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Synthesis of SiC nanowires on biochar surfaces by a simple direct method

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Abstract

Demand for air filters is crucially required due to PM 2.5. So, SiC nanowire networks are directly grown on biochar surfaces that serve strength and filtering purposes. The nanowires were prepared by spraying silicon powder on Lesser bulrush and Grey sedge biochars which acted as carbon sources and heating up to 1300°C to 1500°C for 1 h in Ar atmosphere. As a result, silicon powder evaporated and diffused on biochar surfaces. Meanwhile, CO vapor was generated from biochar reacted with O_2 in the chamber. The evaporated silicon reacted with both C(s) and CO(g) to form cubic SiC nanowires. At the early stage of the reaction, large nuclei of SiC nanowires were formed at 1300°C. Those nuclei became smaller to promote nanowires when a higher temperature was applied before being disappeared at 1500°C. At such temperature, the obtained network structure SiC nanowires with 10 nm to 30 nm diameters on the outer and inner surfaces of biochars were completed, facilitating superior strength and filtering purposes. Comparatively, the Lesser bulrush provided higher strength than those of the Grey sedge ones, thus it was selected for the filtering efficiency test. The results showed that particulate filter efficiency was up to 97% but it was still over the range of pressure drop at 30 in H_2O.

1. Introduction

Silicon carbide (SiC) nanowires exhibit several outstanding characteristics higher than those of the micro SiC, such as ultra-high hardness, high melting point, thermal stability and corrosion resistance, low thermal expansion coefficient [1,2], large specific surface area, high electron mobility [3,4] and photoluminescence properties [5]. Therefore, SiC nanowires play an important role to create nanodevices construction, for example in transmitters, effect transistors and sensor nanoscales [6-8], microwave absorbers, optoelectronic devices [9], and micro-capacitor [10].

In recent years, SiC nanowires have focused many researchers on their synthetic methods which have been used to form β-SiC nanowires, including carbothermic reduction [11], chemical vapor deposition (CVD) [5], microwave [12], arc-discharge [13], thermal evaporation [14] and etc. These methods are very expensive and complicated processes. Also, the metal catalysts and molten salt used in these processes were difficult in order to remove from the resultant SiC nanowires which can affect the product properties. Other studies had conducted by infiltration of silica sol to charcoal (Tatia amurensis wood) and heat up to obtain β and α-SiC phases with bulk struts and whisker structures due to gas-solid (SiO(g) + C(s)) and gas-gas (SiO(g) + CO (g)) reactions, respectively [15]. Hexing Liu *et al.* [16] fabricated a porous SiC/C ceramic from the reaction of biomass C-template, Si powder, and $Fe(NO_3)_3{\cdot}9H_2O$ as a catalyst. When heating up to 1300°C in Ar, SiC nanowires were grown on the surfaces and holes giving rise to improve superior oxidation resistance. Two mechanisms could be explained by the growth of nanowires 1) SiO(g) reacted with 2C(s) and 2) SiO(g) and CO(g) dissolved in Fe-Si alloy until reaching its optimum condition to form SiC nucleation and further growth in perpendicular to (111) plane. Many researchers reported the synthesis of a high yield of SiC nanowires through thermal evaporation without adding any metal catalyst from an exterior [17,18]. However, the source of carbon was methane gas (CH_4), a kind of synthetic gas, that was quite expensive. Therefore, a greener, simpler, and low-cost process need to be developed to synthesize SiC nanowires as investigated in this study.

Particulate Matter (PM) is a mixture of solid and liquid particles suspended in the air. These are categorized into coarse, fine, and ultrafine ones. Coarse particles have a diameter of 2.5 µm to 10 µm and are relatively heavier and thus tend to settle. Conversely, PM 2.5 is referred to particles that have a diameter less than 2.5 µm and remain suspended longer resulting in a serious pollutant and causing respiratory problems. Consequently, the needs for high-performance air filter devices with low operation cost are common for a household. Recently, air filter efficiency defines two main properties including particulate removal efficiency and low airflow resistance (pressure drop). In addition, filter garbage should not be affected by the environment. So, biomass is possible to use as an air filter due to its highly porous network structure in nature and biodegradability. Biomass could be used as the bio-template after carbonization and then converted to bio-ceramic SiC via carbothermic reaction under a controlled atmosphere. It showed both network structures of the vascular system and synthesized SiC nanowires. Therefore, bio-ceramic SiC has been used in many applications such as high-temperature

filters [19], wastewater treatment [20], and microreactor systems [21]. Moreover, the carbon source from biochar and silicon vapor, SiC nanowire [22] have been generated on bio-template resulting in a high surface area that was beneficial to more efficient PM 2.5 filtration.

