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Thermal Effects of Microwave Reduced-Graphene-Oxide Coated Polyester Fabric on a Simulated Human Skin in Cool and Neutral Air Temperatures

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Abstract: Batteryless wearable technology has wide applications. In particular, human body surface temperature controlling fabrics can help regulate skin temperature in heat or cold. This study investigated surface temperature distribution of the fabrics coated with reduced graphene oxide (rGO) on simulated human body skin conditions at 18 °C (cool) and 27 °C (neutral) ambient air temperatures. Polyester fabrics were spin-coated with a graphene-oxide (GO) solution of 0.2 wt%. Preparation of rGO was processed by using a microwave oven (MW-rGO). Non-treated fabric (CON) was compared to GO and MW-rGO. The surface temperature of a hot plate was maintained at 35 °C or 40 °C. The test fabrics were put on the heated hot plate or non-heated-outer portions of the hot plate. Surface temperatures of MW-rGO on the heated hot plate at an air temperature of 18 °C (cool) were higher than those of non-treated fabric (CON) under the same conditions ($p<0.01$). No effects from the graphene treatment were found on non-heated portions of the graphene oxide fabric (GO) or the reduced graphene oxide fabric (MW-rGO). On the non-heated portions, surface temperatures were higher at the location closer to the hot plate compared to the location farther from the hot plate ($p<0.05$). These results partially represent thermal effects of MW-rGO under a specific environment and heat source. Our findings enable an application of reduced graphene oxide to body temperature regulating clothing.

Keywords: Surface temperature, MW-rGO, Polyester fabric, Heat source, Thermal effect

Introduction

Graphene has emerged as a revolutionary material due to its extraordinary properties such as its high electric and thermal conductivity, mechanical properties and easy functionalization [1,2]. These properties have led the application of graphene to fabrics and textiles. Kim and Lee published a series of studies on the electrical characteristics of graphene-coated fabrics or films in terms of applying to clothing [3-5]. They found that the electrical properties of graphene/waterborne polyurethane composite films were improved as the content of graphene increases [3]. Colmar [6], one of representative outdoor clothing brands, has applied graphene to sportswear in collaboration with Directa Plus, which is one of the largest producers and suppliers of graphene-based products [7]. These graphene products transfer generated body heat from the hottest to the coldest body regions [6]. Colma, however, applied graphene not to the whole fabric but only to the hexagonal lining of the clothing surface.

Heat transfer is very important for sportswear or workwear, especially for maintaining physiological comfort and safety [8]. Internal heat production during exercise induces an increase in core temperature and can result in hyperthermia and heat stroke. Heavy sports uniforms or personal protective clothing may also contribute to hyperthermia during physical activity because clothing is a barrier for heat exchange with the environment [9]. If the increase in core temperature could be delayed or suppressed, some sport activities and work performance could be improved. On the other hand, for physical activities performed in the cold, the issue of high temperature gradients between the human body and the outside environment could be mitigated.

Graphene oxide (GO) is mainly employed due to its low cost and easy mass production [10,11]. The negative charge of GO allows it to interact with functional fabrics, thus increasing the fixation of GO [1,12]. The posterior conversion to reduced graphene oxide (rGO) by chemical, electrochemical, thermal or UV methods allows the partial restoration of conductivity [1]. GO's microwave reduction (MW-rGO) increased thermal properties, leading to experiments with long periods of microwave irradiation [11]. The integration of graphene into textiles is the next logical step in achieving not only conductive textiles but also multifunctional fabrics [1]. Further, the current electrical heating textiles require batteries to be operated, which brings about the increase of the total clothing weight as well as other technical problems. In this context, exploring thermal properties of graphene without any battery is worth going forward to the next generation of wearable clothing.

