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Preliminary Study of Preparation and Characterizations of Carbon-Based Materials-Embedded on Nanocomposites Fiber for Smart Textile Applications

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Abstract: Carbon-based materials-embedded on nanocomposites fiber are interesting due their unique properties for versatile application such as smart textile or electro or e-textile. Herein, functionalized multi-walled carbon nanotube (MWCNT)/polyvinyl alcohol (PVA) and graphene oxide (GO)/PVA nanocomposites fibers were developed through rotational wet spinning process. The resultant of nanocomposites fiber was characterized systematically through optical microscopy (OM), scanning electron microscopy (SEM), resistivity test, tensile test, and sensitivity test. Based on SEM observation, the diameter of pristine MWCNT/PVA and GO/PVA nanocomposites fibers were around 260 and 200 μm, respectively. The results show that MWCNT/PVA nanocomposites fibers with the ratio of 1:5 show higher tensile strength and elongation in compare with GO/PVA nanocomposites fibers with the ratio of 1:5. According to the resistivity test, MWCNT/PVA nanocomposites fiber shows the conductivity values in the range of 10^{-2} S/cm. Dispersion and arrangement of carbon-based materials on the polymer matrix are supposed to be the main factor that influenced nanocomposites fiber properties including the mechanical and electrical properties. Therefore, these nanocomposites fiber are anticipated to be developed as a component for smart fabric or smart textiles.

Keywords: Wet spinning; nanocomposites; smart textiles; carbon-based material
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1. Introduction

Nanocomposites fiber exhibits interesting features and properties that could be developed for versatile applications such as smart fabric and smart textiles. Nanocomposites fiber can be developed through incorporation of inorganic materials as fillers on the organic materials (polymer) as a matrix. The combination of these two different materials is designed to take the advantages from their materials properties. As a matrix, polymeric materials provide a flexible and elastic feature, meanwhile as a filler, inorganic materials can be used as a support for mechanical or electrical

characteristics. Various carbon-based materials including carbon nanotube (CNT) [1] or graphene-based materials have been developed as a filler in polymeric system [2]. Previous research conducted by Min Kyoon Shin et al has developed reduced graphene oxide flakes and CNT incorporated in PVA matrix system [3]. Pei Zhang et al prepared MWCNT grafted PVA through alkylation [4].

Wet spinning is an efficient method to develop nanocomposites fiber material [5]. Fibers that are developed from wet spinning process are anticipated to be used for wearable strain sensors [5]. Herein, PVA was used as matrix and carbon-based material (MWCNT or GO) as filler for polymer nanocomposites. PVA is one of hydrophilic polymer that is commonly developed as spun-fiber [6, 7]. Surfactant plays an important role in order to improve the dispersion and interfacial bonding between CNT and polymeric materials which generate the stable colloidal system [8, 9].

In this study, nanocomposites fiber based on PVA and MWCNT or GO as a matrix and filler, respectively, were developed through rotational wet spinning process. The resultant of nanocomposites fiber was evaluated mainly in term of structure, morphology, and electrical properties through OM, SEM, and resistivity measurement, respectively. Their potential application for smart fabric or smart textile would be evaluated.

2. Materials and Methods

2.1 Materials

PVA and sodium dodecyl sulphate (SDS, $C_{12}H_{25}SO_4Na$) were purchased from Sigma Aldrich. Acetone was purchased from Merck. MWCNT was purchased from Chengdu China. Furthermore, MWCNT was functionalized *via* acid treatment. Aqua demineralization was used throughout experiments. All chemical were used as received without any further purification.