The aim of this research was to synthesize SiC nanowires coated on biochar by a simple direct method. Carbon sources from biomass of Lesser bulrush (*Typha angustifolia L.)* and Grey sedge (*Lepironia articalata*) were carbonized and then reacted to Si powder at various temperatures (1300°C to 1500°C) in a controlled atmosphere to form SiC nanowires coated biochar. The effects of two types of carbon sources, Si particle coating methods and synthesis temperatures on the phase, crystal size, and structure of the nanowires were investigated.

2. Experimental

2.1 Materials

Both biomass of Lesser bulrush (*Typha angustifolia L.)* from Pathum Thani province and Grey sedge (*Lepironia articalata*) from Rayoug province were cut, cleaned, and dried at 100°C for 12 h. The samples were wrapped with aluminum foil and placed in a stainless retort to control the amount of oxygen and then carbonized at a temperature of 700°C for 1 h. The fixed carbon as shown in Equation (1) after drying and burning out of moisture (MC) to obtain volatile matters (VM) and ash was calculated. The obtained biochars were stable in shape due to the percent of fixed carbon in the range of 65 to 75. They were used as carbon sources to synthesize SiC nanowires. Silicon (Si) powder (-325 mesh, 99% trace metals basis, product no. 215619, Sigma-Aldrich) was another major material used in this study. When heating up, it was in a gaseous state surrounded and infiltrated to the biochars. Then, it reacted to C-source biochars to generate SiC coated on the outer and inner surfaces of the tube biochars. Such SiC nanowires coated on the surfaces would improve the strength of biochars together with their porous structure originating from the vascular system which was beneficial for filter application.

$$\text{Fixed carbon (\%)} = 100\text{-ASH(\%)-VM(\%)-MC(\%)} \qquad (1)$$

2.2 Fabrication of SiC nanowire

Two coating methods of Si solution, dipping and spraying (See Figure 1), were studied on the effect of SiC surface coating on Lesser bulrush and Grey sedge biochars. The silicon powders were dispersed into a methanol solution by a weight ratio of silicon: methanol of 1:4. The mixture was stirred until the silicon powder was evenly distributed. For dipping, biochars were immersed in the stirred solution one time for 10 min and spraying was performed by keeping a 20 cm distance with 45°of the machine from biochars and rotated biochar 90° for one cycle and repeating for three cycles. Then, the coated biochars were dried at 100°C for 3 h. After drying, the samples were placed in an atmospheric control furnace chamber and air suction in the furnace chamber was operated down to 10^{-5} torr while argon gas was purged at 5 mL·min^{-1} into the furnace chamber. The atmospheric control furnace was heated up to 1300°C to 1500°C for 1 h in the argon atmosphere where the SiC reaction was taking place. Finally, the obtained biochars coated with SiC phase were observed phases by X-ray Diffractometer (XRD 6000, Shimadzu Corporation) at 2θ = 20° to 80°, Cu K_α (λ= 1.5406 Å), 30 kV, 30 mA and scan speed 2°·min^{-1}. The crystal size of SiC was related to measuring the full-width half maximum (FWHM) of SiC main peak and then calculated by Scherrer's Equation (2).

$$D = \frac{K\lambda}{\beta\cos\theta} \qquad (2)$$

Where D = Crystallite size
K = Scherrer constant: 0.89 [23]
λ = X-ray wavelength, CuK_α = 1.5406 Å
β = The line broadening at FWHM in radians
θ = The Bragg's angle in degrees, half of 2θ

The morphologies of SiC-coated biochars at the outer surface and fracture surfaces of the tubes were observed by the mean of Scanning Electron Microscopy (SEM, JSM-5410LV, JEOL). All samples were attached to the studs by carbon tape, put in a sputter coater for vacuum operation, and ionized a gold sheet with a current of 15 mA for 180 s. The schematic of the experimental setup is shown in Figure 2.

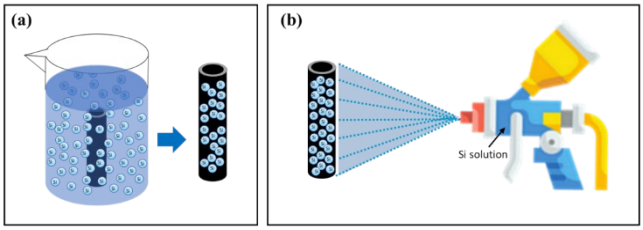

Figure 1. The schematic diagrams of biochar coating methods: (a) dipping, and (b) spraying.

