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Additional Information

Analysis of the influence of graphene and phase change microcapsules on thermal behavior of cellulosic fabrics

RUIZ-CALLEJA Tamara¹, BONET-ARACIL Marilés², GISBERT-PAYÁ Jaime², BOU-BELDA Eva²

¹Universidad Rey Juan Carlos, Lecturer in Fashion and Technology, Calle Tulipán, 28933 Móstoles, Spain

²Universitat Politècnica de València, Textile and Paper Department, Plaza Ferrándiz y Carbonell s/n, 03801, Alcoy, Spain.

Corresponding author: BONET-ARACIL Marilés. Plaza Ferrándiz y Carbonell s/n, 03801, Alcoy, Spain. E-mail: maboar@txp.upv.es

Abstract Thermal management is a critical factor in several areas, such as architecture, computing, and transportation. In the textile field, researchers are developing numerous advances for effective thermal control. One of the most relevant is phase change materials (PCM), which allow temperature regulation due to their ability to absorb or release latent heat through their state changes. Graphene, because of its high thermal conductivity, is considered an exciting alternative for thermal management. In this investigation, we focus on finding new approaches for thermal regulation of cellulosic fabrics combining both technologies, phase change materials and graphene. For this purpose, we compare the thermal behavior of a cellulosic fabric when applying a coating paste containing graphene or phase change materials individually, finding that their performances are similar during heating. Likewise, the synergy produced by using both materials in the same coating paste is studied, proving that the action of graphene and PCM simultaneously allows the dissipation of more heat energy than when acting individually. These results open new paths of research on thermoregulation that may be useful in numerous applications beyond textiles.

Key words: thermoregulation, graphene, phase change materials, coating, textile

1. Introduction

The study and control of thermoregulation are of extreme importance in different fields, including sportswear, underwear, and protective equipment, where user

comfort needs play a significant role [1, 2]. In terms of clothing, body comfort is a combination of four elements: thermal or thermophysiological comfort, sensorial comfort, garment fit, and psychological comfort. Thermophysiological comfort is affected by several factors comprising 'loss (or gain) of heat by radiation, conduction and convection, the loss of heat by the evaporation of sweat, the physical work done by the user, and the environment (ambient temperature, air humidity, and air movement)' [3].

Furthermore, to enhance the thermal regulatory properties of garments, there are numerous products and treatments [4–6]. According to Tyurin et al. [7], they are categorized in five groups, including electrothermoregulated, chemically thermoregulated, clothing made of materials that absorb solar energy, clothing made from materials with new regenerating sources of energy and clothing made from phase change materials.

NASA, whose work around the mid-1960s and early 1970s led to the development of phase change materials as known today, describe them as materials with the unique ability to absorb and liberate large quantities of heat without appreciable temperature change [8]. PCM have a phase change temperature range, above this temperature, they change from solid to liquid, absorbing latent heat; when the temperature decreases below said phase change temperature range, they change from liquid to solid, releasing latent heat [9].

The first step to incorporate phase change materials to textile substrates is to microencapsulate or macroencapsulate them. This encapsulation means there is a physical barrier between PCM and their surroundings, to avoid the leak of the phase changing substances in the event of melting or evaporation. Microencapsulated PCM are usually applied to textile substrates as a coating, using a resin or binder, or when extruding the fiber; macroencapsulated PCM are integrated as a coating or as a thin laminated film [9, 10].

Currently, phase change materials are present in a wide range of textile products such as smart fabrics [11, 12], medical applications [13], protective apparel [14], sportswear [15], denim [16], and nonwovens [17], amongst others [18–20].

Numerous studies demonstrate the influence of phase change materials on the thermal regulation of cellulosic fabrics. For example, the research carried out by Hassabo [21] proves that PCM applied as a coating on cotton fabrics improve the thermal properties of treated fabrics, compared to uncoated fabrics. Furthermore, Salaün et al. [22] demonstrate the thermal behavior of the PCM-PU binder mixture is linked to the thermal properties of the polyurethane.

