

# Fabrication of Phase Change Material Functionalized Regenerated Cellulosic fibres with Thermoregulation Capability for Textile Applications

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**Abstract** - While the viscose fibre properties are suitable for general use, there is scope for improving their thermal management properties in order to actively provide temperature regulation based on the ambient conditions. Functional modification has been done onto viscose by incorporation of phase-change materials (PCMs) into viscose fiber in order to develop thermal regulation capability. The organic compound-based PCMs in two forms viz. as-is (octadecane, lauryl alcohol etc.) and microencapsulated materials (mPCMs) were evaluated in this work. Viscose fibres containing these PCMs were prepared by the regular viscose fibre spinning process. DSC & SEM analysis of these fibres showed that the added PCM compounds had survived the process steps of viscose fibre manufacturing. Dynamic temperature profile by thermal imaging showed that microencapsulated mPCMs displayed more intense cooling effect than the lab emulsified PCM additives. It was shown that, based upon type of phase-change compound added into viscose polymer, thermoregulating viscose fibre with distinct transition temperatures can be prepared which could be utilized for different end-use commercial applications.

**Key Words:** Viscose, PCM, Textiles, Dynamic thermoregulation, Cooling

## 1. INTRODUCTION

Phase-change materials (PCMs) are inorganic or organic compounds which store and release latent heat through a phase transformation i.e. going from a solid to a liquid and vice versa [1]. PCMs are widely used for thermal energy storage in areas such as building insulation due to their ability to effect large heat transfers at near constant temperature. Some of these PCMs change phase in a temperature range close to human skin temperature, making them suitable for use in textile apparel & clothing. The technology for incorporating PCM into textiles was first developed in the early 1980s under a NASA research programme for use in astronauts' space suits & gloves to reduce the impact of extreme temperature fluctuations in outer space. After that fibres & textiles began to be designed in order to produce garments intended to moderate skin temperature variation when wearer experiences varying levels of activity [2]. In recent times such PCM containing textiles have found widespread use in form of various consumer products ranging from t-shirts, undergarments, ski jackets & shoes to blankets, pillows & quilts.

Several types of PCM additives are available & can be roughly grouped [3, 4] as per their chemical nature into following categories - i) Hydrated inorganic salts (sodium carbonate decahydrate, calcium chloride hexahydrate); ii) Fatty acid esters (methyl stearate, glycerol trimyristate, myristyl myristate, erythritol tetrastearate); iii) Linear long chain hydrocarbons/paraffins (n-hexadecane, n-heptadecane, n-octadecane, eicosane); iv) PEGs of different chain length with molecular weight from 400 to 4000; v) Others (dodecyl carbonate, glycerin, lauryl alcohol). In few cases eutectic mixtures of above compounds are also applied in textiles [5, 6]. Often many of the above mentioned compounds are microencapsulated in a core surrounded by polymeric shell material such as acrylate [7]. The microencapsulated PCMs (mPCMs) are commercially available from various suppliers in slurry form with different active content. The mPCMs are generally used for developing PCM textiles via surface coating route, however their use by direct inclusion within the fibre is not widely reported.

### 1.1 Principle of skin micro-temperature management by use of PCM in textiles

The ability of PCMs to latently accumulate energy when changing phases can help regulate the skin microclimate [2, 3]. When the wearer's temperature increases due to either higher ambient temperature or a warmer body, the PCM absorbs heat via melting endotherm as it moves from a solid state to a liquid state. This phase change produces a temporary cooling effect. Also the reverse transition occurs when the ambient temperature drops i.e. the PCM releases heat via crystallization exotherm as the molten PCM goes back to its crystallized form. Thus when wearer is subjected to colder ambient conditions, the PCM textile generates a temporary warming effect. The thermal regulation effect of the PCM lasts only for the duration of phase change and is no longer available when the phase change of all the PCM inside the garment is complete. This type of temporary effect can therefore be called dynamic thermoregulation [2].

Factors affecting the level of thermo-regulating function offered by the PCM textiles mainly include the heat storage capacity of the PCM and their applied quantity [8-10]. The textile substrate construction (woven versus knitted, thickness, density, number of layers etc.) is another major parameter which affects the efficiency of the active thermal regulation effect of the PCM. The garment must be so

designed that its use temperature falls within the phase transition range of the PCM used in it. Also there must be sufficient quantity of the PCM in the garment in order to regulate skin temperature. Thus a specific design approach is necessary to get the maximum benefit out of a textile apparel infused with PCM [11-15]. For instance, the amount of air trapped between the outermost clothing layer & the wearer's skin significantly impacts the moisture transport away from body which is a significant factor in wearer comfort.

