Twisted nylon 6 nanofibre yarns containing microencapsulated phase change materials (MPCMs) were fabricated via electrospinning to prepare thermal regulating nanofibre yarns. Electrospun nanofibre yarns with different contents of MPCMs including 0.375, 0.75, 1.5 and 3 wt% were prepared. The surface morphology, crystallisation and thermal properties of the yarn samples were characterised by scanning electron microscopy (SEM) and differential scanning calorimetry, respectively. SEM results showed that electrospun composite fibres and yarns had good morphology with smooth surface and the MPCMs were randomly distributed on the composite yarn surface, inside the nanofibres and between the fibres in the yarn structure. Additionally, the SEM results suggested that the average diameters of nylon 6/MPCM nanofibres decreased from 0.23 ± 0.03 μm for neat nylon 6 to a minimum of 0.10 ± 0.02 μm for composite yarn containing 3 wt% MPCM. However, nylon 6/MPCM nanofibre yarn diameter displayed a complex behaviour; the average diameters of electrospun composite yarns increased upon addition of MPCM and reached a maximum value of 165.1 ± 5.11 μm for composite yarn containing 0.75 wt% MPCM, then decreased markedly. These changes in nylon 6/MPCM nanofibres and yarn diameters have been discussed in terms of electrospinning solution properties and twist parameter. The melting enthalpy values of MPCM in the composite nanofibre yarns increased as the content of MPCMs increased up to the highest content and higher than 80% of the heat storage capacity of MPCMs was retained after electrospinning.

1. Introduction

Phase change materials (PCMs) store and release high latent heat energy as their physical state changes under the nearly isothermal condition [1–7]. The PCMs are used extensively in many different areas such as building energy conservation, space and water heating, cooling and air-conditioning systems, medical application, cooling of engines, etc. [3–5,8–10]. In the textile industry, PCMs are used for making thermoregulating fibres.
and smart textiles and garments, having applications in various fields such as military, aviation and space, sportswear, home textiles, automotive, medical and health care [11–15].

There are various methods to incorporate PCMs into textiles such as melt spinning [16–20], wet spinning [21–24], coating and lamination [25–27]. Electrospinning is currently an efficient process for the fabrication of ultrafine fibres. Recently, various types of ultrafine PCMs/polymeric fibres were prepared via electrospinning [28–42]. These ultrafine composite fibres of polymer/PCMs have excellent performances in the thermal energy storage and capability to temperature regulation. These ultrafine composite fibres of polymer/PCMs with high surface-area-to-volume ratio have excellent performances in the thermal energy storage and capability of temperature regulation. In typical electrospinning, due to the helix motion and instability of the jet, generally fibres are deposited in a random state and form of non-woven structure. Generally electrospun fibres are too weak for conventional physical manipulation and handling. The relatively low mechanical strength and the difficulty in tailoring such fibrous structure with random fibre orientation have limited their applications in many fields. Comparing to the non-woven mats, the yarns hold more lateral interaction and friction between nanofibres which assist in improving mechanical properties. It can be expected that nanofibre yarns will find more applications [43].

To the best of our knowledge, the preparation of twisted nanofibre yarn containing microencapsulated PCM has not been investigated. This study aimed to develop and characterise electrospun nylon 6/PCM nanofibre yarn as an ultrafine phase change fibres.

2. Experimental

2.1. Materials

Nylon 6 pellets were kindly obtained from Alyaf Co., Iran. Formic acid was purchased from Sigma-Aldrich and used as received without further purification. Microencapsulated PCMs (MPCMs), Microtek MPCM-18D, was purchased from Microtek Laboratories, USA. As quoted by the supplier, the MPCMs are very small bi-component particles consisting of a core material (hexadecane) and an outer shell or capsule wall (melamine–formaldehyde resin).

2.2. Methods

To prepare the solutions for electrospinning, nylon 6 pellets were first dissolved in formic acid at a concentration of 15 wt% and were stirred at room temperature for at least 10 h. The MPCM powder was then added into the solution and the MPCM contents were set at 0.375, 0.75, 1.5 and 3 wt%. Subsequently, the mixtures were magnetically stirred for 3 h to acquire homogenous solutions.

2.3. Electrospinning

The electrospinning set-up for preparing continuous twisted nanofibre yarns consisted of a high-voltage DC power supply, two syringe nozzles with flat-tipped needles (22-gauge, ID = 0.4 mm, OD = 0.7 mm), a neutral surface and a take-up/twister unit.
Electrospinning solutions were fed using two digitally controlled syringe pumps (TOP-5300, Japan). Each needle was charged with a voltage of the same value but opposite polarisation using a high-voltage DC power supply. A neutral cylinder (6 cm diameter × 30 cm length) was vertically placed in the middle of the electric field. The twister unit consists of a rotating plate with adjustable rotation speed (1–440 rpm) and a take-up roller.