After its discovery in 2004 by Andre Geim and Konstantin Novoselov, graphene has gained attention in multiple fields including composites [23–25], energy storage [26–28] and electronics [29–31] due to its outstanding mechanical [32] and electrical [33] properties. According to Dubey et al. [34], graphene is a flat monolayer of carbon atoms tightly packed into a two-dimensional (2-D) honeycomb lattice.

Graphene manufacturing can be carried out, mainly, following one of the following methods according to Koratkar et al. [35]: chemical vapor deposition (CVD), epitaxial growth of graphene on electrically insulating substrates, mechanical exfoliation of graphene from bulk graphite, and reduction of graphene derivatives such as graphene oxide.

There are several methods to incorporate graphene into textile substrates, being coating one of the most widely used [36–38], usually comprising an aqueous solution impregnation and followed by a reduction. Some other methods include graphene yarn/fibers embedding to fabrics and chemical vapor deposition over a fabric-like metallic mesh, which is removed after reduction [39].

Regarding its thermal properties, graphene exhibits superior thermal conductivity [40] and is an excellent material for thermal management [41]. Some investigations confirm its usefulness for textile thermal regulation. For instance, Gao et al. [42] have applied a graphene aerogel to thermal protective clothing with promising flame-retardant results with excellent thermal protective performance and increasing the time required to cause skin damage. Likewise, other researchers have created non-woven fabrics [43] and films [44] with high thermal conductivity suitable for industrial-scale applications.

This research has two objectives: first, we want to compare the influence of graphene and phase change materials on the thermal behavior of a cellulosic textile used individually in a coating paste, to assess the similarities and differences of both materials concerning thermal comfort. Secondly, we want to evaluate the performance of both materials used simultaneously in the same coating paste to determine if the synergy between them could mean a significant improvement in the field of thermoregulation.

There is a wide variety of articles related to phase change materials and about graphene, although documents covering both subjects are not so studied and mainly if we focus on ‘textile’, ‘fiber’, or ‘fabric’. The afore-cited are related to thermal energy storage [45–47] and batteries [48, 49]. However, none of them include the term ‘coating paste’, thus proving this research to open a new path of investigation merging both materials, graphene and phase change material, in the same coating paste for textile purposes. Given the lack of research within this topic, we think it is necessary to investigate in this direction to explore the potential of both products in textile thermoregulation.

2. Experimental

2.1. Materials

For this research, we use a 50% cotton-50% flax twill with a mass per unit area of 210 g/m² and chemically bleached in an industrial process.

Graphene and phase change material are purchased ready-made from specialized suppliers.

Coating paste is obtained using the following products:

- Polyurethane binder,
- Thickener and
- Deaerating agent.

2.2. Fabric coating procedure

Coating paste deposition onto fabric is performed using an adjustable scraper and metal frame set at 1 mm.

The concentrations for the base coating paste, according to binder's supplier instructions, are described in Table 1:

Product	Quantity (g/Kg)
Polyurethane binder	400
Thickener	15
Deaerating agent	1,5

Table 1. Base coating paste composition

Different formulations of the coating paste are tested to analyze the influence of graphene and phase change materials on the cellulosic fabric thermal behavior, as shown in Table 2.

Sample	Graphene (g/Kg)	PCM (g/Kg)
Untreated	Non coated	
PU	0	0
GRF25	25	0
PCM100	0	100
GRF25+PCM100	25	100

Table 2. Coating paste formulation

The precise amount of PCM is determined as a result of several previous internal studies. The quantity of graphene is subjected to the rheology of the printing paste, which does not admit a higher amount of graphene without saturation.

All coated samples are oven-dried at 60 °C followed by a thermal treatment performed during 3 minutes at a temperature of 145 °C to ensure binder curing.

2.3. Sample characterization

The observation of the samples is carried out using a Field Emission Scanning Electron Microscope (FESEM) (ULTRA 55, ZEISS) and Transmission Electron Microscopy (TEM) (TEM-1010 (JEOL)). To analyze samples by SEM each of them is placed on a surface and covered with a layer of gold and palladium to transform them into conductive by using a Sputter Coater and covering them with gold. The

samples are analyzed with the appropriate magnification and with an acceleration voltage of 2 kV in SEM and 80 kV in TEM.

