

Fabrication of Electrospun Nonwoven Mats of Polyvinylidene Fluoride/Polyethylene Glycol/Fumed Silica for Use as Energy Storage Materials

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ABSTRACT: Nonwoven mats were fabricated from a polyvinylidene fluoride (PVDF)/polyethylene glycol 1000 Da (PEG 1000) blend by the electrospinning process. PEG 1000 is a low-temperature phase change material that acts as a heat storage material, while PVDF serves as a supporting material to afford a mechanically strong form-stable structure. Addition of fumed silica to electrospun (e-spun) PVDF/PEG 1000 nanofibers helped prevent the leakage of molten PEG 1000 during the solid–liquid phase change, as confirmed by field emission scanning electron microscopy. It was also found that the addition of fumed silica helps improve the mechanical strength of the PVDF/PEG 1000 nonwoven mats. The thermal properties of PVDF/PEG 1000 and PVDF/PEG 1000/fumed silica composite nonwoven mats were investigated by differential

scanning calorimetry. There were no significant changes in the melting temperature and heat of fusion of PEG 1000 after 100 heating/cooling cycles, thereby confirming that these nonwoven mats have high thermal stability. The fumed-silica-incorporated PVDF/PEG 1000 nonwoven mats had large energy storage capacity and excellent mechanical strength. Hence, the aforementioned types of mats are thought to have various applications in energy storage and in the manufacture of thermoregulating textiles. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 121: 3596–3603, 2011

Key words: fibers; thermal properties; differential scanning calorimetry; phase change material; polyethylene glycol

INTRODUCTION

In the past few decades, there has been a dramatic increase in energy consumption at global level. That is the reason why there is strong demand for the development of energy-saving methods. Thermal energy storage has been shown to be an efficient and economical energy-saving technique. Among the various thermal energy storage techniques of interest, those involving the use of phase change materials (PCMs) are particularly attractive. These materials undergo a reversible phase change, and hence they can absorb energy upon heating and release the absorbed energy during cooling.^{1–3} Because most PCMs undergo a solid–liquid phase change substances, it must be sealed in a special packing to prevent leakage of the material after conversion to the liquid phase. The use of containment methods that entail

storing the PCM in tubes, pouches, spheres, panels, etc., causes a marked decrease in the conductivity of the PCM.⁴ Therefore, many studies have been carried out with the aim of solving this problem. One possible solution would be the enhancement of the thermal conductivity of conventional PCMs. In this method, various porous matrices with different thermal conductivities and porosities are impregnated with PCM.⁵ Graphite and silica are well known to have high thermal conductivity and high porosity. When such a material is mixed with a PCM, the molten PCM is absorbed in the pores of the material, resulting in the formation of a composite. Haillot et al.⁶ reported that the composite materials formed from expanded graphite and PCM are stable at ambient temperature and that the thermal conductivity of these materials can be increased by a factor of 5–100 depending on the density of graphite. Wang et al.⁷ used a polyethylene glycol (PEG)/silica blend to create a new type of composite, form-stable PCM. In the case of the PEG (80%)/SiO₂ (20%) composite, the latent heat was 137.7 J/g, and the thermal conductivity was increased by 21%.

Microencapsulation has been proposed to be an effective means of increasing the heat transfer rate in PCMs. Microcapsules are particles with a wide

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range of sizes, that is, submicron-sized to several micron-sized particles; in these microcapsules, the PCM is enclosed by a thin polymer film.^{8–11} Because of their small diameter of the microencapsulated particles, these microencapsulated particles have a high-surface-area-to-volume ratio, and their thermal conductivity is well enhanced.¹² Sari et al.¹³ reported that *n*-octacosane-containing poly(methylmethacrylate) (PMMA) microcapsules prepared by emulsion polymerization have good thermal and chemical stability and that the phase change temperature and enthalpy show no significant change even after 5000 heating/cooling processes. Similarly, the preparation and characterization of PMMA/docosane microcapsules were reported by Alkan et al.¹⁴ Encapsulated paraffin wax prepared by Hawlader et al.¹⁵ showed a thermal energy storage/release capacity of about 145–240 J/g, indicating that it potential to be used as a solar energy storage material.

