## RESEARCH



# Innovative flexible thermal storage textile using nanocomposite shape-stabilized phase change materials



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## Abstract

A novel flexible thermal storage system based on organic phase change materials (PCMs) deposited on a non-woven polyester (PET) substrate is described in this article. Thermally regulating effects were created via encapsulation of polyethylene glycol (PEG) in carbon nanofibers (CNFs) to manufacture a shape-stable phase change material (SSPCM). Improvement in the thermal conductivity (TC) of the system was obtained by incorporating reduced graphite oxide nanoparticles (rGONP) into the CNFs. A new method was applied to load and secure the manufactured SSPCMs on the fibrous substrate so that an acceptable level of flexibility was preserved (change in bending length less than 30%). The sample performance was evaluated by measuring its thermal properties. The physical properties, wash fastness, abrasion resistance, morphology, and PCM leakage of the samples were also assessed. The results point to a good thermal storage ability of the samples with characteristic phase change temperature ranges of 30.1–31.4 °C and 19.2–24.3 °C for melting and freezing, respectively, and a latent heat of 8.9–22.9 J g<sup>-1</sup> for meting and 11.2–21.4 J g<sup>-1</sup> for freezing. The use of the CNF-rGONP for PEG enhanced the TC of the system by 454%, thus providing a rapid thermal response, and efficiently prevented the leakage of PEG. Finally, the loading and fixation method on the non-woven substrate allowed an acceptable level of durability with less than 4% of weight loss during washing and abrasion tests. This system provides a promising solution for rapid response, flexible thermal storage wearables.

**Keywords:** Flexible thermal storage system, Shape-stabilized phase-change material, Nanocomposites, Thermal conductivity, Rapid thermal response

## Introduction

Clothing plays a crucial role in maintaining thermophysiological balance of human beings (Ganesh et al., 2021). Smart textiles with embedded thermal storage/release materials can preserve thermal comfort during changes in the surrounding conditions (Gu et al., 2021; Zhang et al., 2021). The development of thermal energy storage based on PCMs is a popular research field that involves designing smart, multidimensional networks (Merati, 2021).



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The developed PCM-embedded textile products also have to resist service conditions such as laundering and abrasion. It is thus critical to find an appropriate method for securing PCMs on the fibrous substrate in a durable way while preserving the fabric characteristics such as flexibility and softness depending on the end use.

Organic PCMs such as fatty acids, paraffin, and PEG are the most commonly used compounds due to their interesting properties in terms of high latent heat at the liquid–solid phase transition and physiologically-relevant melting and crystallization temperatures (Koncar, 2016). PEG exhibits the advantages of non-toxicity, biocompatibility, high latent heat, and low cost (Ding et al., 2020; Jia et al., 2020). Vigo and Frost were pioneers in the direct bonding of PEG to fibers via a crosslinking agent and hot catalyst using citric acid to manufacture thermal-adaptable textiles (Vigo & Frost, 1985). However, the samples they prepared had various issues, such as enthalpy reduction, lack of shape stability, and PCM leakage, which limited their use for direct application onto fibrous substrates (Sarier & Onder, 2012).

The most effective strategy to overcome the problem of leakage during the phase change process is to stabilize the shape of PCMs by encapsulating them in supporting materials (Umair et al., 2019). This has led to the development of a new type of PCMs, called SSPCMs, in the late 1990s (Cárdenas-Ramírez et al., 2020).

Studies conducted on the preparation and optimization of SSPCMs have concluded on the suitability of these materials for energy storage and use in solar cells to convert heat energy into electricity (Nian et al., 2021; Zhu et al., 2018). Yet, most research on SSPCMs towards producing thermally adaptable textiles has been limited to the application of microcapsules by conventional coating methods or embedding microPCMs into the fibers during the manufacturing process (Dolez & Mlynarek, 2016). For example, in a study by Shin et al. (2005), polyester knit fabrics were treated with PCM microcapsules by a pad-dry-cure method with a polyurethane binder. The authors observed that the heat storage capacity of the treated fabrics increased with an increase in the microcapsule add-on. The fabric with 22.9% added microcapsules was capable of absorbing 4.44 J/g of heat. The air permeability of the treated fabric decreased with an increase in the amount of microcapsules.

Salaün et al. (2010) reported the manufacture of a thermoregulating textile containing microPCMs. A series of fabrics containing different polyurethane binder-to-microcapsules ratios were prepared by a padding process. The results showed that the polymeric binder played a major role during the 30 s of the cold-to-warm transition, creating a delay in the temperature increase. A good thermoregulating effect without altering the fabric air permeability was obtained for a PCM coating containing 20–40 wt% of binder and a minimum of coating add-on of about 17%.

In another work, Kim et al. (2016) studied the thermal properties of the combat uniform treated with microcapsules containing octadecane. An aqueous acrylic resin (Koplex TF-125) was used as a binder between the microcapsules and the fabric. They observed that the heat of fusion ( $\Delta H_f$ ) and heat of crystallization ( $\Delta H_c$ ) of the specimens increased with an increase in the microcapsule concentration. At 12% and 18% microcapsule concentration,  $\Delta H_f$  and  $\Delta H_c$  decreased as the treatment temperature increased. On the other hand, for specimens with 24% microcapsules,  $\Delta H_f$  and  $\Delta H_c$  increased when the specimens were cured at a higher temperature. This difference in behavior was attributed to the fact that, at a high microcapsule concentration, a high curing temperature can help bind the microcapsules to the fabric.