2.4. Thermal behavior evaluation

Samples listed in Table 2 are exposed to a radiant heat source at 100 °C, for two minutes, inside an enclosed area at a constant temperature of 27 °C. Measurements are taken every fifteen seconds.

After two minutes, the heat source is removed, and measurements are taken every fifteen seconds for a total test time per sample of four minutes.

Temperature fluctuations experienced by the samples are recorded using a thermographic camera, with emissivity (ϵ) set to 0,77 according to the manufacturer instructions.

The experimental setup, as seen in Fig. 1, consists of a frame where the fabric sample is placed; 20 cm below the sample is the heat source, mounted on rails to allow an easy extraction; the thermographic camera is above the frame.

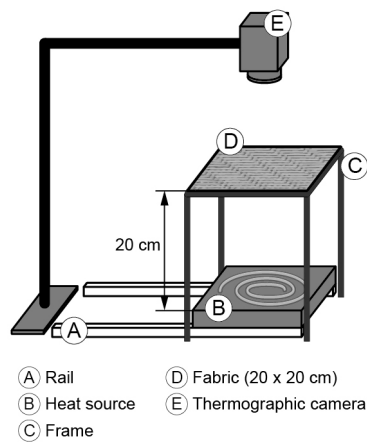


Fig. 1. Experimental setup for thermographic evaluation

3. Results and discussion

3.1. SEM images

Precise details of the distribution of the polyurethane binder, the graphene, and the phase change materials are observed in the FE-SEM images of the samples.

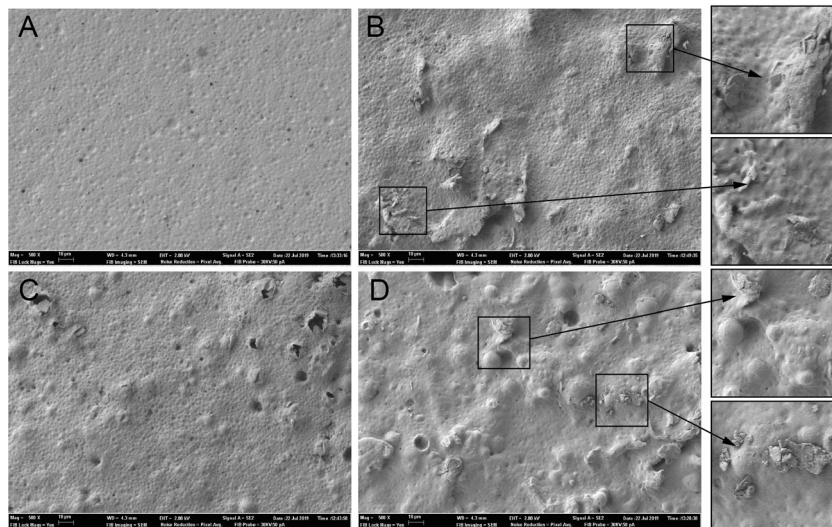


Fig. 2. FE-SEM images of samples at 500X magnification. (A) PU; (B) GRF25; (C) PCM100 and (D) GRF25+PCM100

The polyurethane-coated sample in Fig. 2 a exhibits bubbles in its structure, areas in which air is trapped, and acts as an insulator by itself.

GRF25 sample, in which the coating paste containing graphene is applied (Fig. 2 b), shows an extremely irregular distribution of the graphene flakes. Due to their inherent two-dimensional structure [34], they do not occupy a homogeneous volume in the coating paste.

However, PCM100 sample, coated using phase change materials, shows a higher homogeneity of the PCM microcapsules layout, seen in Fig. 2 c.

GRF25+PCM100 sample, containing both graphene and phase change materials in its coating (Fig. 2 d), demonstrates the expected behavior after analyzing previous samples: PCM microcapsules have a relatively uniform distribution while, in

contrast, graphene flakes have a slightly more irregular arrangement amongst the coating paste.

