5 %, 13.75 %, and 15 % w/v.10 % *w/v*is shown in Figure 2. It can be seen that the produced SiC nanofibers were formed in beaded-structures, which indicated the low concentration of PVA solution. Generally, in electrospinning, the polymer solution has critical value in order to obtain the fibers. Kim *et al*. reported that beaded-structure SiC nanofibers can be overcome by increasing volume concentration of PVA [14]. Figure 2 expresses the SEM results of the SiC nanofibers with the 12.5 %, 13.75%, and 15 % *w/v* variations of PVA solution. The produced SiC nanofibers tended to be straight and to possess a smooth and bead-free surface. An increase in the PVA solution concentration increased the SiC nanofiber diameter. The SiC nanofibers with 12.5 % and 15 % *w/v* PVA concentrations have different diameters. The percentages of SiC nanofiber element contents identified via EDX are shown in Table 2. The existence of the O element and a high proportion of C element resulted from the carbon chain of the PVA.

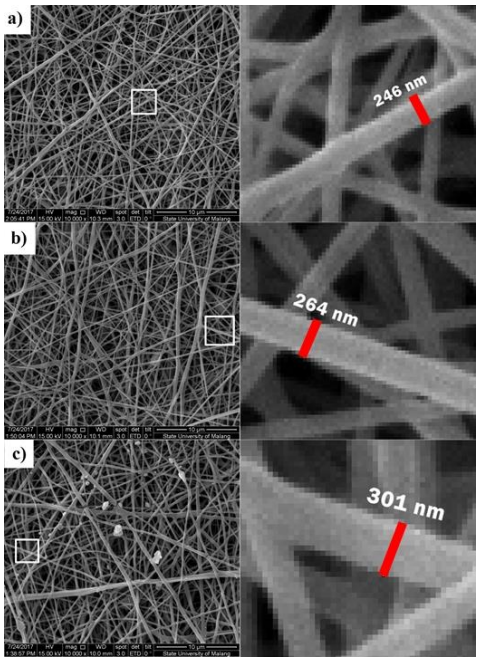

Figure 2. Morphology of SiC nanofibers synthesized by electrospinning method using a PVA solution of (a) 12.5% w/v; (b) 13.75% w/v; and (c) 15% w/v.

Table 2. Elemental contents of SiCNanofibers

No.	PVA Concentration (% w/v)	Element Contents (%)		
		Si	C	O
1.	12.50	1.64	63.20	35.16
2.	13.75	1.58	62.83	35.59
3.	15.00	1.89	64.23	33.88

In this research, we also report the effect of voltage variations on the structures of SiC nanofiber as presented in Figure 3. Theoretically, the voltage plays an important role in producing SiC nanofibers. The figure shows that the voltage gives an effect on the structural change of the SiC nanofibers. From the figure, the higher the voltage, the lower the diameter of the SiC nanofibers. Physically, an increasing voltage causes the increasing strain of the polymer originating from increasing Coulomb force and electric field and finally reducing the diameter of the fibers [8].

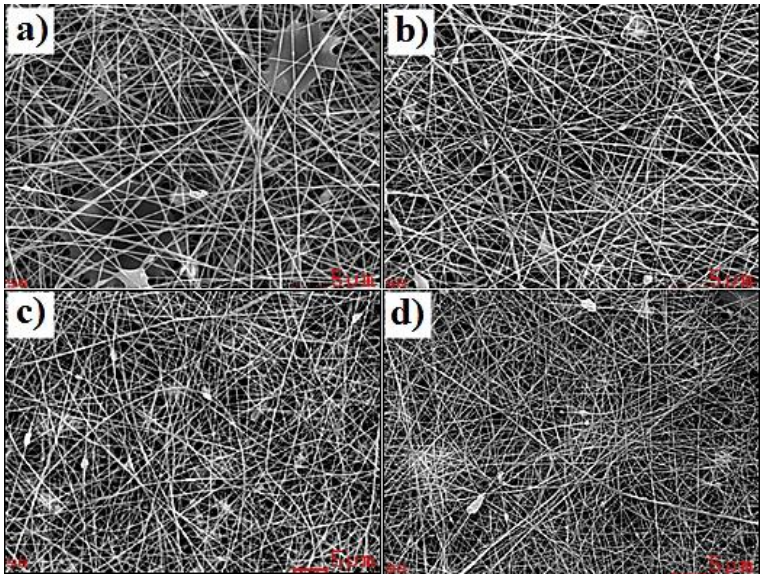

Figure 3. Morphology of the SiC nanofibers with voltage variations of a) 18 kV, b) 20 kV, c) 22 kV; and d) 24 kV.

4. Conclusions

In this research, SiC nanofibers were prepared from a natural resource using electrospinning method. The SiC nanoparticles were dispersed in a polymer solution by the polymer concentration and applied voltage affected the structure and diameter of the SiC nanofibers. The optimum level of the PVA to obtain the proper SiC nanofibers was 13.75 %, and the applied voltage was set up at about 24 kV, which had straight structured SiC fibers with a smooth surface.

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