Regenerated cellulosic fibres such as viscose are used in diverse applications such as garments (men's trousers, women's t-shirts & leggings) and others (home textiles, sportswear & innerwear). The phase-transition temperature of PCM additive must be defined by the end-use of the fibre. Such fibres can be intended to give either a cooling or a warming effect or may provide both the effects in a specified temperature range. For clothing applications, in a very broad sense, the melting heat-absorbing temperature interval can be from 25°C to 40°C and the crystallization heat-releasing temperature interval can be from 24°C to 10°C. Currently there are commercial PCM-based apparel offering distinct peak temperatures of melt/crystallization transitions at 33°C / 27°C or 27°C / 22°C respectively. The phase change enthalpy (i.e. latent heat storage capacity) of the PCM fibres must be sufficiently high e.g. in the range of 9-13 J/g in order to obtain a satisfactory effect at the final garment stage, keeping in mind the loss of said additives during conversion of PCM fibre to garment.

**1.2 Performance evaluation of PCM-based textiles**

While evaluating the performance of thermoregulating textiles, phase change technology presents unique challenges to the standard testing procedures used for determining the thermal characteristics of traditional fabrics (no energy stored in the fabric). Even though a garment may contain PCM but it still might not differentiate itself in human

physiological tests. Standard test methods for characterizing phase change textiles include ASTM-D1518 and KES-F7 ThermoLabo-II (qmax). These steady-state tests may not completely capture the distinction of thermal performance between phase-change and non-phase-change fabrics. While techniques for determining the static thermal comfort properties of clothing are well developed, methods for evaluation of their dynamic thermal comfort properties are much less advanced. Several test protocols and apparatus have been proposed to quantify the dynamic thermoregulatory characteristics of fabrics containing phase-change materials. Table 1 lists the methods developed & reported in literature so far, along with their primary output indices [16-20].

**1.3 Application areas for PCM in textiles**

There are different product segments in which 'smart' thermoregulating textiles are available such as outdoor jackets, ski boot linings, vehicle interiors, underwear, socks, gloves, sportswear, beddings/quilts, expedition grade garments (to provide protection against extreme colds in base layer or insulation layer), mattresses, blankets, pillows, bed linen & so on. Commercial smart textile products are observed to be primarily made from polyester, nylon & elastane in different blend ratios. A cooling effect of -0.3 °C to -2.5 °C is claimed in these products. Cell Solution CLIMA™ and smartcel™ by Cellsolution & Nanonic respectively are lyocell-based PCM fibres [21, 22]. However, to our knowledge, viscose based PCM containing fibres are not widely reported except Kelheim's Outlast® Universe using Outlast technology [23].

In the present article several types of phase-change materials (PCMs) are incorporated into viscose fibre with an objective of investigating their influence on the thermoregulation capability of the PCM textiles made therefrom.

**Table -1:** Test methods developed for comparing thermal comfort properties of PCM textiles to non-PCM textiles

S. no.	Method description	Reference	Key Indices	Apparatus
1.	Dynamic heat transfer measurements	16, 17 (Pause)	Dynamic Thermal Resistance	Custom panel heater/cooler with temperature sensor
2.	Measurement of thermal regulating factor	18 (Hittle & Andre')	TRF	Textile regulation apparatus
3.	Measurement of the thermal transient process	19 (Ying et al.)	static thermal insulation ( $I_s$ ), phase change duration ( $\Delta t_d$ ), mean of the heat flux ( $I_d$ ) and thermal psychosensory intensity (TPI)	Fabric Intelligent Hand Tester (FIHT)
4.	Transient change of the temperature of hot plate surface (which simulates the skin) & the heat flux through the hot plate surface	20 (Wan Fan) &	final 'skin' temperature difference ( $T_p$ ), duration of the phase change ( $t_d$ ) and average reduced heat loss due to the phase change (dH)	Guarded hot plate system

## 2. MATERIALS & METHODS

### 2.1 Ingredients

Lauryl alcohol and n-octadecane, laboratory reagent grade, were purchased from Lobachemie (India) & Alfa Aesar

(India) respectively. The microencapsulated additives mPCM CN-1 & mPCM Benochem were supplied by Beijing YOTO E. S. Tech. Co. Ltd. (China) and Shanghai Benochem Co. Ltd. (China) respectively. And mPCM T32RF was sourced from J&C Microchem Inc. (South Korea). Table 2 lists the properties of PCM additives used in this work.

