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PEG2000@PAN Composite Electrospinned Nanostructures as Shape Stabilised Phase Change Materials for Thermal Energy Storage

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ABSTRACT

In this study, nanostructures were produced through electrospinning from poly(ethylene glycol) 2000 (PEG2000)/polyacrylonitrile (PAN) solutions in N, N-dimethylformamide (DMF). Since PAN is the most easily spun polymer, it acts as a scaffold for nano-dispersed PEG2000 that cannot be electrospinned into a nanofiber. In this way, PEG2000@PAN electrospinned materials can be easily applied. The solutions prepared are 10%, 20%, and 35% PAN by mass in DMF because further ratios showed leakage when PEG2000 was liquid. PEG2000 is dispersed randomly in PAN nanofibrils in the resulting structure, sufficient for shape stability and nanodispersion efficiency. DSC analysis performed in the range of 0-70 °C revealed the melting enthalpies of PEG2000@PAN 10/90, PEG2000@PAN 20/80 and PEG2000@PAN 35/65 nanofibers as averages of 1.8 J.g-1, 39.4 J.g-1, and 46.3 J.g-1, respectively. According to the SEM images, PEG2000@PAN nanofabrics showed increasing aggregation with the PEG2000 content.

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1. INTRODUCTION

Technological thermal energy storage (TES) materials technology develops with new materials and material systems with functional properties. TES materials operate in 3 categories: sensible heat storage, latent heat storage, and reversible chemical reaction heat storage. Latent heat energy storage using phase change materials (PCMs) is effective due to considerably high energy storage capacity and isothermal working [1, 2]. Electrospinning produces functional nanofibers in a laboratory for different purposes [3-5]. Nanofibers have extended property profiles due to the extended surface area, giving TES materials a short response time advantage [6-8].

In the literature, the TES characteristics of PEGs [9], their blends [10,11], and composites [12-14] have been studied widely to understand the mechanism of phase change and to eliminate leakage handicaps. PEGs have also been investigated in polymeric structures for their potential as working blocks of solid-solid PCMs at the backbone or as grafts [15-18]. PEGs cannot be electrospinned into nanofibers directly but can be co-electrospinned with suitable polymers to place their cross sections [19-21]. Yang et al. explained that various ways of incorporating PCM into textiles, including electrospinning, have been detailed [22]. Yibing Cai et al. blended some polymers with fiber characters like Polyamide 6/Lauric acid (LA) fibers, poly(ethylene terephthalate) (PET)/LA fibers, and PET/binary fatty acid fibers systematically as PCMs [23-25]. Nuray Kizidag studied paraffin/polyacrylonitrile (PAN) composites as nanofibers produced by solution electrospinning [26]. Chao Lin et al. produced some ultrafine liquid/aluminum nitride/polyamide mixtures in nanofiber form. The shape-stable composition with the amount of PCM was found in a liquid/aluminum nitride/polyamide composite at 57.0 wt%, and the enthalpy was 86.4 J/g [27]. Changzhou Chen studied the electrospinning of diacid dioctadecyl esters to get better thermal stability than 1-octadecanol in PET via blend electrospinning. The study resulted in a significant reduction in overcooling and encapsulation ratio of 50 wt% [28]. Ning Xie et al. prepared a PCM fiber based on fatty acid eutectic of mystic acid and lauric acid, PAN, via electrospinning. It was found that the leakage was avoided in the PCM fibers [29]. Changzhong Chen et al. revealed that the diameter of the CA/PEG PCM fibers increased with more PEG content, and

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the ultimate tensile strength of the final CA/PEG PCM fibers was reduced with higher PEG content [30]. Esmailzadeh used multi-walled carbon nanotubes to stabilize PEG and searched for information about property developments. In the study, polyacrylonitrile and PEG with carbon nanotubes were coelectrospinned. The solution was homogenized by ultrasonication to reduce the nanotube aggregations. It was found in the study that individualized multi-walled carbon nanotubes within the aqueous media using two nonionic surfactants with different hydrophilic/lipophilic balance values (Triton X-100 and Tween-80) were effective. Electrospinning conditions were optimized, and nanofibers, so-called shape-stable nanofibers with the lowest possible diameter, were produced by electrospinning [31]. Noyan et al. produced electrospinning fibers of PEGs by coaxial electrospinning to avoid possible viscosity problems caused by low molecular weight. They studied some low molecular weight PEGs (400–2000 gmol−1) due to their thermal properties, especially relatively low working temperatures, chemical stabilities, and biodegradability. They also studied three methyl ethers of PEGs for the first time as shape-stable thermal energy storage materials. The coaxial electrospinning brought about uniformly shaped bicomponent nanofibers as shape-stabilized nanocomposites [32].