3.2. TEM images

TEM images of the samples allow an accurate evaluation of the size and distribution of both the PCM microcapsules and the graphene flakes. Microcapsules show regularity in their shape and size (Fig. 3), with their diameter being around 1 μm and without perceptible aggregates. However, regarding graphene flakes (Fig. 3 and Fig. 4), their dimensions are smaller than PCM and extremely irregular, as well as their shape. Besides, there are areas where graphene flakes are concentrated and some others in which their presence is not observable.

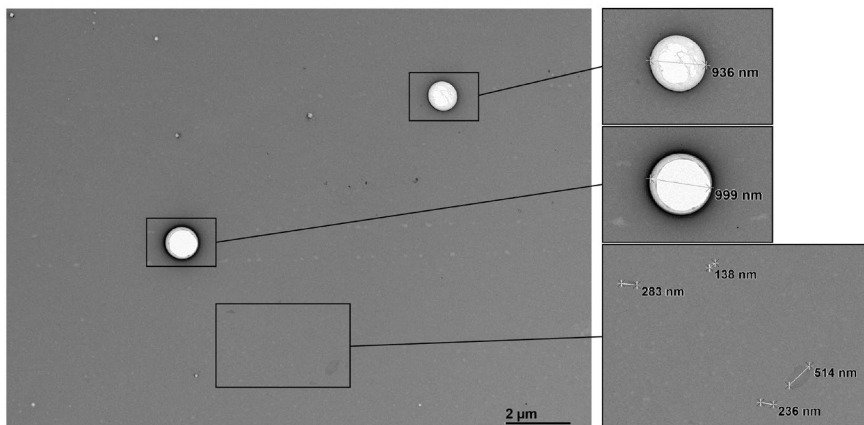


Fig. 3. TEM image of sample GRF25+PCM100

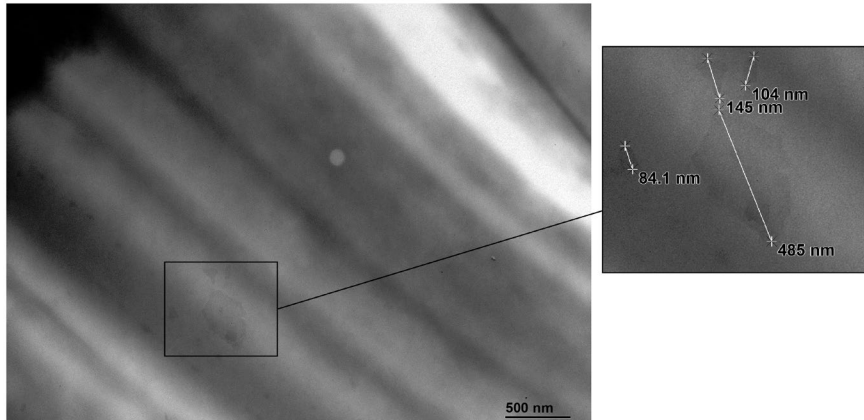


Fig. 4. TEM image of sample GRF25

3.3. Thermal behavior evaluation

All temperature data obtained during thermal tests of the samples are registered, as seen in Fig. 5.