Silva et al. (2012) applied polystyrene microcapsules containing Rubitherm<sup>®</sup> RT31 paraffin on seven types of textile substrates by means of a coating technique using the WST SUPERMOR<sup>®</sup> commercial binder. The PCM microcapsules had a  $\Delta H_f$  of 98.72 J g<sup>-1</sup> and an average particle size of 4.1 µm. The PCM-coated fabrics exhibited a  $\Delta H_f$  between 11.1 and 19.4 J g<sup>-1</sup>, depending on the textile substrate.

Alternatively, PCMs can be embedded into the body of polymer fibers (Dolez & Mlynarek, 2016). The composite fibers are further spun into a yarn, which can then be knitted or woven. Different techniques can be utilized to prepare the PCM composite fibers, viz., wet spinning microPCMs dispersed in a polymer matrix or bulk polymerization of the PCM and matrix compounds, followed by melt spinning. PCM fibers are now commercially available with cellulose (e.g., Smartcel<sup>™</sup> clima and Alceru<sup>®</sup> thermosorb fiber) and synthetic polymer (e.g., Outlast<sup>®</sup> microPCM-containing acrylic, rayon, and polyester fibers) matrices.

Selecting the appropriate supporting material and preparation method for the SSPCM is critical to tackle the other challenge for the application of PCMs in textiles that is related to obtaining a sufficiently high TC and acceptable response time to temperature changes (Umair et al., 2019; Zeighampour et al., 2022a). Studies have revealed that the TC of SSPCMs can be significantly improved by incorporating carbon supporting materials with 1D, 2D, and 3D structures (Ding et al., 2020; Shah, 2018). However, only limited research has been conducted on the synergistic effect of carbon hybrids with different structures and pores used as supporting materials to improve the performance of SSPCMs, especially their TC (Yu et al., 2022; Zeighampour et al., 2022b). In addition, none of these prior work with carbon hybrid SSPCMs involved textile applications. To the best of our knowledge, it is the first time such endeavor has been attempted. In addition, only a few studies have been carried out to stabilize the shape of low molecular weight PEG, which is not completely solid at ambient temperature and results in low loading rates (Frigione et al., 2020; Song et al., 2019).

This study thus aimed at preparing a SSPCM flexible thermal regulation fibrous structure with improved TC, suitable phase transition temperature, desirable thermal endurance, and leakage-proof property. To achieve this goal, a carbon hybrid supporting matrice of rGONPs embedded into CNFs was used to hold PEG. It was coated on a polyester (PET) non-woven fibrous structure. The effect of the concentration of the carbon-based SSPCMs on the thermal properties of the coated fabric was investigated. The serviceability of the SSPCM-coated fabric was also considered; it was assessed in terms of preservation of the fabric flexibility and resistance of the SSPCM coating to abrasion and laundering.

#### Methods

#### Materials

A non-woven polyester fabric with a 300 g/m<sup>2</sup> weight and 2.3 mm thickness was obtained from Negin Rose Sepahan Co. in Isfahan, Iran. A non-ionic detergent (Sera Wet CNR) was supplied by DyStar, Turkey. The vinyl acetate/acrylic resin (Subratex 100<sup>®</sup>) used as the binder and the thickener (Ultra Thickener ER Conc.) were received from Subranco

Co. in Isfahan, Iran. The chemicals used for the preparation of the PEG/CNF-rGONP SSPCMs are: polyethylene glycol (PEG) (melting point: 32 °C,  $M_w = 1000$  g/mol, density: 1.1 g/cm<sup>3</sup>) and N, N-dimethylformamide (DMF, 99.97%) were sourced from Merck, Germany. PAN powder ( $M_n = 7 \times 10^4$  g mol<sup>-1</sup>) containing acrylonitrile (94 wt%), methyl methacrylate (4 wt%), and methacrylic acid (2 wt%) was obtained from Polyacryl Company, Iran. rGONP (140 nm, 458 m<sup>2</sup> g<sup>-1</sup>, 99.8%) was prepared using ball milling followed by thermal process (Zeighampour et al., 2022b).

#### Sample preparation

The PEG/CNF-rGONP SSPCMs were prepared and characterized according to the protocol developed by Zeighampour et al. (Zeighampour et al., 2022b) (detailed preparation method in Supporting Information). Polyacrylonitrile (PAN) nanofibers containing rGONPs with a weight ratio of 50:50 were prepared by electrospinning and then carbonized and activated under specific conditions in a furnace. At higher weight fractions of rGONPs to PAN (e.g. 60:40 rGONP/PAN), electrospinning was not possible due to the increased viscosity of the solution (Zeighampour et al., 2022b). The resulting rGONPcontaining CNFs played the role of carbon hybrid supporting material to trap and hold the 1000 g/mol PEG selected for this study. The vacuum impregnation method was used to prepare the desired PEG-rGONP/CNF SSPCMs.

