Fashion and Textiles

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Characterization of carbon nanofiber (CNF)/polymer composite coated on cotton fabrics prepared with various circuit patterns

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Abstract

Fabric heating elements with carbon nanofiber (CNF)/Poly(vinylidene fluoride-*co*-hexafluoropropylene) (PVDF-HFP) composite coated cotton fabrics were prepared with various circuit patterns with the aim of providing more flexible and uniform heating performance compared to conventional fabric heating elements. To investigate the properties of the fabric heating element according to the pattern condition, patterns consisting of 3, 5, and 7 horizontal lines, i.e., P3, P5 and P7, were respectively used; and subsequently, vertical lines were added to the horizontal lines, i.e., 2P3, 2P5 and 2P7, respectively. P0 was used as the referencesample. P0 showed a surface resistance of 1.2×10^3 Ω/sq at a current of 0.85 A and an electric heating temperature of 76.9 °C. P3 and 2P3 showed better electrical and electric heating properties than other samples, showing surface resistance values of 1.0×10^3 and 1.2×10^3 Ω/sq at the current values of 0.20 and 0.25 A, and surface temperatures of 71.8 and 75.7 °C, respectively. Although the currents applied to P3 and 2P3 were lower than that applied to P0, the electrical heating properties were modified to be similar. In terms of mechanical properties and water repellency, it was shown that the coated fabrics had higher values compared to the uncoated fabric. It was thus suggested that a small amount of CNF/PVDF-HFP composite can be used to manufacture an electric heating element with excellent performance.

Keywords: Carbon nanocomposite, Carbon nanofiber (CNF), Poly(vinylidene fluoride-*co*-hexafluoropropylene) (PVDF-HFP), Circuit patterns, Fabric heating element

Introduction

Recently, e-textiles have been receiving significant interest in terms of their applicability to wearable smart devices, because textiles are breathable, flexible and lightweight, yet strong and robust (Åkerfeldt et al. 2014; Asghar et al. 2016). Wearable smart devices incorporating e-textiles are used in healthcare, sports, military, wearable displays, bio-monitoring and power storage devices and so on. For these various applications, efforts are continuing to integrate functional nanomaterials with common textiles to provide flexible, lightweight materials and to improve electrical properties. The fabric heating element is one of the application fields of e-textiles used for the maintenance of body temperature such as work clothes used in extreme environments, protective clothing, military, immersion suit for winter season, etc. Preparation methods for heating elements include methods for making a fiber or fabric (Ilanchezhiyan et al. 2015; Park and Lee 2015; Roh and Kim 2016), forming a film (Jee et al. 2013; Park et al. 2015; Wang et al. 2015; Wang and Ruan 2016) and coating fabric (Kang and Lee 2015; Pahalagedara et al. 2017; Tian et al. 2016).

A number of studies on the preparation of electric heating elements using conductive nanocomposites have also been carried out. Such materials have electrical conductivity based on the types of the functional composites, like metal particles such as silver (Ag) (Stempien et al. 2016; Wang et al. 2016b), and copper (Cu) (Roh and Kim 2016; Wang and Ruan 2016), and conductive polymers such as polyaniline (Wang et al. 2015), and polypyrrole (Wang et al. 2016a). Research of conductive nanocomposites based on carbon nanomaterials has being actively pursued with the aim of improving flexibility and reducing weight. Carbon nanocomposites are high-performance carbon-based materials in which carbon fiber (Park and Lee 2015), graphite (Jee et al. 2013; Park et al. 2015), carbon nanotube (CNT) (Cipriano et al. 2008; Ilanchezhiyan et al. 2015; Jee et al. 2013), and carbon nanofiber (CNF) (Lee 2011, 2012; Das et al. 2012; Kang and Lee 2015) are charged in the polymer. CNF is a carbon-based material that has chemical stability, thermal stability and facilitates ease of handling like distribution. In addition, due to their availability in large quantities with a consistent quality, their cost is significantly lower than that of carbon nanotubes (Al-Saleh and Sundararaj 2009).

In a previous study, the electrical properties of nonwoven (Lee 2011) and silk fabric (Lee 2012) coated with CNF/PVDF-HFP composites were reported. This study found that the electrical properties of CNF/PVDF-HFP coated nonwoven fabrics increased with increasing CNFs content, and of silk fabric increased with 4% CNFs content. As well, Kang and Lee (2015) reported that in a preparation of carbon nanofiber composite-coated nylon fabric heating elements, surface temperatures of 16 wt% carbon nanofiber composite-coated fabrics reached 80 °C when 20 V was applied. When coating the entire surface, the coating solution was used in a large amount, but the problem of heating only in the localized region was confirmed. Therefore, the purpose of this study was to fabricate a fabric heating element coated with a carbon nanocomposite with various linear circuit patterns in order to resolve the economic and localized electric heating characteristics by using a smaller amount of carbon nanocomposite than the conventional planar heating element. To achieve this goal, we prepared a CNF/PVDF-HFP composite with electric heating properties, and the carbon nanocomposite was coated onto cotton fabric designed with two types of circuit patterns developed in our laboratory. The samples with various circuit patterns were analyzed by considering surface morphology, electrical property, electrical heating property, mechanical property, and water repellent property.