Ji et al. coelectrospinned PEG and poly(vinyl pyrrolidone) because they are adaptive to PEG. There was also graphene oxide as a thermal conductivity enhancer in the system. The resultant material was proposed as a novel thermo-regulated smart nanofiber. The influences of graphene oxide ratio on the microstructure, TES properties, thermal conductivity, and thermal stability were revealed [33].

Liu et al. produced PEG8000@PAN nanofibers from dimethyl formamide (DMF) solutions at different composition ratios. PEG8000 has a considerably higher molecular weight and suitable character to electrospin than the low molecular weight PEG2000 used in this work. PAN/PEG composite nanofibers were shape-stable PCMs for thermal energy storage. Their findings were noteworthy because of the simplicity of blending and cost efficiency compared to the systems with emulsifiers [34].

Electrospinning uses electric force to draw charged threads of polymer solutions or sometimes polymer melts up to several hundred diameter nanosizes. It produces a conventional solution of dry spinning of fibers by electrospraying. The process operates at room temperature in general and does not need the use of coagulation chemistry. This enables the spinning of large and complex molecules. Electrospinning is also performed from molten precursors to ensure that there is no solvent in the final product. A sufficiently high voltage is necessary to charge the body of the liquid for electrostatic repulsion that counteracts the surface tension, and the charged droplet is stretched. After a threshold, a stream of liquid erupts from the surface to the mat (the point is known as the Taylor cone). A possible high cohesion may result in stream breakup not occurring, and the charged liquid jet and electrospraying occur [35].

The jet dries during flight, and the current flow mode turns from ohmic to convective. Electrostatic repulsion causes whipping, which drives the jet to elongate until it is accumulated on the grounded collector. The thinning of the fiber by elongation instability leads to uniform fiber formation at nanometer scales [36].

There are some critical parameters for material production through electrospinning. The polymer's molecular weight, molecular-weight distribution, and architecture (branched, linear, etc.) are effective in electrospinning the solution viscosity of the polymers. Polymer type also effectively determines solution properties (viscosity, conductivity, and surface tension). Also, conditional parameters, electric potential, flow rate, and concentration result in some effects on the fiber structure. The distance between the capillary and collection screen may determine the nanofiber structure. Furthermore, ambient parameters (temperature, humidity, and air velocity in the chamber) may differ in the fiber's physical state. In some applications, the collector can be mobile, in which case the collector's movement and the target screen's size have an effect. Needle gauge is another factor on electrospinned fibers. The parameters were general to many electrospinning studies [37].

The used solvent evaporates during the transfer, and the polymer accumulates in nanofiber form on a collector material [38-39]. Electrospinned nanofibers have interesting properties like high surface-area-to-volume ratio, low density, high pore volume, and functionalization possibilities [40-42]. Therefore, they are used in miscellaneous applications such as filtration [43], energy harvesting and storage [44], tissue engineering [45], drug delivery [46], wound healing [47], sensors [48], reinforcing some polymer reinforcement [49], etc. Electrospinned composites are produced to combine properties of the constituents in a single nanofiber. Some polymers are added to bear functionality, like antistatic nanofibers with polyaniline [50], antibacterial nanofibers with silver nanoparticles [51], and tensile properties with the addition of carbon nanotubes [52].

PAN and its derivatives are widely used in producing nanofibers using the electrospinning method. Electrospinning of PAN in DMF solvent is a prevalent method, and in many studies, it is considered the essential component to obtain nanofiber structures with different contents. Fig.1 shows the apparatus used in this work with an SEM photograph taken. PAN is a widely used material with a wide range of uses. It is usually produced by adding a comonomer such as vinyl acetate, methyl acrylate, methyl methacrylate, and itaconic acid to the recipe of the main ingredient, acrylonitrile, to improve its applicability. PAN in electrospinned materials is always beneficial because it is readily available at desired amounts and reasonable costs [53]. In the literature, carbon nanofibers were obtained by the carbonization process after obtaining nanofibers using a PAN/Novolac mixture [54]. Poly(vinyl alcohol) was transformed into the ultrathin layer by the electrospinning method [55]. Cyclodextrin-functionalized poly(methyl methacrylate) nanofibers were produced by electrospinning as new functional nanowebs [56]. The antibacterial properties of polyethylene oxide and chitosan nanofibers were investigated by electrospinning [57]. Ambient parameters of poly(trimethylterephthalate) nanofibers were studied by İcoglu and Ogulata [58].

