

Electrical Conductivity of Vapor-Grown Carbon Nanofiber/Polyester Textile-Based Composites

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ABSTRACT: The main objective of this study was to investigate the capability of vapor-grown carbon nanofibers (VGCNFs) to improve the electrical conductivity of textile-based composites. A combination of mechanical stirring and ultrasonication was used to disperse VGCNFs at various weight fractions (2, 4, 6, 8, and 10 wt %). Textile-based composites were fabricated with a hand-layup method with the application of three fabric types, including carbon, Kevlar, and polyester fabrics. The electrical conductivity of the samples was measured with a four-point probe method, and morphological analysis was performed with field emission scanning electron microscopy. The electrical conductivity of the composite samples was investigated from the standpoint of the VGCNFs' weight fraction, fabric type, cure process temperature, and sonication time. We found that with increasing VGCNF weight fraction, the conductivity increased. Also, the optimum conductivity was obtained at a sonication time of about 2 h. A higher conductivity was observed in the carbon fabric-based composites than in the Kevlar- and polyester-fabric-based composites. Nevertheless, there was no significant difference among the electrical conductivities of the VGCNF/polyester-textile-based composites prepared at room temperature and 60°C. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2013

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INTRODUCTION

In recent years, conductive vapor-grown carbon nanofiber (VGCNF)/polymer nanocomposites have been widely considered among scientists because of their exceptional multifunctional properties compared to conventional conductive polymer composites.^{1,2} Thus, the use of VGCNFs could lead to significant improvements in the electrical, mechanical, and thermal properties of composites.

VGCNFs are produced by a vapor-deposition process. Their morphology is similar to that of multiwalled carbon nanotubes (CNTs), where hollow-core nanofibers contain a single or double layer of graphite planes stacked parallel or at a certain angle to the fiber axis. This kind of nanofiber can be prepared with diameters from 15 to 200 nm and with lengths of a few tens of micrometers; this results in high aspect ratios.^{3–5} VGCNFs are distinguished by an extraordinarily high tensile modulus, tensile strength, and electrical and thermal conductivity. Because of their high electrical conductivity, they are suitable fillers for the development of new conductive composites with many applications in the nanoelectronics field transistors, autoelectron emitters, diodes, supercapacitors, sensors, electromagnetic shielding materials, and electrostatically paintable materials.^{6–10} In particular, most studies have concentrated on epoxy and others

polymers, such as polypropylene, nylon, and vinyl ester systems,^{11–14} and less attention has been focused on the electrical conductivity of VGCNF/polyester textile nanocomposites.

Unsaturated polyester resins (UPRs) are one of the most important thermoset resins and are used extensively in the composite industry because of their good mechanical properties, ease of processing, low density, and low cost.^{15,16} They are useful for applications such as water pipes, chemical containers, buildings construction, electrical appliances, and automotive.^{17,18} The use of VGCNFs, CNTs, and carbon black as conductive nanofillers could promote the electrical conductivity of UPR composites. Although most studies have been concerned with CNT/UPR composites, few studies have been reported on the electrical properties of nanocomposite-based UPR and VGCNFs. Torre et al.¹⁹ employed a calendering process to disperse carbon nanofibers in a UPR. Their investigation on the electrical conductivity of the nanocomposites at different CNF concentrations revealed that the electrical percolation threshold was obtained around 0.3 wt %, where the electrical conductivity switched from 10^{-13} to 10^{-7} S/cm. Natsuki et al.²⁰ described the temperature dependence of electrical resistivity for CNF/UPR nanocomposites prepared by a solvent evaporation method. They found that the CNF/UPR nanocomposites exhibited quite a low

electrical percolation threshold because of a large aspect ratio of CNFs and its dispersion in the UPR matrix. The influence of the CNF content on the electrical resistivity was investigated in detail as a function of the temperature. The nanocomposites showed a positive temperature coefficient effect for the resistivity and a strong temperature dependence near the percolation threshold.

