Environmentally friendly and highly productive bi-component melt spinning of thermoregulated smart polymer fibres with high latent heat capacity

Ch. Cherif¹, N. H. A. Tran^{1*}, M. Kirsten¹, H. Brüning², R. Vogel²

¹Technische Universität Dresden (TU Dresden), Institut für Textilmaschinen und Textile Hochleistungswerkstofftechnik (ITM), 01062 Dresden, Germany

²Leibniz-Institut für Polymerforschung Dresden e. V. (IPF Dresden e. V.), Hohe Straße 6, 01069 Dresden, Germany

Received 24 August 2017; accepted in revised form 12 October 2017

Abstract. A stable and reproducible bi-component melt spinning process on an industrial scale incorporating Phase Change Material (PCM) into textile fibres has been successfully developed and carried out using a melt spinning machine. The key factor for a successful bi-component melt spinning process is that a deep insight into the thermal and rheological behaviour of PCM using Difference Scanning Calorimetry (DSC), Thermogravimetric Analysis (TGA), and an oscillatory rheological investigation. PCM is very sensitive to the temperature and residence time of the melt spinning process. It is found that the optimal process temperature of PCM is 210 °C. The textile-physical properties and the morphology of the melt spun and further drawn bi-component core and sheath fibres (bico fibres) were investigated and interpreted. The heat capacities of PCM incorporated in bico fibres were also determined by means of DSC. The melt spun bico fibres integrating PCM provide a high latent heat of up to 22 J/g, which is three times higher than that of state-of-the-art fibres, which were also obtained using the melt spinning process. Therefore, they have the potential to be used as smart polymer fibres for textile and other technical applications.

Keywords: smart polymers, thermal properties, latent heat, phase change material, bi-component melt spinning

1. Introduction

Due to the increasing demand for technical textiles, particularly in terms of functional textiles for clothing and home textiles, further developments have focused mainly on the investigation and fabrication of high performance and functional chemical textile fibres. Textile fibres with integrated Phase Change Material (PCM) that can dynamically and proactively manage heat have been investigated and improved rapidly, even compared to other high functional textile materials due to their broad range of application possibilities; ranging from apparel, shoes, bedding, to carpets and car seats [1–8]. Outlast^{TM} and ComforTemp® are well-known trademarks for such functional textile

materials incorporating PCM, which have the ability to absorb and store heat energy, and can subsequently release it for optimal comfort as well [9]. Figure 1 shows how the textile integrating PCM works. In principle, when the ambient and/or body temperature increase, the skin produces heat and consequently, PCM melts (i.e. it changes from a solid to a liquid state). Excess body heat and hot ambient air are absorbed and stored by PCM as latent heat. When it cools down and/or the body temperature decreases, PCM solidifies (i.e. it changes from a liquid to a solid state), and the stored heat is released back into the body to maintain thermal balance and keep the body warm. As a result, a constant microclimate between

*Corresponding author, e-mail: nguyen_hoai_an.tran@tu-dresden.de
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Figure 1. Schematic diagram of the basic working principle of PCM or textiles including PCMt

body and textile is actively and dynamically controlled and balanced by PCM.

The incorporation of PCM into textiles has attracted increasing attention, and has recently come into the focus of industry [10, 11] and academic research [12–14]. There are various available methods for the integration of PCM into textiles, such as coating, lamination, finishing, injection moulding, foam techniques, and fibre technologies (dry, wet, and melt spinning) [14–16].