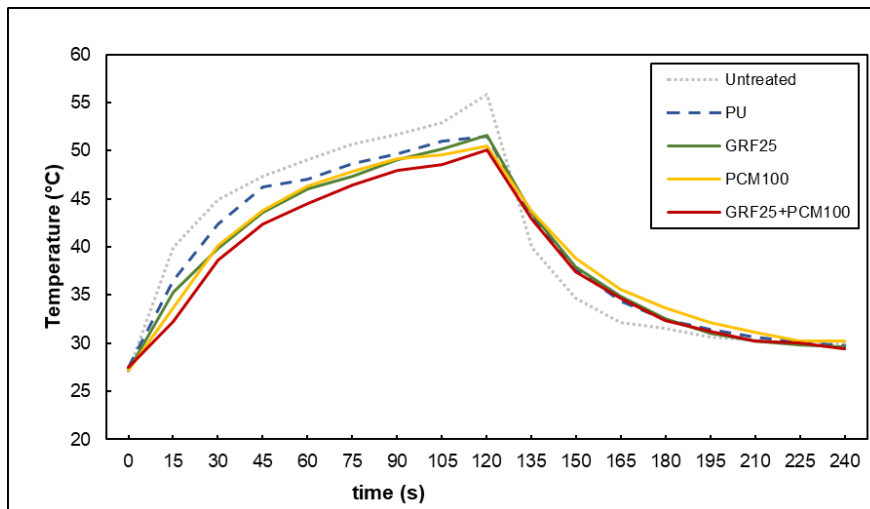


Fig. 5. Behavior of samples during thermal test.

Temperature differences are calculated after testing all samples, both in the heating and the cooling phase, as listed in Table 3.

	ΔT Heating (°C)	ΔT Cooling (°C)
Untreated	28,8	-25,9
PU	24,0	-21,8
GRF25	24,4	-22,0
PCM100	23,3	-20,3
GRF25+PCM100	22,6	-20,7

Table 3. Temperature variations of samples

When heating, the surface of the untreated fabric rapidly increases its temperature experiencing a variation of 28,8 °C compared to the initial temperature value. Cooling occurs abruptly during the first 45 seconds and then slowly decreases.

In the sample with polyurethane coating the temperature variation when heating is 24,0 °C, significantly lower than ΔT obtained for the untreated fabric, proving the influence of the polyurethane binder in the thermal regulation of cotton textiles as stated by Salaün et al. [22]. While cooling, there is a notable difference compared to the untreated sample, since the surface tends to recover room temperature in a much more gradual way without significant temperature drop.

Curves of samples PCM100 and GRF25 are remarkably similar at the beginning of the heating stage, when their inclinations have almost identical angles. The sample with phase change materials shows a slightly lower temperature increase than the sample with graphene, 24,4 versus 23,3 °C. The similarity between both behaviors is due to the ability of graphene to dissipate heat thanks to its high thermal conductivity [40] and the ability of PCM to absorb thermal energy [9]. It can be stated that, while the mechanisms that produce said results are different, the performance in terms of thermoregulation is similar in both cases. When cooling occurs, the temperature of sample PCM100 decreases more smoothly than that of sample GRF25, which does it in a more accentuated way.

Finally, the curve of sample GRF25+PCM100 is the most interesting in terms of thermoregulation. Its temperature increase occurs much more gradually than in the other samples, reaching $\Delta T = 22,6$ °C at 120 seconds, the smallest increase of all the samples tested, and 6,2 °C less than the variation achieved by the untreated sample. When cooling, GRF25+PCM100 behavior resembles the curve of sample

GRF25, and not the one of PCM100, due to the ability of graphene to dissipate the latent heat stored by phase change materials microcapsules during heating, that is released during cooling.

3.4. Thermal images

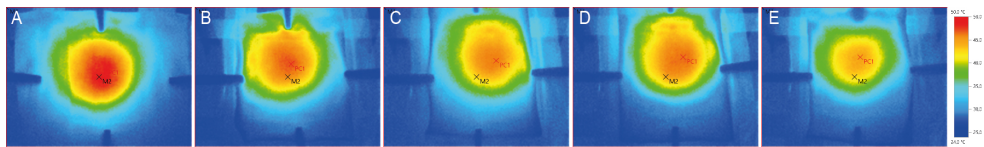


Fig. 6. Thermographic images of samples (A) Untreated; (B) PU; (C) GRF25; (D) PCM100 and (E) GRF25+PCM100 after 60 seconds