The application of graphene to fabrics and textiles has been recently reported [1,10]. To the best of our knowledge, however, no studies have reported on the heat transfer of

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fabrics coated with GO and rGO in terms of surface temperature, despite the fact that this is an easy, useful and accessible indice for examining heat level. Considering that humans wear clothes, the effectiveness of graphene textiles for temperature distribution should ultimately be examined through human wear trials. According to the standard analysis of the physiological properties of textiles and garments [13], however, analysis of textiles separately comes first. Therefore, the present study explored textile properties by examining surface temperature as the first step in terms of heat transfer. In our study we explored the possibility of thermal effects in polyester textiles with GO and MW-rGO by spin-coating them. We hypothesized that MW-rGO fabric would have greater thermal distributive properties in terms of surface temperature compared to non-treatment fabric or GO fabric.

Experimental

Materials

Fabric

This study explored the applicability of graphene oxide to fabrics which could be used to make sportswear or personal protective clothing with greater thermal insulation in cold or heat distribution in hot environments. To that purpose, 100 % polyester fabric was used as the base fabric (Plain stitch knit, 232 g/m^2, 0.46 mm in thickness).

Preparation of Graphene Oxide (GO) and Reduced Graphene Oxide (rGO) on Fabrics

The graphene oxide (GO) sheets were synthesized from graphite powder using the following Hummers method: The natural graphite powder (1.5 g) was added to a mixture of sulfuric acid (180 m*l*), phosphoric acid (20 m*l*) and potassium permanganate ($KMnO_4$, 9 g). The mixture was heated at 50 ^{o}C and stirred for 24 h. After that, hydrogen peroxide (70 m*l*) was slowly injected into the solution in ice bath. Finally, the reaction mixture was purified by centrifugation [14]. The concentration of the GO solution was 0.2 wt% and it was provided by Graphene Square Inc., Korea. The bottle containing this solution was shaken because graphene tends to precipitate in aqueous solutions and then spun in a centrifuge. The polyester fabric (10×10 cm) was placed on the film and then the GO solution (7 m*l*) was added on the upper surface of the fabric, allowing it to spread evenly by using spinning coater (ACE-200, iNexus Inc., Korea) for 20 s: 500 rpm for 5 s, 1,000 rpm for 10 s and then 500 rpm for 5 s. The reduced graphene oxide fabric was obtained by using a conventional microwave oven (M-M209EW, LG electronics, Korea) at 700 W for 2 min. The fabric was then placed at the room temperature for more than 12 h.

Experimental Conditions

Three experimental conditions were tested: non-treatment fabric (CON), graphene oxide fabric (GO) and reduced graphene oxide fabric (MW-rGO). To examine the effects of human's body skin temperature under the fabric, two levels of temperatures were set on the hot plate: 35 ^{o}C and 40 ^{o}C. In general, mean skin temperature of the human body in thermal comfort is 33±1 ^{o}C [10]. The reason why the higher temperatures were set in the present study was the situation assumption using heating devices or heating panels inside clothing to keep the body warm. Also, all the three fabric conditions were tested under two air temperatures (18 ^{o}C and 27 ^{o}C) to investigate effects of air temperature (Table 1). The air temperatures of 18 ^{o}C and 27 ^{o}C were considered as a cool and neutral environment, respectively. The preparation of the fabric samples were conducted at thermo-neutral room temperature (23-25 ^{o}C). Air flow in the climate chamber was maintained at ~0.01 m/s. Ambient humidity was also maintained at 40±5 %RH.

Table 1. Experimental conditions

Fabric condition	Air temperature (^{o}C)	Setting temperature on heat source (^{o}C)
CON GO MW-rGO	18 27	35 40

CON: fabric with non-treatment, GO: fabric with graphene oxide solution by a spin coater, MW-rGO: reduced GO fabric obtained by using a microwave oven; Total experiment conditions=three fabric conditions×two air temperatures×two setting temperatures of the hot plate=12 conditions. All measurements were repeated 5 times. Total data set=12 conditions * 5 repetitions=60 sets.