Among the above mentioned methods, melt spinning is considered as an economical and environmentally friendly process due to its high productivity and the absence of any additional toxic solvents. In the melt spinning process, there are two main ways to integrate PCM into filaments: the single component melt spinning of polymer blends and the bi-component melt spinning. The first one is a relatively simple method, because it is similar to the melt spinning process for producing single polymer fibres: PCM pellets are mixed with thermoplastic polymers, and melt spun into blended fibres including PCM. Unfortunately, the PCM in the blended fibres can be easily removed by washing, wiping or abrasion. To avoid these disadvantages, therefore, the melt spinning of bi-component fibres with core-sheath structures is preferred, since PCM is securely enclosed within the core component of the fibres by the sheath component. Magill *et al.* [17] have developed bi-component fibres including PCM having latent heat ranging from 8.4 to 17.1 J/g, depending on the used polymers. Recently, with a press release on ‘Advanced Textile Source’, Hågström [18] informed that they have successfully integrated PCM into fibres with a very high latent heat of up to 60 J/g through the melt spinning of bi-component fibres. This latent heat is significantly higher than that of

state-of-the-art viscose and acrylic yarns, which was obtained by means of the solution spinning with latent heats in the range of 1–20 J/g [10, 16]. However, this short report [18] does not provide further basic information regarding melt spinning process parameters, such as machine type, spinneret, geometry of the spinning die, etc. Furthermore, a few fundamental characteristics of melt spun fibres, such as fineness, tensile strength, and elongation are missing as well. Hence, numerous questions and issues for the integration of PCM still remain unanswered. For instance, how does the thermal behaviour of PCM change during melt spinning? What are the optimal spinning parameters for a stable and reproducible process resulting melt spun bi-component fibres including PCM (PCM bico fibres) with a high latent heat? How do the textile-physical properties (tensile strength and elongation) of these PCM bico fibres behave depending on various PCM contents?

Within this research project, in addition to the integration of PCM into textile fibres, we aimed at answering the above mentioned questions, starting with the investigation of the thermal behaviour and rheological properties of PCM pellets to select an optimal set of spinning process parameters on an industrial-scale melt spinning machine. The morphological and textile-physical properties of melt spun PCM bico fibres were then investigated. The tensile strength of melt spun PCM bico fibres containing the highest PCM content was improved by means of the off-line post-drawing process. Finally, we compared the latent heat of the produced PCM bico fibres to that of melt spun blended fibres according to state-of-the-art work.

2. Experimental

2.1. Materials

Phase Change Material (PCM, $\rho_{\text{PCM}} = 0.65 \text{ g/cm}^3$) was kindly supplied by our research partner, S Textil Tech, Hof, Germany. This PCM has been developed using physical bonding of a paraffin wax with general chemical formula $\text{C}_n\text{H}_{2n+2}$ in a network structure of polymer. It has a melting temperature of approx. 44°C and a latent heat of 108.2 J/g (Figure 2). Polypropylene (PP) Moplen HP561R ($\rho_{\text{PP}} = 0.91 \text{ g/cm}^3$, MFR = 25 g/min at 230°C and 2.16 kg) was kindly provided by LyondellBasell, Netherlands. Before melt spinning, the PCM was dried at a temperature of 30°C for 8 h.

Figure 2. DSC curves of the second heating run of PCM and PP

2.2. Differential scanning calorimetry (DSC) measurements

DSC was used to characterize the glass transition, melting temperature, and heat capacity of PCM, PP, and the melt spun PCM bico fibres. All DSC analyses were carried out using a DSC Q2000 calorimeter (TA Instruments, Delaware, USA) under a nitrogen atmosphere. Calibration was performed with an indium standard. Samples of about 5 mg were encapsulated in aluminium pans. Heating-cooling-heating scans were performed at various temperatures, ranging from 100, 150, 200, and 220 °C at a scan rate of 10 K/min.

2.3. Thermogravimetric analysis (TGA)

All TGA analyses were conducted using a TA Instruments Modulated TGA Q500 Thermal Analyser under a nitrogen atmosphere. The sample weight was around 5 mg in all experiments. Within this study, isothermal TGA measurements were also performed with two different procedures: the samples were heated up to 200, 210, 220, and 230 °C at a slow heating rate of 10 K/min, and also at a higher heating rate of 100 K/min; they were examined isothermally for 30 and 50 minutes, respectively.