It is seen from the literature that PEG at low molecular weights has not been reported to be electrospinning in PAN solution. It can be attributed to the insufficient solution dynamic properties of low molecular weight PEGs to electrospin. The study focuses on fabricating a shape-stable fabric form that can ease application and react more quickly to thermal stimuli due to the expanded surface area by electrospinning PEG2000 in PAN solution. The structure does not need to be an ideal nanofiber because these fabrics are placed constantly and do not require important mechanical constraints. As a PCM, PEG did not disperse homogeneously into the matrix structure of nanofibers in previous studies but formed its heterogeneous domains within the fiber crosssection that do not distort the appearance. For TES applications, electrospinning PAN materials with colloidally dispersed low molecular weight PEG polymers can be applicable if shape stability is maintained. Low molecular weight PEG-PAN solutions have insufficient solution dynamic properties for electrospinning. These materials can be used in geotextiles, electronic cooling systems, etc. In this study, PEG2000 dispersed structure in PAN matrice was achieved by simple electrospinning in DMF.

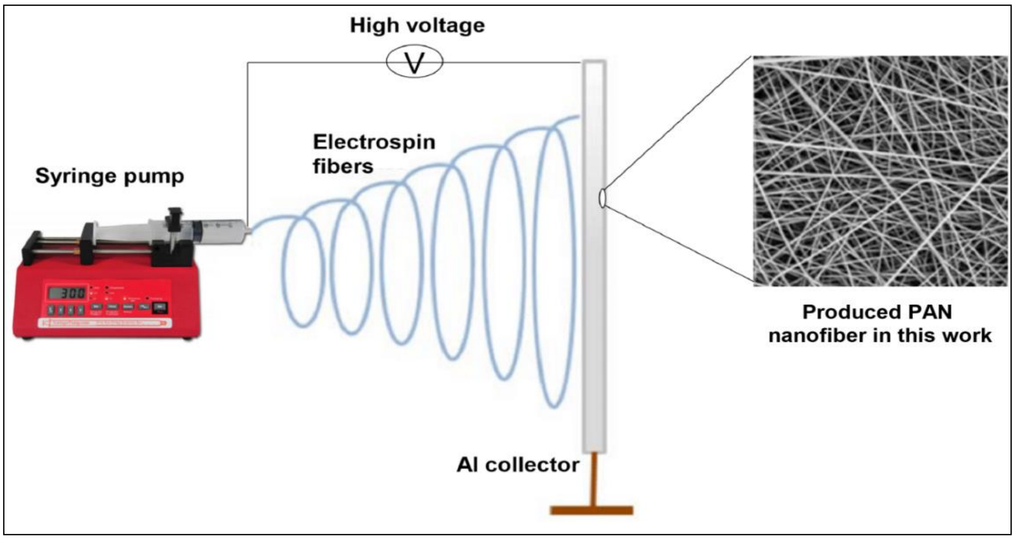

Figure 1 Schematic diagram of the electrospinning device with original SEM photo of electrospinned fiber produced in this study

2. MATERIAL AND METHOD

PEG2000 purchased from Sigma Aldrich, 5 mL Ayset brand syringe, DMF purchased from Merch, PAN obtained from AKSA Acrylic Chemicals Company, an Electrospinning Device (Electrospin 100) operating between 0-25 kV and purchased from Nanoweb Instruments were used for PEG2000@PAN electrospinned fibers production. The electrospinning apparatus is shown in Figure 2. PEG2000 and PAN are dissolved with different compositions in DMF solvent and left to stir for 5 hours. Electrospinning nanofibers with higher ratios than 35% PEG2000 showed seepage and, therefore not further characterized, while nanofibers with 35% PEG2000 and less were classified as shape-stable nanofibers. For electrospinning, the polymer solutions were filled into a 5 ml syringe, taking care not to leave any air bubbles. The syringe was mounted on the electrospinning system (Fig. 2). A distance between 7 to 20 cm was maintained between the collection screen and the syringe tip. While the applied voltage was changed in the range of 10 to 20 kV, the pump flow rate was changed in the 1.0-3.0 ml/hour range.

Solution parameters were measured for each solution prepared, and dynamic viscosity measurements of the shell and core solutions were performed at 25 °C. SEM images show that beaded fibers are obtained mostly instead of smooth fibers in the nanoweb samples. The polymer blend solutions were electrospinned at a voltage of 15 kV with a solution feeding rate of 1 mL/h, and at a receiving distance of 15 cm.

2.1 FT-IR Spectroscopy Analysis

FT-IR spectroscopy was used to analyze the presence of different functional groups in the PEG2000@PAN electrospinned nanofibers. For this purpose, the Jasco 430 model FT-IR spectrophotometer was used to analyze the samples between 4000 cm^{-1} and 400 cm^{-1}.