Methods

Materials

Carbon nanofiber (CNF) was obtained from PR-24-XT-LHT (Pyrograf III Carbon nanofiber, Pyrograf Products, Inc., USA). The Poly(vinylidene fluoride-*co*-hexafluoropropylene) (PVDF-HFP) chip was supplied by Solvay (SOLEF 21508, Solvay Co., Ltd.). The density was 1.78 g/cm^3. Acetone (1st grade, Junsei chemical Co., Ltd., Japan) was used as the solvent. The fabric used was cotton (Sombe Co., Ltd., Korea) with a thickness of 0.28 mm, and a weight of 117 g/m^2. The size of the prepared samples was 10 cm × 10 cm.

Preparation of CNF/PVDF-HFP composite solution

For the obtained CNF/PVDF composite solutions, a 15 wt% PVDF-HFP solution was prepared in acetone and the content of CNF in the PVDF-HFP solution was 16 wt%. The prepared CNF/PVDF composite solutions were stirred for over 1 week with a hotplate stirrer digital control (MSH-20D, Daihan Scientific, Korea), digital orbital shaker (SHO-1D, Daihan Scientific, Korea) and dispersed for 24 h at 25 °C using power sonic (Power sonic 410, HST, Co., Ltd., Korea).

Preparation of CNF/PVDF-HFP composite coated on cotton fabrics with various circuit patterns

Various conductive circuit patterns with CNF/PVDF composite coated on textiles were obtained through a knife-edge coating method (Kang and Lee 2015). Uncoated cotton fabric had a thickness of 0.28 mm, and the thickness of the coating with CNF/PVDF-HFP solution was 0.08 ± 0.01 mm. The total thickness of the coated parts of the linear heating element is 0.36 ± 0.01 mm. As shown in Fig. 1, six different circuit patterns with the same concentration of CNF/PVDF composite solution were designed to compare heating performances: (1) one with three parallel coated lines, (2) one with five parallel coated lines, (3) one with seven parallel coated lines, (4) two with three parallel coated lines, (5) two with five parallel coated lines, (6) two with seven parallel coated lines. The sample size was 100 mm × 100 mm, and the coated line size was 100 mm × 10 mm. Weight increases of each of the circuit patterns are summarized in Table 1. After the

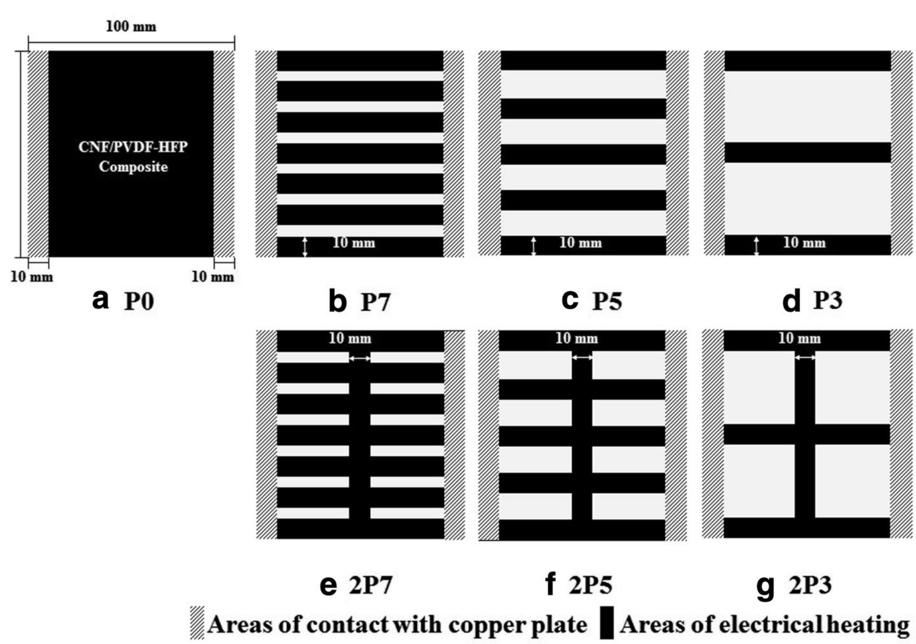

Fig. 1 Design of CNF/PVDF-HFP composite coated on cotton fabrics with various circuit patterns

Table 1 Characteristics of CNF/PVDF-HFP composite coated on cotton fabrics of various circuit patterns