2.4. Rheological measurements

The rheological properties of polymers were determined using an ARES-G2 rheometer from TA Instruments. Specimens were measured by means of a parallel plate disc geometry of 25 mm diameter, and the gap between the plates was set to 2 mm. The frequency sweep experiments were performed at a strain of 5% in a frequency range of 0.1 to 100 Hz at 200 °C. In this study, isothermal rheological measurements

were also conducted at different temperatures from 200 to 230 °C, rising by 10 °C at a constant frequency of 10 rad/s for 50 minutes. All measurements were carried out using nitrogen as heating gas.

2.5. Melt spinning

Melt spinning was carried out on an industrial-scale bi-component melt spinning machine employed for the fabrication of synthetic fibres at the Institute of Textile Machinery and High Performance Material Technology (ITM). This melt spinning machine can be used in applications requiring mass flow rates, such as in industrial scales ranging from 3 up to 15 kg/h, and take-up speeds from 750 to 3500 m/min. The number of spinneret holes varied from 40 to 400. The bi-component spinneret selected for this research project contained 60 holes. The diameter and length of each capillary hole for the sheath component was 0.45 mm and 1.80, respectively, and 0.30 mm with an aspect ratio L/D of 3 for the core component. The temperature profile in the twin-screw extruder for the core component (PP/PCM blends) began at 140 °C at the feeding zone and increased to 210 °C at the die. The temperature profile in single extruder for the sheath PP component was 200, 210, and 210 °C. The spinning speed was kept constant at 1000 m/min with a constant volumetric flow rate for each component of 30 cm³/min (ratio of core and sheath 50:50). Within the core component, which is a mixture of PCM and PP blends, the amount of PCM was changed to various volume percentages of 22, 30, 36, 44, 50, and 64% (Figure 3).

2.6. Post-drawing process

To improve the textile-physical properties of melt spun PCM bico fibres, the PCM32 bico fibres, which

Figure 3. Scheme of a cross-section of PCM bico fibres (left) and a photographic image of PCM bico fibres on 6 spools with different volume percentages [vol%] of PCM (right), e.g. PCM11 (spool number 1) and PCM 32 (spool number 6) representing for PCM bico fibres, which contain 11 and 32 vol% PCM, respectively

contain 32 vol% of PCM, were then drawn off-line at various draw ratios of 1.50, 1.75, 2.00, and 2.10, at a temperature of 50 °C on a laboratory drawing machine at the Leibniz-Institut für Polymerforschung Dresden e. V. (IPF Dresden e. V.).

The drawing process was performed by stretching the PCM32 bico fibres between two rollers (the input and first output feed rollers), whereas the first output feed roller rotated faster (Figure 4). These fibres were supplied to the input feed roller at a constant feeding velocity, V_{in} , and stretched by the first output feed roller running at a constant drawing velocity, V_{1out} , equalling $DR \times V_{in}$, where DR is the Draw Ratio. For this study, the drawing speed (the velocity of the winder), $V_{take-up}$ ($V_{take-up} = V_{1out} = V_{2out}$), was kept at 30 m/min, while the feeding speed (the velocity of the input feed roller), V_{in} , was continuously adjusted in order to achieve the desired variety of draw ratios mentioned above.

2.7. Mechanical testing

The tensile and elongation properties of the filaments were determined by tensile testing using a Zwick/Roell Z 0.5 Universal Tester controlled with TestXpert software (Zwick/Roell, Ulm, Germany) according to DIN EN ISO 2062. The gauge length was 62.5 mm and the test speed was 200 mm/min. Twenty tests were performed for each multi-filament yarn, which is made from 60 bi-component core and sheath filaments. The fineness (titer) and diameter of fibres were measured by weighing and using an optical microscope/SEM. The measurements were carried out in an air-conditioned laboratory at 22 °C and 65% relative humidity.

Figure 4. Scheme of the drawing process (above) and drawing device at IPF Dresden e. V. (below)

2.8. Morphological characterization

The cross-section of fibres was evaluated using a light microscope Carl Zeiss Axio Imager M1m with a digital colour camera AxioCam MRc5, Germany. The samples were observed at 200× and 1000× magnifications. The fibre surfaces were investigated with a Scanning Electron Microscope (SEM) DSM 982 Gemini Carl Zeiss AG, Jena, Germany. All samples were recorded at 1000× magnifications. The sample discs were prepared by sputtering a thin layer of 3 nm platinum.