Test Apparatus and Methods

A climatic chamber (FLC-5000S, Fuji Medical Science, Japan) maintained at a constant room-air temperature and

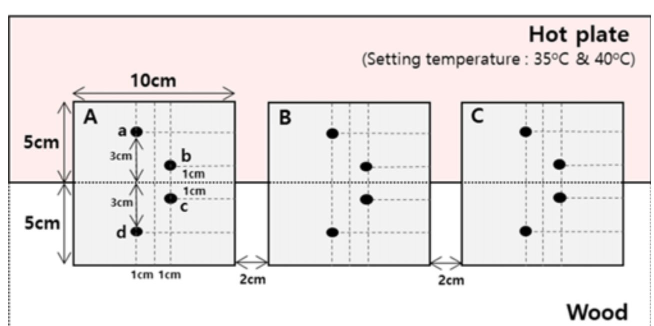

Figure 1. Measurement location of surface temperature on the polyester fabric (10×10 cm). Half of the fabric was on the hot plate and the other half was on the unheated wood. Black dots indicate the locations of surface temperature sensors. The letters of a and b indicate locations on the hot plate while c and d indicate locations on the wood. The CON, GO and MW-rGO fabrics were placed randomly on A, B and C locations.

humidity during preparation. A hot plate (HPLP-C-P, DAIHAN Scientific, Korea) was used as a heat source. The first-half (5 cm) of the fabric was placed on the hot plate while the second-half (5 cm) of the fabric was placed on the unheated surrounding wood. The sensors for measuring surface temperature were attached to the fabric (Figure 1) and then transparent plastic boxes (breadth 10.5 cm×length 21 cm×height 6 cm) were placed over them to cover them and prevent direct heat exchange between the fabric and the air. Surface temperatures on the fabric, air flow in and out of the plastic boxes, and the microclimate (temperature and humidity) inside plastic boxes were measured: four locations (a, b, c and d) on each fabric using a data logger (LT-8A, Gram Corporation, Japan) for surface temperature (Figure 1), two locations using an airflow measurement system (Model 6244 system, Kanomax, Japan) for air flow and one location using the equipment (TR-72U, T&D Corporation, Japan) for microclimate air temperature and humidity. All variables were measured every 5 s or 1 s for 45 min. All conditions were repeated 5 times. Three fabrics were measured in each test and the fabric location on the hot plate was randomly chosen to exclude the effects of temperature differences on it.

Data Analysis

Data were expressed as mean with standard deviation (SD). Statistical analyses were performed using IBM SPSS for Windows version 25 (IBM SPSS Statistics, USA). All data were averages of the 45 min. Five-repeated values were averaged as a representative value. To test group differences among the three fabrics, one way ANOVA was conducted with Tukey's post hoc test. Statistical significance was set at $p<0.05$.

Results and Discussion

We hypothesized that the temperature gradient on the fabric surface would be reduced due to that of the MW-rGO coating under thermal imbalance situations because of its high thermal conductivity. However, contrary to our expectation, instead of a thermal equalizing effect, a heat-holding effect was found for MW-rGO in a cool environment. This finding provides supports the possibility of applying the reduced graphene oxide to winter clothing rather than for summer clothing.

Comparing Surface Temperatures and Microclimates of the Three Fabrics

Statistical differences in surface temperature between CON, GO and MW-rGO were found only on the 40 ^{o}C hot plate at an air temperature of 18 ^{o}C (Figure 2, Table 2, 3). Some variations in surface temperature on MW-rGO were higher than those on CON, and there was no thermal effect on GO (Figure 2). The surface temperature of MW-rGO at the b location on the hot plate was higher than that of CON ($p<0.01$, Figure 2(A)) and the thermal effect of reduced graphene oxide without any battery on the heated part was found. On the other hand, no graphene treatment effects were found on non-heated locations (c and d in Figure 1). The fact that average surface temperatures on the hot plate on the MW-rGO fabrics were higher than those on non-heated parts means that heat transfer from the heated part to non-heated part is inefficient and that a thermal effect of MW-rGO was on the heated surface part only. As the present study evaluates textiles in isolation, there is a possibility of different results when such textiles are worn and the whole body is heating element.