**Table -2:** Additives explored as options of PCM inclusion in viscose fibre

Additive name	Typical Characteristics	Chemical formula	Solubility
n-octadecane	Alkane hydrocarbon, white crystalline solid	C <sub>18</sub> H <sub>38</sub>	Insoluble in water
Lauryl Alcohol	Colourless liquid with floral odor	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>10</sub> CH <sub>2</sub> OH	Insoluble in water
mPCM T32RF	Emulsion of microencapsulated material (capsule size ~ 1-2 μm)	Shell: Melamine plastic Core: nonadecane wax mixture Active content: ~ 30%	Dispersible in water
mPCM CN-1	Emulsion of microencapsulated material (capsule size ~ 1-2 μm)	Shell: Acrylate Core: octadecane Active content: ~ 40%	Dispersible in water
mPCM Benochem	Emulsion of microencapsulated material (capsule size ~ 3-4 μm)	Shell: Acrylate Core: paraffin wax Active content: ~ 30%	Dispersible in water

### 2.2 Methods

#### 2.2.1 Emulsification

The water insoluble PCM additives e.g. octadecane must be converted into an oil-in-water emulsion to facilitate their addition into viscose. The additive was melted and then added to a dispersant solution (water + non-ionic surfactant) under high speed stirring at 10-15 thousand rpm for 5 minutes at a temperature of 60°C. The dispersion thus obtained was subjected to ultrasonication for 12 minutes in a probe ultrasonicator & then cooled to obtain the final stable dispersion. The average particle size of thus obtained dispersion was measured by HELOS particle size analyser & found to be 2-3 μm.

#### 2.2.2 Fibre formation

Spinning of viscose staple fibres without & with PCM additives was carried out via a standard regenerated cellulosic fibre formation route i.e. extrusion of viscose dope (cellulose solution) through fine capillaries into an acid regeneration bath and stretching the resulting filament bundle. In the next step the filament bundle was cut into 'staple fibre' of 38±2 mm length. Staple fibre thus obtained was then washed in several after-treatment washing steps to obtain finished viscose fibres of two types, regular (non-PCM) and modified (PCM) fibres.

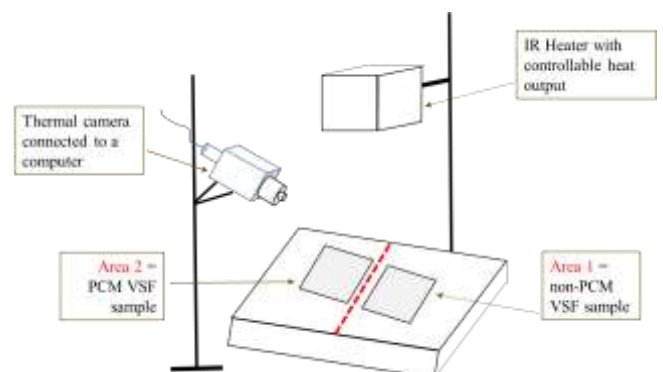
#### 2.2.3 Differential Scanning Calorimeter (DSC) study

Measurements of phase change temperature and energy storage capacity (enthalpy) of different materials employed were performed in a differential scanning calorimeter model DSC Q200 V24.11 of TA Instruments.

Measurements were done at temperature 0-200°C with a heating & cooling rate of 10°C/min.

#### 2.2.4 Dynamic temperature profile by infrared thermography

An in-house test method was developed, based on the principle of infrared thermal imaging, to evaluate the performance of PCM functionalized viscose fibres (Fig. 1). The thermoregulating textile samples were examined with a thermal camera Micro-Epsilon TIM<sup>2</sup> while being heated by an IR lamp operating at stabilized conditions in a controlled environment (23±2°C, 63±5 %RH). The camera is calibrated with the help of software provided along with it. Images were downloaded using *TIM Connect* software for analysis.