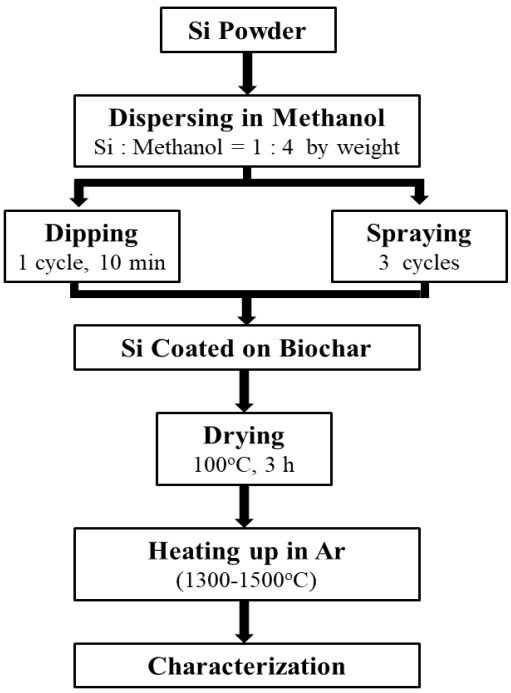

Figure 2. The schematic of the experimental setup.

2.3 Method of air filter measurement

For air filter application, the particulate filtration efficiency method (the particle filtration and pressure drop) was adapted from medical mask testing ASTM F2299 and 42 CFR 84 using Electrostatic classifier equipment (TSI classifier). The atomizer aerosol generator (3076, TSI Inc., USA) generated NaCl and Emery oil particles in the size range of 15 nm to 750 nm. Those particles would be dried, concentration adjusted, and aerosol neutralized. Then the aerosols were sent to mix with air to obtain the air flow rate at 8.5 $L \cdot s^{-1}$ and face velocity of 10.6 $cm \cdot s^{-1}$. All adjusted aerosols were flown to a test chamber with SiC-coated biochar tubes inside (filter area of 17.35 cm^2) and counted by an Electrostatic size classifier (Model 3788, TSI Inc., St. Paul, MN, USA) of the upstream particles (particle/cm^2) and downstream particles (particle·cm^{-2}). Filter efficiency was calculated by Equation (3)

$$\text{Filter efficiency} = \left[\frac{\text{Cintel-Coutlet}}{\text{Cinlet}}\right] \times 100\% \qquad (3)$$

C_{inlet} = Particle concentration before passing SiC-coated biochar filter

C_{outlet} = Particle concentration after passing SiC-coated biochar filter

The pressure drop, a value to determine the capability and lifetime of a filter, was measured by a manometer (Model 8380, TSI Inc., St. Paul, MN, USA) before and after aerosols passed through the SiC filter [24].

3. Results and discussion

3.1 The effect of the Si coating method

To investigate the effect of Si coating on biochar surfaces, Si powder was dispersed in a methanol solution and coated on the biochar surfaces by dipping and spraying. Then, they were heated up to 1400°C for 1 h in the Ar atmosphere. Figure 3 shows the biochar (Figure 3(a)) and coated biochar after firing (Figure 3(b-c)). It could obviously notice that two coating methods, spraying and dipping, showed different morphologies of grey color on the samples. That was, spraying Si solution on biochar provided more uniform SiC nanowires coating with homogeneous grey color (Figure 3(b)) while the dipped biochar surface showed uneven grey throughout the surface (Figure 3(c)). As a result, the spraying technique, Si particles were well dispersed together with better Si particles alignment and high adherence force from the nozzle pressure due to the smooth coating layer. Meanwhile, in the dipping method, Si particles were possibly agglomerated along with uneven thickness coating occurred resulting in uneven color. Furthermore, the Si-methanol solution was in a gas-liquid phase during spraying, so it was easily evaporated. Conversely, in dipping, the Si-methanol solution was in powder-liquid form. When biochar was immersed in Si solution, it could absorb liquid methanol until saturation affected the particles' attachment to the biochar surface. However, the two different coating methods did not affect to SiC structure but it did to the amount of SiC phase which could notice from different colors in grey (SiC) and black (C). Therefore, in the next experiment, the spraying method was applied for the fabrication process to synthesize SiC nanowires on biochar surfaces.