In the last few years, the electrical characteristics of textile structures, such as fibers, yarns, and fabrics, have caught the attention of many researchers to increase the applications of these structures in electronic devices. Textile substrates such as fabric-based networks provide opportunities to improve the efficiency of sensors, batteries, solar cells, and other pieces of electronic equipment. Naturally, most fabrics have a low electrical conductivity or have the characteristics of insulator materials. Accordingly, to use fabrics as a part of electronic devices, their conductivity should be promoted. Many research works have been performed on the development of textile structures with proper electrical conductivity properties with conductive polymers such as polypyrrol^{21–25} and polyaniline^{26–32} or coatings with metals and metallic salts. In a previous study, Joshi and Bhattacharyya³³ used carbon nanofibers as a reinforcement in carbon fabric/polymer composites, and then, the thermal conductivity and mechanical properties of the produced composites were investigated. However, no effort has been reported on the effect of conductive polymer nanocomposites (VGCNF/polyester) on the improvement of the electrical conductivity of textile-based composites. Thus, we decided to use conductive polymer nanocomposites (VGCNF/polyester) at a lower percolation threshold value along with textile carbon, polyester, and Kevlar fabrics to enhance the electrical conductivity of textile-based composites as a new class of conductive fabric-based composite materials.

We conducted this study to characterize the efficiency of VGCNF/polyester nanocomposites to promote the electrical conductivity of textile-based composites. With this aim, a combination of mechanical stirring and ultrasonication was used to prepare nanocomposites. The dispersion level for various VGCNF weight fractions was analyzed with field emission scanning electron microscopy (FESEM). Finally, the electrical conductivity of supported fabrics with the VGCNF/polyester nanocomposite was studied in terms of the VGCNF weight fraction, fabric type, cure process temperature, and sonication time.

EXPERIMENTAL

Materials

The VGCNF material was purchased from Carbon Nanomaterial Technology Co. The VGCNF properties are listed in Table I, and their morphology is shown in Figure 1. The polyester resin was a low-viscosity orthophthalic UPR supplied by Boytek (Turkey) with the commercial name BRE 300. Methyl ethyl ketone peroxide as an initiator and cobalt octoate as an accelerator were used in this study. Three types of textile fabrics, including carbon, Kevlar, and polyester fabrics (PET), from Sazeh Morakab Co. were selected to use in the textile composite structures. Today, carbon fabrics are commonly used to promote the

Table I. Properties of the VGCNFs Used in This Study

Property	VGCNFs
Diameter (nm)	<200
Length (μm)	20
Density (g/cm^3)	2.0
Aspect ratio	100
Purity (wt %)	90

electrical conductivity of textile composite materials, particularly for lightweight structures. Polyester fabric as an isolated material was used in this work, however, to control and compare the electrical conductivity results. In addition, Kevlar fabrics were also used in the textile composite structure to enhance the thermal conductivity and the impact properties of the composite structures. The physical properties of these fabrics are listed in Table II.

Nanocomposite Preparation Procedure

The nanocomposite preparation procedure is shown in Figure 2. For production of the VGCNF/polyester nanocomposites, different weight fractions of the VGCNFs (2, 4, 6, 8, and 10 wt %) were mixed with polyester resin with the mechanical stirrer at 1600 rpm for 10 min, and this was followed by a dispersion process for 2 h with sonication. This process was carried out with an ultrasonic apparatus (Hielscher, Germany). To prevent a temperature increase, the sonication process was carried out in an ice bath and was stopped at regular intervals (every 10 min). Consequently, the temperature during the dispersion process was held at less than 40°C. In the next step, the mixture was placed at room temperature for about 30 min to remove air voids. Afterward, the accelerator and initiator were added to the mixture, respectively, and then, it is mixed for 10 min with the mechanical stirrer. In the last step, the mixture was aged at room temperature until the gelling reaction started.

Formation of Textile-Based Composites

The composite formation procedure is depicted schematically in Figure 3. We used two glass plates to give adequate pressure to nanocomposite materials (VGCNFs) in the fabric and hence to spread the VGCNFs into the fibers as well. In this method, a



Figure 1. FESEM micrograph of the VGCNFs.

Table II. Characterization of the Fabrics Used

Fabric	Mass per unit area (g/m ²)	Thickness (mm)	Fabric type	Courses (cm)	Wales (cm)	Number of weft threads (cm)	Number of warp threads (cm)
Polyester	140	0.10	Knitted (type of crepe)	9	9	—	—
Kevlar	75	0.11	Plain (JSWK 0201P)	—	—	15.94	15.94
Carbon	200	0.22	Plain (YC-3K200)	—	—	5.31	4.92

very thin layer of resin (nanocomposite) was placed over the fabric surface, and the nanocomposite materials (VGCNFs) gradually penetrated into the fibers because of induced pressure. However, in other methods, such as the dipping of fabric into the nanocomposite, a dense resin layer is placed on the fabric that creates a barrier to conductivity.