2.2 Preparation of MWCNT/PVA and GO/PVA nanocomposites fibers

Briefly, water was chosen as dispersing medium for MWCNT or GO through probe sonication and as solvent for PVA. SDS was used as surfactant-aided carbon-based dispersion in order to enhance the MWCNT dispersion on the water system. The spinning dope composed of carbon-based dispersion such us MWCNT or GO dispersed in PVA solution was loaded into 1 mL disposable syringe installed in a syringe pump, which was continuously injected into acetone coagulation bath. After injection, nanocomposites-based fiber were immersed in water for 30, 60, 90, and 120 minutes at 50^{o}C and drying overnight on the vacuum condition at 50^{o}C prior to characterization. These process are expected to remove any residual surfactant [10]. In this study, the characteristics of resultant nanocomposites fiber was also evaluated through immersion in water at room temperature for 24 h and 48 h and commercially available detergent solution for 30 min at room temperature and 50^{o}C. Eventually, after washed with water and drying, the resistance values of the nanocomposite fiber were measured. GO/PVA (1:5) nanocomposites fibers was prepared as the aforementioned process. The resultant of GO/PVA nanocomposites fibers was evaluated in water immersion at room temperature for 2h and 4h and water immersion at 50^{o}C for 30, 60 and 90 min.

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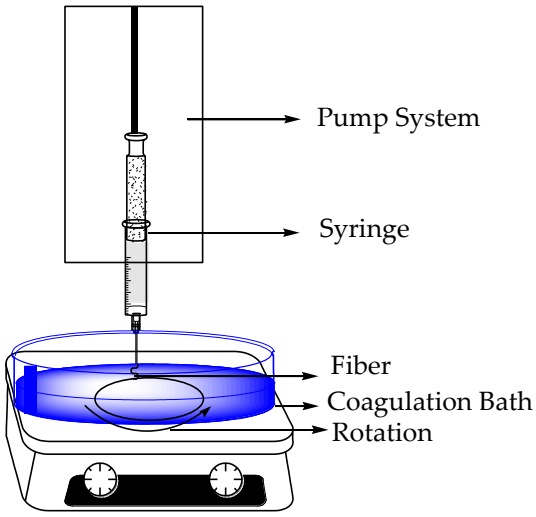

Figure 1. Schematic image and the set-up of the rotational wet spinning machine used to produce the MWCNT/PVA or GO/PVA nanocomposites fiber

2.3 Characterizations

Structure and morphology of nanocomposites fiber were evaluated through SEM and OM observation. Single filament tensile test (DMA Q 800 V20.26) was used to evaluate the mechanical properties of the nanocomposites fiber. Resistivity of the resultant nanocomposites fiber was measured by using digital multimeter. Preliminary studies of humidity sensing was evaluated through humidity bottles containing metal salts saturated solutions of sodium chloride (NaCl) sealed in glass bottles at 20 °C to obtain relative humidity (RH) conditions of 75%. The resistivity of nanocomposites fiber during the sensitivity test was measured using a probe DC method with a Keithley 2000 multimeter.

3. Results

3.1 Macroscopic observation of nanocomposites fiber

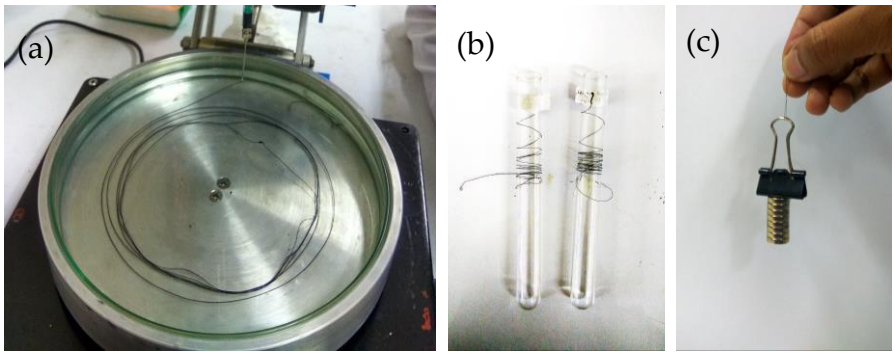

Figure 2. Production of nanocomposites fiber (a), as-spun fibers (b), schematics representation of a weight (25 gram) was lifted using MWCNT/PVA (1:5) nanocomposites fiber