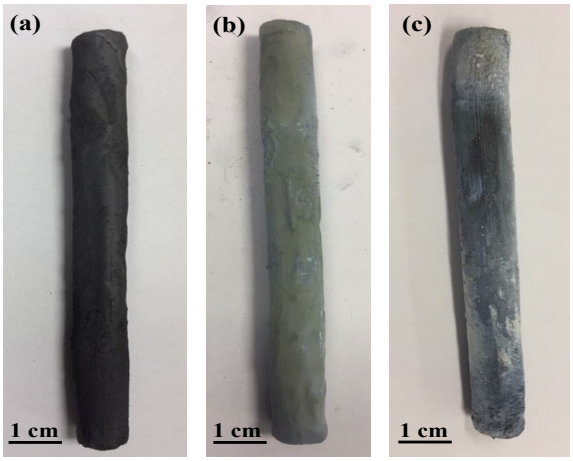

Figure 3. Morphologies of biochar fired at 700°C coated with different methods Biochar without coating, (b) coated by spraying, and (c) coated by dipping.

3.2 The effect of synthesis temperatures

To observe the effect of synthesis temperatures, Si-coated biochars were heated at different temperatures, 1300°C, 1350°C, 1400°C, 1450°C, and 1500°C for 1 h in Ar atmosphere. The results are reported in 3.2.1 and 3.2.2. for both types of biochars, Lesser bulrush, and Grey Sedge, respectively.

3.2.1 Synthesis of SiC nanowires on the Lesser bulrush (*Typha angustifolia L.)* biochar surface

Figure 4 shows phase analysis of Lesser bulrush biochar and Si-coated biochars fired at 1300°C to 1500°C in the Ar atmosphere. From XRD patterns, Lesser bulrush biochar showed broad spectra of the C-amorphous phase along with JCPDS card number 75-1621. For Si-coated biochars, they demonstrated three main peaks at 2θ (Cu k_α) = 35.63°, 60.28°, and 71.82°, which were in good agreement with reflections of (111), (220) and (311) planes, respectively, as cubic β-SiC corresponding to JCPDS card number 73-7108. The obtained SiC phase was observed at all synthesis temperatures (1300°C to 1500°C). Furthermore, it was found that SiC nanowire crystallite sizes varied depending upon synthesis temperatures calculating full-width half max (FWHM) of SiC main peak at 35.63°. When the synthesized temperature was increased, the FWHM of SiC peaks were narrowed. Thus, the SiC nanowire crystallite sizes were increased at increasing temperatures (Figure 5).

Figure 6 shows the morphologies of SiC nanowires on the surface of Lesser bulrush at 1300°C (Figure 6(a)), 1400°C (Figure 6(b)), and 1500°C (Figure 6(c)) at low (2500X) and high magnifications (10,000X). At 1300°C, SiC nanowires were generated with a lot of nuclei and then disappeared when increasing to 1400°C and 1500°C. This could be explained that the reaction of cubic SiC nanowires was completed at higher temperatures and provided the nanowires in the range of 10 nm to 30 nm in diameter.

Figure 7 is the cross-section of Lesser bulrush biochar-coated SiC which demonstrated the network-built structural morphologies of biological materials and coated by SiC nanowires on the surfaces. Those networking structures might show potential for air filter application which will be observed in the next experiment.

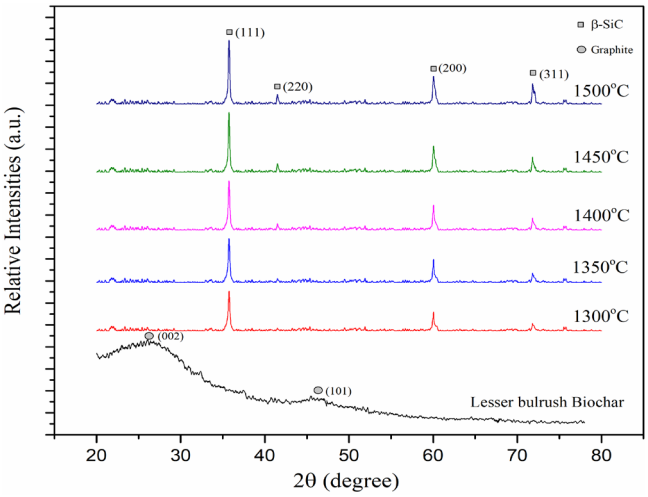

Figure 4. XRD patterns of Lesser bulrush biochar and biochars coated SiC from 1300°C to 1500°C with an interval of 50°C.