2.2 DSC Analysis

The physicochemical properties of nanofiber fabrics as TES materials were investigated using data obtained from the Netzsch DSC214 Polyma model DSC instrument. Commonly investigated properties are phase change (melting and crystallization) temperatures and enthalpy values. The DSC device calibration was done with an indium standard. The conditions of measurements were experienced in the previous studies. Temperature interval of measurements, heating/cooling rate, atmosphere, and gas flow rate were 0-70 °C, 5 °C min^{-1}, high purity nitrogen, and 60 ml/min, respectively, during the measurements. Melting and freezing points were onset temperatures given by the computer software. It was well known that the onsets were placed by crossing points of base and signal lines. Overcooling is the difference between melting and crystallization onsets. The latent heat capacity was the area of the phase transition peak determined by numerical integration utilizing computer software. Experiments were performed in 3 replicates and reported as an average. Total enthalpy curves were produced using the DSC data according to our previous work [10].

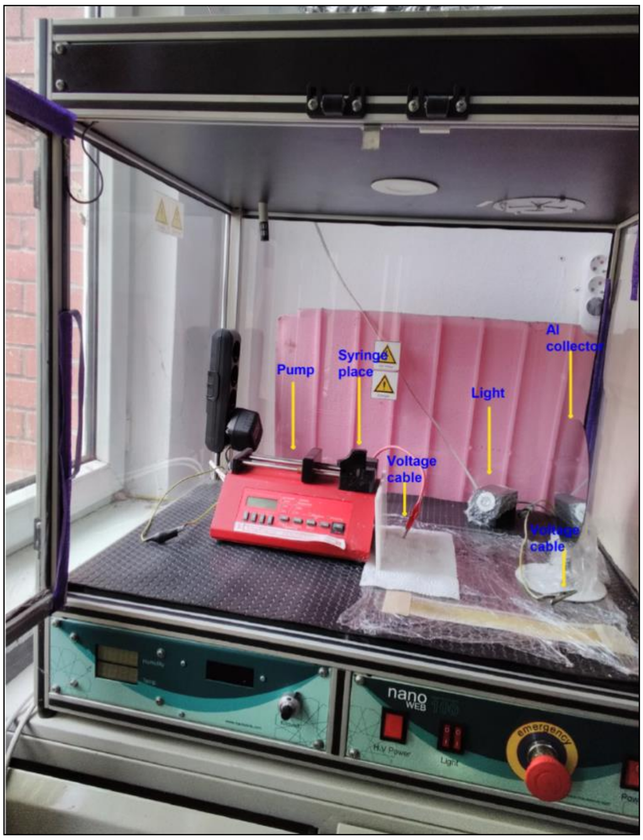

Figure 2 Electrospinning system

2.3 T- History Analysis

Researchers have frequently used the T-History method to measure the temperature-time dependency curves of PCMs by cooling and heating samples at a predetermined rate. This study held samples at -12 °C in the refrigerator (Samsung RT50H6360WW) for 30 min. and immersed into 70 °C using a Polyscience model constant temperature circulation bath. The graphs drew 0-60 ºC periods to eliminate prior and after thermal effects during heating and cooling. The temperature change of the samples was monitored using a Nova 5000 model datalogger. The cooling turn was applied oppositely using a constant temperature circulation bath from 70 °C to 0 °C.

2.4 SEM analysis

SEM analysis of nanostructures is critical to reveal the average nanofiber diameter, diameter distribution, and PEG2000@PAN nanofiber system's morphology, which differs from that of joint structures. SEM analyses were obtained by using the Mira 3 Tescan SEM instrument.

3. RESULTS AND DISCUSSIONS

3.1. Structured characterization by FT-IR spectroscopy analysis

The PEG2000@PAN 35/65sample was analyzed by FT-IR spectroscopy to reveal the copresence of the polymers (Fig.3). The analysis is unique for some shape-stable materials due to its efficiency and possibility to be applied to low soluble systems [33]. Aliphatic carbon ($-CH_2CH_3$) peak (2800 -2900 cm^{-1}) was observed in all samples. Polymeric oxygen-carbon (O-C) peak (2200-2300 cm-1 and 1600-1700 cm-1) was observed for PEG and PEG2000@PAN 35/65. It indicates that the PEG 2000 polymer was dispersed within the PAN nanofibers.