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3.2 SEM observation of pristine MWCNT/PVA (1:5) and (b) GO/PVA (1:5)

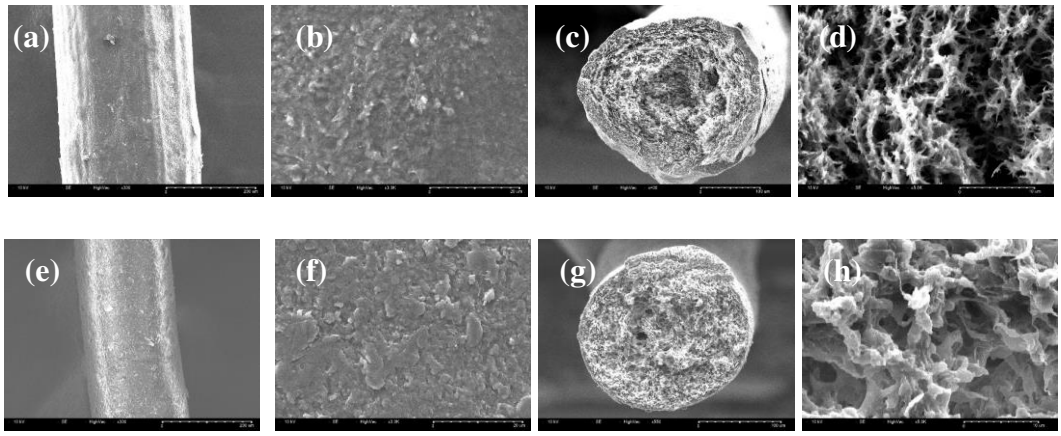

Figure 3. SEM and cross section SEM images of MWCNT/PVA (ratio of 1:5) (a-d) and GO/PVA (ratio of 1:5) (e-h) nanocomposites fiber

3.3 Single filament tensile test measurement of pristine MWCNT/PVA (1:5) and (b) GO/PVA (1:5)

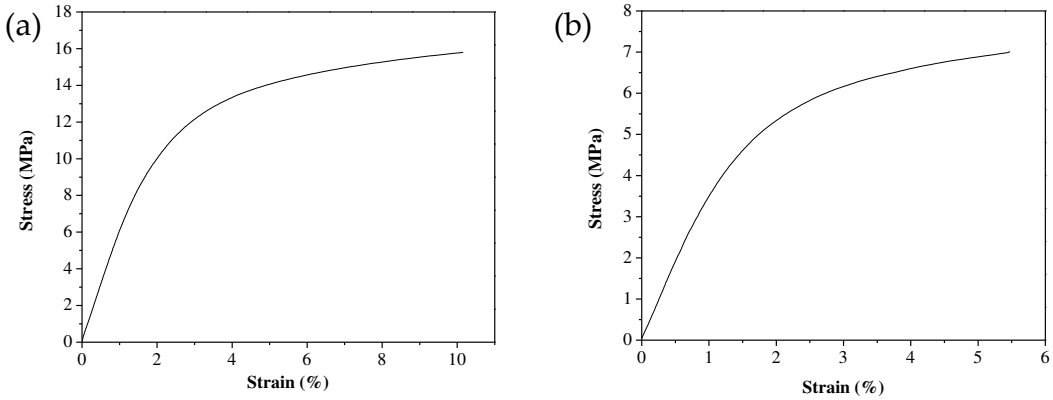

Figure 4. Stress-strain curve of (a) MWCNT/PVA (1:5) and (b) GO/PVA (1:5) nanocomposites fiber filament