Sample code	Weight increase (%)	Electrical properties		Tensile properties	
		Surface resistivity (Ω/sq)	Power-law exponent values (*n*)	Load (N)	Elongation (%)
Uncoated	–	$4.22 \times 10^{11} \pm 0.16$	–	7.33 ± 0.26	4.76 ± 2.83
P0	76.5	$1.15 \times 10^{3} \pm 0.41$	1.06	10.10 ± 0.15	13.48 ± 1.20
P7	54.6	$1.28 \times 10^{3} \pm 0.42$	1.09	8.31 ± 0.09	14.24 ± 1.44
P5	48.1	$1.36 \times 10^{3} \pm 0.19$	1.08	8.25 ± 0.40	14.08 ± 0.82
P3	37.2	$1.01 \times 10^{3} \pm 0.27$	1.08	7.98 ± 0.15	13.48 ± 1.20
2P7	60.4	$1.21 \times 10^{3} \pm 0.47$	1.10	8.91 ± 0.33	15.28 ± 0.24
2P5	49.0	$1.65 \times 10^{3} \pm 0.46$	1.06	8.40 ± 0.18	15.08 ± 0.69
2P3	40.0	$1.17 \times 10^{3} \pm 0.73$	1.08	8.28 ± 0.58	14.86 ± 1.07

coating, samples were stored at room temperature for 24 h and maintained in a desiccator before characterization.

Characterization

Surface morphology of CNF/PVDF-HFP composite coated on textiles was analyzed using a fabric image analysis system (Nex measure Pro5 NTZ-6000, Bestec Vision Co., Ltd.) at 6.5 magnification and a field emission scanning electron microscope (FE-SEM, JSM-6700F, Joel, Japan) at 200, 5000 and 10,000 magnification. The CNF/PVDF-HFP composite coated on textiles was visualized after a platinum coating process.

The electrical properties of the CNF/PVDF-HFP composite coated on textiles were measured based on surface resistivity. The surface resistivity measurements were performed using a four-probe resistivity meter (MP-8111, TMA Co., Ltd., Korea). Five samples were tested and the measured results were averaged.

The electric heating property of the CNF/PVDF-HFP composite coated on textiles was analyzed in order to investigate the heating property when a voltage was applied. The electrical heating property was measured using a heating pad clamping device (Dong-Woo Int. Ltd., Korea) connected to a DC power supply (CPS-2450B, CHUNGPAEMT. Co., Ltd., Korea) at an applied voltage interval of 5 V from 0 V to 20 V under a thermal imaging camera (FLIR i5, FLIR Systems INC, USA). We measured the current generated when a voltage was applied to the sample. Five samples were tested and three randomly selected parts of the heating area of each sample were measured. The average was obtained and then the value was used.

The mechanical properties of the CNF/PVDF-HFP composite coated on textiles were measured with a constant rate of extension machine (Autograph AGS-500D, Shimadzu Co., Ltd., Japan). The tensile strength and elongation of the CNF/PVDF-HFP composite coated on textiles with various circuit patterns were measured. The sample size was 10 cm × 10 cm, the clamp length was set at 50 mm, and the test speed was 50 mm/min. Three specimens were tested to determine the mean values.

Contact angles (CA) were measured and analyzed to determine the water repellency of the CNF/PVDF-HFP composite coated on textiles. CAs were measured using a contact angle meter (Phoenix 300, SEO Co., Ltd., Korea). Liquid drops were placed on the CNF/

PVDF-HFP composite coated on textiles with a pipette, and the image of each drop was captured as quickly as possible after the liquid drops were deposited onto the sample surface. The average of nine measurements obtained from each sample was reported.

Results and discussion

Surface morphology of CNF/PVDF-HFP composite coated on cotton fabrics with various circuit patterns

The surface morphology of the CNF/PVDF-HFP composite coated on cotton fabric is shown in Fig. 2. At 6.5× magnification, we confirmed that the surface of samples formed