Next, the composite samples were fabricated on the basis of a hand-layup method. First, one layer of the nanocomposite was poured onto the bottom glass plate, and then, the fabric was mounted on the nanocomposite layer. In the next step, another nanocomposite layer was spread onto the fabric, and the top glass plate was put on this structure. Finally, the samples were put in a vacuum oven to be cured at 60°C under pressure with

a dead weight for 8 h. This process was done very fast because when the gelling reaction began, the nanocomposite would solidify within few seconds, and after that, the spread of the nanocomposite layer and its penetration in to the fabric would be impossible.

Characterization

The morphology of the nanocomposites and the VGCNFs was evaluated by FESEM (S-4160, Hitachi, Japan). A four-point probe was used to measure the resistivity of the samples at room temperature and under ambient conditions (at 24°C and at a relative humidity of 64%). The electrical current was measured with a Keithley 224 electrometer, and measurements of the voltage were performed with a Keithley 196 instrument.

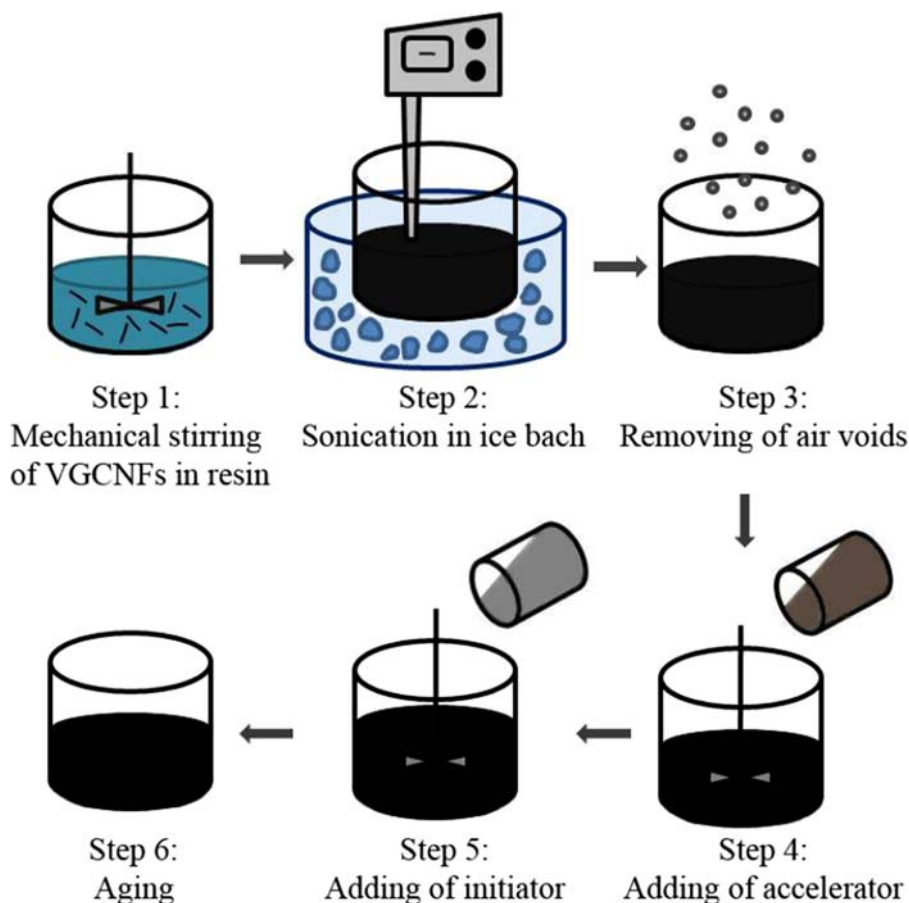


Figure 2. Schematic of the VGCNF/polyester nanocomposite fabrication procedure. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

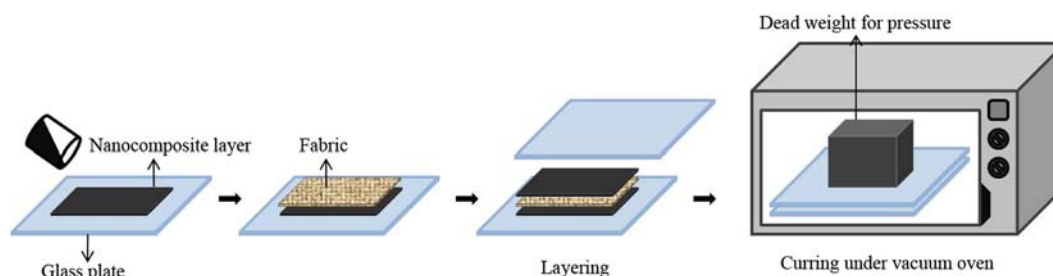


Figure 3. Schematic of the textile-based composite fabrication. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