3. Results and discussion

3.1. Thermal behaviour of PCM

In contrast to solution spinning, where polymers dissolve in solvents, in the case of melt spinning, polymers are processed at significantly higher temperatures and pressures. Almost all polymers are temperature sensitive and degrade considerably at high temperatures to a certain degree depending significantly on the process temperature. PCM is a type of material that undergoes thermal degradation and begins to degrade rapidly at just above 200 °C, even it already starts losing mass earlier at around 150 °C (Figure 5). It is seen that the decomposition temperature of PCM, at which it lost 5% of its mass, is around 235 °C. At temperatures above 250 °C, PCM lost up to 10% of its mass. Therefore, PCM or PCM blends with other thermoplastic polymers should be processed at a maximum temperature of 230 °C, at which PCM would lose 5% of its mass.

Unlike TGA measurements, the process temperatures in the case of the melt spinning are more complex. Polymers undergo certain residence times at various temperature profiles during mixing and

Figure 5. Mass loss vs. temperature in TGA measurement of PCM

Figure 6. Non-isothermal TG curves with a heating rate of 10 K/min (up to set-up isothermal temperatures: 200, 210, 220, and 230 °C) and isothermal TG curves at different isothermal temperatures for 30 min

extrusion in extruder and spinning head. It is commonly known that the time necessary to completely transport polymers from the beginning/feeding zone of the twin-screw extruder to spinning head usually ranges from a few minutes up to about 20 minutes depending on the length of the twin-screw extruder, speeds of extruder, and speeds of gear spinning pump. By means of the TGA technique, the decomposition of PCM was determined, and its thermal stability was predicted. Figure 6 suggests that the mass loss of PCM increases over time; moreover, the higher the temperature, the less time needed for the same mass loss to occur. The thermogravimetric (TG) curves in Figure 6 were obtained by TGA measurements starting at 30 °C with a heating rate of 10 K/min up to the desired temperatures of 200, 210, 220, and 230 °C. The samples were then maintained at isothermal temperatures for 30 minutes. From Figure 6, it is found that PCM lost 5% of its mass (decomposition point) after 20 to 22 minutes and 10% of its mass after 23–27 minutes.

In the melt spinning process, the temperature process in twin-screw extruder is more similar to the second experimental procedure, in which the samples were quickly heated to the desired temperatures at a heating rate of 100 K/min. Subsequently, the samples were isothermally exposed for 50 minutes. Figure 7 illustrates the isothermal TG curves of PCM at temperatures of 200, 210, 220, and 230 °C. In comparison to the first experimental procedure, the mass loss of PCM took place earlier: within 9.1 to 19.1 minutes, PCM lost 5% of its mass, and up to 10% just in a few minutes, for instance 6.8, 8.2, 8.4, and 11.5 minutes for isothermal temperatures of 200, 210, 220, and 230 °C, respectively (Figure 7b). At a temperature of 230 °C, it is seen from Figure 7a that the mass loss of PCM occurred earlier and more rapidly.

Therefore, it is recommendable to process blends of the PCM with other thermoplastic polymers at a maximum temperature of 220 °C. Temperatures above this limit are considered as critical temperatures for the melt spinning process.

3.2. Rheological behaviour of PCM

The complex viscosity of PP and PCM decreases with the increase of the shear rate as shown in Figure 8. PP and PCM melts exhibit a typical behaviour for non-Newtonian fluids, and thus demonstrate shear-thinning behaviour, i.e. they belong to the class of pseudoplastic materials. In the range of shear rates from 0.1 to about 30 rad/s and at a temperature of 200 °C, all measured points fitted well to the Carreau fluid model [19]. However, for the higher shear rates from 30 rad/s, PCM melts no longer appeared to fit the Carreau fluid model. Unlike PP, also known as a typical spinning polymer, PCM exhibits a stronger shear thinning-behaviour, therefore, the complex viscosity rapidly dropped at high shear rates. For instance,