The PEG/CNF-rGONP SSPCMs were coated onto a polyester non-woven fabric. Impregnation was carried out by padding; the amount of material loaded was controlled with the concentration of the compounds in the impregnation solution and the pressure of the pad rollers. Three samples were prepared (labelled S5, S15, and S25) with three percentages of SSPCM (5, 15 and 25 wt%) in the fabric impregnation solution (Table 1). Even though the amount of additives used in the textile industry can reach 40%, excessive dusting of the SSPCMs from the fibers was observed at weight fractions of 30% and above. A higher binder content may have allowed increasing the SSPCM content; however, since it would have had a negative effect on the flexibility and breathability of the fabric, it was not considered. For comparison purposes, untreated fabric samples and fabric samples only treated with the binder (labelled as binder-coated) were also assessed. By setting the pressure to 3.5 bar and the roll speed to 3.02 m/min, a loading (or pick-up rate) of 70 wt% was attained. The fixation method for SSPCMs on the fabric was dry heat; the samples were dried at a temperature of 70 °C for 7 min (Warner Mathis AG, Niederhasli/Zurich, Switzerland). Then, curing was performed at a temperature of

Sample	Composition of the impregnation solution (wt%)									
	Vinyl acetate/acrylic resin	Thickener	Water	SSPCM						
Uncoated	-	_	_	_						
Binder-coated	20	6	74	_						
S 5	20	6	69	5						
S 15	20	6	59	15						
S 25	20	6	49	25						

Table	1	Comp	position	of the	tested	samples
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120 °C for 3 min. The drying and curing times and temperatures provided appropriate conditions for the even fixation of the SSPCMs in the whole structure of the substrate. They prevented the thermal migration of the chemicals on the fabric surface and the deterioration of the fastness properties.

#### **Characterization methods**

#### Morphological analysis of the fabrics

The surface topography and cross-section of the specimens were examined before and after coating with a stereo microscope (2CE-MU, HP, USA) operating at a magnification of 10–40 and a field emission scanning electron microscope (FESEM, Quanta FEG 450, FEI, USA) operating at 15 kV acceleration voltage and magnification of 100–60,000. Prior to FESEM analysis, the samples were coated with a thin layer of gold.

#### Weight change measurement

The weight of the fabric specimens was measured using a laboratory balance (GR-120, A&D, Japan). The specimens were conditioned at 20 °C and 65% relative humidity (RH) for 24 h before the weighting, which was carried out in a conditioning room. The change in weight was calculated according to Eq. (1).

Add - on (%) = 
$$\frac{W_b - W_a}{W_b} \times 100$$
 (1)

where  $W_a$  is the initial weight of the specimen and  $W_b$  is the weight of the specimen after coating or being subjected to the abrasion or washing cycles.

When assessing the coating add-on ratio (wt%), the measurement was repeated at least five times and the average was reported. In the case of the abrasion resistance (2.3.9) and washing fastness (2.3.10), five replicates were measured.

#### Thickness measurement

The thickness of the specimens was measured in accordance with the ASTM D5729-97 (2004) test method at constant pressure level of 1 kPa. For each sample, the measurement was performed at least five times at different locations of the different specimens and the average was reported.

#### Air permeability measurement

The sample's ability to allow air flow was tested according to the BS 9237–1995 test method using an air permeability tester (MO 215, SDL Atlas, USA). The test surface area was 0.785 cm<sup>2</sup>, and the pressure drop was 100 Pa. 10 measurements were performed for each sample. The air permeability (R, in mm/s) was calculated according to Eq. (2).

$$R = q_{\nu}/A \times 167 \tag{2}$$

Here,  $q_{\nu}$  stands for the arithmetic mean of the air flow rate in cm<sup>3</sup>/m. The number 167 refers to the conversion factor from dm<sup>3</sup>/min.cm<sup>2</sup> to mm/s. *A* is the area of the samples in cm<sup>2</sup>. Ten replicates were tested for each sample.

#### Bending property measurement

The bending properties were measured according to the ASTM D 1388–96 (2002) test method using a Shirley fabric stiffness tester (Stiffness Tester, SDL, UK). The specimen bending rigidity (G) and bending modulus (q) were calculated according to Eqs. (3) and (4), respectively.

$$G = WC^3 \times 10^3 \tag{3}$$

$$q = \frac{12 \times G \times 10^{-6}}{d^3} \tag{4}$$

In Eq. (3), *C* refers to the bending length in cm, *W* stands for the weight per unit surface of the samples in  $g/cm^2$ , and *G* is the bending rigidity in mg.cm. In Eq. (4), *d* stands for the average thickness of the samples in cm, and *q* refers to the bending modulus of the samples in kg.cm<sup>2</sup>. Five replicates were tested for each sample.

#### Porosity analysis

The characterization of the porosity of the CNF-rGONP nanofibers was conducted using a sorptometer (BELSORP MINI II, BEL, Japan) at 77 K. The nitrogen adsorption/ desorption isotherms were analyzed according to the Barrett-Joyner-Halenda (BJH) and Brunauer–Emmett–Teller (BET) models, leading to the determination of the specific surface area, pore volume, and average pore size of the CNF-rGONP nanofibers.

## Thermal conductivity

A togmeter (IUT, IRAN) was used for determining the thermal conductivity of the samples according to ISO 5085-1:1989 (part 1, low thermal resistance). The togmeter comprises a forced-air cabinet, a hotplate having a circular form, and a cold plate which is designed to lie on the top of the sample with a pressure of 6.9 Pa over the whole area of 855 cm<sup>2</sup> to ensure good contact with the plates. Prior to testing, the 330-mm diameter specimens were conditioned at 20 °C and 65% RH for 24 h. The protocol described in ISO 5085-1:1989 (part 1, low thermal resistance) was applied to determine the thermal resistance ( $R_f$ ). The TC (k, in W/m K) was calculated according to Eq. (5) (Standardization, 1989).

$$k = \frac{d \times 10^{-3}}{R_f} \tag{5}$$

Here, *d* refer to the thickness of the specimen (mm) under a pressure of 6.9 Pa and  $R_f$  stands for the thermal resistance of the samples in m<sup>2</sup>.K/W. Three replicates were measured for each sample and the average was calculated.