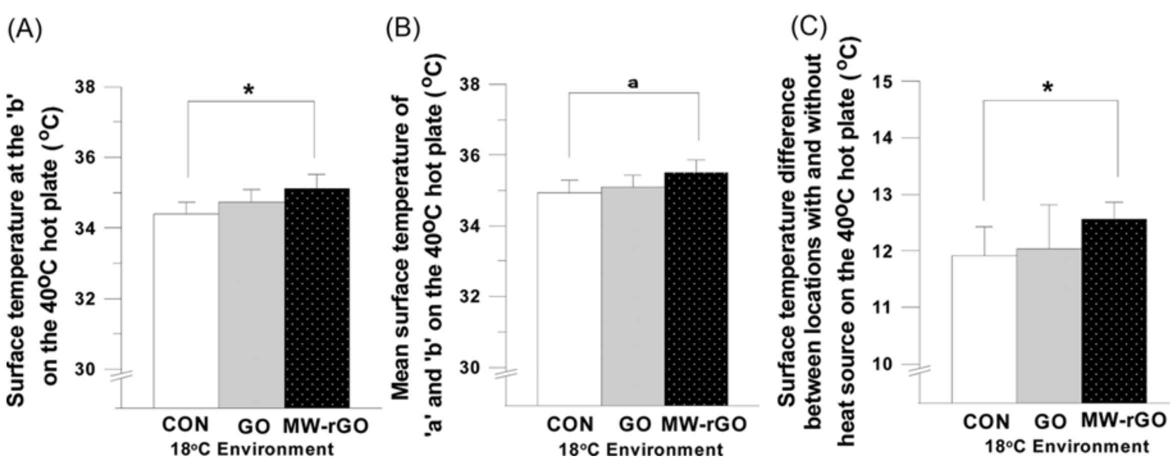

Figure 2. Comparisons of surface temperature between CON, GO and MW-rGO fabrics on 40 ^{o}C hot plate condition in 18^{o}C air temperature environment; (A) surface temperature on the b, (B) mean surface temperature of the hot plate, (C) difference in surface temperature between heated and non-heated parts. CON: fabric with non-treatment, GO: fabric with graphene oxide solution by a spin coater, MW-rGO: reduced GO fabric obtained by using a microwave oven. *: $p<0.05$, ^{a}: $p<0.1$.