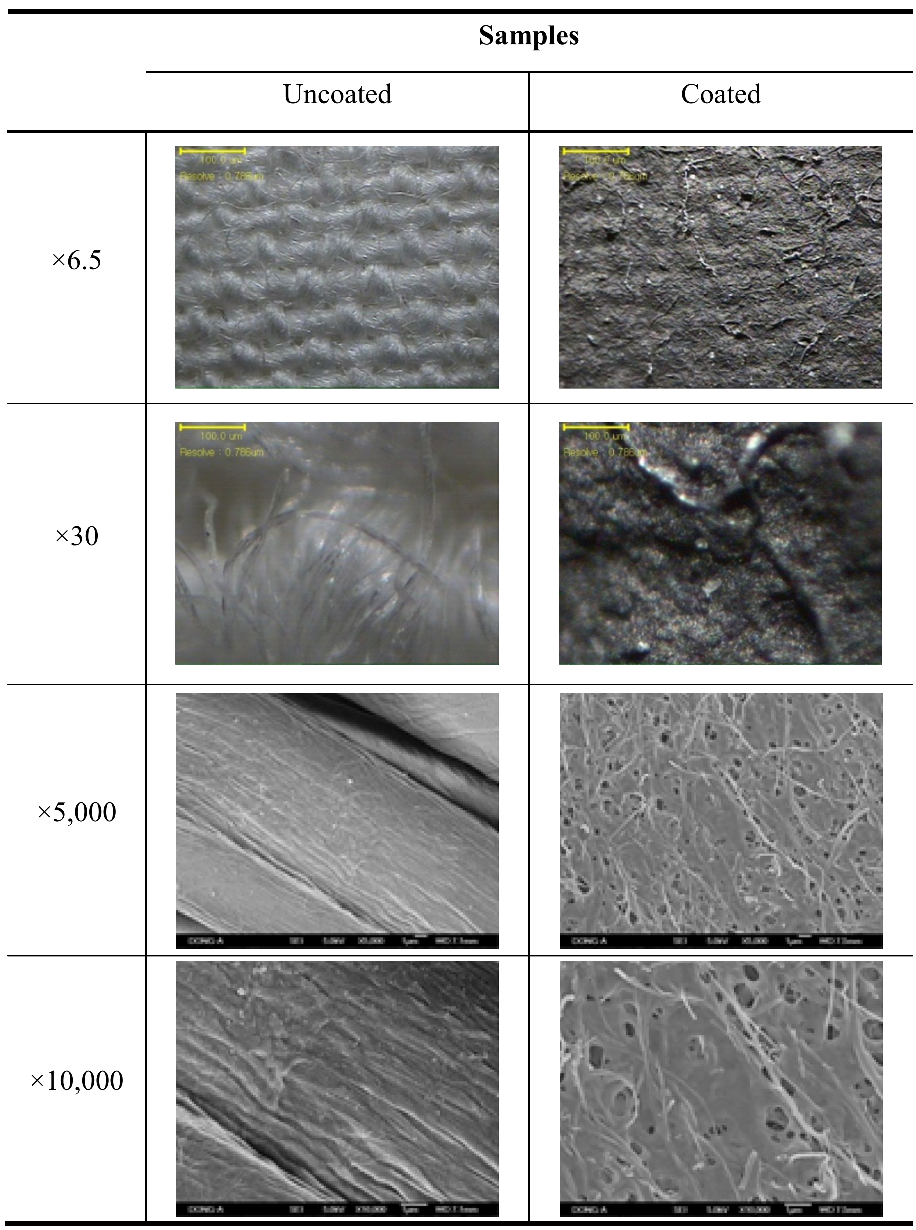

Fig. 2 Surface morphology of the CNF/PVDF-HFP composite coated on cotton fabrics

a CNF/PVDF-HFP membrane. At 30× magnification, the CNF bundles were observed, and on closer inspection, these may have been CNF. At the 5000× and 10,000× magnifications, the samples were confirmed to have a CNF/PVDF-HFP membrane with pores and CNF. The results indicated the presence of CNF/PVDF-HFP membrane and CNF on cotton fabric.

Electrical properties of CNF/PVDF-HFP composite coated on cotton fabrics with various circuit patterns

The surface resistivity values of CNF/PVDF-HFP composite coated on cotton fabric with various circuit patterns are presented in Table 1. In general, the characteristics and applications can be classified according to the surface resistance value of the material, with the resistivity value of 10^1–10^6 classified as conductive materials (Al-Saleh and Sundararaj 2009).

The surface resistivity value of P0 was $1.15 \times 10^3 \pm 0.41$ Ω/sq; the surface resistivity values of P7, P5, P3 were $1.28 \times 10^3 \pm 0.42$, $1.36 \times 10^3 \pm 0.19$ and $1.01 \times 10^3 \pm 0.27$ Ω/sq, respectively. The surface resistivity values of 2P7, 2P5 and 2P3 added vertical circuit lines were $1.21 \times 10^3 \pm 0.47$, $1.65 \times 10^3 \pm 0.46$ and $1.17 \times 10^3 \pm 0.73$ Ω/sq, respectively. The surface resistivity was 1.01×10^3 to 1.65×10^3 Ω/sq, and it was confirmed that the samples with various circuit patterns exhibit excellent electrical characteristics. In terms of the number of circuit lines, the resistances of P3 and 2P3 with three lines were lower than those of fabric heaters with 5 and 7 lines, and it is shown that the effect of the resistance is less affected by the decrease in the number. By pattern, P7, P5 and P3 showed slightly lower resistance than 2P7, 2P5 and 2P3, indicating that the line in the vertical direction slightly increased the resistance.