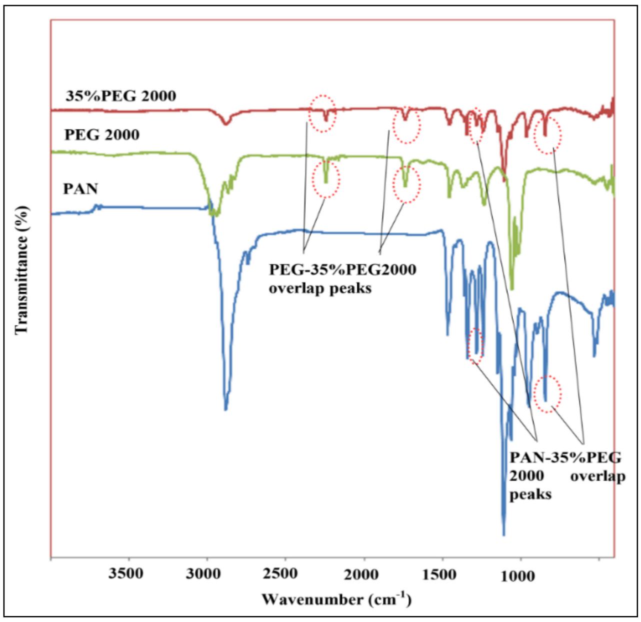

Figure 3 FTIR spectra of PAN, PEG2000, and PEG2000@PAN 35/65 electrospinned nanofibers

3.2. Physicochemical properties by DSC analysis

TES materials have some common characteristics that are generally determined by DSC instruments. Among all phase changes, temperature and enthalpy determine the application temperature and suitability of the materials for TES applications. Thermal structural and performance stability are also determined by utilizing DSC measurements. For the determination of the physicochemical properties of the nanostructures produced through electrospinning, DSC curves, and their evaluations have been performed. Fig. 4 shows the nature of the curves drawn for the heating and cooling operations. It is seen from the curves that when the ratio of PEG2000 is low, the storage capacity is deficient. It may be attributed to the fact that some PEG2000 is miscible at low concentrations in the PAN matrix. The situation is consistent with the miscibility window theory of polymer blends. When the ratio is increased to 20% by mass and then 35%, the excessive amount increases and the phase is separated in the system. This causes the enthalpy to increase. PCM composites are not miscible systems; miscible systems are mixtures close to ideal, which means no crystallinity of each of the constituents. The data are also summarized in Table 2. PEG 2000@PAN35/65 system showed the highest melting and crystallization enthalpy.

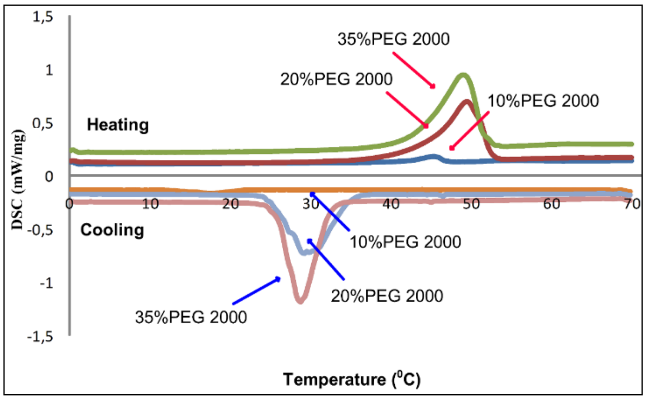

Figure 4 DSC heating-cooling thermograms forPEG2000@PAN nanofibers including PEG2000@PAN 10/90, PEG2000@PAN 20/80 and PEG2000@PAN 35/65

Fig. 5 shows the DSC graph obtained after heating and cooling 10 times for PEG2000@PAN 35/65 polymer. According to the graph, heating and cooling 10 times did not cause any significant changes in PEG2000@PAN 35/65 thermal properties. This shows that the obtained PEG2000@PAN 35/65 sample is sustainable in terms of melting and crystalizing properties.

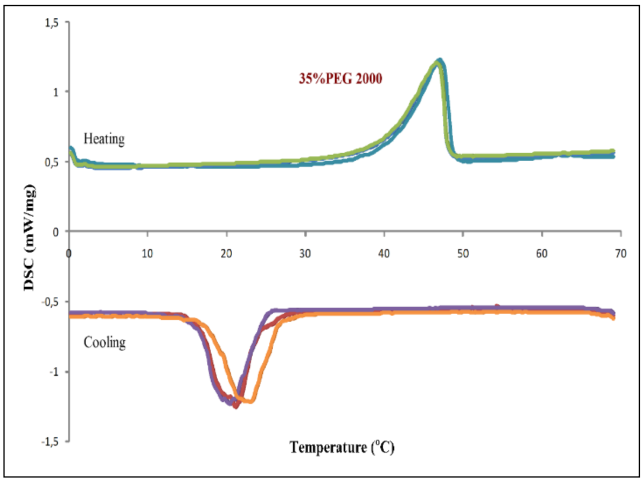

Figure 5 DSC curves of PEG2000@PAN 35/65 electrospinned nanofibers (10 heating-cooling cycles)