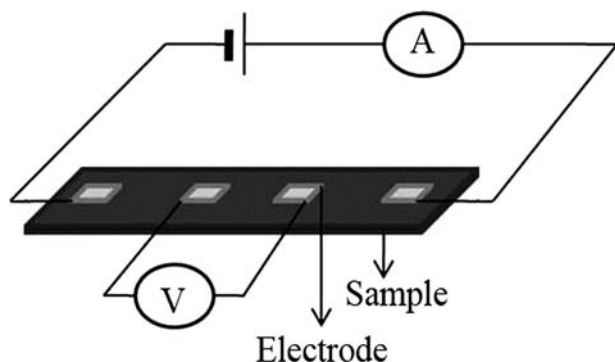


Figure 4. Schematic of the electrical circuit for the measurement of resistivity.

Figure 4 illustrates a schematic electrical circuit for the measurement of resistivity. The electrical conductivity (σ) could be calculated with eq. (1) on basis of the sample resistivity:

$$\sigma = \frac{1}{\rho} = \frac{d}{RS} \quad (1)$$

where R is the electrical resistivity (Ω), d is distance between the two electrodes (cm), and S is the cross-sectional area perpendicular to the current direction in the specimen (cm^2).³⁴ Each sample was measured three times, and average values are reported.

RESULTS AND DISCUSSION

Electrical Conductivity

VGCNF Content and Fabric Type Effects. The electrical conductivity and electrical resistivity of the nanocomposite and

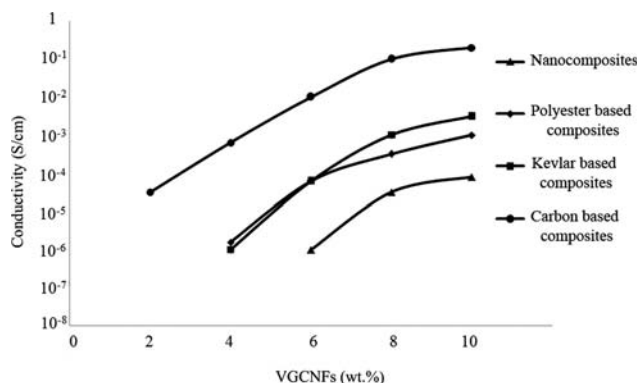


Figure 5. Electrical conductivity at various weight fractions of the VGCNFs.

textile-based composites at various weight fractions of VGCNFs are shown in Figure 5 and Table III, respectively. As shown, as compared to the pure polyester resin (0 wt % VGCNFs), the nanocomposite had more conductivity at all contents of VGCNFs. With increasing VGCNF concentration up to 4 wt %, the conductivity was enhanced. On the other hand, samples containing the fabrics, particularly in case of the carbon fabric, exhibited a higher conductivity compared to that of the nanocomposite sample. These results were attributed to the material transfer from the isolative condition into the conductive condition with the VGCNFs. With the addition of the carbon nanofibers at a critical percentage, which is known as the *percolation threshold*, a conductive path was formed in the resin, which caused the material to shift from an isolating state to a conducting state.^{12,35–38} In fact, nanofibers are conductive fillers

Table III. Electrical Resistivity at Various Weight Fractions of the VGCNFs

VGCNFs (wt %)	VGCNF/polyester nanocomposites	Polyester-fabric-based composites	Kevlar-fabric-based composites	Carbon-fabric-based composites
0	—	—	—	—
2	—	—	—	0.04×10^5
4	—	0.25×10^6	0.25×10^6	0.06×10^4
6	0.25×10^6	0.12×10^5	0.08×10^5	0.12×10^3
8	0.03×10^5	0.05×10^4	0.12×10^4	0.08×10^1
10	0.08×10^5	0.12×10^4	0.03×10^3	0.06×10^1