The current–voltage (I–V) characteristics of the CNF/PVDF-HFP composite coated on cotton fabric with various circuit patterns are presented in Fig. 3. Each sample showed a tendency to have an increased current as the applied voltage increases. The

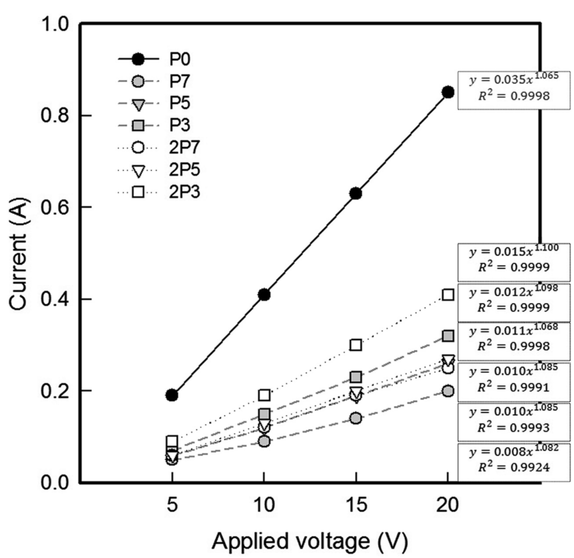

Fig. 3 Current–voltage characteristics of the CNF/PVDF-HFP composite coated on cotton fabrics with various circuit patterns

current increased with increasing horizontal circuit lines, and further increased when the vertical line was added.

I–V characteristics can be expressed as

$$I = KV^{n} \tag{1}$$

where K is a constant and n is the exponent, which is determined from the slope of the curve in the log–log scale. Ohm's law is fulfilled in the system at $n = 1$ (Choudhury 2011; Srivastava and Mehra 2008).

Linear *I–V* relationship at Ohm's law indicates that direct contact between the filler particles is the dominant conduction mechanism. However, the tunneling mechanism is the dominant mechanism for composites characterized by a power law *I–V* relation (Choudhury 2011). The nonlinear behavior in I–V curves has a value of $n > 1$. It represents nonlinear conduction due to tunneling of charge carriers (Srivastava and Mehra 2008).

In a sample coated with a concentration of 16 wt% CNF/PVDF-HFP composite solution, the sheet resistance value of samples with various circuit patterns was P3 < 2P3 < P5 < 2P5 < P7 < 2P7 < P0. The power-law exponent values of the CNF/PVDF-HFP composite coated on cotton fabric with various circuit patterns are indicated in Table 1. All the samples satisfy $n > 1$ and thus it is considered to follow tunneling conduction.

Electrical heating properties of CNF/PVDF-HFP composite coated on cotton fabrics with various circuit patterns

IR thermal images of the surface temperatures of samples with various circuit patterns are shown in Fig. 4. The surface temperature of each sample was measured after various voltages from 5 to 20 V were applied for 90 s. When a voltage of 5 V was applied, the temperature difference before and after the applied voltage showed a negligible value to ± 2.2 °C. The surface temperature of P0 was 38.4 ± 2.3, 56.9 ± 3.1 and 76.9 ± 6.4 °C with voltages of 10, 15, and 20 V applied, respectively. And the surface temperatures of P7, P5, P3 with 10 V voltage applied were 31.3 ± 1.6, 31.3 ± 1.5 and 33.4 ± 2.1 °C, respectively. At an applied voltage of 15 V, the surface temperatures of P7, P5, P3 were 41.7 ± 3.6, 41.7 ± 4.6 and 48.9 ± 5.2 °C, respectively. When 20 V was applied to P7, P5 and P3, they reached 60.6 ± 8.5, 58.4 ± 8.7 and 71.8 ± 9.0 °C, respectively. The P0 was the best in terms of the electric heating performance, but non-uniform heat distribution was shown as the result of the previous study (Kang and Lee 2015). However, IR thermal images of fabric heating elements with various circuit patterns coated with carbon nanocomposite showed that the heating temperature was lower than that of the P0, but the coating was more uniform.

The resultant surface temperatures of 2P7, 2P5 and 2P3 were 31.9 ± 3.7, 32.0 ± 5.1 and 35.2 ± 2.8 °C, respectively with an applied voltage of 10 V. At an applied voltage of 15 V, the surface temperatures of 2P7, 2P5 and 2P3 were 44.6 ± 9.2, 44.7 ± 7.2 and 46.9 ± 5.1 °C, respectively. The surface temperatures of 2P7, 2P5, 2P3 with an applied voltage of 20 V were 63.5 ± 9.1, 62.0 ± 9.1 and 75.7 ± 7.7 °C, respectively. The circuit with the added vertical line showed a similar trend and a slightly higher surface temperature than P7, P5, and P3. The surface resistances of 2P7, 2P5 and 2P3 were found to be slightly higher than those of P7, P5 and P3.