Figure 7. Mass loss vs. time during isothermal TG measurement of PCM at various temperatures of 200, 210, 220, and 230 °C (a), mass loss of 5% and 10% vs. time at different isothermal temperatures (b)

Figure 8. Complex viscosity vs. frequency of pure PP and PCM at a temperature of 200 °C

while the viscosity of PCM at lower shear rates is much higher compared to typical spinning polymer PP, at higher shear rates, the viscosity of PCM is lower than that of PP (Figure 8). In other words, PCM melt exhibits an unusual rheological behaviour in comparison to any common thermoplastic spinning polymers.

To gain an in-depth knowledge of this rheological behaviour of PCM, the effects of temperature and time on the thermal stability of PCM and PP were determined by continuously recording the dynamic moduli at a constant frequency as a function of time at the process temperature. It is seen from Figure 9 that while the viscosity of PP decreases with increasing temperature (Figure 9b), the viscosity of PCM does not follow this tendency at temperatures exceeding 220 °C (Figure 9a). Furthermore, the viscosity of PP slightly dropped during the residence time. Moreover, the higher is the temperature, the stronger the decrease of viscosity. This behaviour is typical for nearly all common polymers, which slightly degrade

more at high temperatures and with the residence time. This tendency did not apply to PCM, especially at a temperature of 220 °C, the viscosity of PCM rapidly increased for the first 10 minutes, and then rose continuously. This behaviour may be accounted for by the crosslinking of PCM. At temperatures of 200 and 210 °C, the viscosity of PCM is almost stable within 50 minutes of heating, although there was a slight increase in viscosity for the first few minutes. It is worth noting here that, at a temperature of 230 °C, the rheological measurements could no longer be performed because the PCM melt started to develop thick fog and became swollen after around 10 minutes of testing (Figure 9a).

In summary, the above analysed results of the thermal and rheological behaviour of PCM indicated that PCM is very sensitive to temperatures and residence times. To avoid high mass loss, crosslinking, and the viscosity instability of PCM during the melt spinning, PCM should be processed in a short time (not longer than 20 minutes) at a maximum spinning temperature of about 210 °C.

3.3. Textile-physical properties of PCM bico fibres

Effect of PCM content

Figure 10 shows the tensile strength and elongation properties of PCM bico fibres for different amounts of PCM. With the increase in PCM content from 11 (PCM11) to 32 vol% (PCM32), the tenacity σ_R [cN/tex] (maximum specific stress) decreases from 18.2 to 6.8 cN/tex, i.e. the use of PCM led to a considerable reduction in tensile strength of PCM bico fibres (Figure 10). Unlike the tenacity, the elongation of fibres slightly increases linearly with the increase in PCM content from 11 to 25 vol%. However,

Figure 9. PCM (a) and PP (b) melt stability followed in oscillation at different temperatures at constant frequency of 10 rad/s

Figure 10. Tensile and elongation properties of PCM bico fibres with different PCM contents: stress-strain curves (a), tenacity and elongation at break vs. PCM content (b)

at a high amount of PCM (PCM32 with 32 vol% of PCM in fibres), the elongation of fibres drops from 306 (PCM25) to about 260%, which is below that of PCM11 bico fibres (Figure 10b). The reason for this may be that the PP amount in PCM32 bico fibres dropped to a lower limit value, which is equal to 68 vol% of PP. It was also found that, when the PCM content is higher than 32 vol% (or the PP content is less than 68 vol%), the melt spinning process is not reproducible and stable anymore. Recently, Iqbal and Sun [16] have presented that the PCM can be integrated into fibres with up to 8% to develop multifilament yarns containing PCM using the melt spinning of PCM and PP blends with their newly constructed spinneret. These results analysed above enabled the conclusion that the incorporation of PCM into fibres leads to a reduction in tensile strength, but simultaneously results in an increase in elongation, unless the amount of PCM exceeds certain limits.