## Thermal behavior

Thermal analyses of the samples were conducted using a differential scanning calorimetry analyzer (DSC, DSC 2010, TA Instrument, USA). The measurements were made using a Heat/Cool/Heat program between -10 to 50 °C, with a heating and cooling rate of 10 °C/min and an environment of 100%  $\rm N_2$  gas. The DSC diagrams were used to determine the temperatures of the phase change and the latent heat of solidification and melting of the samples. Three replicates were tested for each sample.

## Dynamic heat transfer

To predict the thermo-regulating performance of the substrate, a dynamic heat evaluation test method under simulated body conditions was used (Borhani et al., 2010). It involves a sweating guarded hot plate (model 431–213, MTNW, USA) used in dry mode inside an environmental chamber, two temperature sensors (SMT172, Smartec, Germany), and a data acquisition system (Arduino, NCD, USA). The conditions inside the environmental chamber were 20 °C and 65% RH. A schematic of the setup is shown in the supporting information (Additional file 1: Fig. S1). The guarded hot plate serves as source of heat and is equipped with a membrane to simulate human skin and sensors to determine the temperature. The fabric specimen is positioned at a distance of 5 mm above the membrane. Two temperature sensors measure the temperature on each face of the specimen. The variation of the temperature over time is captured by the data acquisition unit. The dynamic heat transfer of the samples is given by Eq. (6).

Heat transfer (%) = 
$$(\Delta T_1 / \Delta T_2) \times 100$$
 (6)

Here,  $\Delta T_1$  corresponds to the temperature difference on the upper face of the specimen between the beginning and the end of the experiment and  $\Delta T_2$  refers to the temperature difference between the upper and lower faces of the specimen at the beginning of the experiment. Five replicates were tested for each sample.

#### Abrasion resistance

The abrasion resistance of the samples was tested following ASTM D4966-89 using a Martindale Wear and Abrasion tester (Martindale 1440, James Heal, UK) and the standard wool abrader. The weight change, TC, and HT of the specimens after abrasion were assessed after 3000 cycles. Five replicates were tested for each sample.

#### Washing fastness

The resistance of the samples to repeated launderings was evaluated according to the AATCC test method 61(2A)-1996. Each washing was done using 1 mL/L of non-ionic detergent and 0.2 g/L sodium carbonate (pH 8–9), considering a 30:1 liquor-to-fabric ratio, at the temperature of 55 °C for 45 min. The drying process of the washed specimens was done at 50 °C for 30 min following each washing. The weight change of the washed specimens was determined after 1, 3, and 5 laundering cycles. The TC and the HT of the washed specimens were determined after 5 laundering cycles. Five replicates were tested for each sample.

#### Leakage test

Leakage tests were performed by placing the SSPCM-coated specimens on a filter paper and keeping them at 75  $^{\circ}$ C in an oven for 2, 12, 24, 48, and 72 h. An assessment was also done at time 0. The sign of PEG leakage on the filter paper was captured with a digital

camera. For comparison purposes, pure PEG was subjected to the same conditions and the staining of the filter paper recorded the same way.

#### Statistical analysis

When relevant, statistical analysis was performed with the SPSS 26.0 statistical package and carried out using one-way analysis of variance (ANOVA). A value of p < 0.05 was interpreted as an indication of statistically significant differences.

#### **Results and Discussion**

#### Porosity of the CNF-rGONP nanofibers

The porosity of the CNF-rGONP nanofibers was determined by analysing the N<sub>2</sub> absorbtion/desorption curves (Additional file 1: Fig. S2a) using two methods: BET and BJH. As shown in Additional file 1: Fig. S2a, the CNF-rGONP nanofibers display the shape of the IV characteristic adsorption isotherms typical of micro/mesoporous structures with the H<sub>4</sub> type hysteresis loop. This observation is in agreement with the pore diameter distribution results obtained (see Additional file 1: Fig. S2b). Additional file 1: Table S1 gives the surface area, pore volume and average pore diameter of the CNF-rGONP nanofibers. The CNF-rGONP nanofibers exhibited a specific surface area of 657.7 m<sup>2</sup> g<sup>-1</sup> and a total pore volume of 0.59 cm<sup>3</sup> g<sup>-1</sup>.

#### Surface characteristics

The sample surface morphology was characterized with a stereo microscope and a FESEM. Stereo microscope images were taken for uncoated, binder-coated, and SSPCM-coated specimens (S5, S15, and S25) (Additional file 1: Fig. S3a–e). The SSPCM-coated specimens displayed a coarser surface, thus indicating that the PEG/CNF-rGONP SSPCM finish was successfully applied onto the surface of the fabric. The surface roughness appears to increase with the increase in the SSPCM concentration in the impregnation solution. This observation can be related to the increasing sample thickness and weight per unit area recorded as the SSPCM concentration in the impregnation solution is increased (Table 2). Table 2 also provides the add-on weights for the coating (SSPCM+Vinyl acetate/acrylic resin+thickener) as well as the SSPCM add-on values for each sample.