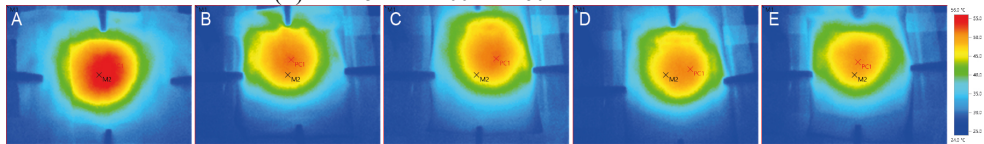


Fig. 7. Thermographic images of samples (A) Untreated; (B) PU; (C) GRF25; (D) PCM100 and (E) GRF25+PCM100 after 120 seconds

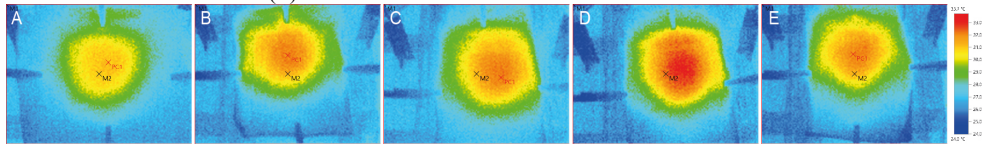


Fig. 8. Thermographic images of samples (A) Untreated; (B) PU; (C) GRF25; (D) PCM100 and (E) GRF25+PCM100 after 180 seconds

Thermographic images of untreated cellulosic fabric, Fig. 6 a, Fig. 7 a, and Fig. 8 a, show it heats abruptly and cools off promptly with hardly any thermal energy conserved.

PU sample images, Fig. 6 b, Fig. 7 b and Fig. 8 b, demonstrate the fabric treated with polyurethane binder gets less warmed up than the untreated sample [22].

In Fig. 6 c, Fig. 7 c and Fig. 8 c, the heat-conductive effect of graphene due to its superior thermal conductivity [40] is noticeable since the area of the fabric that shows a temperature increase is larger than in samples a and b, untreated and polyurethane-coated respectively, managing to distribute the thermal energy in a wider area and heating less the whole sample, as described in textile [42, 43] and non-textile studies [44].

Regarding PCM100 sample, the most significant behavior occurs during the cooling stage, Fig. 8 d. In said image, it is clear how phase change material microcapsules release the latent heat they have stored during the heating stage, result in accordance with previous researches within this topic [9, 21].

GRF25+PCM100 sample images, Fig. 6 e, Fig. 7 e and Fig. 8 e, exhibit the promising behavior that occurs when both products are used simultaneously due to the absorption or release of thermal energy by phase change materials and the high thermal conductivity inherent to graphene. This performance leads to lower temperatures than other samples during the heating stage and a similar result to GRF25 sample in the cooling stage due to the conductive effect of graphene previously discussed.

4. Conclusions

This research demonstrates that phase change materials and graphene, applied individually on a cellulosic textile as a coating paste, have similar behavior regarding their ability to dissipate heat energy when subjected to the action of a heat source, PCM due to their ability to store latent heat, graphene due to its superior thermal conductivity.

When cooling occurs, graphene continues to dissipate thermal energy while phase change materials release previously stored energy, so their behavior in this situation can be said to be the opposite.

Also, this research demonstrates that using both materials together in the same coating paste leads to higher dissipation of thermal energy during the heating stage.

The present work opens new lines of research on thermoregulation in textiles, combining these two materials in the same coating paste. Future research is proposed to evaluate the use of other means of application on textiles, such as a pad-dry process or the manufacture of filaments that include both materials in their cores. The use of other textile substrates, such as hollow fibers, is also suggested for greater tunability of thermal comfort. In conclusion, these findings may be

useful in other applications where thermoregulation is essential such as batteries development or energy harvesting.

CRedit authorship contribution statement

Ruiz-Calleja Tamara: Methodology, Investigation, Writing - original draft, Writing - review & editing. **Bonet-Aracil Mariles:** Conceptualization, Resources, Supervision, Writing - review & editing. **Gisbert-Paya Jaime:** Conceptualization, Resources, Supervision, Writing - review & editing. **Bou-Belda Eva:** Conceptualization, Resources, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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