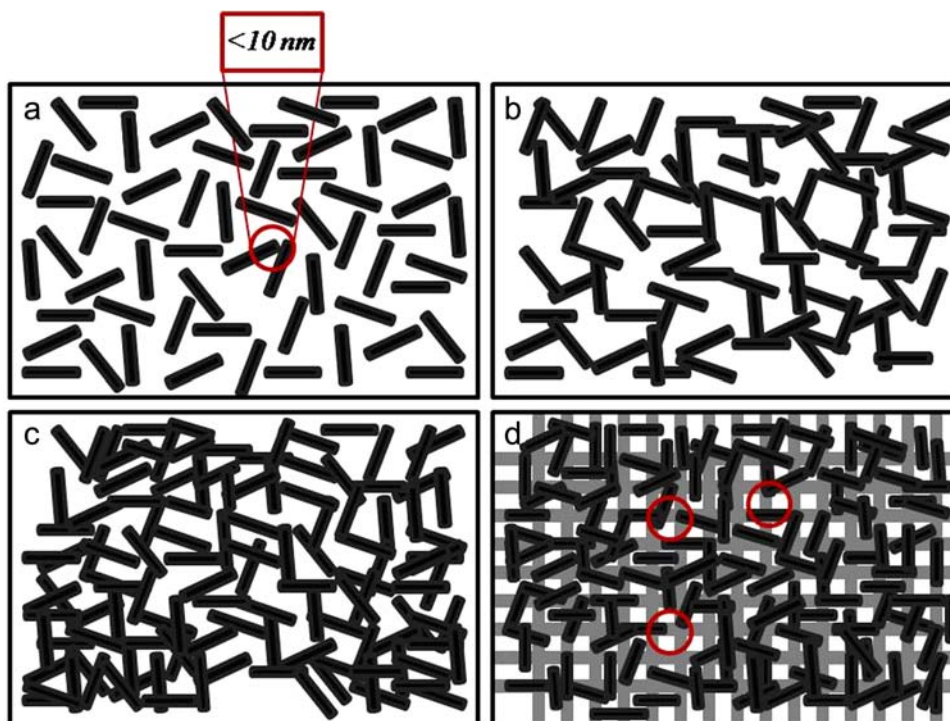


Figure 6. Schematic of the formation of the conductive network: (a) nanocomposite at percolation concentration, (b) nanocomposite after percolation concentration, (c) nanocomposite at a high percentage of VGCNFs, and (d) fabric containing nanocomposite. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

that contact each other and create a conductive network inside the resin. Usually, at the percolation concentration, the conductive network has not yet been formed, and the conductivity is due to tunneling between the fillers. Conduction through a tunneling mechanism occurs when the distance between the filler particles are lower than the hopping distances of the conducting electrons (<10 nm).^{35,39} With increasing filler content above the percolation threshold, the conductive network forms, and the conductivity increases.

As shown in Figure 5, at certain critical weight fractions of VGCNFs around 6 wt % in the nanocomposite sample, the conductivity began to increase rapidly. In fact, at this VGCNF concentration, a tunneling mechanism occurred, which is shown schematically in Figure 6(a). By increasing the weight fractions of the VGCNFs, the conductive network was formed [Figure 6(b)]; this led to a higher conductivity; the conductivity increased from about 10^{-6} to 10^{-5} S/cm as the VGCNF weight fraction increased from 6 to 8 wt %. However, a further increase in the VGCNF content (>8 wt %) had little effect on the nanocomposite electrical conductivity because of the formation of a more compact conductive network [Figure 6(c)]. To clarify the reason for the conductivity trend, we performed a morphological analysis with FESEM (Figure 7). As shown in these pictures, aggregations took place in the sample containing 10 wt % VGCNFs compared to the sample with 2 wt % VGCNFs. On the other hand, the carbon nanofibers were better distributed in samples with 2 wt % VGCNFs. It is quite conceivable that more contact points took place between the

nanofibers at higher weight fractions of VGCNFs, and also, the nanofibers could have been closer to each other. Furthermore, the formation of a conductive network did not necessarily need a very good distribution and dispersion because the nanofibers in a single-by-single position did not create an efficient conductive network. Hence, the occurrence of slight aggregations and also improper distribution at high VGCNF weight fractions could have diminished the space between the nanofibers or created direct contact between them and led to more conductivity.⁴²

In comparison with the nanocomposite, the samples reinforced with textile fabrics showed higher levels of conductivity. A possible explanation for this trend was the location of the VGCNFs on the fabric during the production process. During the composite fabrication process, the pressure insertion caused the nanocomposites penetrate into the fabric, and thus more volume fraction was available over the fabric surface [Figure 6(d)]. This structure not only created more direct connections between the nanofibers but also produced bridges of the fibers on the gaps between the VGCNFs. This event is shown in FESEM photographs in Figure 7. In other words, the presence of the fabric amended the foibles of the conductive network between the VGCNFs, and this resulted in an increase in the electrical conductivity.