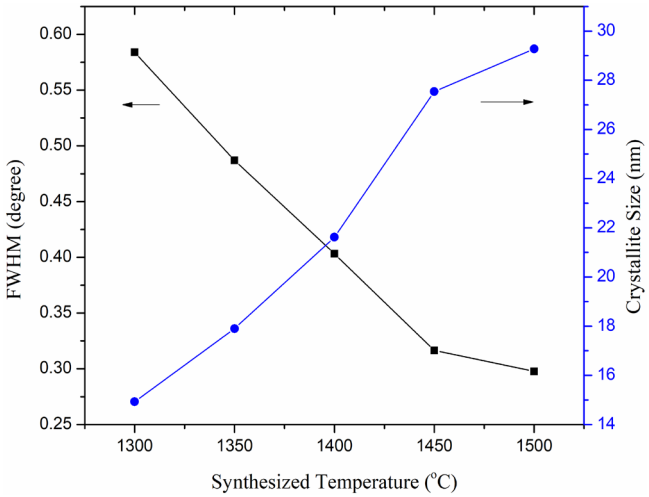

Figure 5. FWHM and crystallite size of the SiC nanowires of Lesser bulrush from 1300°C to 1500°C with an interval of 50°C.

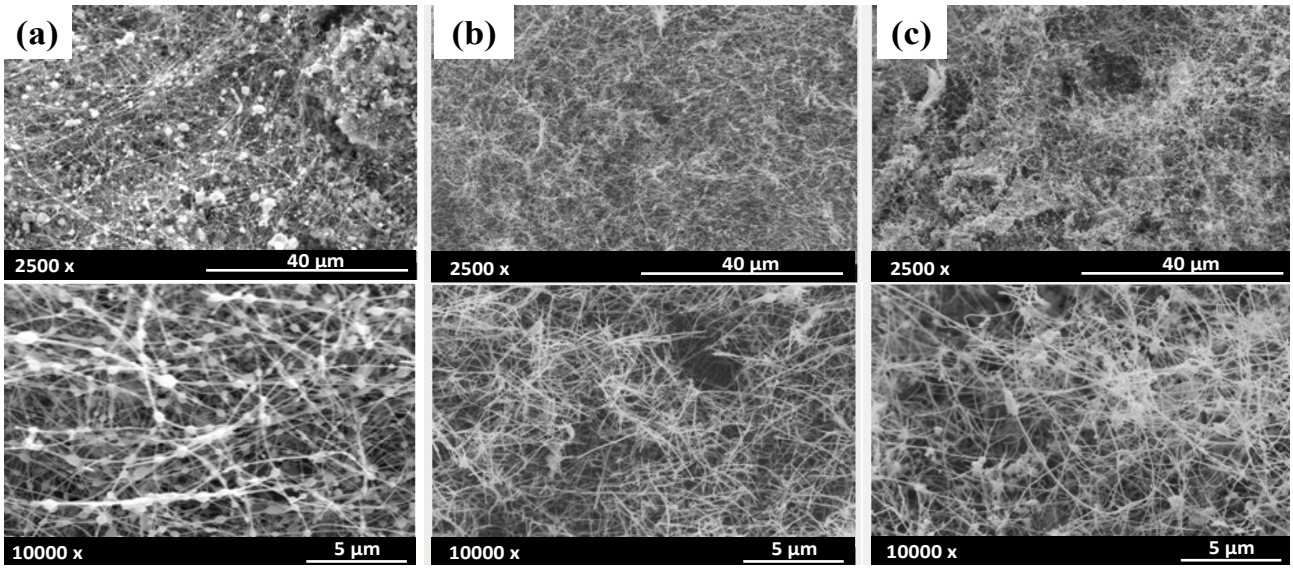

Figure 6. SEM photographs of the synthesized nanowire surfaces of Lesser bulrush at (a) 1300°C, (b) 1400°C, and (c) 1500°C.