**Fig -1:** Schematic of infrared thermal imaging based dynamic temperature profile test apparatus

The test specimen of equal weight are taken (10x10 cm<sup>2</sup> carded fibre web, each of PCM & non-PCM sample) and then kept for at least 24 hours in the conditioned room. Both

samples are brought under an IR lamp to heat them simultaneously & changes in their temperature are recorded with the thermal imaging camera as a function of time (maximum temperature in a focused area of 5x5 cm<sup>2</sup>). The temperature vs. time data profile was exported to a data processing software.

The difference in temperature  $\Delta T$  between the PCM & non-PCM samples was calculated as below:

$$\Delta T = \text{Area 2 maximum temperature (PCM sample)} - \text{Area 1 maximum temperature (non-PCM sample)}$$

The  $\Delta T$  was calculated either as an average over a given time period of test (e.g. from 46-90 seconds) or as a single point value at a given instant of the test. The mean & instantaneous temperature difference were defined as  $\Delta T_{av}$  &  $\Delta T$  respectively in this work.

The measurement error of thermal profile test was determined by observing the temperature difference  $\Delta T$  when the same sample has been kept in area 1 and area 2 of the test apparatus. This  $\Delta T$  was termed baseline error of the test since ideally the  $\Delta T$ , when same sample is tested in areas 1 & 2, should be equal to zero. The measurement error of

mean temperature difference  $\Delta T_{av}$  and instant temperature difference  $\Delta T$  was determined as  $\pm 0.3^\circ\text{C}$  &  $\pm 0.4^\circ\text{C}$  respectively.

### 2.2.5 SEM study

The scanning electron microscope imaging of fibers was done with Phenom XL Scanning Electron Microscope at a working potential of 5-10 kV.

## 3. RESULTS & DISCUSSION

### 3.1 Thermal properties of PCMs & viscose staple fibre (VSF) made therefrom

The thermal properties of different PCM compounds used in this study were evaluated by DSC at scan rate of 10°C per minute. The results (average of 2 tests) are shown in Table 3. All of these PCM additives exhibit phase transitions in a temperature range which is suitable for textile applications of viscose fiber as discussed in section 1. Notably the commercial encapsulated PCMs (mPCMs) have lower enthalpies when compared with non-encapsulated PCM chemicals.

**Table -3:** DSC thermal transitions of as-received PCM additives

PCM name	Melting			Crystallization		
	T <sub>onset</sub> (°C)	T <sub>peak</sub> (°C)	ΔH <sub>m</sub> (J/g)	T <sub>peak</sub> (°C)	T <sub>onset</sub> (°C)	ΔH <sub>c</sub> (J/g)
n-octadecane	28	34	221	25	21	217
Lauryl alcohol	23	28	205	17	15	190
mPCM CN-1 <sup>^</sup>	25	29	72	24 & 13	22 & 10	45 & 9
mPCM Benochem	31	33	28	30	28	20
mPCM T32RF	28	42	28	36	33	31

<sup>^</sup> shows two crystallization peaks

Viscose staple fibres were made from all of the above compounds by adding them into the viscose polymer before fiber formation. After fibre spinning the PCM and regular VSF samples were conditioned (23±2 °C, 63±5 %RH) for 24 hours & then their thermal behaviour was analysed by DSC. The results shown in Table 4 are average of 3 tests on different samples (% indicates the loading of the additive on weight of fibre).

From Table 4 it can be inferred that the PCM additives are physically entrapped in the finished viscose fibres (there are

no DSC peaks for non-PCM regular viscose fibre). The PCM viscose fibre's melting and crystallization enthalpies (ΔH<sub>m</sub> & ΔH<sub>c</sub>) are, expectedly, lowered relative to the corresponding PCM compound (Table 4). The latent heat storage capacity of 10-14 Joules per gram of viscose fiber is satisfactory for its intended applications and is at par with the Outlast® fibre. Exceptions to this are the additives lauryl alcohol, mPCM Benochem & mPCM T32RF which possess enthalpies as low as 1.8 Joules per gram.