A reduction in the percolation concentration was another noticeable benefit that could be achieved with textile fabrics in the nanocomposite structure. As illustrated in Figure 5, in the presence of the fabrics, a smaller percentage of VGCNFs (ca. 2

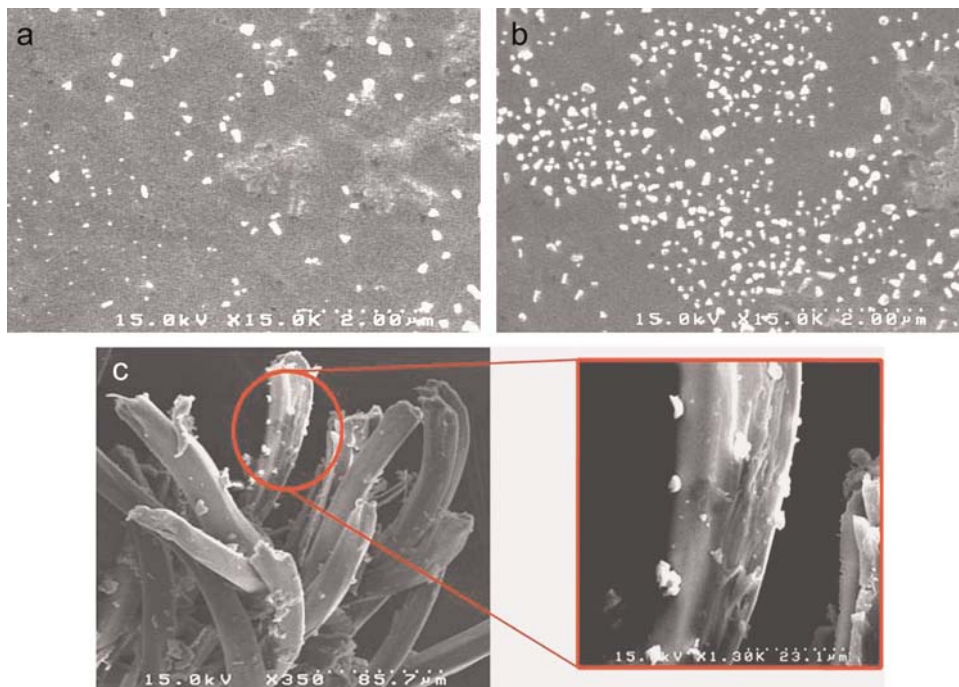


Figure 7. FESEM micrographs of the (a) polyester resin with 2 wt % VGCNFs, (b) polyester resin with 10 wt % VGCNFs, and (c) nanofibers on the carbon fabric fibers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

wt % in the sample containing carbon fabric and 4 wt % in the samples containing the Kevlar and polyester fabrics) was necessary to increase the electrical conductivity and thus caused the specimens to change from the insulated phase into the semiconductive or conductive condition. This decline in the percolation concentration was valuable for making the nanocomposites economically practicable because VGCNFs are very expensive. The higher electrical conductivity of the sample containing carbon fabric compared to those containing the two other fabrics is apparent in Figure 5. This was believed to be because of the inherent conductivity of the fabrics. The electrical conductivity

of the carbon fabric measured in this study was about 10^{-3} S/cm. However, in the case of the Kevlar and polyester fabrics, their electrical resistivity was high and could not be measured by the used apparatus.

Curing Temperature Effect. To investigate the effect of the curing temperature on the conductivity of the composites, the curing temperature was varied, and the conductivity was measured at room temperature. In the specimens including fabrics, there was no appreciable difference between the conductivity of the samples produced at 60°C and the samples prepared at room temperature. However, in the case of the nanocomposite, a significant decrease in the conductivity was found (Figure 8). This may have been due to the stability of the VGCNFs through the resin during the curing process. With regard to the long curing time (ca. 48 h) in the samples produced at room temperature,

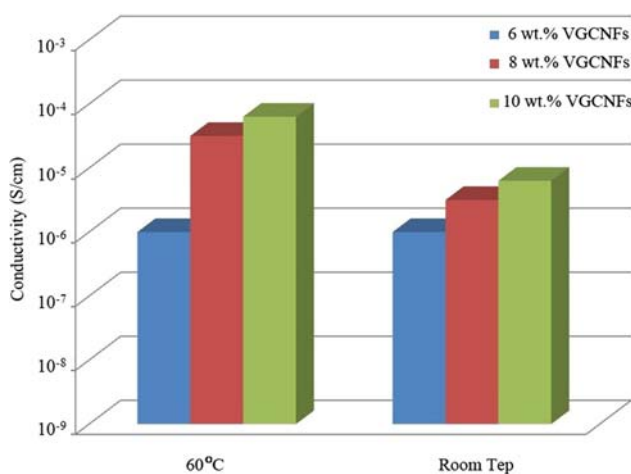


Figure 8. Comparison between the electrical conductivity of the samples cured at room temperature and 60°C . [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