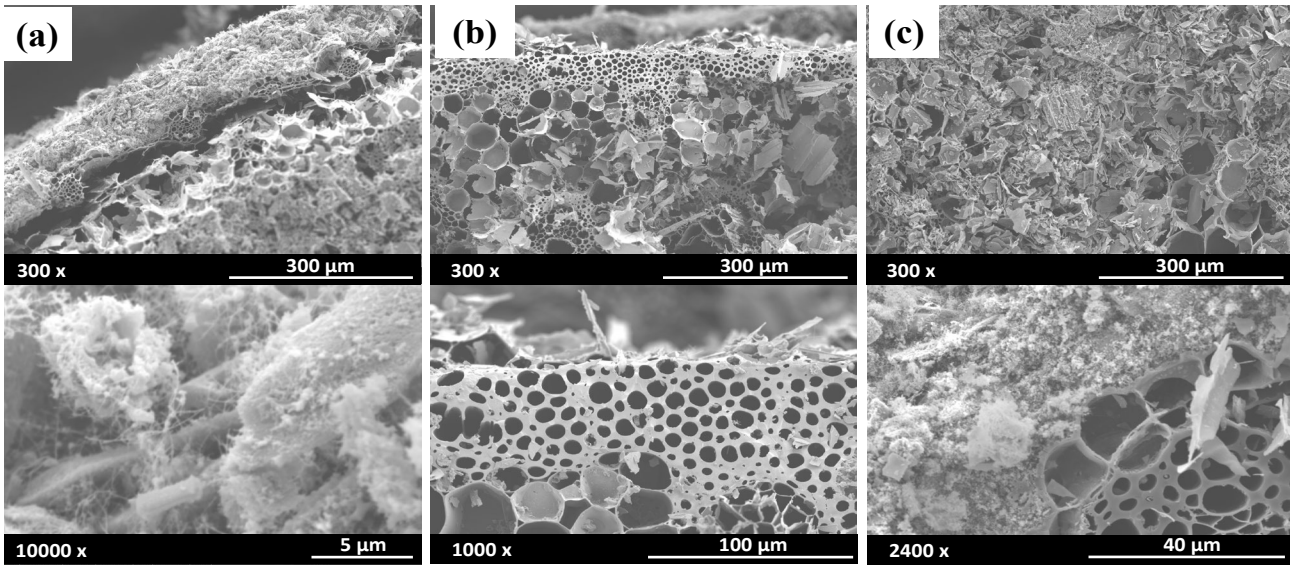

Figure 7. SEM photographs of the fracture surfaces of Lesser bulrush at (a) 1300°C, (b) 1400°C, and (c) 1500°C.

3.2.2 Synthesis of SiC nanowires on Grey sedge (*Lepironia articalata*) biochars surface

Figure 8 exhibits phases of Grey sedge biochar and biochars coated Si after firing at 1300°C to 1500°C. Grey sedge biochar showed a broad peak of the C-amorphous phase as well. Biochars coated Si was found SiC and one more phase of $CaNa_3B_5O_{10}$ as JCPDS card number 78-0295 in all synthesis temperatures due to the effect of NaCl molecule in the seawater where Krajood was grown near the seaside area. Therefore, the obtained phases in Grey sedge biochar were different from Lesser bulrush biochar which showed only SiC nanowire. Also, the Grey sedge had a smaller tube, thinner wall area of networking porous structure, and lower strength compared to Lesser bulrush biochar. Thus, the Grey sedge was not proper to observe filter properties. Figure 9 shows the crystallite sizes calculated from full-width half max (FWHM). It was shown that when the synthesized temperatures were increased, the FWHM of SiC peaks were narrowed. Thus, the SiC nanowire sizes were increased with increasing temperatures.

Morphologies of SiC nanowires on the surface of Grey sedge (Figure 10(a-c)) were synthesized from 1300°C to 1500°C. The results showed that nanowires on the Grey sedge surface were smaller than those on the Lesser bulrush surface with the same magnification (Figure 6(a-c)). It was because, in Grey sedge biochar, there were some kinds of salts such as Ca and Na, etc, which could act as a catalyst to promote the reaction between Si and carbon source from biochar and finally small SiC nanowires generated. Figure 11 reveals the fracture surfaces of SiC-coated Grey sedge biochars, they were found to have a thin membrane at the center of a tube (Figure 11(c)) which is a typical characteristic structure of Grey sedge.

Based on the experimental result mentioned previously, the SiC nanowires formation process was seemingly involved with the SiO vapor phase. As the temperature increased, SiO vapor is generated through the evaporation of Si. Especially, when the furnace is heated up until close to the melting point of Si (1414°C), the evaporation of Si is accelerated. Simultaneously, C-source biochar reacted with oxygen to form CO and CO_2 vapor which still existed in the system, and then reacted to SiO vapor to form SiC as the following equations.

$$2C(s) + O_2(g) \rightarrow 2CO(g) \quad (4)$$

$$C(s) + O_2(g) \rightarrow CO_2(g) \quad (5)$$

Three main reactions of SiC nucleation might be as equations below.

$$SiO(g) + 3CO(g) \rightarrow SiC(s) + 2CO_2(g) \quad (6)$$

$$Si(s) + 2CO(g) \rightarrow SiC(s) + CO_2(g) \quad (7)$$

$$SiO(g) + 2C(s) \rightarrow SiC(s) + CO(g) \quad (8)$$

The clusters of SiC nuclei gathered to form nanowires. The nanowires in a preferred orientation rapidly grew as more SiO and CO vapor co-existed in the system which was in agreement with the previous works [17,18].