In a recently reported method, it was found that a PCM could be permanently locked within polymer fiber by direct electrospinning of a PCM/polymer blend solution.^{16,17} Electrospinning is a simple, convenient technique for producing ultrafine fibers with diameters ranging from a few nanometers to several micrometers.^{18–20} Thin electrospun (e-spun) nonwoven mats comprising a PCM and a polymeric materials have desirable dimensions, high latent heat, excellent mechanical strength, and large specific surface area. Hence, they can be directly used as effective energy storage material in various applications, especially for the manufacture of thermoregulating textiles. Chen et al.¹⁶ reported the successful fabrication of ultrafine fibers of a PEG/cellulose acetate composite, in which PEG acted as the PCM, and cellulose acetate acted as the polymer matrix, via electrospinning. The composite fibers had high latent heats of melting and crystallization (85.91 J/g and 64.95 J/g, respectively). The e-spun PEG/cellulose acetate composite fibers were crosslinked for further improvement of the thermal stability and the water resistance.²¹ However, this crosslinking resulted in a significant decrease in the enthalpy of the e-spun fibers. Chen et al.¹⁷ also reported the development of a novel polymer-based shape-stabilized PCM from the ultrafine fibers of a lauric acid/PET composite. Until date, however, there are very few reports on the preparation of PCM/polymer nonwoven mats by electrospinning.

In this study, polyvinylidene fluoride (PVDF)/PEG blend nonwoven mats containing fumed silica were prepared by electrospinning. PEG is a polyether compound that is widely used as a PCM. The thermal storage capacity and melting point of PEG are dependent on its molecular weight.^{22,23} PVDF is a supporting polymer that can help maintain the shape of the e-spun fibers and enhance the mechanical

strength of the nonwoven mats obtained from these fibers. Fumed silica is added to the blend solution of PVDF and PEG before electrospinning in order to prevent the leakage of the melted PEG during phase change. Furthermore, the addition of fumed silica helps increase the mechanical strength of the mats. The morphology and thermal properties of the nonwoven mats prepared from the e-spun fibers are characterized by field emission scanning electron microscopy (FE-SEM) and different scanning calorimetry (DSC), respectively. In addition, the tensile properties of these nonwoven mats are evaluated.

EXPERIMENTAL

Materials

PEGs with average molecular weights (M_w) of 600 and 1000 Da were purchased from Samchun Co. (Korea), while PEGs with M_w of 2000 and 4000 Da were supplied by Daejung Chemicals and Metals Co. (Korea). Hohnson Matthey Co. (USA) supplied PEGs with M_w of 8000 Da. Kynar@761 PVDF was used. *N,N*-Dimethylacetamide (DMAc) and acetone solvents were purchased from Samchun Co. (Korea). Fumed silica (average particle size: 7 nm) was obtained from Sigma Aldrich. All the chemicals were used as such without further purification.

Electrospinning process

PVDF was dissolved at a concentration of 20 wt % in a mixture of DMAc and acetone (60 : 40, w/w). PEG and fumed silica were then added in the polymer solution. The amount of PEG was calculated from the weight percentage of PEG in the polymer solution, while the amount of fumed silica was equal to 2 wt % of the PEG. These solutions were stirred at 50°C until the mixture became clear. Electrospinning was carried out using these blended solutions at 25°C. Each of the prepared solutions was poured into a standard 5-mL plastic syringe that was attached to a blunt 22-gauge stainless steel hypodermic needle. The solution flow rate was controlled using a syringe pump (KD scientific, Model KDS200, USA). An aluminum sheet was wrapped around a rotating collector that was connected to the negative electrode. A high supply voltage (Chungpa EMI, Korea) was applied to the hypodermic needle as a positive electrode. The polymer solution was electrospun at a positive voltage of 11 kV, needle tip-to-collector distance of 17 cm, and solution flow rate of 1 mL/h.

Characterization

The ionic conductivity of the polymer solution was measured using a microprocessor conductivity meter

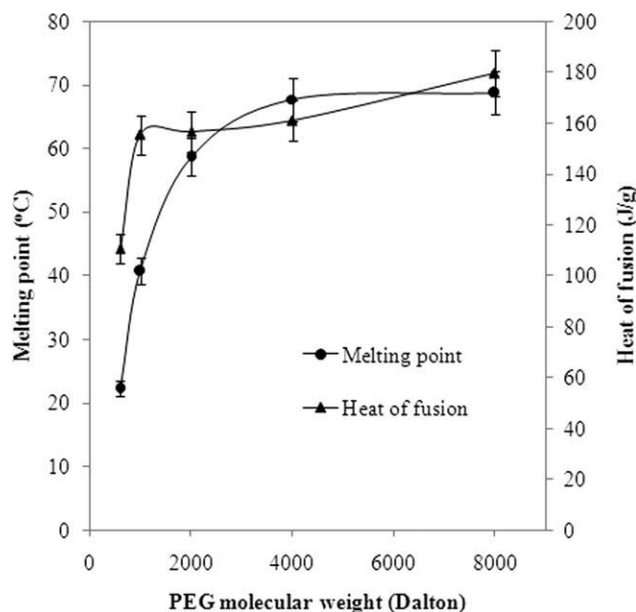


Figure 1 Relational curves between melting point, heat of fusion, and different molecular weights of PEGs.