Table 2. DSC data for PEG2000@PAN 10/90, PEG2000@PAN 20/80 and PEG2000@PAN 35/65 electrospinned nanocomposite structures

	Melting temperature (℃)	Melting enthalpy (J.g^{-1})	Crystallization temperature (℃)	Crystallization enthalpy (J.g^{-1})
PEG2000@PAN 10/90	42.8	1.81	20.7	2.3
PEG2000@PAN 20/80	44.9	39.4	34.3	39.0
PEG2000@PAN 35/65	43.5	46.3	32.3	45.5

The molecular weight of PEG is very much effective in the solution dynamics of PEG-PAN in DMF. PEG2000 is a good alternative to use in solar applications in spite of weak solution dynamics.

3.3 Total enthalpy of storage and release

Heat capacitances in the sensible heat regions (below and above the phase transition temperature) are used for the selected temperature range when calculating the total enthalpy. DSC data is used for heat capacity calculation [57]. That is, DSC gives a graph of heat transfer power versus temperature. The temperature axis is also the time axis as the temperature is increased/decreased at constant heating/cooling rates. The product of energy and time is the heat taken in or given off. Here, the integral graph can be drawn using a simple graph program.

Total enthalpies of electrospinned nanocomposite structures from DSC data are given in Fig. 6. Total enthalpies of shape stable PEG2000@PAN 10/90, PEG2000@PAN 20/80 and PEG2000@PAN 35/65 electrospinned nanocomposite structures had 105.0 J.g^{-1}, 162.1 J.g^{-1}, and 258.0 J.g^{-1} respectively during heating for the temperature interval as the total enthalpies of them were -110. J.g^{-1}, -184.4 J.g^{-1}, and -246.5 J.g^{-1}, respectively, for the exact temperature interval during cooling.

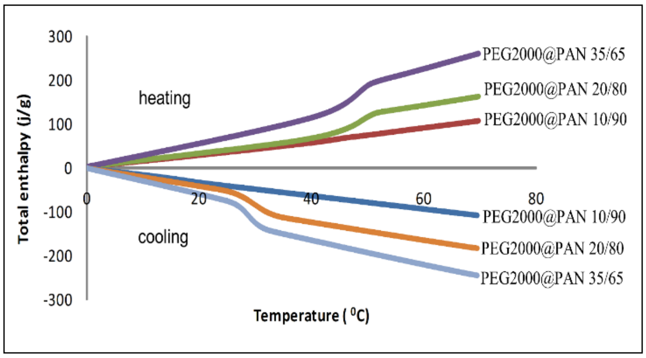

Figure 6 Total enthalpies of PEG2000@PAN 10/90, PEG2000@PAN 20/80, PEG2000@PAN 35/65 electrospinned nanofibers during heating and cooling between 0 and 70℃

3.4. T-history analysis

T-history is a method in which all thermophysical properties of materials work together. This proves the applicability of the materials. It is accomplished with much more than the substance used in DSC. The effectiveness of this method also varies according to the practitioner because the system is a relative analysis system created by each working group with its design. Scientists working on TES are familiar with this method, and everyone interprets the temperature-time graph similarly. In this study, since a suitable system is used to increase the heat transfer rate, it is possible to determine the material efficiency precisely. Fig. 7 shows T-history graphs of PEG2000@PAN 10/90, PEG2000@PAN 20/80, and PEG2000@PAN 35/65 electrospinned nanocomposite structures. Measurements are highly compatible with DSC analyses for both heating and cooling periods. These analyses show the extent to which the supercooling behavior is realized. T-history analyses are repeated with new optimizations until the same graphs are drawn for each material.

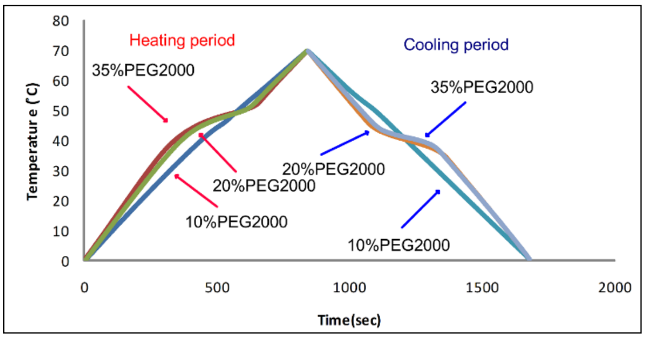

Figure 7 T-history results of PEG2000@PAN 10/90, PEG2000@PAN 20/80 and PEG2000@PAN 35/65 electrospinned nanofibers

T-history analysis showed that as the amount of PEG2000 increased, there was an improvement in the warm-up and cooling times. Especially for electrospinned fabrics with 20% and 35% PEG content, the heating remained constant for a significant period during the phase transitions. The same trends were followed for the cooling behavior. T-History reveals the temperature behavior that can occur during the use of materials and has shown that electrospinned fabrics responded to the temperature changes during phase transition.