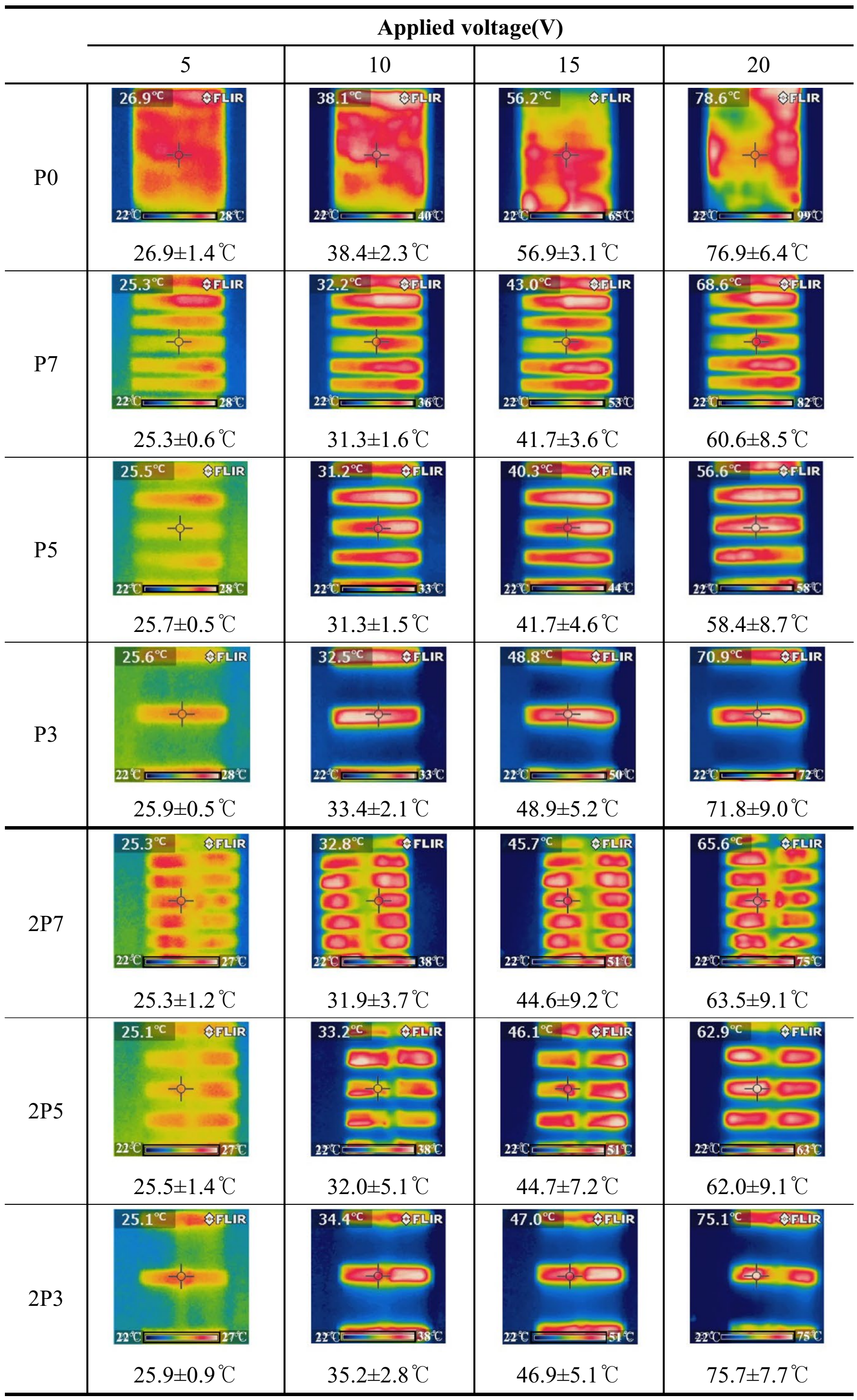

Fig. 4 IR thermal image of surface temperatures at various applied voltages of the CNF/PVDF-HFP composite coated on cotton fabrics with various circuit patterns

Kang and Lee (2015) reported on the electrical heating property of a fabric heating element with CNF nanocomposite coated on nylon fabric. A fabric heating element with carbon nanofiber composite was prepared in the size of 12 × 12 cm. The surface temperature indicated that the fabric heating element with 16 wt% CNFs/PVDF-HFP composite at 20 V was up to 89.1 °C, but fluctuations were present. Ilanchezhiyan et al. (2015) studied electrical heating properties with CNT coated cotton fabrics for flexible/wearable heating applications. CNT based cotton heaters were considered with short and long dimensions (1 × 2 and 2 × 4 cm). For the CNT based cotton heater with short dimensions, the steady-state temperature at 20 V reached 55.0 °C, and for the one with long dimensions it was up to 35 °C. In this study, when a voltage of 20 V was applied, the CNF/PVDF-HFP composite coated on a cotton fabric with various circuit patterns showed a surface temperature of more than 60 °C. In particular, P3 and 2P3 presented a uniform heating distribution of the surface temperature of 71.7 ± 9.0 and 75.7 ± 7.7 °C at an applied voltage of 20 V. Thus, it was confirmed that the CNF/PVDF-HFP composite coated on a cotton fabric with decreasing conductive lines exhibited an excellent electrical heating property, and the use of a smaller amount of the CNF/PVDF-HFP composite than that of the plane fabric heating element can help achieve a reduction in cost.

