



Research Paper

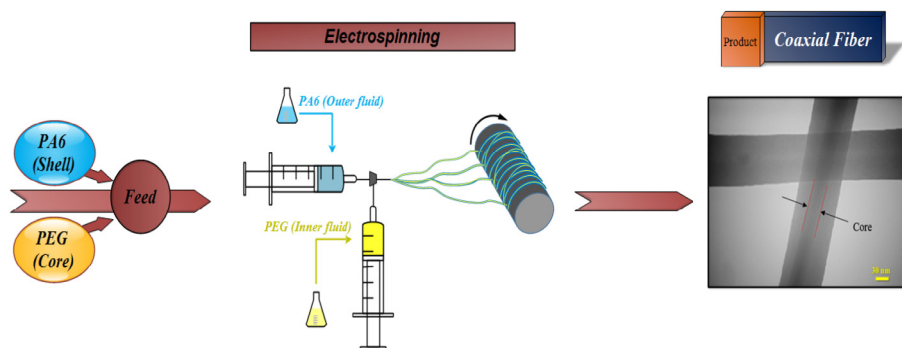
Coaxial electro-spun PEG/PA6 composite fibers: Fabrication and characterization

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HIGHLIGHTS

- Core-shell PCM nanofibers are fabricated by coaxial electrospinning.
- PEG1000 (core) and PA6 (shell) are used to fabricate nanofibers.
- The peak temperature is increased by raising the PEG concentration.
- The shell structure can prevent PEG leakage at high temperatures.

GRAPHICAL ABSTRACT



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ABSTRACT

Energy storage systems have been recognized as one of the most important technologies for conservation and utilization of renewable energy sources. In this study, core-shell phase change material (PCM) nanofibers were fabricated by using coaxial electrospinning of polyethylene glycol (PEG1000) as the core material (i.e., PCM) and polyamide 6 (PA6) as the shell (supporting) material. The effects of inner core solution flow rate and PEG content on the morphology, structure, and phase change behavior of the produced composite fibers were studied thoroughly by scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR) and differential scanning calorimetry (DSC). The experimental results indicated that by increasing the flow rate of the core solution, slightly thicker fibers can be produced, and the onset temperature of melting is reduced. Also, as the PEG concentration rises, the peak temperature increases and higher amounts of latent heat enthalpy are achieved. The results indicate that the fabricated core-shell structure has almost resolved the leakage instability normally associated with other types of PCM fibers and hence, has the potential to improve thermal storage capacity.

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Abbreviations: Bio-PCM, bio-based phase change material; C-PCM NFW, coaxial phase change material nanofibers web; CA, capric acid; DOE, design of experiments; DSC, differential scanning calorimetric; FTIR, Fourier transform infrared spectroscopy; PA6, polyamide 6; PCM, phase change material; PEG, polyethylene glycol; PU, polyurethane; PVB, polyvinyl butyral; RSM, response surface methodology; RT, temperature range; SEM, scanning electron microscopy; TEM, transmission electron microscopy; TGA, thermogravimetric analysis.

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1. Introduction

Phase change materials (PCMs) are used in many energy conversion systems primarily because of their capability for storing/releasing high amounts of thermal energy in the form of latent heat during transition from one thermodynamic state to another [1–9]. During the phase change process, the PCM temperature remains almost constant and hence, a uniform temperature can be maintained [9–11].

Nomenclature

RT	temperature range
t	time (s)
T	temperature (°C)
T	transmittance
T_m	melting point (°C)
wt.	weight fraction

<i>Greek letters</i>	
γ	latent heat (kJ/kg)
ρ	density (kg/m ³)

<i>Subscripts</i>	
m	melting
w	weight

Among different commercially available PCMs, polyethylene glycols (PEGs) have shown a great potential for use in energy systems. They are of particular interest due to their high energy storage capacity, chemical stability, negligible volume change during phase change, non-corrosiveness, non-toxicity, and low super-cooling behavior. Despite having all these advantageous features, PEGs suffer from some limitations such as low thermal conductivity and leakage issues often observed during the solid-to-liquid phase change process. In order to enhance their thermal conductivity, highly conductive materials are normally added to the phase change materials [4–6,9–14]. To alleviate leakage problem, PCMs must be encapsulated within or supported by a matrix such as expanded graphite, silicon dioxide, and/or other polymers.