(HI2300, Hanna Entrustments). The viscosity of the polymer solution was obtained with a Brookfield DV-II+ viscometer (USA).

The morphology of the nonwoven mats was determined by FE-SEM (HITACHI S-4700, Japan) using a coating system (BAL-TEC MED020). From the FE-SEM photographs, the ranges of the fiber diameters and the average fiber diameters of the nonwoven mats were measured using visualization software (TOMORO ScopeEye 3.6).

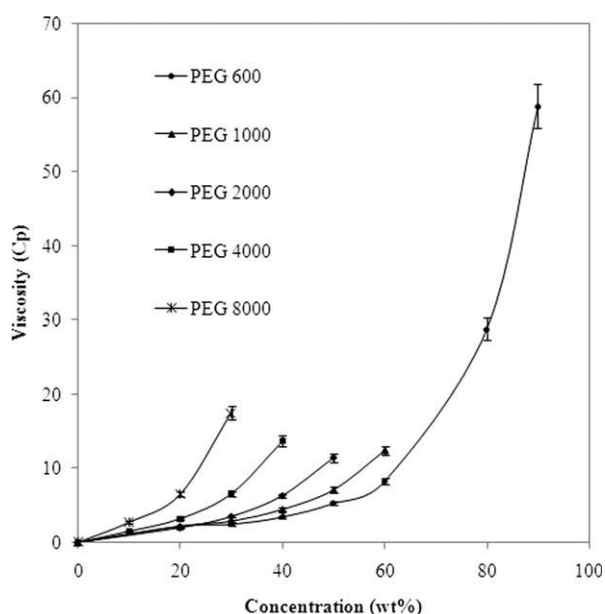


Figure 2 Relational curves between the viscosities and concentrations of PEG 600, PEG 1000, PEG 2000, PEG 4000, and PEG 8000. The experiments were conducted at 25°C.

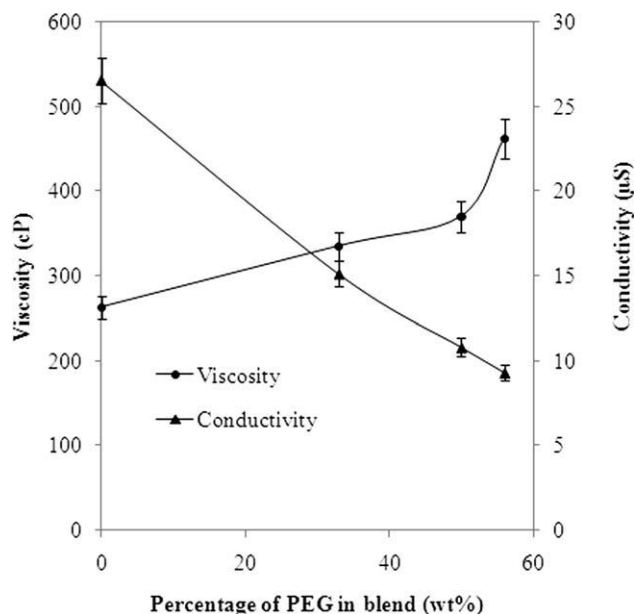


Figure 3 Relational curves between the viscosity and conductivity of the PVDF/PEG 1000 solution with different weight percentages.

The thermal properties of the nonwoven mats, including the melting and crystallization points and the latent heat, were measured by DSC (SH IN2920, TA Instruments, USA). The analyses were carried out at a 10°C/min heating rate under a constant stream of nitrogen at a flow rate of 70 mL/min. A thermal-cycling test consisting of 100 consecutive melting and freezing processes was conducted by DSC to determine the thermal reliability of the nonwoven mats.

The mechanical performance of the nonwoven mats was characterized by tensile testing on a tensile tester (LR 5K, LLOYD Instrument) with a load cell of 10N.