Joule's law (Jee et al. 2013) also states that the power of heating generated by an electrical conductor is proportional to the product of its resistance and the square of the current. Thus, the surface resistance value of the patterns with added vertical circuit lines was larger than that of the patterns with horizontal circuit lines, and the surface temperature of samples was improved due to the resistance heat generation.

The current-dependent temperature of CNF/PVDF-HFP composite coated cotton fabric with P0, P3, and 2P3 is indicated in Fig. 5. The R^2 values were 0.9664, 0.9524 and 0.9703 for the P0, P3 and 2P3, respectively. And the temperature-current characteristic indicated a tendency toward linear increases with increased current. As shown in Figs. 4 and 5, if the voltage was increased there was an increase in the current, and accordingly, there was an increase in the surface temperature. As per Joule's law, the power is

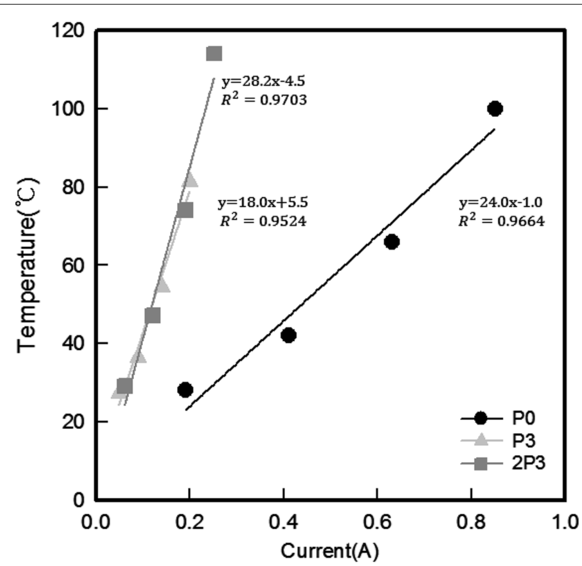

Fig. 5 Current-dependent temperature of CNF/PVDF-HFP composite coated cotton fabrics with various circuit patterns

proportional to the square of the current, so the surface temperature increases proportionally as the current increases.

Mechanical properties of CNF/PVDF-HFP composite coated cotton fabrics with various circuit patterns

The mechanical properties of CNF/PVDF-HFP composite-coated cotton fabric with various circuit patterns are indicated in Table 1. The tensile strength of uncoated cotton fabric was indicated as 7.33 ± 0.26 N, P0 was 10.10 ± 0.15 N. The results showed that the CNF/PVDF-HFP coated on cotton fabrics had higher tensile strength than the uncoated cotton fabric. Comparing the various patterns, the tensile strengths of P7, P5, and P3 were 8.31 ± 0.09, 8.25 ± 0.40, and 7.98 ± 0.39 N, respectively; for 2P7, 2P5, and 2P3 it was shown as 8.91 ± 0.33, 8.40 ± 0.18, and 8.28 ± 0.58 N, respectively. It is confirmed that the ultimate strength of carbon nanocomposite coated on cotton fabrics tends to be similar. The tensile strength of 2P7, 2P5, and 2P3 was slightly larger than that of P7, P5, and P3, indicating that the tensile strength increases as the number of lines coated with the CNF/PVDF-HFP composite increases. And the elongation of uncoated cotton fabric was 4.76 ± 2.83% and P0 was 13.48 ± 1.20%, which was about 3 times increased. Comparing it by pattern, P7, P5 and P3 are 14.24 ± 1.44, 14.08 ± 0.82 and 12.08 ± 1.24% respectively, and 2P7, 2P5 and 2P3 were 15.28 ± 0.24, 15.08 ± 0.69 and 14.86 ± 1.07%, respectively. The elongation tended to be better when CNF/PVDF-HFP was coated than for uncoated cotton fabric, and 2P7, 2P5, and 2P3 were more increased than that of P0.

Generally, when the tensile strength is large, the elongation shows a small value. It is reported that when conductive nanoparticles such as CNT and graphite are well-dispersed in polymer matrix, it can enhance the inherent mechanical properties of polymeric materials (Coleman et al. 2006). In this study, the tensile strength and elongation of patterns of added vertical circuit lines, which had a large area coated with conductive composite solution, showed a large value. Therefore, it was confirmed that the highest work of rupture was 2P7 and as the number of circuit pattern lines coated with a CNF/PVDF-HFP composite decreased, it became a more flexible fabric heating element than plane fabric heating elements.