**Table -4:** DSC thermal transitions of VSF made with PCM additives

VSF name	Melting			Crystallization		
	T <sub>onset</sub> (°C)	T <sub>peak</sub> (°C)	ΔH <sub>m</sub> (J/g)	T <sub>peak</sub> (°C)	T <sub>onset</sub> (°C)	ΔH <sub>c</sub> (J/g)
Regular (non-PCM)	-	-	-	-	-	-
10% Octadecane	26	28	13	20	13	11.5
10% Lauryl alcohol	19	22	7.7	10	7	7.7
20% mPCM CN-1	25	27	10	18	13	10

20% mPCM Benochem	30	32	2	27	23	1.8
15% mPCM T32RF	33	36	1.9	28	24	1.9
Outlast® #	25	27	10.3	24	22	10.4

# commercial viscose fibre with PCM technology

A textile garment worn in tropical climates is desired to give warming effect at around 20-22°C in typical use situations e.g. user entering into an air-conditioned room from outdoors. On the other hand cooling effect must be provided at around 27-30°C. The commercial PCM viscose fibre (Outlast®) shows peak melting & freezing temperatures of 27°C and 22°C respectively. Thus the difference between peak melt & freeze temperatures for Outlast® fibre is 5°C, however this difference is as large as 15°C for the viscose fibres evaluated in this study. As a consequence, the freeze temperatures of few PCM fibres prepared in this work are too low (7 to 13°C) for practical use. Although the peak crystallization temperature is higher for mPCM Benochem & mPCM T32RF fibres, their melt transition also occurs at higher temperature. Thus it can be said that two different types of PCM viscose fibres were obtained- the first type (octadecane and mPCM CN-1) which show melt transition at 27-28°C but have very low freezing transition of 13°C & the second type (mPCM Benochem & mPCM T32RF) with freezing transition at 23-24°C but melting at higher temperatures 32-36°C.

Furthermore, since the lauryl alcohol fibre showed very low melt & crystallization temperatures which are useful neither for cooling nor warming application, it was excluded from further evaluation in this work.

### 3.2 Dynamic temperature profile of PCM VSF

The performance of thermoregulating textiles was evaluated by temperature profiling test method described in section 2.2.4. The fibres were tested in the form of carded web and results (average of 2 tests) are listed in Tables 5 & 6.

It can be inferred from Table 5 that PCM viscose fibres exhibit a slower rate of heating compared to the non-PCM viscose, resulting in large negative  $\Delta T_{av}$  values at different time intervals. The externally supplied heat energy during the test is partially used for melting of the PCM contained within the fiber, thus the rate of temperature rise of the PCM viscose is slower than a non-PCM viscose fibre. The Outlast VSF maintains a  $\Delta T_{av} \leq -1^\circ\text{C}$  even up to the later stages of test (91-150 seconds), whereas VSF with mPCM CN-1 & mPCM T32RF exhibit slightly less intense effect (higher, i.e. less negative  $\Delta T_{av}$  values) than Outlast®.

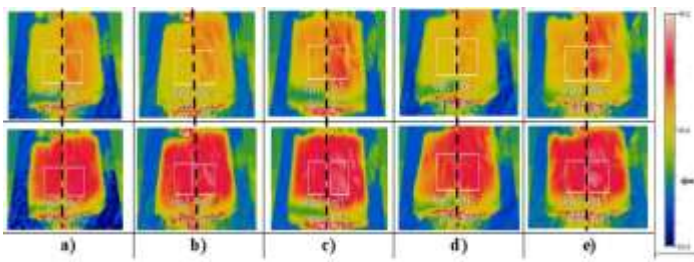
As seen from Table 6 most of the additives explored show high negative instantaneous  $\Delta T$  at the start of test after which the  $\Delta T$  becomes less negative as time increases, implying a decreasing intensity of thermoregulation effect. This may be explained by continued melting of the PCM inside the fiber. Also note that while Outlast fibre shows instantaneous  $\Delta T \leq -1^\circ\text{C}$  even at t=150 seconds only mPCM CN-1, and mPCM T32RF VSF could show similarly intense effect. For octadecane VSF, the  $\Delta T_{av}$  values are positive after 90 seconds of test meaning its temperature rises faster than regular VSF, thus showing a drop in its cooling capability.