RESULTS AND DISCUSSION

Selection of PEGs with a suitable average molecular weight

PEG is a crystalline polymer with $(\text{CH}_2\text{—O—CH}_2)_n$ units in the chain and hydroxyl groups at two ends. The physical properties of PEG, including viscosity, melting, and crystallization points and latent heat vary depending on its molecular weight. The main requirements for developing a material with high-energy storage capacity by electrospinning are low viscosity and high latent heat. In this study, five PEGs with different molecular weights were evaluated: PEG 600, PEG 1000, PEG 2000, PEG 4000, and PEG 8000. The numbers that are included in the names indicate the average molecular weights, that is, the PEG with an average molecular weight of ~ 600 Da was labeled PEG 600. Figure 1 shows the

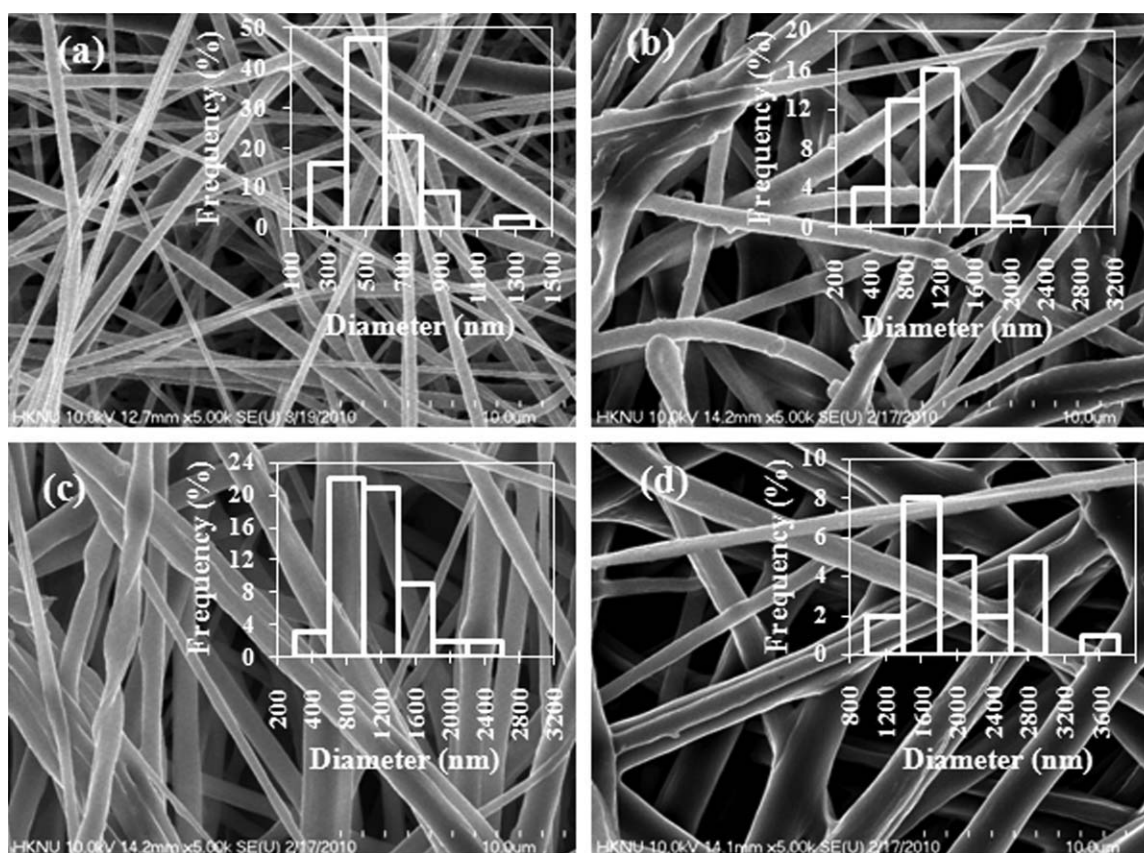


Figure 4 FE-SEM images of e-spun fibers of PVDF/PEG 1000 blend with different percentage of PEG 1000: (a) 0 wt %, (b) 33 wt %, (c) 50 wt %, and (d) 56 wt %. The inserted graphs show the diameter distribution of nanofibers.

melting point and heat of fusion of the PEGs with different molecular weights, as measured by DSC. The melting point and heat of fusion of the PEG increased sharply with an increase in the molecular weight from 600 to 1000 Da. The heat of fusion of PEG 1000 was 155.2 J/g, while that of PEG 8000 reached 179.4 J/g, indicating that there was not significant change in the heat of fusion in this molecular weight range.