The nanofibre yarn electrospinning process was explained in more details in previous research works [44,45]. Briefly, after the two nozzles started to electrospin, a piece of yarn was placed at the convergence point to collect the nanofibres. Next, the other end of the yarn was pulled towards the take-up roller. Finally, nanofibres were twisted by rotating the yarn around its axis by the take-up/twister unit. All electrospinning experiments were performed in environmental conditions. The optimal conditions for fabricating continuous nanofibre yarns were determined and the results are presented in Table 1.

### 2.4. Characterisation

The morphology of MPCM, nanofibres and twisted nanofibre yarns was observed with a scanning electron microscope (KYKY-EM 3200, KYKY Technology, China). All samples were coated with gold under vacuum prior to scanning electron microscopy (SEM) observation.

The thermal properties of MPCM and nanofibres were investigated using differential scanning calorimetry (DSC; DSC 2010 machine, TA Instruments, New Castle, DE). The heating and cooling rates were 10 °C/min in nitrogen atmosphere. The crystallinity of samples ($X_c$) was calculated by the following equation:

$$X_c = \frac{\Delta H_m}{\Delta H_{m}^{c}} \times 100$$

where $\Delta H_m$ is the measured melting enthalpy and $\Delta H_{m}^{c}$ is the enthalpy of fusion of a 100% crystalline polyamide 6, it was taken as 167.2 J/g [46].

### 3. Results and discussion

#### 3.1. Spinnability

First, a set of preliminary experiments were performed to obtain the optimised content of MPCM for yarn production with uniform and bead-free fibres. The trial tests indicated that at MPCM contents higher than 3 wt%, the viscosity of the solution was not high enough to electrospin fibres and it was observed that the electrospinning jet broke up into small droplets and yarn formation was impossible. Therefore, the MPCM contents of 0.375, 0.75, 1.5 and 3 wt% were chosen to produce electrospun nylon 6/MPCM nanofibre yarn.

| Applied voltage (kV) | Feeding rate (mL/h) | Twister speed (rpm) | Take-up speed (m/h) | Distance between needles tips (cm) | Needle centre/take-up unit distance (cm) |
|----------------------|---------------------|---------------------|---------------------|------------------------------------|----------------------------------------|
| 12                   | 0.3                 | 200                 | 2.4                 | 30                                 | 30                                     |

Table 1. Electrospinning conditions and set-up parameters for preparing twisted nanofibre yarn.
3.2. Morphology

The SEM images of MPCMs are shown in Figure 1. As can be seen, the MPCMs had a broad size distribution with a mean diameter of 17–20 μm. Some large MPCMs were the aggregates of small MPCMs. The MPCMs had smooth surface and spherical shape.

Figure 2 represents the SEM images of neat nylon 6 and nylon 6/MPCM nanofibre yarns with different magnifications and corresponding fibre diameter distribution. As can be seen, both neat nylon 6 nanofibres and composite nanofibres were quite uniform and smooth with cylindrical shapes. In addition, SEM images showed that the majority of MPCMs retained their spherical shape with a smooth surface morphology without any damage and crack. Moreover, MPCMs were dispersed randomly in nylon 6 polymer matrix and no MPCM agglomeration was observed in the nylon 6/MPCM nanofibre yarn.

The average diameters of nanofibres and twisted nanofibre yarns are presented in Figure 3. The statistical analysis confirmed that the presence of the MPCM had a significant effect on the average diameter of nanofibres and yarns \( (p \leq 0.05) \). Electrospun nanofibre diameters in composite yarns showed a decrease from 0.23 ± 0.03 μm for neat nylon 6 to a minimum of 0.10 ± 0.02 μm for composite yarn containing 3 wt% MPCM.

The previous studies have revealed that the diameter and surface morphology of the electrospun nanofibres were affected by various factors, including solution properties, process parameters and environmental conditions \([47–50]\). The changes in nanofibre diameter can be attributed to the variations of nylon 6/MPCM electrospinning solution properties such as decreased viscosity due to reducing macromolecule entanglements and intermolecular forces of nylon 6 polymer as suggested by the others \([28,33,35]\). Moreover, formation of hydrogen bonding interactions between amino (–NH) groups in the melamine formaldehyde shell in MPCM and the carbonyl (C=O) and/or amino (–NH) groups in nylon 6 fibre might also have complicated effects on electrospinning solution properties, further resulting in morphological changes of electrospun nylon 6/MPCM fibres and their twisted nanofibre yarn.