FESEM images of the surface and cross-section of the binder-coated and SSPCMcoated specimens (S25) are shown in Additional file 1: Fig. S4. The presence of the

Table 2	Physical	properties	of	the	uncoated,	binder-coated,	and	SSPCM-coated	non-woven
polyeste	r substrat	e (standard	dev	iatior	n in parenth	esis)			

Sample	Mass per unit area (g/ m <sup>2</sup> )	Thickness (mm)	Coating add-on weight (wt%)	SSPCM add-on (wt%)	Air permeability (m/s)	Thermal conductivity (W/m K)
Uncoated	268 (14)	2.28 (0.03)	-	-	20 (1)	0.14 (0.02)
Binder-coated	298 (5)	2.31 (0.02)	7.30 (0.64)	-	10 (1)	0.13 (0.02)
S 5	306 (14)	2.32 (0.02)	12.48 (0.01)	4.58 (1.80)	9 (2)	0.19 (0.07)
S 15	335 (15)	2.39 (0.46)	19.99 (0.07)	13.69 (2.00)	2.7 (0.8)	0.22 (0.05)
S 25	371 (7)	2.44 (0.02)	27.85 (0.50)	22.17 (0.40)	2.7 (0.5)	0.74 (0.05)



Fig. 1 Stereo microscopic photographs of the upper surface of the a uncoated, b S5, and c S25 samples

Sample	Bending length (cm)	ΔL/L <sub>o</sub>	Bending rigidity (mg.cm)	Bending modulus (Kg cm <sup>2</sup> )		
Uncoated	5.1 (0.2)	_	3574.9 (0.1)	3.60 (0.03)		
Binder-coated	5.4 (0.2)	0.05	4450.6 (0.3)	4.3 (0.40)		
S 5	6.19 (0.09)	0.23	7260.69 (0.02)	6.98 (0.02)		
S 15	6.4 (0.3)	0.24	8574.0 (0.7)	7.49 (0.01)		
S 25	6.5 (0.2)	0.28	10,387.1 (0.4)	8.5 (0.30)		

**Table 3** Bending properties of the uncoated, binder-coated, and SSPCMs-coated non-woven polyester substrate (standard deviation in parenthesis)

coating can be observed over a depth of 200  $\mu$ m in the cross-section images of the binder-coated samples (Additional file 1: Fig. S4a). The fibers of the binder-coated samples (Additional file 1: Fig. S4b) show a smooth surface, which indicates that the binder mixture applied was able to create an even film on the fiber surface. For the SSPCM-coated samples, the coating penetration depth into the non-woven substrate is also around 200  $\mu$ m (Additional file 1: Fig. S4c). The surface of the SSPCM-coated fibers looks much rougher compared to the binder-coated samples (Additional file 1: Fig. S4d vs. Additional file 1: Fig. S4b). The interstices between the fibers also appear to be partially filled with the SSPCM coating applied.

#### Air permeability

Air permeability is a large contributor of fabric comfort. Table 2 shows the results of the measurement of the uncoated, binder-coated and SSPCM-coated sample air permeability. The binder-coated samples experienced a 50% reduction in air permeability compared with the untreated fabric. The air permeability kept decreasing with the addition of SSPCMs to the finish solution. The reduction was 56.5%, 86.2%, and 86.4% for S5, S15, and S25, respectively, compared with the untreated fabric. Previous research has shown the relation between air permeability and the porosity of fabric structures (Oğlakcıoğlu et al., 2021). The reduction in the fabric air flow measured can thus be attributed to the obstruction of the gaps between the fibres in the non-woven structure that can be observed in the stereo micrographs of the uncoated and coated samples in Fig. 1a–c.

#### **Bending properties**

Fabric flexibility also has a large effect on the functionality and comfort of textile products. The average bending length, bending rigidity, and bending modulus of the uncoated, binder-coated and SSPCM-coated samples are provided in Table 3. The binder-coated sample experienced an increase in the bending length (by 6%), rigidity (by 25%) and modulus (by 20%). A further significant increase (p=0.02) was obtained when SSPCMs were present in the impregnation solution. The bending length, rigidity and modulus of the S25 sample, with the highest incorporated amount of SSPCMs, increased by 20%, 133%, and 98%, respectively. The changes in the bending length for the coated samples compared to the value for the uncoated fabric ( $\Delta L/L_o$ ) are also provided in Table 3. The changes in bending stiffness values ( $\Delta L/L_o$ ) of the SSPCM-coated fabrics was less than 1 (0.23–0.28). The sample coated with the highest amount of SSPCMs (S25) could be bent, creased, and twisted without any issues, similar to the uncoated fabric. Therefore, it is estimated that the SSPCM-coated specimens prepared have acceptable formability/flexibility properties for a wide variety of applications such as protective clothing, wall covers, transportation vehicles, and packaging.

## Thermal performances of the SSPCM-coated samples

In this study, the thermal properties of the prepared SSPCM-coated fabrics were evaluated. They included the phase transition temperature range, latent heat enthalpy, thermal conductivity (TC), heat transfer (HT), and thermal energy storage performance.

## Thermal conductivity

The TC of organic PCMs has a large effect on their energy storage and discharge rate. The low TC of PEG is one of the main issues still limiting its wide range application. The presence of the binder and polyester nonwoven fabric, both materials with a low thermal conductivity, also has a negative effect on the thermal properties. The TC of the samples was measured using a togmeter. The values of TC of the uncoated, binder-coated, S5, S15, and S25 were  $0.14 \pm 0.02$ ,  $0.13 \pm 0.02$ ,  $0.19 \pm 0.07$ ,  $0.22 \pm 0.05$ , and  $0.74 \pm 0.05$  W/m K, respectively (Table 2). The TC strongly increased with the added SSPCM. Larger concentrations of SSPCMs in the coating led to higher values of TC. In particular, with a value of 0.74 W/m K, the S25 samples exhibited a TC more than three times higher compared to the S5 sample. This result can be attributed to the high TC of the CNF-rGONP (2.390 W/m K) (Zeighampour et al., 2022b).