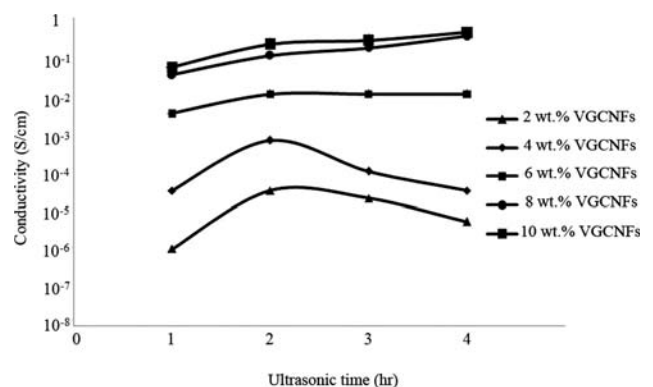


Figure 9. Electrical conductivity of the carbon fabric-based composites at various sonication times.

Table IV. Electrical Resistivity of the Carbon-Fabric-Based Composites at Various Sonication Times

Sonication time (h)	2 wt % VGCNFs	4 wt % VGCNFs	6 wt % VGCNFs	8 wt % VGCNFs	10 wt % VGCNFs
1	0.25×10^6	0.06×10^5	0.05×10^3	0.05×10^2	0.04×10^2
2	0.04×10^5	0.06×10^4	0.12×10^3	0.08×10^1	0.06×10^1
3	0.08×10^5	0.25×10^4	0.12×10^3	0.06×10^1	0.06×10^1
4	0.03×10^6	0.05×10^5	0.12×10^3	0.05×10^1	0.04×10^1

the VGCNFs had an adequate time to move before the resin became solidified. This movement led to inappropriate dispersion and destroyed the conductive network formed through the resin. However, in the samples fabricated at 60°C, the nanocomposite cured in a short time (ca. 8 h), and there was no sufficient time for the VGCNFs to move. Consequently, we deduced that the curing time had a prominent effect on the acquired electrical conductivity of the nanocomposite.⁴¹ In case of the samples containing fabrics, as mentioned before, the fabric acted like a substrate, and VGCNFs collected on the fiber surface became stable during this time; this means a similar conductivity could be achieved with variations in the curing time.

Dispersion Time Effect. To reach optimum dispersion conditions and to study their effect on the electrical conductivity, the sonication process was carried out for different periods of time. Figure 9 and Table IV show the electrical conductivity and electrical resistivity of the nanocomposite sample with carbon fabric prepared for various sonication times. As shown, at low weight fractions of the VGCNFs (2 and 4 wt %), the conductivity improved up to 2 h, and it diminished with increasing sonication time. At other weight fractions of the VGCNFs, the conductivity was improved.

Ultrasound sonication is a type of vibration that provides energy to the agglomerated nanofibers to escape from the surrounding resisting force. When there is not enough energy available to the mixture of the resin and the nanofibers, the agglomerates cannot escape. On the other hand, if too much energy is available to the agglomerations to move around, the frequency of collision between each single nanofiber will be

increased. Hence, the chances of each single nanofiber of tangling up and forming a larger nanofiber agglomeration would increase.⁴²

Therefore, as discussed previously, an optimum sonication time must be selected to obtain a good dispersion. In the presence of 2 and 4 wt % VGCNFs, the increase of the sonication time above 2 h presumably led to the formation of new agglomerations and, consequently, a lower conductivity. Furthermore, a reduction in the nanofiber length could be another drawback of using a longer sonication time. Figure 10 shows the FESEM micrographs of the nanocomposites at various sonication times. As illustrated, smaller nanofibers were achieved with increasing sonication time. Previous studies have also shown that the nanofiber length decreased with increasing sonication time.^{43,44} Shorter nanofibers have a lower chance of touching each other and ultimately result in a reduction in conductivity.