Water repellent properties of CNF/PVDF-HFP composite coated cotton fabrics with various circuit patterns

Contact angle images of CNF/PVDF-HFP composite coated cotton fabrics with various patterns are shown in Fig. 6. To enhance the water repellent properties of carbon-based materials, studies have focused on increasing the surface roughness by introducing a geometric surface area (Meng and Park 2014). The contact angle value of the uncoated cotton fabric was indicated as 90.24° and the cotton fabric coated with conductive composite solution was presented 99.93°. This result shows that when coated with a conductive composite solution, contact angle is increased by about 10°. Therefore, this shows that the contact angle was improved because the CNF formed a rough surface and introduced a geometric surface area.

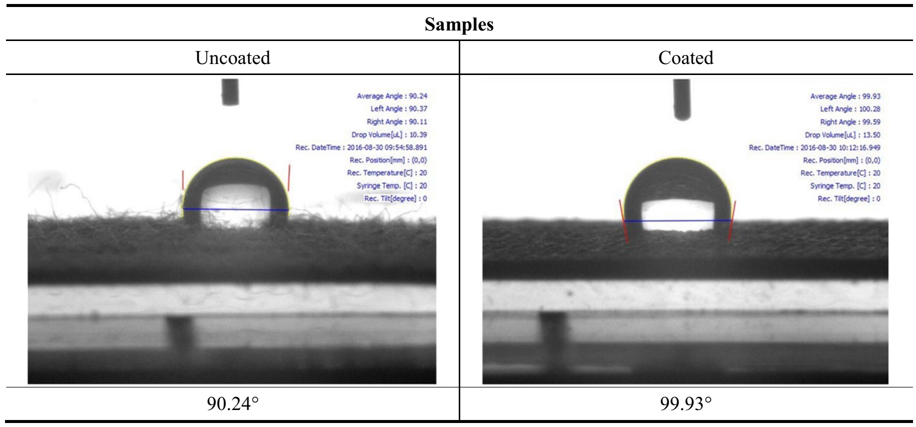

Fig. 6 Contact angle images of CNF/PVDF-HFP composite coated cotton fabrics

Conclusion

The purpose of this study was to develop fabric heating elements using CNF/PVDF-HFP composite coated cotton fabric with various circuit patterns with the aim of providing low cost, flexible, light, and uniform heating performance compared to conventional fabric heating elements. We prepared a carbon nanocomposite with electrical properties and it was coated cotton fabric with two types of circuit patterns. The sample code of P0 designates a reference sample, while P3, P5 and P7 were used when the number of horizontal lines was 3, 5, and 7, respectively; patterns in which a vertical line was added to the horizontal lines were named 2P3, 2P5, and 2P7, respectively. The surface resistivity values ranged from 1.01×10^3 to 1.65×10^3 Ω/sq, and it was thus confirmed that the samples with various circuit patterns exhibit excellent electrical characteristics. The resistances of P3 and 2P3 were lower than those of fabric heaters with 5 and 7 lines, and it is thus presented that the effect of the resistance is less affected by the decrease in the number of lines. Comparing patterns, P7, P5 and P3 showed slightly lower resistances than 2P7, 2P5 and 2P3, indicating that the line in the vertical direction slightly increased the resistance. For electrical heating temperature, P7, P5 and P3 showed more uniform heating performance than P0 and the circuit with the added vertical line had a higher surface temperature than P7, P5, and P3 in terms of joule heating. 2P3 had the best heating performance, and the surface temperature was similar to P0 at about 76 °C. In terms of mechanical properties and water repellency, it was confirmed that the coated fabrics had higher values compared to the uncoated fabric. Therefore, we confirmed that the electrical heating performance of the fabric heating element coated with carbon nanocomposite in three lines is improved compared to that of the plane fabric heating element, and it is also confirmed that it is more economical, flexible and lightweight than the conventional products because of its use of a smaller amount of carbon nanocomposite. Thus, it is suitable for use as a functional material for electric heating. Fabric heating elements that employ a CNF/PVDF-HFP composite with various circuit patterns have potential applicability in extreme work environments, for active winter sports, cold military training, and wet suits that must maintain body temperature in water.

Authors' contributions
SL conceived the work and HK prepared the samples and performed the experiments. HK and SL are participated in the sequence alignment and drafted the manuscript. Both authors read and approved the final manuscript.

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Acknowledgements
This research was part of a master's thesis. This research was supported by Basic Science Research Program through the National Research of Korea (NRF) funded by the Ministry of Education, Science and Technology (No. NRF-2014R1A1A2057445).

Competing interests
The authors declare that they have no competing interests.

Ethics approval and consent to participate
Not applicable.

Received: 17 May 2017 Accepted: 16 November 2017
Published online: 28 March 2018

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