As mentioned in the introduction, the main objective of our work was to develop a stable and reproducible melt spinning process that can incorporate PCM into textile fibres with high latent heat storage capacities. It was found that PCM32 bico fibres containing 32 vol% of PCM provide the highest latent heat in comparison to other PCM bico fibres. However, these PCM32 bico fibres possess a low tenacity of 6.8 cN/tex and a relatively high elongation of over 200%, which complicates their further processing by various textile technologies, such as knitting and weaving. Furthermore, the stress-strain curve of PCM32 bico fibres (continuous curve in Figure 10a) reveals that these fibres still possess a rather large plastic deformation area. Unlike elastic deformation, this type of deformation is irreversible, and therefore

these fibres may not be suitable for common textile products. The textile-physical properties of the PCM32 bico fibres was improved using the off-line post-drawing process, which we already described in the experimental part.

Effect of off-line draw ratio

Figure 11 illustrates that the post-drawing process can significantly improve the textile-physical properties of PCM32 bico fibres. By increasing the draw ratio, the tenacity of fibres increases and the elongation decreases. Furthermore, most of plastic area in the stress-strain curve of fibres without drawing (PCM32) has been transformed into an elastic deformation area.

Figure 12 plots the values of tenacity and elongation of PCM32 bico fibres as a function of the draw ratio. It is seen that by increasing the draw ratio from 1.50 to 2.00, the tenacity of fibres increases, whereas the elongation at break decreases. These results exhibit a typical textile-physical behaviour shared by the majority of most common textile fibres. However, this tendency does not occur at a higher ratio of $DR = 2.10$. There is a linear, continuous decrease in elongation at break with the increase in draw ratio, but the tenacity of fibres slightly decreases from 13.5 cN/tex at $DR = 2.00$ to 13.4 cN/tex at $DR = 2.10$ after the draw ratio reaches a limit value. It is well-known that any semi-crystalline thermoplastic polymer such as the PP used in this study, has definite fatigue limits after reaching a maximum value of their molecular orientation.

These results regarding textile-physical properties of PCM bico fibres suggest that the optimal draw ratio for the post-drawing process is $DR = 2.00$ because

Figure 11. Tensile and elongation properties of melt spun and drawn PCM32 bico fibres for various draw ratios: stress-strain curves (a), tenacity and elongation at break (b)

Figure 12. Tenacity and elongation at break vs. draw ratio

the PCM bico fibres possessed the highest tensile strength and satisfying (low) elongation. Moreover, it was also found that the post-drawing process at a draw ratio of $DR = 2.00$ was more stable than that at a draw ratio of $DR = 2.10$. The tenacity of fibres considerably increased from $\sigma_R = 6.8$ to 13.5 cN/tex (has doubled) by increasing the draw ratio from $DR = 1.00$ to 2.00 and the elongation of fibres was reduced from 200 to 87%.

Table 1 shows a comparison between the tenacity and elongation at break of these PCM32 bico fibres with a $DR = 2.00$ and that of various commercial fibres

Table 1. Textile-physical properties of PCM32 bico fibres and selected commercial textile fibres

Fibre	Tenacity [cN/tex]	Elongation at break [%]
PCM32 bico fibre $DR = 2.0$	13.5	87
Wool	11–14	30–43
Acetate	13.0	24
Viscose rayon	18.0	27

Figure 13. Stress-strain curve of PCM bico fibres with draw ratio of $DR = 2.00$ in comparison to the stress-strain curves of various common textile fibres (modified following [20])

that have similar values of tenacity. It is seen from Figure 13 that the textile-physical properties of PCM32 bico fibres with a $DR = 2.00$ are comparable to those of acetate and wool fibres. However, these fibres are much more elongated than acetate (24%), viscose (27%) and wool fibres (43%).