CNF-rGONPs in a 50–50 wt. ratio thus seems a promising solution to increase the TC of the PCMs when applied to fabrics. This interesting performance can be attributed to the synergistic effect observed in hybrid carbon materials. Since the geometry and size of pores play a role in molecular thermal mobility, researchers have shown that carbon supporting materials with various sizes of pores and 1D, 2D, and 3D geometric structures result in higher TC in SSPCMs compared to metal and metal oxide matrices (Amaral et al., 2017; Shah, 2018). Large pores in porous supporting materials result in high energy storage capacity in SSPCMs (Umair et al., 2019). Meanwhile, adsorption in smaller pores by capillary forces preserves the shape of the PCM during the phase change process, thus preventing it from leaking; however, it limits the movement of the

chains of the PCM and prevents it from being crystallized. Therefore, it is expected that the use of carbon porous materials consisting of a set of diverse structures (1D, 2D, and 3D) and pores with micro, meso and macro sizes can result in excellent energy storage performance due to the synergistic effect (Dutta et al., 2014). Other examples of hybrid carbon materials with similar synergetic effect on TC found in the literature include multi-walled carbon nanotubes/graphene nanoplates (MWNTs/GNPs) (Jen et al., 2020), porous graphene foam/carbon nanotubes (GCNTs) (Yu et al., 2021), and expanded graphite/carbon nanofiber (EG/CNF) (Qu et al., 2020).

TC values reported in the literature for TC-enhanced SSPCMs applied on fabrics ranged between 0.13 and 0.15 (Rezaie & Montazer, 2018, 2019, 2020). The combination of 1D CNFs and 2D rGONPs thus appears to lead to much higher values of TC ( $0.74 \pm 0.05$  W/m K).

## Phase change temperature and energy storage capacity

The DSC curves for the binder-coated and SSPCM-coated (S5, S15, and S25) samples are shown in Fig. 2a and b. The binder-coated samples display no phase transition. This indicates that the vinyl acetate acrylic resin used here as a binder to fix in a durable manner the SSPCMs on the polyester fabric does not have any negative impact on the thermal properties of the SSPCMs-coated samples.



Fig. 2 Examples of typical DSC curves measured for the binder-coated, S5, S15, and S25 samples: a crystallization and b melting

On the other hand, the DSC curves for the SSPCM-coated samples show well defined melting and solidification peaks. The properties extracted from the DSC curves for the SSPCM-coated samples are shown in Additional file 1: Table S2. Additional file 1: Table S2 also includes the melting and solidification latent heat and temperature of PEG ( $\Delta H_m = 192 \text{ J/g}$ ,  $\Delta H_c = 141.6 \text{ J/g}$ , with melting and solidification temperature of 31.7 and 19.9 °C). The melting temperatures of samples S5, S15 and S25 (between 30.1 and 31.4 °C) are slightly below the temperature of the skin and their solidification temperatures (between 19.2 and 24.1 °C) are around room temperature. These values are similar to that of PEG and make the PEG/CNF-rGONP composite system suitable for applications in the low-temperature range.

As seen in Additional file 1: Table S2, the SSPCM-coated samples experienced a reduction in the melting and solidification latent heat with values of 8.9–22.9 and 11.2–21.4 J/g, respectively, compared to PEG alone (melting and solidification enthalpies of 192.0 J/g and 141.6 J/g, respectively). The high latent heat values of PEG can be attributed to its linear polymeric chain  $(CH_2-CH_2-O)_n$  with two hydroxyl groups on both ends and strong hydrogen bindings. Therefore, it can easily crystallize with a high enthalpy due to its simple structure. The lower values of latent heat for the the SSPCM-coated samples can be attributed to the fact that the addition of CNF and rGONPs to PEG, less material is available for the phase transition. In addition, with their small pore size, the rGONPs may have acted as an impurity and hindered PEG crystallization process, thereby reducing the enthalpy of the PCM. These findings align with previously reported data on nano-enhanced organic PCMs (Leong et al., 2019; Zeighampour et al., 2022b). The presence of the nonwoven PET fabric also negatively affected the measured latent heat.

In comparison with other works on PCMs applied to fabrics found in the literature, the values of latent heat obtained in the present study are similar to the best results reported (Table 4). When comparing the three series of SSPCM samples, an increase in the melting and solidification latent heat is obtained at a higher loading of SSPCM (Additional

Fabric	PCM compound	Supporting material	SSPCM add-on weight (wt%)	Melting temperature (°C)	Melting latent heat (J/g)	References	
Polyester knit- ted fabric	n-Octadecane	Melamine–for- maldehyde	40	29.4	7.4	(Choi et al., 2004)	
Polyester knit- ted fabric	n-Octadecane	Melamine–for- maldehyde	8.8	29.1	13.5	(Chung & Cho, 2004)	
Polyester/cot- ton (40/60)	Paraffin	Styrene	18.9	31.6	14.3	(Sánchez-Silva et al., 2012)	
Cotton/poly- propylene (60/40)	n-Octadecane	Melamine– urea–formal- dehyde	18	30.1	15.7	(Xing-xiang et al., 2005)	
Nonwoven polyester	PEG	CNF-rGONP (50:50 wt%)	22.2	31.4	22.9	Present study (S25)	
Cotton	n-Hexa- decane, n-eicosane	Melamine-for- maldehyde	30	30	23.03	(Salaün et al., 2010)	

Table 4	Comparison	of the	e melting	latent	heat	of	SSPCM-coated	sample	(S25)	with	the	other
results fro	om the literat	ure										

file 1: Table S2). With the addition of CNFs-rGONPs to the PCM compound, the current solution has the major advantage of an improved TC, thus a higher response rate.