The incorporation of MPCM also affected the diameter of composite nanofibre yarns. However, electrospun nylon 6/MPCM nanofibre yarn diameter displayed a complex behaviour. The average diameters of electrospun composite yarns increased upon the addition of MPCM and reached the maximum value of 165.1 ± 5.11 μm. The yarn contains 0.75 wt% MPCM. At higher MPCM contents, the yarn diameter decreased significantly. As presented in Figure 3, the average diameter of the nylon 6 yarn was 103 μm.

**Figure 1.** SEM images of microencapsulated PCMs (MPCMs) with different magnifications.
and when loaded with 3 wt% MPCM reached 31 ± 4.88 μm. It seems that in the lower contents of MPCM (up to 0.75 wt%), compared to neat nylon 6 yarn, no significant change has occurred in the nanofibre diameter and the presence of MPCM spherical particles within the fibres in the yarn structure prevented the nanofibres being twisted, resulting in an increase in the yarn diameter. However, by further increasing the MPCM

| MPCM content (wt%) | SEM Images | Diameter Distribution |
|-------------------|------------|-----------------------|
| 0                 | ![SEM Image] | ![Diameter Distribution] |
| 0.375             | ![SEM Image] | ![Diameter Distribution] |
| 0.75              | ![SEM Image] | ![Diameter Distribution] |
| 1.5               | ![SEM Image] | ![Diameter Distribution] |
| 3                 | ![SEM Image] | ![Diameter Distribution] |

**Figure 2.** SEM images of electrospun nylon 6/MPCM yarns and corresponding nanofibre diameter distributions at different MPCM contents.
Figure 3. Effect of MPCM content on the diameter of electrospun yarns and nanofibres.
contents, due to the decrease in solution viscosity, the average diameter of the nanofibres decreased and as the dominant factor led to a reduction in the yarn diameter. Moreover, by decreasing nanofibre diameter, they were easily twisted, and thereby the diameter of nylon 6/MPCM yarns decreased because of generating a more compact structure.

SEM images also showed that MPCMs distributed homogenously over different locations in the composite nanofibre yarn. As shown in Figure 4, by considering MPCM sizes and also nanofibre diameters, the MPCMs were located inside the nanofibres or between them (Figure 4(a, b)) and actually trapped within the yarn body (Figure 4(c, d)) or they were attached to the outer nanofibres (Figure 4(e, f)).

**Figure 4.** Distribution of MPCMs in the nanofibre yarn structure: (a) MPCM inside the nanofibre; (b) MPCM located inside the nanofibres and also between them; (c) MPCM trapped between the nanofibres within the yarn body; (d) the image of (c) with higher magnification. In (e) and (f), the MPCMs are attached to the outer nanofibres of yarn.
3.3. Thermal analysis

Figure 5 represents the DSC thermograms of neat nylon 6 nanofibre and all nylon 6/MPCM nanofibre yarns. The thermal property data from the DSC curves are summarised in Table 2. The phase transition temperatures were taken as the peak value of the DSC curves. From Figure 5, only DSC curves of nylon 6/MPCM containing 3 wt% MPCM showed both clear melting and crystallisation process peaks of the MPCM in the fibres, while the others just had a clear melting peak of the MPCM and a weak signal of crystallisation peak. The $\Delta H_c$ value for MPCM just appeared for the sample containing 3 wt% MPCM. The phase change behaviour of MPCMs in the composite nanofibre yarns suggested that the thermal energy storage property of MPCMs was significantly affected after a thermal cycle, probably relating to higher heating range in the DSC test.

As shown in Table 2, both $T_m$ and $T_c$ values of MPCM in nylon 6/MPCM composite yarns were lower than those of neat MPCM powder which is in agreement with the transition point downward tendency of PCM in the composite fibres due to relatively high melting point of nylon 6 as proposed by Chen et al. [31]. Moreover, increasing MPCM content in the nylon 6/MPCM composite yarn had no role in melting temperature of MPCM. Similar results have also been reported in the literatures on thermoregulating fibre [28,51].

Compared to those of neat nylon 6 nanofibres, the values of $\Delta H_m$, crystallinity and $\Delta H_c$ of nylon 6 in nylon 6/MPCM composite fibres first increased and then decreased with the increasing MPCM content (Table 2). It seems that low contents of MPCM could promote heterogeneous nucleation, resulting in increasing crystallinity of nylon 6 nanofibres, while in a higher content of MPCM, hydrogen bonding interactions between MPCM and nylon 6 chains might decrease the nucleating role of MPCM.