Table 2. Surface temperature between CON, GO and MW-rGO

Setting temperature of hot plate (°C)	Room-air temperature (°C)	Temperature variable (°C)	CON	GO	MW-rGO	*p*-value
35	18	Surface temperature at ‘a’ location (a)	31.64±0.38	31.79±0.71	31.90±0.35	NS
		Surface temperature at ‘b’ location (b)	30.83±0.48	31.10±0.79	30.85±0.57	NS
		Surface temperature at ‘c’ location (c)	23.40±0.49	23.15±0.43	23.05±0.29	NS
		Surface temperature at ‘d’ location (d)	21.60±0.56	21.58±0.46	21.48±0.17	NS
		Mean of ‘a’ and ‘b’ (A)	31.23±0.42	31.45±0.65	31.37±0.44	NS
		Mean of ‘c’ and ‘d’ (B)	22.51±0.51	22.37±0.44	22.27±0.19	NS
		Difference between A and B	8.73±0.49	9.08±0.42	9.11±0.49	NS
		Difference between ‘a’ and ‘b’ (within heating area)	0.81±0.20	0.69±0.74	1.05±0.35	NS
		Difference between ‘c’ and ‘d’ (within non-heating area)	1.80±0.26	1.57±0.18	1.57±0.28	NS
	27	Surface temperature at ‘a’ location (a)	33.47±0.06	33.46±0.26	33.50±0.32	NS
		Surface temperature at ‘b’ location (b)	33.13±0.11	33.05±0.43	33.21±0.40	NS
		Surface temperature at ‘c’ location (c)	29.23±0.14	29.29±0.25	29.19±0.11	NS
		Surface temperature at ‘d’ location (d)	28.34±0.20	28.38±0.18	28.34±0.14	NS
		Mean of ‘a’ and ‘b’ (A)	33.30±0.08	33.25±0.34	33.35±0.33	NS
		Mean of ‘c’ and ‘d’ (B)	28.78±0.17	28.83±0.21	28.76±0.12	NS
		Difference between A and B	4.51±0.16	4.42±0.30	4.59±0.31	NS
		Difference between ‘a’ and ‘b’ (within heating area)	0.33±0.07	0.40±0.21	0.29±0.28	NS
		Difference between ‘c’ and ‘d’ (within non-heating area)	0.89±0.07	0.91±0.14	0.85±0.08	NS
40	18	Surface temperature at ‘a’ location (a)	35.47±0.43	35.46±0.58	35.91±0.49	NS
		Surface temperature at ‘b’ location (b)	$34.38{\pm}0.34^{a}$	$34.73{\pm}0.35^{ab}$	$35.10{\pm}0.41^{b}$	0.043
		Surface temperature at ‘c’ location (c)	24.03±0.26	24.09±0.30	23.92±0.43	NS
		Surface temperature at ‘d’ location (d)	21.98±0.26	22.01±0.36	21.96±0.30	NS
		Mean of ‘a’ and ‘b’ (A)	$34.92{\pm}0.36^{a}$	$35.09{\pm}0.35^{ab}$	$35.51{\pm}0.35^{b}$	0.072
		Mean of ‘c’ and ‘d’ (B)	23.01±0.26	23.05±0.31	22.94±0.35	NS
		Difference between A and B	$11.92{\pm}0.51^{a}$	$12.04{\pm}0.78^{ab}$	$12.56{\pm}0.30^{b}$	0.026
		Difference between ‘a’ and ‘b’ (within heating area)	1.10±0.28	0.72±0.67	0.81±0.59	NS
		Difference between ‘c’ and ‘d’ (within non-heating area)	2.05±0.08	2.07±0.21	1.95±0.23	NS
	27	Surface temperature at ‘a’ location (a)	37.22±0.44	37.35±0.43	37.53±0.33	NS
		Surface temperature at ‘b’ location (b)	36.81±0.39	36.98±0.48	36.98±0.41	NS
		Surface temperature at ‘c’ location (c)	30.58±0.45	30.37±0.15	30.38±0.24	NS
		Surface temperature at ‘d’ location (d)	29.10±0.05	29.06±0.15	29.08±0.24	NS
		Mean of ‘a’ and ‘b’ (A)	37.01±0.39	37.08±0.44	37.25±0.37	NS
		Mean of ‘c’ and ‘d’ (B)	29.84±0.25	29.72±0.14	29.73±0.24	NS
		Difference between A and B	7.17±0.22	7.37±0.38	7.52±0.19	NS
		Difference between ‘a’ and ‘b’ (within heating area)	0.41±0.31	0.53±0.22	0.55±0.10	NS
		Difference between ‘c’ and ‘d’ (within non-heating area)	1.47±0.42	1.31±0.12	1.30±0.09	NS

NS means no statistical difference. CON: fabric with non-treatment, GO: fabric with graphene oxide solution by a spin coater, MW-rGO: reduced GO fabric obtained by using a microwave oven.

Our results of MW-rGO on fabric support the claim that MW-rGO is the main contributor to the thermal effect [11, 15-17]. Most studies on the thermal properties of graphene oxide (GO) have reported its extraordinary high thermal conductivity. Further, Ruan *et al.* [18] reported that thermally conductive coefficient, thermal diffusion coefficient and heat-resistance index of the obtained thermal reduced graphene oxide/polystyrene nano-composites were all improved with the increasing addition of thermal reduced graphene. However, an interesting question in the present study is why the thermal effect of MW-rGO was found only for the condition of the 40 ^{o}C surface temperature at the air temperature of 18 ^{o}C, and not for the 35 ^{o}C surface temperature nor for the 27 ^{o}C air temperature. These results indicate that MW-rGO would conserve body heat under cold stress by reducing heat dissipation to ambient air.