The form-stable PCMs can be prepared by different methods such as sol-gel, microencapsulation, and electrospinning [15–17] among which, electrospinning is known as a simple, convenient, economic, and versatile technique for generating ultrafine (sub-micron) fibers. A wide range of polymers and/or polymer blends with nanoparticles or even drug-impregnated polymers [18,19] have been used in electrospinning.

The electro-spun fibers can be generated using either single- or double-nozzle methods [20]. However, the double-nozzle technique, also referred to as coaxial method, is an advanced, relatively new method of fabricating core-shell micro/nanofibers [21]. In this technique, two different material solutions (core and shell solutions) flow through a coaxial capillary in the presence of an electric field to generate the coaxial structures at micro/nano levels [22]. Depending on the materials used, the electro-spun core-shell structures have great potential applications in microfluidics, photonics, and energy storage systems [23].

Electro-spun PCM nanofibers have attracted the attention of many researchers in various science and engineering fields in recent years. Hu and Yu [24] encapsulated bio-based phase change material (bio-PCM) in ultrafine fibers via coaxial electrospinning technique. Natural soy wax was used as the bio-PCM for thermal storage and polyurethane (PU) as the shell material for encapsulation. The coaxial electrospinning has resulted in uniform fiber morphology with core-shell structures and homogeneous wax distribution throughout the core of the fibers. Also, the thermal analysis results show that the composite enthalpy has increased with wax content. Sun et al. [25] fabricated core-shell octadecane/polyvinyl butyral (PCM/PVB) nanofibers via coaxial electrospinning using ethanol as solvent. They found that the morphology of the core-shell structure could be improved at the higher PVB concentrations at the cost of degradation in the size uniformity. McCann et al. [26] fabricated phase change nanofibers based on the composites of long-chain hydrocarbons as the core material and TiO₂-poly vinyl pyrrolidone as the shell structure implementing melt coaxial electrospinning. Chen et al. [27] prepared the ultrafine fibers of polyethylene glycol/cellulose acetate composites through electrospinning of PEG and capric acid (CA)

mixture solutions in which CA acted as the PCM and PEG as the supporting material. They observed a high level of thermal stability for the composite fibers due to the supporting and/or shielding effect of the PEG matrix. Cai et al. [28] prepared electro-spun ultrafine composite fibers comprising of PEG4000 and PA6 via single-nozzle electrospinning method. Although the differential scanning calorimetry (DSC) results showed an increasing trend in the melting enthalpies of the fibers with increasing PEG content, the enthalpies were always lower than those of neat PEG powder. Seifpoor et al. [29] fabricated nylon6-PEG 6000 nanofibers by single-nozzle electrospinning approach. DSC tests were used to characterize the composite thermo-regulating properties. Chen et al. [30] have implemented single-nozzle electrospinning method to fabricate PA6-PEG 4000 nanofibers and performed a feasibility study of the electro-spun blended fibers as thermo-regulated materials.

Although there are a wide range of potential applications for coaxial nano-PCM fibers, there are currently few reports available on the fabrication procedure, structure, and thermal properties of the core-shell nanofiber composites. In the present study, coaxial electro-spun nanofibers of PEG 1000-PA6 were produced and examined for the first time. As the supporting material, PA6 benefits from some features, such as, good mechanical properties and thermal stability and hence, it is able to prevent PEG leaking out from the composite during solid-to-liquid phase transition. Thermal and morphological properties of fabricated nanofibers were investigated by DSC, TGA, SEM and FTIR. The fabricated coaxial phase change material nanofiber web (C-PCM NFW) composites (also called thermo-regulated fibers or temperature-adaptable fabrics) can be potentially used in sensitive thermal management applications mostly due to their thermal stability, large cycling capacity, and excellent rate of performance at an affordable price.

2. Experimental description

2.1. Materials

PEG 1000 having melting point range of 35–51.1 °C and PA6 ($T_m = 210$ °C) were purchased from Merck and Sigma Aldrich, respectively. Formic acid having the concentration of 85%, provided from Scharlau Co., was used as the PA6 solvent and 1,2 dichloro ethane purchased from Merck, was used as PEG solvent.

2.2. Sample preparation

Response surface methodology (RSM) (Design Expert software-DOE) was implemented in such a way that the best performance is reached by adjusting the key design and operating parameters. The optimum concentration ratio for PEG/PA6 was calculated to be 50/100 w/v [6]. This ratio has been used as the base concentration for all experiments.

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