3.4 Optical microscopy, nanocomposites fiber diameter measurement and conductivity measurement

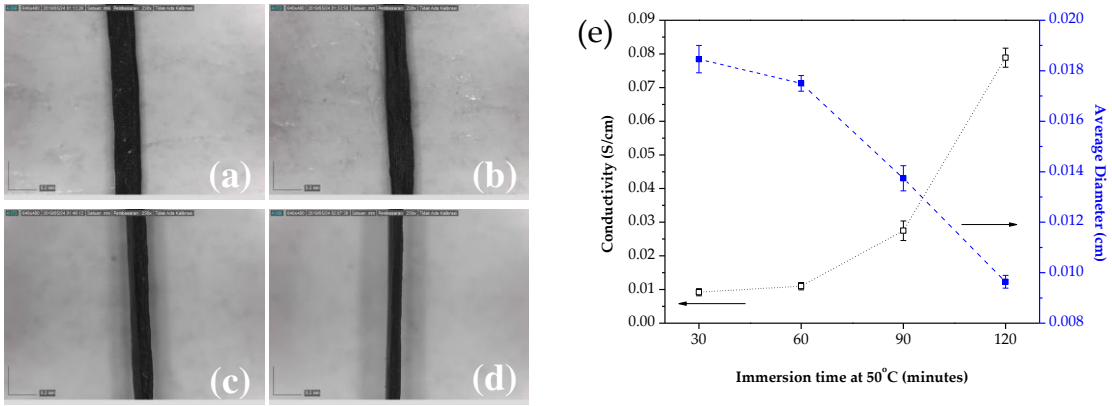

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Figure 5. Optical images of MWCNT/PVA (1:5) nanocomposites fiber with immersion 50°C with the immersion time of 30 (a), 60 (b), 90 (c), and 120 (d) min, and their average diameter and conductivity values (e)

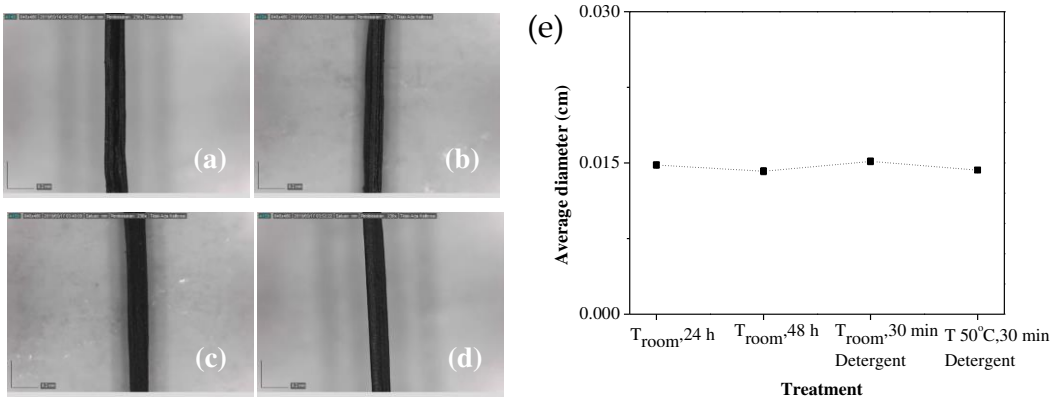

Figure 6. Optical images of MWCNT/PVA (1:5) nanocomposites fiber with immersion at room temperature for 24 h (a) and 48 h (b) and with addition of commercial detergent at room temperature (c) and at 50°C (d) and their average diameter (e)