Thus the heat conserving effect would be greater with higher skin temperature in cold environments (e.g., during winter sports). When considering the skin temperature of the human body in thermal comfort ranges of 33-34 ^{o}C [13], the MW-rGO heat conserving effect could be developed by using other heating devices that function over 40 ^{o}C inside

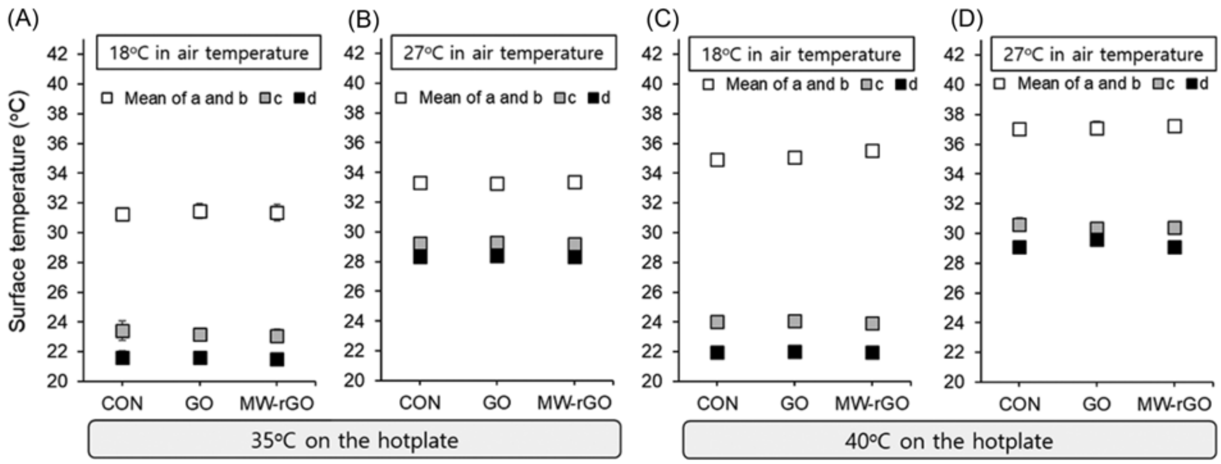

Figure 3. Surface temperatures according to the distance from the heat source and room-air temperature (Mean±SD); (A) 18 ^{o}C in air temperature and 35 ^{o}C on the hotplate, (B) 27 ^{o}C in air temperature and 35 ^{o}C on the hotplate, (C) 18 ^{o}C in air temperature and 40 ^{o}C on the hotplate, (D) 27 ^{o}C air temperature and 40 ^{o}C on the hotplate. CON: fabric with non-treatment, GO: fabric with graphene oxide solution by a spin coater, MW-rGO: reduced GO fabric obtained by using a microwave oven.

Table 3. Microclimates between CON, GO and MW-rGO

Setting temperature of hot plate (^{o}C)	Room-air temperature (^{o}C)	Microclimate under plastic cover	CON	GO	MW-rGO	*P*-value	Mean
35	18	Temperature (^{o}C)	23.26±0.37	23.34±0.27	23.34±0.19	NS	23.31±0.27
		Humidity (%RH)	47±4	45±4	46±3	NS	46±4
		Air flow (m/s)	0.05±0.04	0.01±0.01	0.02±0.01	NS	0.02±0.02
	27	Temperature (^{o}C)	29.24±0.17	29.22±0.18	29.28±0.15	NS	29.25±0.16
		Humidity (%RH)	53±2	52±1	53±3	NS	53±2
		Air flow (m/s)	0.01±0.01	0.00±0.00	0.01±0.01	NS	0.01±0.01
40	18	Temperature (^{o}C)	24.52±0.22	24.60±0.14	24.60±0.24	NS	24.57±0.19
		Humidity (%RH)	46±2	46±2	45±2	NS	46±2
		Air flow (m/s)	0.05±0.04	0.03±0.01	0.03±0.03	NS	0.04±0.02
	27	Temperature (^{o}C)	30.62±0.08	30.70±0.28	30.72±0.24	NS	30.68±0.21
		Humidity (%RH)	50±2	51±2	51±2	NS	51±2
		Air flow (m/s)	0.02±0.03	0.01±0.01	0.02±0.02	NS	0.01±0.01