3.4. Morphology of PCM bico fibres

Figure 14 presents a microscopic image of cross sections (left) and an SEM image of surface morphologies of PCM32 bico fibres. It is clearly visible that the PCM and PP blend in the core component securely enclosed by the PP sheath component (left), which has a smooth and featureless appearance (right). The core component including PCM was still trapped after the off-line post-drawing process, even at draw ratios up to $DR = 2.10$ (Figure 15).

Figure 14. A microscopic image of the cross sections (a) and an SEM image of the surfaces (b) of melt spun PCM32 bico fibres

Figure 15. Microscopic images of the cross-sections of drawn PCM32 bico fibres with various draw ratios: $DR = 1.50$ (a), $DR = 1.75$ (b), $DR = 2.00$ (c), $DR = 2.10$ (d), scale bars: 20 µm

3.5. Latent heat of the produced PCM bico fibres

Latent heat of PCM pellets

Figure 16a shows the DSC curves of PCM pellets obtained using DSC measurements at a heating rate

of 10 K/min from 0 to 220 °C. The melting enthalpy ΔH_m , which is considered as latent heat, is 108.2 J/g for the second heating run. The latent heat of PCM was almost constant when the samples were heated up to different upper temperature limits of 50, 100,

Figure 16. DSC curves of PCM pellets (a) and heat enthalpy of PCM for different upper temperature limits obtained from DSC measurements (b)

Table 2. Melting enthalpy ΔH_m and cold crystallization enthalpy ΔH_c obtained from second heating runs and cooling runs, respectively, for different upper temperature limits

Upper temperature limits [°C]	Melting enthalpy, ΔH_m [J/g]	Crystallization enthalpy, ΔH_c [J/g]
50	110.4	111.7
100	103.6	106.1
150	106.7	110.6
200	104.6	111.5
220	108.2	109.5

150, 200, and 220 °C, as shown in Figure 16b and also summarized in Table 2. It can be concluded that the latent heat is almost conserved during heating and cooling, and thus it is independent of the upper testing temperatures. The melting and cooling process of DSC measurements did not have any significant effect on the latent heat.

Latent heat of the melt spun and drawn PCM bico fibres

Table 3 summarizes the heat enthalpies of melt spun PCM bico fibres with different amounts of PCM (11, 18, 25, and 32 vol%). Figure 17 shows DSC curves of PCM32 bico fibres with 32 vol% of PCM. Similar to the heat enthalpy of PCM pellets presented above in Figure 16, the heat enthalpies of the first heating and cooling, as well as second heating run slightly differ from each other. It is commonly known that results obtained from the first heating run depend considerably on the heat history of the polymers, and the results achieved by the second heating run are often used to directly compare materials to each other. Hence, in this section, only the melting en-

thalpies for the second heating runs of various PCM bico fibres obtained using DSC measurements are summarized in Table 3. It is obvious that the melting enthalpy of fibres increases from 8.1 to 22.2 J/g with the increase in PCM content from 11 to 32 vol%, respectively. It is also found that there is a difference between calculated and measured melting enthalpies. The loss rate of the melting enthalpy increases with an increase in PCM content. This phenomenon occurs due to the mass loss of PCM during the melt spinning under the high processing temperatures (as presented in the sections 3.1 and 3.2 ‘thermal and rheological behaviour of PCM’).

In summary, the melt spun PCM32 bico fibres provide a relatively high latent heat of 22.2 J/g, which is nearly three times as high as that of state-of-the-art fibres with a latent heat of 8.08 J/g, obtained by Iqbal and Sun by means of melt spinning [16]. It was also found that the melting enthalpy of PCM bico fibres from the second heating run did not change significantly after post-drawing, and the heat enthalpy of the drawn PCM32 bico fibres with different draw ratios from 1.50 to 2.10 ranged from around 21 to 23 J/g.