The relational curves between the viscosities and the concentrations of the different PEGs are presented in Figure 2. The highest concentrations of PEG 600, PEG 1000, PEG 2000, PEG 4000, and PEG 8000, which were completely dissolved in the mixture of DMAc and acetone (60 : 40, w/w) at around 25°C, are 90, 60, 50, 40, and 30 wt %, respectively. It

can be seen from Figure 1 that the viscosity of the PEG solution increased with an increase in its molecular weight and concentration in the same solvent. One of the most important requirements for the electrospinning process to form fibers is that the solution must consist of a polymer of sufficient molecular weight and viscosity.²⁴ When the viscosity is too high, the solution may dry at the tip of the needle; hence the electrospinning process can be discontinued.²⁵ It can be seen that the higher the molecular weight of PEG, the lower is the PEG concentration required to obtain a polymer blend with sufficient viscosity for electrospinning process. Consequently, there is a decrease in the energy storage capacity of the fabricated nonwoven mats. PEG 600 had the lowest viscosity in solution; however, it also had the

TABLE I
Thermal Properties of PEG 1000 and E-Spun Fibers of PVDF/PEG 1000 Blends with Different Weight Percentages of PEG 1000

Type of sample	Percentage of PEG 1000 (wt %)	Melting point (°C)	Heat of fusion (J/g)	Crystallization temperature (°C)	Heat of crystallization (J/g)	Enthalpy ratio (%PEG 1000)
PEG		40.7	155.2	21.6	152.2	100
PVDF/PEG 1000 blend	33	38.1	37.0	24.0	35.9	24
	50	38.1	56.0	23.5	51.1	36
	56	38.5	72.2	24.8	68.8	47

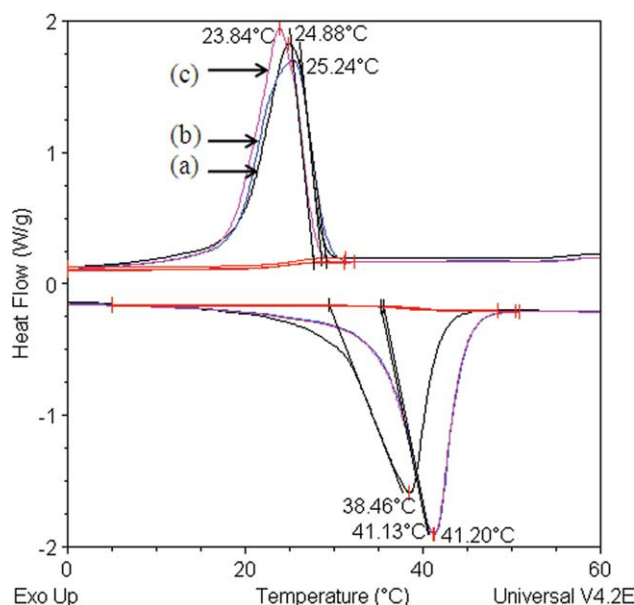


Figure 5 DSC thermogram of e-spun fibers of the PVDF/PEG 1000 blend after (a) the first cycle, (b) 50 cycles, and (c) 100 cycles. The nonwoven mats contained 56 wt % of PEG 1000. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

lowest heat of fusion. The heat of fusion was comparable within the range of PEG 1000 to PEG 8000. In this study, PEG 1000 was selected as a PCM on the basis of its thermal properties and viscosity results, which indicated low melting point, high heat of fusion, and low viscosity in the solution.

Characterization of PVDF/PEG 1000 blend e-spun fibers

Morphology

PVDF 1000 was used as the supporting material because of its great mechanical strength and ease of electrospinning. It provides a form-stable structure of PEG 1000 nonwoven mats. Figure 3 shows the relationship between the viscosity and the ionic conductivity of the blend solutions with different percentages of PEG 1000.

The viscosity of the blend solutions increased with an increase in the percentage of PEG 1000 in blend solution. On the contrary, the ionic conductivity of the blend solutions had an opposite trend. Figure 4

shows the morphology of the e-spun fibers of PVDF and PVDF/PEG blends. It was found that increasing the percentage of PEG 1000 in the blend solutions led to a significant increase in the average diameter of the e-spun fibers. This can be attributed to the changes in both the solution viscosity and the ionic conductivity of the polymer solutions, which were electrospun under a high electric field. The increase in the viscosity of the polymer solutions led to the greater resistance of the solution to be stretched. Meanwhile, a decrease in the ionic conductivity of the polymer solutions resulted in a decrease in the charge density of the ejected jets of the blend solutions; hence weaker elongation forces were exerted on the jets under a high electrical field. Both these factors cause the diameter of the e-spun fibers to be larger.²⁵