NS means no statistical difference. CON: fabric with non-treatment, GO: fabric with graphene oxide solution by a spin coater, MW-rGO: reduced GO fabric obtained by using a microwave oven.

clothing in cold winter. That is, we can propose the MW-rGO based fabric as textiles covering wearable heating elements for winter clothing. Unlike MW-rGO, GO fabric had no additional heat conserving qualities pertinent to the thermal conductivity of GO which is very tightly the coverage of oxygen groups [15].

Effects of Distance from a Heat Source and Room-air Temperature

Surface temperatures of the two heated locations (a and b in Figure 1) on the hot plate were higher than those of non-heated locations (c and d in Figure 1) (all $p<0.01$). Of these two spots on the non-heated part, surface temperatures were higher on the spot closer to the hot plate (c) than on farther one (d) (Figure 3). This shows that heat transfers through all fabrics. Even, surface temperature on the ‘d’ was about 1-2 ^{o}C higher than room-air temperature in respective conditions (Figure 3). From this we can see the heat transfer within an enclosed space.

However, there were no statistically significant differences in surface temperatures between CON, GO and MW-rGO according to the room-air temperature. The surface temperature between heated and non-heated parts of the fabrics differed according to the room-air temperature. As expected, the surface temperature differences between the heated and non-heated spots were greater, as the differences between air temperature and surface temperature were greater: 40 ^{o}C surface at T_{air} of 18 ^{o}C (22 ^{o}C diff.) > 35 ^{o}C surface at T_{air} of 18 ^{o}C (17 ^{o}C diff.) > 40 ^{o}C surface at T_{air} of 27 ^{o}C (13 ^{o}C diff.) > 35 ^{o}C surface at T_{air} of 27 ^{o}C (8 ^{o}C diff.) (Figure 3, Table 2). These differences were identical among the three fabrics. The temperature differences between the non-heated spots c and d were greater for the 18 ^{o}C condition than for the 27 ^{o}C condition (Figure 3), which indicates that temperature gradients on the non-heating fabric were greater for cooler environments irrespective of the graphene coating.

There were no statistical differences in microclimate (air temperature, humidity and air velocity) under the plastic cover due to the three fabrics and setting temperature of hot plate (Table 3). Also, no reciprocal actions among fabrics, setting surface temperature of hot plate or air temperature were shown. However, we found a difference in air temperature and humidity of microclimate between 18 ^{o}C and 27 ^{o}C environmental conditions: The microclimate air temperature and humidity were higher, and air velocity was lower at the 27 ^{o}C environmental condition compared to the 18 ^{o}C environmental conditions ($p<0.05$, Table 3).

The limitation of this study was that we did not investigate the next-to-skin microclimate under fabric. The heat-holding effect of MW-rGO should be examined at lower air temperature than 18 ^{o}C. Also, effects of GO solution on thermal conductivity should be examined in comparison with its film type.

Conclusion

We evaluated the applicability of GO and MW-rGO to polyester fabric in terms of heat transfer over the human skin. Originally, we hypothesized that the thermal gradient of the fabric surface would be reduced due to MW-rGO when put on the heat source that was simulated as the human skin. However, no difference in thermal equalizing effect was found between GO and MW-rGO for the fabric on the heat source, whereas a heat-holding effect was found for MW-rGO, especially at the air temperature of 18 ^{o}C. This finding supports the possibility of applying graphene to winter clothing to maintain a warm body temperature in cold environments. In particular, MW-rGO fabrics might accelerate the efficiency of heating elements inside winter clothing when using as cover textiles of the heating elements. This study is original in that we examined the thermal gradient of MW-rGO fabric under the simulated conditions of heated human skin in a cool environment. In order to actually apply MW-rGO to sportswear, the thermal dynamics of mock-up MW-rGO clothes under various degrees of cold stress, through human wear trials should be evaluated.

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