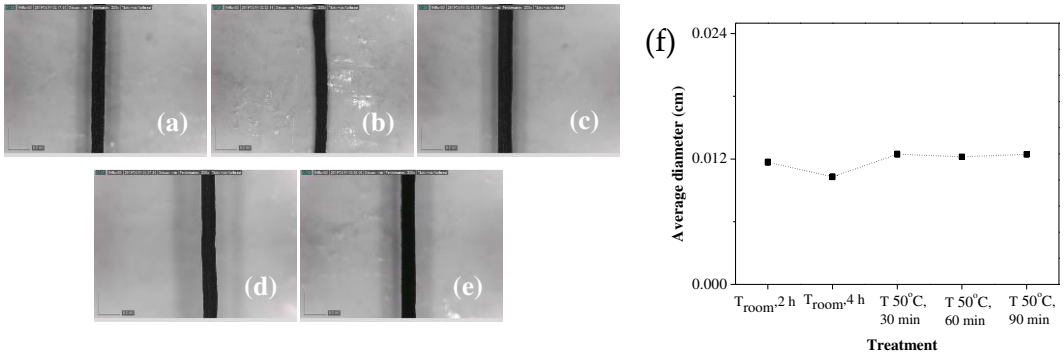

Figure 7. Optical images of GO/PVA (1:5) nanocomposites fiber with immersion at room temperature for 2 h (a) and 4 h (b) and at 50°C with immersion time of 30 (c), 60 (d), and 90 (e) minutes and their average diameters (f)

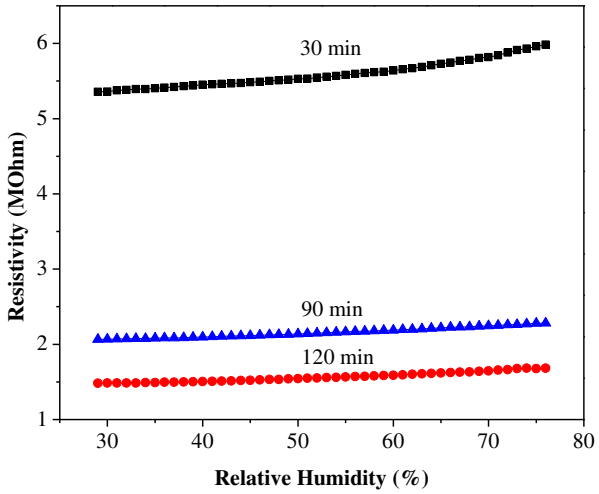

Figure 8. Resistivity of MWCNT/PVA (1:5) nanocomposites fiber as a function of immersion time

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4. Discussion

4.1 Wet Spinning of MWCNT/PVA and GO/PVA Nanocomposites Fibers

Herein, wet spinning process was used to develop bulk nanocomposites fiber containing polymeric material and carbon-based material as matrix and filler, respectively. Morphology transformation was generated during the coagulation process in which spinning dope solution was transformed into bulk MWCNT/PVA or GO/PVA nanocomposites fiber on acetone coagulation bath. Immersion of nanocomposites fibers in water was conducted in order to remove any un-reacted surfactant and impurities, followed by drying at room temperature. Their influence to fiber morphology and electrical properties were investigated through OM observation and resistivity measurement, respectively. The result shows that the morphology and electrical properties of nanocomposites fiber were influenced by the immersion or washing process of nanocomposites fiber in water system and drying process. Furthermore, SEM observation were conducted to evaluate the structure and morphology of pristine nanocomposites fiber. Figure 3 shows selected SEM images of MWCNT/PVA (1:5) and GO/PVA (1:5) nanocomposites fiber. According to the SEM observation, the diameter of MWCNT/PVA (Figure 3a) was slightly larger than GO/PVA (Figure 3e) nanocomposites fiber. The nanocomposites fiber surface was contoured due to the effect functionalized MWCNT (Figure 3b) or GO sheets (Figure 3f) on the nanocomposites fiber. Cross section SEM images revealed the connection between filler MWCNT (Figure 3c) or GO (Figure 3g) on the PVA matrix. The connection of MWCNT and GO on the nanocomposites fiber appears tube-like ((Figure 3d) and sheets-like connection (Figure 3h), respectively. Moreover, the arrangement of MWCNT and GO on the nanocomposites fiber will affect the conductivity and mechanical properties of the nanocomposites fiber.