3.5 Leakage proof property of PEG2000@PAN materials

The leakage test was carried out by placing the samples in an oven at 70 °C and waiting 1 hour. As a result of this test, while PEG2000 was completely liquefied at 70 °C (melting point range of 48-52), no leakage was observed in PEG2000@PAN samples (Fig. 8). PEG2000 was placed on the board for comparison.

The studies determined the highest rate of PCM remaining without liquid leakage in the fiber. The same has been aimed before in shape stable polymer PCM composites. Here, the ratio of PEG islands dispersed in the fiber form without leakage was 35%.

3.6 Analysis of PEG2000@PAN nanofibers

Electron microscopy analysis is the most widely used and essential method for morphologically characterizing micro/nano-scale materials. In this study, the nanofiber structure was proven by SEM. Fig. 9 shows the SEM images of PAN and PEG2000@PAN electrospinned nanocomposite structures. According to the SEM images, PEG2000 was dispersed in the matrix with increasing aggregation instead of fiber diameter expansion, while PAN provided perfect fiber formation. This is consistent with the energy storage capacity of nanofibers increasing with PEG2000 content.

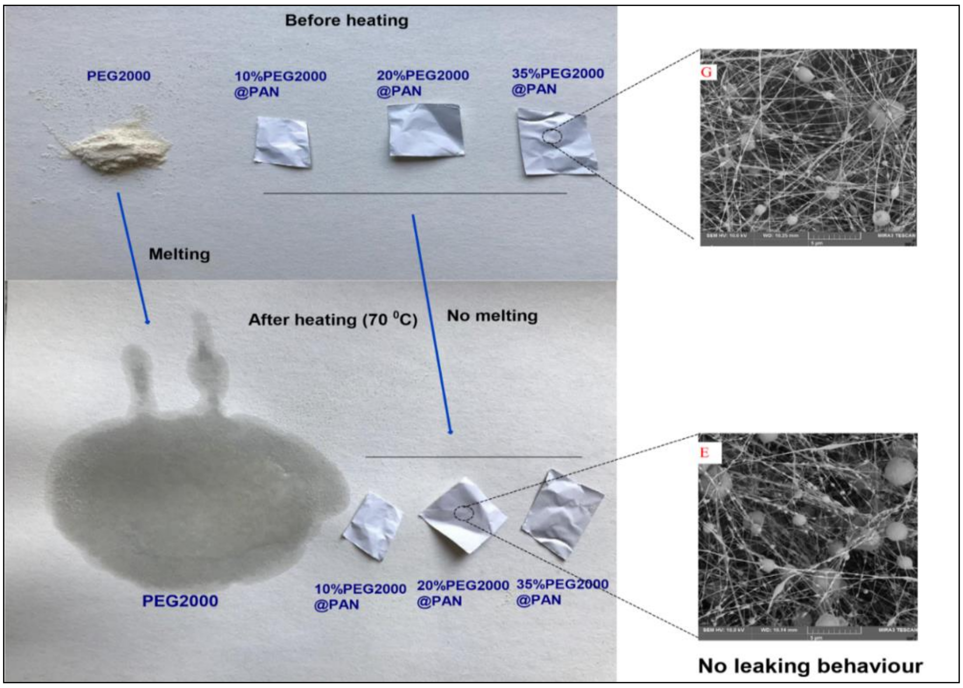

Figure 8 Leakage test of PEG2000 and PEG2000@PAN nanofiber samples

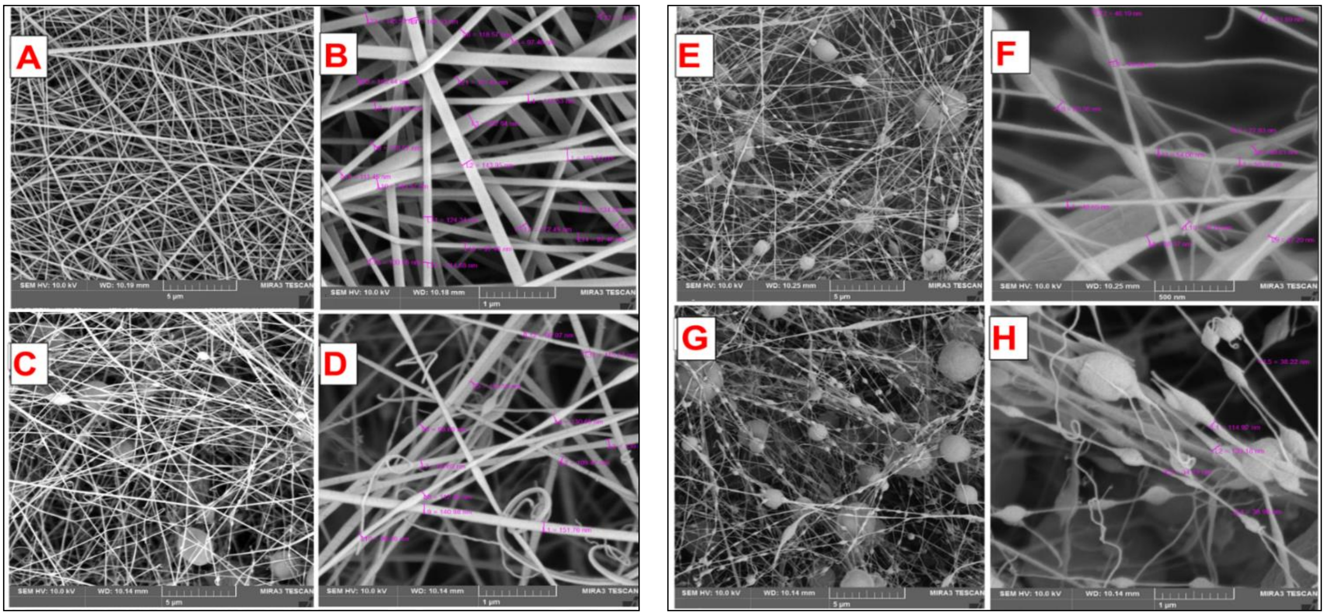

Figure 9 A)10K magnification PAN **B)** 50K magnification PAN **C)** 10K magnification 10% PEG2000 **D)** 50K magnification 10% PEG2000 **E)** 10K magnification 20% PEG2000 **F)** 50K magnification 20% PEG2000 **G)** 10K magnification 35% PEG2000 **H)** 50K magnification 35% PEG2000 for SEM images

The bead formation is not preferable in electrospinned nanowebs resulting in heterogeneity in the properties. However, PEG2000 has never been electrospinned due to viscosity properties in the past, but in the presented heterogeneous form, it was shape stabilized with a very extended surface area and in fabric form for ease of application. Furthermore, the mechanical constraint of fiber or smooth morphology is not a requisite for TES applications. Fixing samples of electrospinned materials to a building wall or use in an electronic device cooling can be examples of the applications for the presented materials.

4. CONCLUSION