#### Heat transfer and thermal energy storage performance of the SSPCM-coated samples

The results provided by the dynamic heat transfer measurement in terms of HT computed using Eq. 6 are reported for the uncoated, binder-coated, and SSPCM samples. The HT of the uncoated, binder-coated, S5, S15, and S25 were  $60.86 \pm 0.28$ ,  $59.13 \pm 0.11$ ,  $63.82 \pm 0.25$ ,  $74.65 \pm 0.17$ , and  $80.49 \pm 0.20\%$ , respectively. While the values of HT for the uncoated, binder-coated, and S5 samples were similar (p=0.06), a significant increase in HT was measured for sample S15 (p=0.03) and S25 (p=0.04). This larger value of HT for S15 and S25 can potentially be attributed to the higher TC at higher SSPCM concentration in the fabric coating. These results are in agreement with the findings of a previous study on carbon-based SSPCM (Zeighampour et al., 2022b).

Dynamic heat transfer was also used to assess the thermal storage and release behavior of the PEG/CNF-rGONP composite coating on the polyester non-woven fabric. Figure 3 shows the melting and crystallization temperature profiles measured by the thermocouple on the side of the specimen facing the hot plate for the PEG, binder-coated, and S25 samples. In both instances, the curves corresponding to the PEG and S25 samples show a two step-process, which can be attributed to the heat storage/release by the PCM material, while the melting and crystallization curves for the binder-coated samples display a



Fig. 3 Temperature variation curves of binder-coated, and S25 in the a heat storage and b release process

monotonous trend. The effect of the heat storage/release by the PCM material can also be observed on the heating/cooling rate. While it took 274 s for the fabric temperature to go from 10 to 45 °C in the case of the binder-coated samples, this time increased to 630 s with the SSPCM sample S25 (Fig. 3a). A similar trend was observed during cooling even though the difference between the binder-coated and S25 samples was not as large (Fig. 3b). The delay in the heating/cooling provided by the SSPCM-coated fabric gives the opportunity to the human body or building environments to adapt in the case of a rapid change in temperature (Shi & Li, 2020). Figure 3 also shows that the delay in cooling and heating further increased for pure PEG. This result can be attributed to the larger value of the melting/crystallization latent heat for pure PEG compared to the PEG/CNF-rGONP system (Additional file 1: Table S2).

## Effect of service conditions

Fastness properties are of important criteria that determine the practical application of materials according to their end-use. In this study, the effect of abrasion and laundering was assessed on the specimen weight, HT and TC.

#### Abrasion resistance

The residual performance of the uncoated, binder-coated and SSPCM-coated samples was assessed after 3000 cycles applied using the Martindale abrader. The results are shown in Additional file 1: Table S3. The weight loss for the binder-coated and SSPCM-coated samples was less than 3 wt%. By comparison, a larger weight loss was observed with the uncoated samples. This difference in terms of weight loss between the uncoated and coated samples can be attributed to a protective effect provided to the non-woven structure of the fabric by the binder. Indeed, in the uncoated samples, the fibers are only held together by mechanical entanglement and thus prone to the damaging effect of abrasion. Stereo microscope images of the uncoated, binder-coated and SSPCM-coated samples after abrasion are displayed in Fig. 4. Freestanding fibres can be seen protruding



Fig. 4 Stereo microscopic photographs of the upper surface of the **a** uncoated, **b** binder-coated, **c** S5, **d** S15, and **e** S25 after 3000 abrasion cycles

from the surface of the abraded uncoated specimen (Fig. 4a). The presence of PEG may also have contributed to lowering the effect of abrasion of the coated fabric as PEG can act as a plasticizer and improve the abrasion resistance of substrates (Valentini et al., 2022).

The TC and HT values for the SSPCM-coated samples were in the range of 0.18–0.68 W/m K and 60.9–76.4% after 5 washing tests, respectively (Additional file 1: Table S3); the reduction was about 5% for HT and below 8% for TC. This reduction in HT and TC after abrasion could be due to the partial removal of the SSPCMs from the sample surface.

The results in Additional file 1: Table S3 also that the weight loss upon abrasion increased as the amount of SSPCM applied on the fabric increased from 1.17 to 2.12%. A similar trend is observed on the HT and TC values, with the largest effect obtained with sample S25 (Fig. 5). As the same amount of binder was used for all samples irrespective to the amount to SSPCM incorporated, this may indicate that a larger amount of binder may be needed for the highest SSPCM concentrations.

#### Washing fastness

The sample washing fastness was evaluated after 1, 3 and 5 laundering cycles. The results are shown in Additional file 1: Table S4. An increase in the weight loss with the number of cycles can be observed in all instances with the exception of the uncoated samples. The effect is stronger in the presence of CNFs-rGONPs in the coating as well as with the increase in the amount of SSPCM coating on the fabric. This indicates that the fabric



Fig. 5 Change in **a** TC and **b** HT for the SSPCM-coated samples after 3000 abrasion cycles and 5 washing cycles



Fig. 6 Result of leakage test at different times with sample S25 (first row) and pure PEG (second row) maintained at 75  $^\circ C$ 

itself has a good resistance to repeated launderings; the effectobserved is possibly caused by the sensitivity of the binder to the damaging action of the laundering process. In addition, as the same amount of binder was used for all samples irrespective to the amount to SSPCM incorporated, this may indicate that a larger amount of binder may be needed for the highest SSPCM concentrations. However, the weight loss experienced by sample S25 after five laundering cycles was below 5%.

A reduction in the TC and HC values after five laundering cycles was also observed (Additional file 1: Table S4). TC and HT values varied from 0.17 to 0.67 W/m K and 60.5 to 73.5% after 5 washing tests, respectively. This effect can be directly attributed to the loss of SSPCM matter due to laundering evidenced by the change in weight of the specimens. The reduction in TC and HC was more pronounced for Sample S25. However, the changes remained lower than 10% (Fig. 5).