4.2 Mechanical Properties

Single filament tensile test (DMA Q 800 V20.26) was conducted to evaluate the mechanical properties of the nanocomposites fiber. Typical strain-stress curve of nanocomposites MWCNT/PVA (1:5) and GO/PVA (1:5) nanocomposites fiber are shown in Figure 4. The maximum strain-to-failure for MWCNT/PVA (1:5) and GO/PVA (1:5) nanocomposites fiber was about 10% and 5.5%, respectively. The yield strength of MWCNT/PVA (1:5) and GO/PVA (1:5) nanocomposites fiber was around 11 and 4.5 MPa, respectively. Based on the single filament tensile test, MWCNT/PVA (1:5) exhibits higher tensile strength and elongation in compare with the GO/PVA (1:5). This might be due to the characteristics of MWCNT that strengthen the nanocomposites fiber along with the tensile direction. Moreover, the dispersion homogeneity of MWCNT on the PVA matrix plays an important role in term of efficient stress-transfer between MWCNT as a filler and PVA as a matrix [11].

4.3 Morphology and Electrical Properties of MWCNT/PVA and GO/PVA Nanocomposites Fibers

Figure 5 shows the OM images of the MWCNT/PVA (1:5) nanocomposites fiber with variation of immersion process at 50^{o}C for 30, 60, 90, and 120 minutes. After immersion the fiber was dried to remove any water. Nanocomposites fiber spun with MWCNT loading exhibits electrical conductivity properties. Figure 5 shows the average diameter and conductivity value of MWCNT/PVA (1:5) nanocomposites fiber against immersion time in water at 50^{o}C. Increasing the immersion time to 120

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minutes will enhance the conductivity value of MWCNT/PVA (1:5) nanocomposites fiber and decrease the average diameter of nanocomposites fiber. This might happen because during the immersion time the unreacted surfactant will be removed [6], thus decrease the average diameter of nanocomposites fiber and lower the resistivity barrier between MWCNT which then enhance the conductivity value of nanocomposites fiber.

In this study, immersion of MWCNT/PVA (1:5) was also conducted at various parameter such as water immersion at room temperature for 24 h and 48 h and also immersion at commercial detergent at room temperature and at 50^{o}C (Figure 6) with the average diameter results of 0.0148, 0.0141, 0.0151 and 0.0143 cm respectively. Moreover, this MWCNT/PVA (1:5) nanocomposites fiber still maintains the fiber after being washed in water and drying. Therefore, it is anticipated that the nanocomposites fiber can be developed as washable fiber or textiles.

On the other side, with the same process as the aforementioned method, the diameter of GO/PVA nanocomposites diameter was 0.0117, 0.0103, 0.0124, 0.0122, and 0.0124 cm for immersion at room temperature for 2h and 4 h and immersion at 50^{o}C for 30, 60, and 90 minutes, respectively (Figure 7).

4.4 Sensing Characteristic

Herein, preliminary research about sensing characteristics of the selected nanocomposites fiber was conducted for MWCNT/PVA (1:5) nanocomposites fiber with immersion time of 30, 90, and 120 minute. Figure 8 shows the relative humidity against resistivity value of MWCNT/PVA (1:5) nanocomposites fiber. Increasing immersion time decrease the measured resistivity. The measured resistivity at the relative humidity of 74% (for saturated sodium chloride at 20^{o}C) was 5.93 ± 0.17, 2.27 ± 0.27 and 1.68 ± 0.62 M ohm for the nanocomposites fiber with immersion time of 30, 90, and 120 minutes, respectively. These characteristics was in accordance with the conductivity test in which the MWCNT/PVA (1:5) nanocomposites fiber with the immersion time of 120 minutes presents in the highest conductivity.

5. Conclusions

We have developed nanocomposites fiber based on the polymeric material PVA with the incorporation of carbon-based material including MWCNT or GO. The conductivity value of MWCNT/PVA was about 10^{-2} S/cm, and increasing the immersion time enhance the conductivity. The presence of conductive properties can be developed for the e-textile or textile-based sensing. After washing with commercial detergent, the nanocomposites fiber still maintains its morphology which represented their washability. According to the results, this nanocomposites fiber are anticipated to be developed for smart textiles or smart fabric.

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Conflicts of Interest: The authors declare no conflict of interest

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