3.2.3 Air filtration result

The results above were related to the carbonization reaction of biochar and the carbothermic reaction to form SiC of Lesser bulrush and Grey sedge biochars. It was found that they hold a tube shape, β-SiC nanowire on the surfaces, and networking-built structure in nature to facilitate for filtration. When consideration of the two biochars coated SiC, we obtained that Lesser bulrush was suitable for evaluation PM 2.5 filter efficiency owing to its physically higher strength and more porous area due to its bigger and thicker wall tube. The measured porosity was 52% with a median pore diameter of 1.95 µm and an average pore diameter of 4.44 µm. The results of particulate filtration efficiency of Lesser bulrush was 97%, however, the pressure drop was too high in a value of 30 in H_2O (normally for N95 Masks < 30 inH_2O and medical Mask < 6 inH_2O). Also, this value was over the range in the capability of the manometer equipped in the instrument. Consequently, optimal conditions have been further studied to decrease a pressure drop and also to increase filter efficiency.

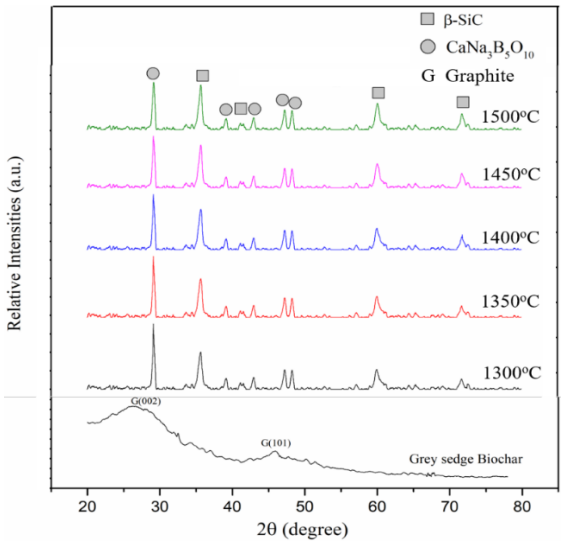

Figure 8. XRD patterns of Grey sedge biochar and biochars coated SiC from 1300-1500°C with an interval of 50°C.

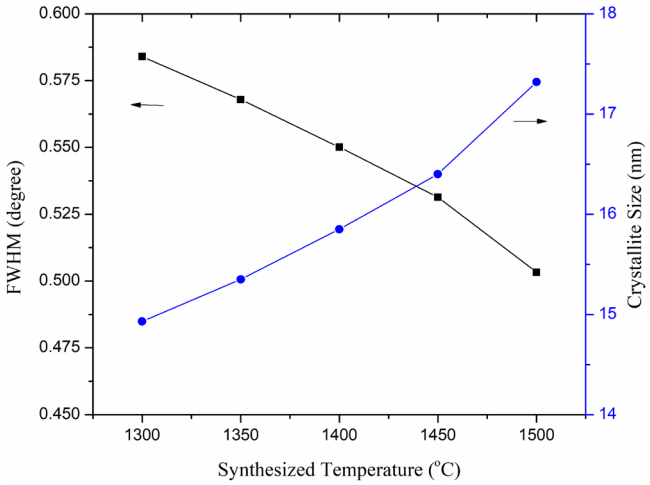

Figure 9. FWHM and crystallite size of SiC nanowires of Grey sedge from 1300-1500°C with an interval of 50°C