## Efficiency of shape stabilization

The leakage test was performed to assess the efficiency of the shape-stabilizing function provided by the CNF-rGONP composite structure at containing PEG upon melting. Figure 6 shows the images recorded by the digital camera for sample S25 at different times for up to 72 h at 75 °C. The first row in Fig. 6 display the front side of sample S25. No sign of leakage is seen for up to 72 h on the filter paper in contact with sample S25. On the other hand, heavy staining of a filter paper is observed for a drop of pure PEG deposited on the filter paper at the beginning of the test. The absence of leakage with the SSPCM samples could be due to intermolecular and capillary forces associated the porous CNF-rGONP structure (Zeighampour et al., 2022b).

## Conclusions

A novel thermo-responsive shape-stable nanocomposite PCMs is proposed to overcome two main concerns with PEG as a PCM material, namely its low TC and the presence of leakage. It takes advantage of a porous composite structure formed by combining CNF with rGONPs. The fabricated SSPCM-coated fabric has an increased TC without PCM leakage during the thermal cycles, which would be essential for practical thermal energy storage use. A suitable phase transition temperature, proper energy storage capacity, desirable thermal endurance, and leakage-proof property are the other advantages of the prepared samples. A method was also developed to apply the PEG/CNF-rGONP on a non-woven PET by impregnation, coating, and dry-fixation using a vinyl acetate acrylic resin as a binder. This process could advantageously be employed to manufacture this SSPCM fabric at an industrial scale without any changes in production layout.

The results show that the vinyl acetate acrylic resin provides acceptable laundering and abrasion durability with no adverse impact on the thermal properties of the SSPCM-coated fabric. Samples prepared with different concentrations of the SSPCM in the coating display phase change temperatures in the thermophysiological range with energy storage capacities similar to the best performing solutions found in the literatures. However, the major advantages of the new PEG/CNF-rGONP non-woven fabric are its high TC, more than four times the best values reported for other SSPCM solutions applied to fabrics, and its high leakage resistance, with no sign of PEG leakage observed after 72 h at 75 °C. In addition, the SSPCM-coated fabric retained an acceptable level of bending flexibility (change in bending length less than 30%).

The results thus show that the incorporation of PEG into a CNF-rGONP (50:50 wt%) supporting matrices offers a promising thermoregulating, dynamic insulation, and energy storage solution for a wide variety of applications. This includes protective clothing and consumer apparel, buildings, transportation vehicles, packaging, electronics, and energy harvesting devices.

#### Abbreviations

PCMs	Phase change materials
PET	Polyester
PEG	Polyethylene glycol
CNFs	Carbon nanofibers
rGONP	Reduced graphite oxide nanoparticles
SSPCMs	Shape-stable phase-change materials
TC	Thermal conductivity
HT	Heat transfer
FESEM	Field emission scanning electron microscope
DSC	Differential scanning calorimetry
$W_a$	Weight of specimen before coating (g)
$W_b$	Weight of specimen after coating (g)
$q_v$	Air flow rate (L/m)
Α	Area (cm <sup>2</sup> )
G	Bending rigidity (mg cm)
9	Bending modulus (Kg cm²)
С	Bending length (cm)
W	Weight per unit area (g/cm²)
d	Thickness (cm and mm)
k	Thermal conductivity (W/m K)
$R_{f}$	Thermal resistance (m <sup>2</sup> K/W)
$\Delta T_1$	Temperature difference on the upper face of the sample between the beginning and the end of the experiment
$\Delta T_2$	Temperature difference between the upper and lower faces of the sample at the beginning of the
	experiment.
VV <sub>0</sub>	Initial weight (g)
W <sub>f</sub>	Final weight (g)
$\Delta L/L_{o}$	Bending stiffness values

#### **Supplementary Information**

The online version contains supplementary material available at https://doi.org/10.1186/s40691-023-00363-7.

Additional file 1: Fig. S1. Schematic representation of the setup used to measure the dynamic heat transfer. Fig. S2. (a) N<sub>2</sub> adsorption/desorption isotherm, and (b) the corresponding pore diameter distribution of CNF-rGONP nanofibers. Fig. S3. Stereo microscopic photographs of the upper surface of the (a) uncoated, (b) binder-coated, (c) S5, (d) S15, and (e) S25. Fig. S4. FESEM photographs of the binder-coated sample and S25 fabric sample: (a and c) cross-section (100 and 250 x magnification), (b and d) upper surface (1, 5, and 60 kx magnification). Table S1. Characteristics in terms of porosity of the CNF-rGONP nanofibers. Table S2. Latent heat and characteristic temperature

extracted from the DSC curves for PEG and S5, S15, and S25 samples (standard deviation in parenthesis). **Table S3.** Abrasion resistance of the uncoated, binder-coated, SSPCM-coated samples after 3000 abrasion cycles (standard deviation in parenthesis). **Table S4.** Washing fastness of the uncoated, binder-coated, and SSPCM-coated samples after 1, 3, and 5 laundering cycles (standard deviation in parenthesis).

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#### Authors' contributions

FZ: Conceptualization, Methodology, Formal analysis, Writing—original draft, Investigation. AKh: Supervision, Resources, Conceptualization, Validation, Writing—review & editing. PID: Conceptualization, Visualization, Writing—review & editing.

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#### Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

#### Declarations

#### Ethics approval and consent to participate

Not applicable.

#### **Competing interests**

The authors declare that they have no competing interests.

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