Thermal properties and thermal stability

The thermal properties of PEG 1000 and e-spun fibers of the PVDF/PEG 1000 blend with different percentage of PEG 1000 were measured by DSC. The data of melting point, heat of fusion, and enthalpy ratio data are summarized in Table I. As shown in table, the ratio of the PEG 1000 incorporated in the e-spun fibers of the PVDF/PEG 1000 blend was calculated with the ratio of the heat of fusion values of the PVDF/PEG 1000 blend nonwoven mats and that of pure PEG 1000 [eq. (1)].¹³ This ratio is a measure of the effective energy storage of the nonwoven mats.

$$\text{Enthalpy ratio} = \frac{\Delta H_{\text{PEG/PVDF}}}{\Delta H_{\text{PEG}}} \times 100 \quad (1)$$

where $\Delta H_{\text{PEG/PVDF}}$ and ΔH_{PEG} are the measured heat of fusion of the PVDF/PEG 1000 nonwoven mats and the pure PEG when they are melting, respectively.

Table I shows that pure PEG 1000 had a melting point of 42.6°C, which was higher than that of the e-spun PVDF/PEG 1000 blend (38.1°C). Meanwhile, the crystallization temperature of PEG 1000 was lower than that of the polymer blends. The difference in the melting and crystallization temperatures between pure PEG 1000 and PEG 1000 in the polymer blends was a result of the presence of PVDF,

TABLE II
Thermal Properties of E-Spun Fibers of PVDF/PEG 1000 Blend After the First Cycle, 50 Cycles, and 100 Cycles

Type of sample	Cycle No.	Melting point (°C)	Heat of fusion (J/g)	Crystallization point (°C)	Heat of crystallization (J/g)
PVDF/PEG 1000 blend	1 cycle	38.5	72.2	24.9	68.8
	50 cycles	41.2	76.1	25.2	71.3
	100 cycles	41.1	76.5	23.8	71.2

The nonwoven mats contained 56 wt % of PEG 1000.