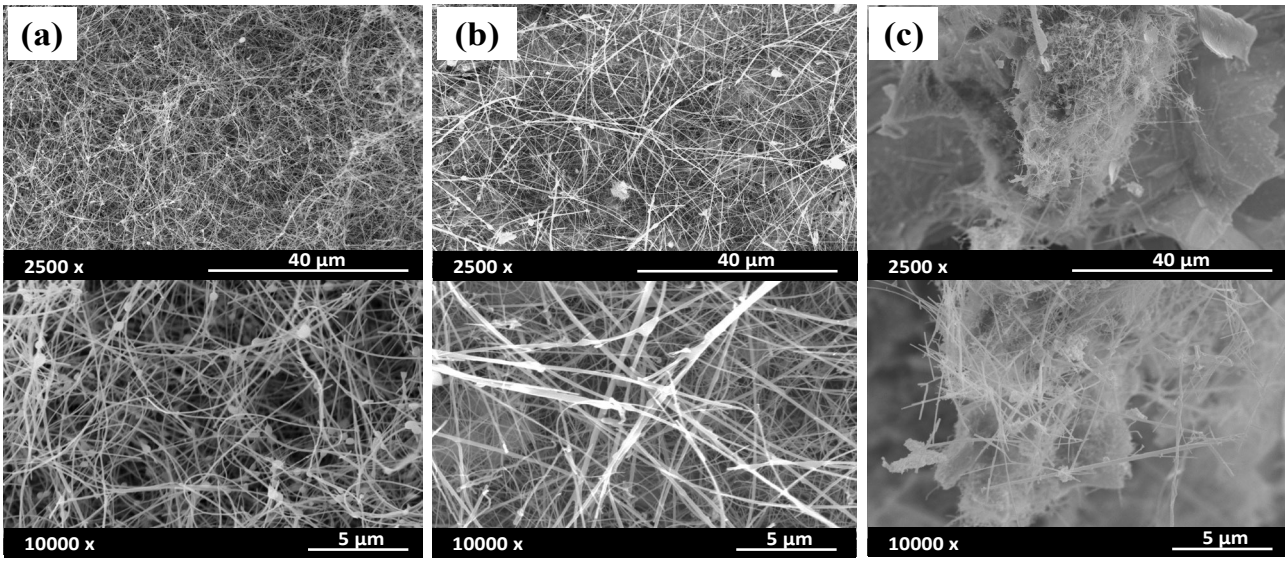

Figure 10. SEM photographs of the synthesized nanowire surfaces of Grey sedge at (a) 1300ºC, (b) 1400ºC, and (c) 1500ºC.

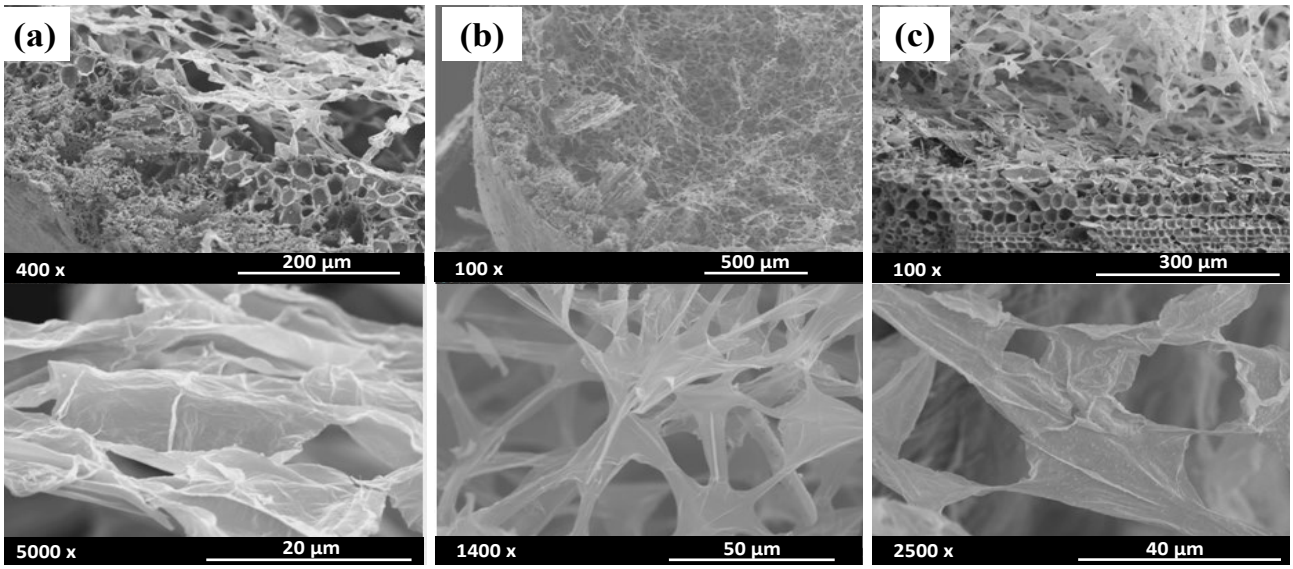

Figure 11. SEM photographs of the fracture surfaces of Grey sedge at (a) 1300ºC, (b) 1400ºC, and (c) 1500ºC.

4. Conclusions

A simple direct method that was very easy and low-cost to fabricate SiC nanowires on the surface of Lesser bulrush (*Typha angustifolia L.)* and (*Lepironia articalata*) biochars from low-cost carbon sources was proposed. Based on the experimental results of the current work, SiC nanowires with a small size of 10 nm to 30 nm in diameter were generated by a carbothermic reaction between C-source from natural resources and Si particles at temperatures from 1300°C to 1500°C. Their porous and network structure did draw much attraction for air filter application. Only Lesser bulrush was tested for filter efficiency because it provided satisfactory strength and proper porous structure. The result of the particulate filter was 97% however, a high-pressure drop over 30 in H_2O was still obtained and needed to be improved.

Acknowledgements

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