Table 3. Measured and calculated melting enthalpy of PCM bico fibres

Fibre sample No. [–]	Measured melting enthalpy, ΔH_{Mm} [J/g]	Calculated melting enthalpy, ΔH_{Cm} [J/g]	Loss rate of melting enthalpy [%]
PCM11	8.1	8.8	7.95
PCM18	13.2	14.7	11.40
PCM25	17.5	20.8	15.90
PCM32	22.2	27.3	18.70

Figure 17. DSC Curves of PCM32 bico fibres with 32 vol% of PCM

Figure 18. DSC Curves of PCM32 bico fibres obtained by thermal cycle test

Stability of the heat enthalpy of PCM bico fibres

In principle, the heat enthalpy of almost Phase Change Materials decreased after a certain time [21]. Therefore, it is necessary to study the stability of the heat enthalpy (melting and cold crystallisation enthalpy) of PCM32 bico fibres when these fibres undergo the repeated process of heating and cooling. In that regard, the thermal cycle test of melt spun PCM32 bico fibres was conducted using DSC technique. Table 4 shows the thermal properties of PCM32 bico fibres for different heating and cooling cycles. Figure 18 represents DSC curves of five different selected cycles of heating and cooling. It is seen that the DSC curves for all cycles are almost identical, except the first heating curve due to the heat history of polymeric fibres. The melting enthalpies of PCM32 bico fibres are similar for all thermal test cycles and ranging from 21.79 to 22.93 J/g, except a higher melting enthalpy of 25.20 J/g obtained from the first heating run (Table 4). Thus, once can conclude that the heat

enthalpy of PCM bico fibres is stable and conserved after at least 300 repeated cooling and heating cycles.

4. Conclusions

Innovative smart bi-component fibres with a high latent heat capacity of 22.2 J/g, – a value almost three times higher than that of state-of-the-art fibres – have been successfully developed and fabricated at an industrial-scale with a high production rate of 3.6 kg/h using the melt spinning process. Unlike solution spinning, melt spinning is a solvent-free process and considered very environmentally friendly. Unfortunately, the polymers used for this process must be processed at a temperature higher than their melting temperatures. The applied Phase Change Material (PCM) for the current study lost its mass very early at around 150 °C, which is a temperature lower than the melting temperatures of almost all thermoplastic polymers. Therefore, the most challenging task of this research project was to understand the thermal and rheological properties of PCM for its use on an industrial-scale bi-component melt spinning machine. By analysing and considering the best potential combinations of thermal and rheological behaviours of PCM, the melt spinning process parameters, particularly the temperature profile along the twin-screw extruder for the PCM and PP blends, were optimized. It was found that the process temperature for a stable and reproducible melt spinning process with a high PCM content of 32 vol% should not exceed 210 °C. The produced as-spun bi-component fibres with 32 vol% of PCM (PCM32 bico fibres) possessed low tenacity and high elongation. However, following post-drawing process, these properties were improved: the textile-physical properties of

Table 4. The thermal properties of PCM32 bico fibres for different thermal test cycles

Number of test cycles [–]	Melting enthalpy, ΔH_m [J/g]	Cold crystallization enthalpy, ΔH_c [J/g]
1	25.20	22.51
2	21.83	22.15
3	22.04	22.73
4	22.03	22.65
5	21.99	22.87
20	21.79	22.88
50	22.60	23.21
100	22.90	22.87
200	22.77	23.07
300	22.93	22.94

PCM32 bico fibres with a draw ratio of 2.00 are similar to those of acetate fibres, which have sufficiently strong and elongation for further processing in various textile processes, such as knitting and weaving. The morphological results showed that PCM is safely enclosed within the core PP component, even after post-drawing at a high draw ratio of 2.10 under heat and mechanical treatments. The produced as-spun and drawn PCM32 bico fibres with heat capacities ranging from 21 to 23 J/g could be promising candidates for thermoregulated smart textiles.

Acknowledgements

The authors thank to the Deutsche Forschungsgemeinschaft (DFG), the Dresden University of Technology (TU Dresden), and the Excellence Initiative ‘Support the Best’, for funding bi-component melt spinning plant, and thus enabling this study. We would also like to thank Mrs. Martina Dziewiencki, Mrs. Tina Anders, Mr. Michael Posselt (ITM, TU Dresden) and Mr. Mathias Häschel (IPF Dresden e. V.) for their technical support.

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