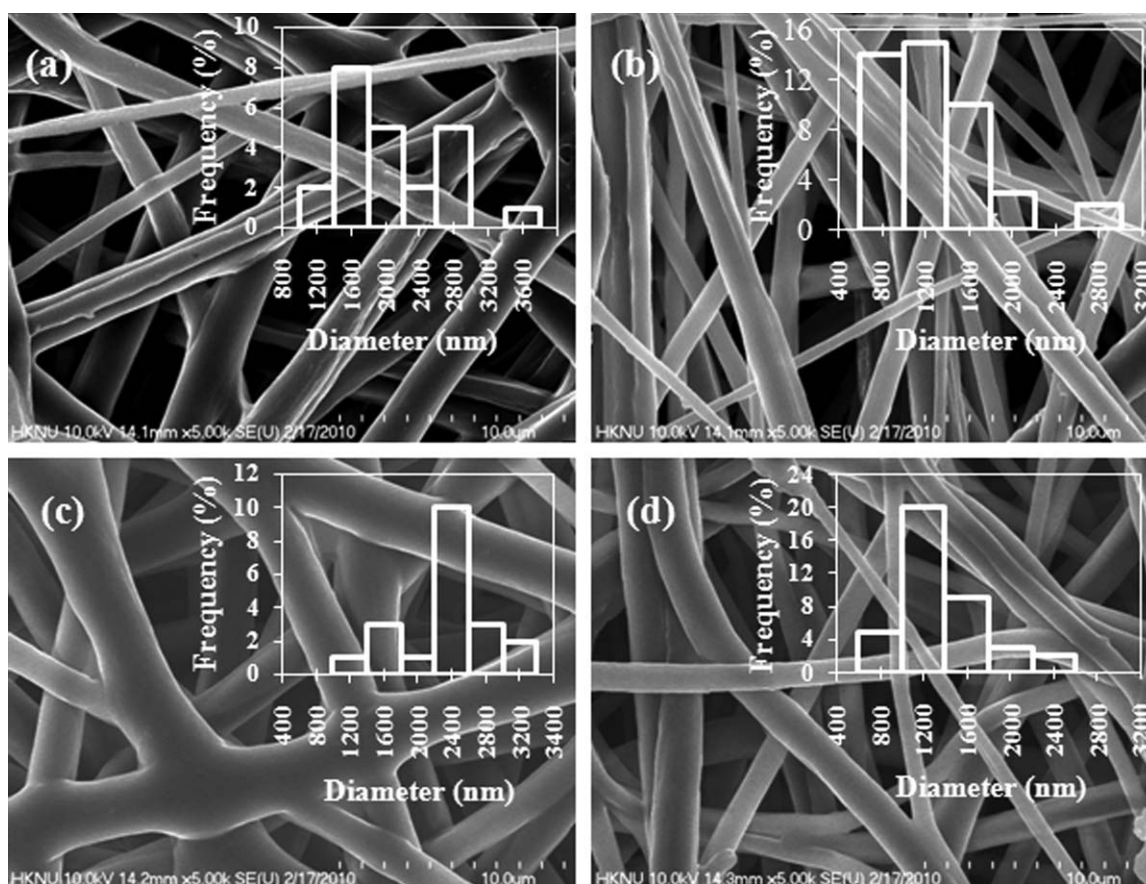


Figure 6 FE-SEM images of e-spun fibers of (a) PVDF/PEG 1000 blend, (b) SiO₂/PVDF/PEG 1000 composite, (c) PVDF/PEG 1000 blend after heat treatment, and (d) SiO₂/PVDF/PEG 1000 composite after heat treatment. All samples contained 56 wt % of PEG 1000. The heat treatment was conducted at 80°C for 24 h. The inserted graphs show the diameter distribution of nanofibers.

which led to an imperfect of crystal structure for PEG 1000. Moreover, PVDF is in a solid state in the phase change temperature range of PEG 1000, and it may cause an obstruction during the melting and solidification of PEG 1000. It can be seen that with an increase in the PEG 1000 content in the polymer blends, the heat of fusion of the polymer blends increases, and hence the ratio of the enthalpy value is increased. Polymer blends, which possess a higher enthalpy ratio, have a higher heat storage capacity. The highest enthalpy ratio of the polymer blends is 47% when the content of PEG 1000 is 56 wt %. This shows that PEG incorporated in e-spun fibers has a

TABLE III
Viscosity and Conductivity Data of the PVDF/PEG 1000 and the SiO₂/PVDF/PEG 1000 Blends, Respectively

Blend solutions	Viscosity (cP)	Conductivity (μS)
PVDF/PEG 1000 blend	462	9.3
SiO ₂ /PVDF/PEG 1000 composite	397	9.3

All blends contained 56 wt % of PEG 1000. The experiments were conducted at 25°C.

lower crystallinity than pure PEG. Many researchers have investigated the crystalline structure and crystallinity of the e-spun polymer fibers from the standpoints of size effects, processing parameters, and solution properties.²⁵ It has been reported that a higher applied voltage may lead to a greater electrical force, which strongly stretches the polymer jet during electrospinning and might result in a more ordered polymer chain structure. An increase in the tip-to-collector distance, rotation speed, and solution conductivity also results in more oriented molecular order in the nanofibers. However, the crystallinity of the e-spun fibers was relatively low when compared with the bulk polymers although they did have an ordered structure.²⁵ The enthalpy ratio value of the e-spun fibers of PVDF/PEG 1000 can be compared to that of the PCM-containing material, which was fabricated using the microencapsulation method. Alkan et al.¹⁴ reported the enthalpy ratio of PMMA/docosane microcapsules to be 28%. Meanwhile, PMMA/octacosane microcapsules fabricated by Sarl et al.¹³ had an enthalpy ratio of 43%.

The thermal reliability of e-spun fibers of the PVDF/PEG 1000 blend was investigated using a 100

TABLE IV
Thermal Properties of E-Spun Fibers of SiO₂/PVDF/PEG 1000 Composite After the First Cycle, 50 Cycles, and 100 Cycles

Type of sample	Cycle No.	Melting point (°C)	Latent heat of melting (J/g)	Crystallization point (°C)	Latent heat of crystallization (J/g)
SiO ₂ /PVDF/PEG 1000 composite	1 cycle	38.6	59.2	24.8	55.4
	50 cycles	41.9	63.7	23.6	57.0
	100 cycles	41.9	63.6	23.5	56.8

The nonwoven mats contained 56 wt % of PEG 1000.

thermal cycling test by DSC, as shown in Figure 5 and summarized in Table II. It can be seen that there were slight differences in temperature and latent heat of the phase change in first 50 melting/solidification cycles. This can be attributed to the impact of the thermal history on the orientation and relaxation of the polymer blend.²⁶ However, the thermal properties of the e-spun fibers were stable after 100 cycles, indicating that this material has good thermal reliability.