One other reason for the preference of a shorter sonication time is the resin performance. The resin gel times at the various sonication times are shown in Table V. It was obvious that with increasing sonication time, the gel times increased. This result may have been related to the destruction of the polymer molecular chain during the sonication process, as mentioned in a previous article by Shi et al.⁴⁵

Considering these results and the importance of a low percolation threshold in the VGCNF/polymer composites, we chose a 2-h period for the sonication process so that it was possible to produce VGCNF/polymer nanocomposites with an acceptable electrical conductivity.

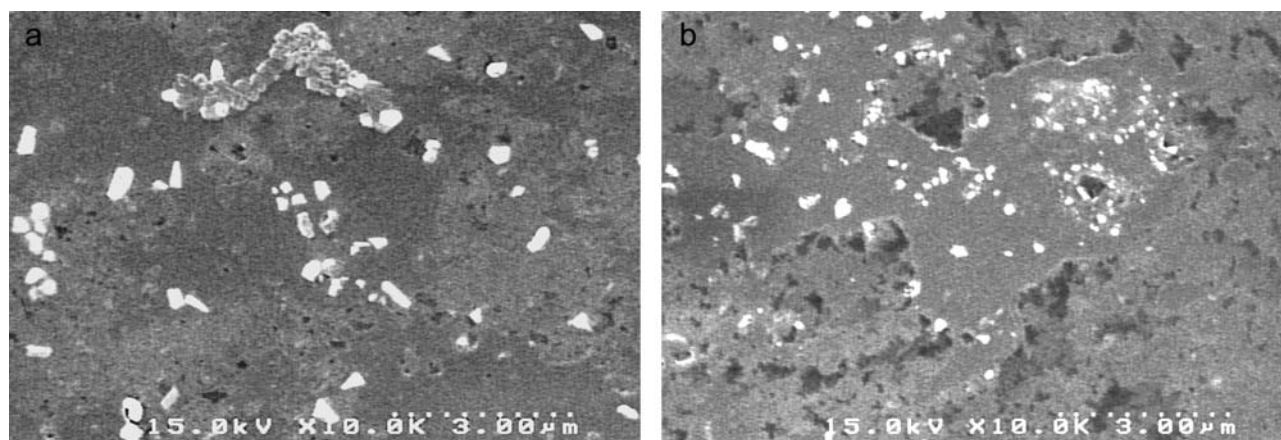


Figure 10. FESEM micrographs of the polyester resin with 4 wt % VGCNFs under (a) 1 and (b) 4 h of sonication. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table V. Alterations of the Resin Gel Time with Increasing Sonication Time

Ultrasonic time (h)	Gel time (min \pm 2 min) at various VGCNF contents				
	2 wt %	4 wt %	6 wt %	8 wt %	10 wt %
1	12	15	20	23	23
2	13	17	23	24	25
3	16	19	24	27	27
4	18	20	26	27	28

CONCLUSIONS

The electrical conductivity of VGCNF polyester-textile-based composites was investigated in this study. VGCNF/polyester nanocomposites were prepared with mechanical stirring and ultrasonication at various weight fractions of the VGCNFs. Textile-based composites were fabricated with a hand-layup method with the application of three types fabric, including carbon, Kevlar, and polyester fabrics. The electrical conductivity of the samples was measured with a four-point probe method. The morphology of the nanocomposites was studied with FESEM to interpret the improvement in conductivity. The effects of some parameters, such as the weight fraction of the VGCNFs, fabrics type, cure process temperature, and sonication time, on the electrical conductivity were considered. We concluded that the use of the VGCNF/polyester nanocomposites led to an improvement in the conductivity of the textile-based composites. Moreover, an increase in the VGCNF weight fraction resulted in a higher electrical conductivity. The sample containing carbon fabric showed a higher conductivity compared to the polyester and Kevlar fabrics. We found that at lower VGCNF values (i.e., 2 and 4 wt %), increasing the sonication time above 2 h decreased the electrical conductivity, but at a high VGCNFs concentration (i.e., up 4 wt %), an increase in the sonication time had not noticeable effect. The curing time had an obvious effect on the electrical conductivity of the VGCNF/polyester nanocomposites, but in the case of the textile-based composites, the same conductivity was acquired with the variation of the curing time. The results of this research suggest that the use of VGCNF/polymer nanocomposites as susceptible materials could create a promising method for achieving a noticeable improvement in the electrical properties of textile-based composites. Nevertheless, the effects of the VGCNF concentration on the mechanical properties of the VGCNF/polyester-textile-based composites is the subject of further research that will be reported in another article.

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