PEG2000@PAN nanocomposite structures were produced as shape stabilized PCMs for TES applications by electrospinning method in DMF solvent. The nanocomposite structures produced are structurally stable, i.e., show no leakage at high temperatures, have an expanded surface area to respond to thermal stimuli, and are easy to apply due to their solid properties. Although PEG polymers do not allow electrospinning according to literature findings and as a result of the studies carried out in this study, systems with PEG2000 dispersion produced have been obtained with various advantages in terms of PCMs that can store heat energy. The study observed that as the ratio of PEG2000 increased, the ability to store heat energy also increased. Still, it was observed that the nanoclusters formed by PEG also developed dimensionally. Therefore, PEG2000 nanostructures can be produced in compositions that can be evaluated as optimum by electrospinning. The produced nanostructures have been successfully proven by analysis methods such as SEM and DSC regarding structural stability and performance continuity and morphologically in terms of their heat energy storage capabilities. Shape stable PEG2000@PAN 10/90, PEG2000@PAN 20/80, and PEG2000@PAN 35/65 nanocomposite structures are characterized in terms of total enthalpy of sensible and latent heat, with significant values such as 105.0 J.g-1, 162.0 J.g-1, and 258.3 J.g-1 in a specific temperature range repeated in both heating and cooling. In addition, its current properties are also proven by T-history analyses that pointed to its applicability. However, the PEG2000@PAN electrospinned nanocomposite structures melted around 48-52 °C and were heated up to 70 °C to prove no leakage.

This study will form a basis for electrospinning studies to be carried out by adding thermal conductivity agents that will not cause problems to the syringe system for evaluation in the electrospinning method.

In addition, it is expected that this study will be validated in heating systems in buildings to be more effective than microencapsulated PCMs or to solve some problems in TES systems. The nano webs produced in this research have a fabric body that can be fixed onto building walls or used in electronic equipment. The presented materials are easy-to-use systems in TES systems, and they do not need common mechanical properties or smooth fiber morphology. In addition, extended surface area and networks of the interlinks will help energy transfer and fixation to different media.

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