**Table -5:** Transient temperature rise of PCM VSF relative to a non-PCM regular VSF: temperature difference averaged over specified time interval of test

Sample detail	Mean temperature difference $\Delta T_{av}$ (°C)		
	0-45 s	46-90 s	91-150 s
10% Octadecane	-1.9	-0.4	+0.3
20% mPCM CN-1	-1.2	-0.8	-0.6
20% mPCM Benochem	-1.4	-0.7	-0.1
15% mPCM T32RF	-1.3	-1.0	-0.8
Outlast®	-1.8	-1.4	-1.1

**Table -6:** Transient temperature rise of PCM VSF relative to a non-PCM regular VSF: temperature difference at specified time instant of test

Sample detail	Instantaneous temperature difference $\Delta T$ (°C)						
	6 s	12 s	44 s	70 s	90 s	120 s	150 s
10% Octadecane	-2.85	-2.50	-1.00	-0.60	0.20	0.15	0.70
20% mPCM CN-1	-1.15	-1.35	-1.25	-0.65	-0.60	-0.80	-0.70
20% mPCM Benochem	-1.55	-1.20	-1.25	-0.10	-0.45	-0.05	-0.20
15% mPCM T32RF	-1.05	-1.25	-1.60	-0.55	-0.85	-0.90	-0.45
Outlast®	-1.90	-1.60	-1.45	-1.25	-1.20	-1.05	-1.05



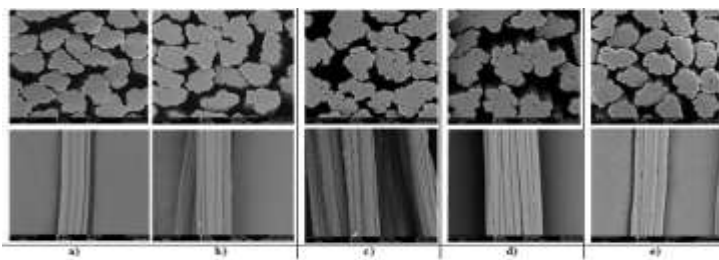
**Fig -2:** Thermal camera images at  $t = 6$  seconds (top row) & 90 seconds (bottom row) after start of the dynamic temperature profile test for: a) 10% Octadecane, b) 20% mPCM CN-1, c) 20% mPCM Benochem, d) 15% mPCM T32RF and e) Outlast<sup>®</sup> PCM VSF. In all images PCM & non-PCM VSF fiber webs are placed on the left and right side of the dotted vertical line respectively. Reference bar in  shown on far right.

The corresponding infrared thermal images recorded during the dynamic heating profile test at  $t = 6$  & 90 seconds (top & bottom row, respectively) are shown in Fig. 2 for a visible illustration of the thermoregulating capacity of PCM viscose fibres.

### 3.3 Morphology of fibres

As observed from scanning electron microscopy, Fig. 3 (a)-(e) (top row), the serrated cross section of viscose remains unaffected on inclusion of PCM. However there are clearly visible islands or holes distributed across the cross section of the PCM viscose fibers. The presence of holes in the PCM viscose fibre may be an indication that the PCM compounds which were added before spinning are physically entrapped in the fibre and have survived the spinning & after-treatment processes. Moreover the distribution intensity of these holes is not same in different PCMs, least being seen in case of n-octadecane.

Longitudinal section of non-PCM viscose fibre is shown in Fig. 3 (a) bottom row, the fiber surface appears to be uniformly serrated and free from any visible particle. However, few indentations & localized agglomerates are observed in longitudinal sections of PCM modified viscose fibres, as seen in Fig. 3 (b)-(e) bottom row.



**Fig -3:** Scanning electron micrographs in transverse (top row) & longitudinal (bottom row) sections of VSF with a) no PCM, b) 10% n-octadecane, c) 20% mPCM CN-1, d) 20% mPCM Benochem and e) 15% mPCM T32RF. All images are at 5000 x magnification.

## 4. CONCLUSION

Functional viscose fibres with thermal regulation capability were produced by incorporation of phase-change materials into viscose polymer before spinning. DSC & SEM analysis of the functional viscose fibres indicated that the added PCM compounds could be sustained inside the fibre even after undergoing the steps involved in viscose fibre manufacturing. It was found from dynamic temperature profile tests that encapsulated mPCMs possessed better cushioning ability to temperature changes than the lab emulsified additives. From the results of this study it can be concluded that thermoregulating viscose fibres with distinct phase-change temperatures can be obtained according to their targeted end-use. This can be achieved by choosing additive PCM compounds or eutectic mixtures thereof with varying melt & crystallization temperatures.

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