The results in Table 2 indicated that $\Delta H_m$ values of MPCM in the composite nanofibre yarns increased as the addition of MPCMs increased up to the highest content. Theoretically, the heat enthalpies of the melting/crystallisation process of MPCM in nylon 6/MPCM nanofibres can be calculated by multiplying the latent heat of neat MPCM powder and the weight fraction of MPCM in the nylon 6/MPCM nanofibre. Figure 6 shows that the theoretical and experimental values of the heat enthalpies for the nanofibre yarns containing different contents of MPCM. The results in Figure 6 indicated that $\Delta H_m$ values of MPCM in nylon 6/MPCM nanofibre yarn were slightly smaller than the corresponding theoretically $\Delta H_m$, MPCM. This may be related to hindering effect of the molecular movements during the phase change of paraffin in the MPCM. Moreover, another possible reason might be that the hydrogen interaction bonding between carbonyl groups of the MPCM shell and carbonyl/amino groups of nylon 6 polymer and also three-dimensional networks in the electrospun composite yarn substantially limited the free movement of MPCM during the melting and crystallisation process, leading to the reduced crystallinity of MPCM in the composite fibres and decreased value of enthalpies. Furthermore, by considering twisted nanofibre yarn fabrication mechanism, part of MPCMs might be lost during the electrospinning process and twist insertion, resulting in actual MPCM contents lower than the calculated values.

Based on the heat enthalpy of neat MPCM powder (164.6 J/g), higher than 80% of the heat storage capacity of MPCMs was retained after electrospinning. It is suggested that the ultrafine diameter of electrospun fibre was probably one of the contributing factors to the better thermal permeability as mentioned by the others [28].
Figure 5. DSC curves for neat nylon 6 nanofibre and nylon 6/MPCM nanofibre yarns with different contents of MPCM: (a) heating and (b) cooling.
| MPCM content (wt%) | Heating cycle | Cooling cycle |
|-------------------|---------------|---------------|
|                   | PCM           | Nylon 6       | PCM           | Nylon 6       |
|                   | $T_m$ (°C)    | $\Delta H_m$ (J/g) | $T_m$ (°C)    | $\Delta H_m$ (J/g) | $T_c$ (°C)    | $\Delta H_c$ (J/g) |
| 0 (neat nylon 6)  | –             | –             | –             | –             | 184.7         | 46.68          |
| 0.375             | 15.39         | 0.461         | 217.9         | 54.75         | 185.8         | 65.73          |
| 0.75              | 15.33         | 1.01          | 219.0         | 71.68         | 183.9         | 72.63          |
| 1.5               | 15.47         | 2.1           | 218.4         | 81.13         | 185.5         | 58.04          |
| 3                 | 15.67         | 4.286         | 220.3         | 70.02         | 183.8         | 56.19          |
| 100 (neat MPCM)   | 18.96         | 164.6         | 217.4         | 59.08         | 2.93          | 3.24           |

$T_m$, melting temperature; $\Delta H_m$, melting enthalpy; $T_c$, crystallisation temperature; $X_c$, crystallinity calculated from enthalpy of melting; $\Delta H_c$, crystallisation enthalpy.
4. Conclusion

A series of twisted nylon 6/MPCM nanofibre yarns with different contents of MPCM were prepared using electrospinning set-up with two oppositely charged needles. An instability and failure of the spinning jet were observed during production process when the MPCM content was higher than 3 wt% due to variation of solution properties in the presence of MPCM. SEM images indicated that the MPCM were randomly distributed on the surface and in the core of the electrospun nylon 6/MPCM nanofibre yarn and no MPCM agglomerations were observed. When the content of MPCM in the solution increased up to 0.75 wt%, the yarn diameter increased. At higher MPCM contents, the yarn diameter decreased significantly. \( \Delta H_m \) values of MPCM in nylon 6/MPCM composite yarn were slightly smaller than the corresponding theoretically \( \Delta H_{m,MPCM} \). This could be explained by the combination hydrogen bonding between MPCM carbonyl groups and carbonyl/amino groups of nylon 6 polymer and three-dimensional networks in the electrospun nanofibre yarns. Part of MPCM might be lost during the electrospinning process and twist insertion, resulting in actual MPCM contents were lower than the calculated values. DSC results showed that low contents of MPCM could promote heterogeneous nucleation, resulting in increasing crystallinity of nylon 6 nanofibres, while in a higher content of MPCM, hydrogen bonding interactions between MPCM and nylon 6 chains might decrease the nucleating role of MPCM. The fabricated electrospun nylon 6/MPCM nanofibre yarns are promising candidate as form-stable phase change nanofibre yarns with great potential applications in thermal energy storage and temperature regulations.

Disclosure statement

No potential conflict of interest was reported by the authors.
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