Characterization of fumed silica/PVDF/PEG 1000 composite e-spun fibers

Morphology

It was thought that the addition of fumed silica (SiO₂) in the e-spun fibers of the PVDF/PEG 1000 blend might prevent the leakage of melted PEG during the heating/cooling process. Figure 6 shows a comparison of morphology between PVDF/PEG 1000 blend fibers and SiO₂/PVDF/PEG 1000 composite fibers before and after heat treatment at 80°C for 24 h. Figure 6(a,b) shows that the addition of SiO₂ to PVDF/PEG 1000 nonwoven mats decreased its average diameter (from 1432 to 1079 nm). This is

probably due to the change in viscosity of the solution caused by the addition of SiO₂. As can be seen in Table III with the inclusion of fumed silica in the polymer solution, the viscosity decreased, while the ionic conductivity was not changed. It was observed that the SiO₂/PVDF/PEG 1000 composite fibers had a smooth surface, indicating that SiO₂ was well dispersed in the PVDF/PEG 1000 nanofibers.

Figure 6 also shows the behavior of the PVDF/PEG 1000 and SiO₂/PVDF/PEG 1000 nonwoven mats after heating. The morphology of the PVDF/PEG nonwoven mats changed significantly after heat treatment. Their average diameter increased from 1432 to 2221 nm. It can be seen from Figure 6(c) that PEG distributed in the fibers melted and led to the fibers combining with each other. However, Figure 6(d) shows that the SiO₂/PVDF/PEG 1000 composite nonwoven mat maintained its shape in the solid state without leakage of the melted PEG after heating. This may be because of the dispersion of PEG into the network of solid SiO₂. Hence, the transition of the SiO₂/PVDF/PEG 1000 composite nonwoven mat during the heating/cooling process could be a solid-solid phase change behavior.

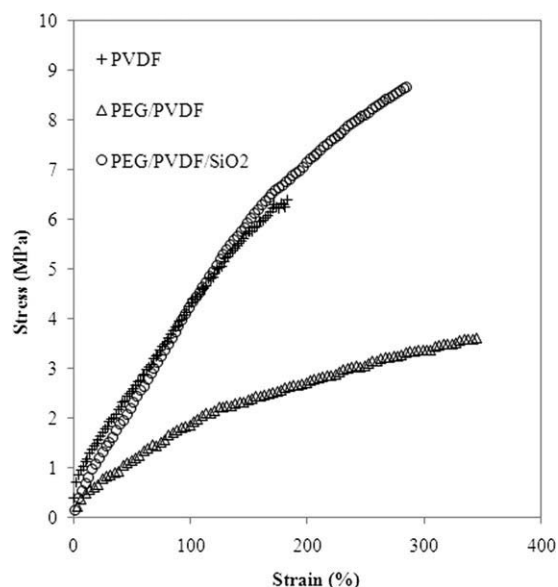


Figure 7 Mechanical properties of nonwoven mats of PVDF, PVDF/PEG 1000 blend and SiO₂/PVDF/PEG 1000 composite.

Thermal properties and thermal stability

The thermal properties of the e-spun fibers of the SiO₂/PVDF/PEG 1000 composite were investigated. The results are summarized in Table IV.

The addition of a small amount of SiO₂ in the PVDF/PEG 1000 blend decreased the heat of fusion of the e-spun fibers from 72 to 59.2 J/g. This can be attributed to the dilution and abstraction effects of fumed silica in the blend. However, it should be mentioned that the melting and crystallization points did not change with the addition of fumed silica. The thermal behaviors of the e-spun fibers in the SiO₂/PVDF/PEG 1000 composite after 100 heating/cooling processes showed the similar trend with those of e-spun fibers PVDF/PEG 1000 blend.

Mechanical properties

In this material, PVDF was a constituent that provided a form-stable structure and mechanical strength. The strain-stress curves of the e-spun

nonwoven mats of PVDF, the PVDF/PEG 1000 blend, and the SiO₂/PVDF/PEG 1000 composite are shown in Figure 7. The result indicates that the nonwoven mat of the PVDF/PEG 1000 blend had a lower tensile stress but higher elongation than that of PVDF. In other words, PEG acted as a plasticizer that reduced the tensile strength and increased the tensile strain of the PVDF nonwoven mat. The same result was reported by Lim et al.²⁷ They found that PEG with different molecular weights had a different effect on the plasticization of poly(vinyl butyral) binder in green BaTiO₃ sheets. The inclusion of fumed silica in the nonwoven mat of the PVDF/PEG 1000 blend increased the tensile strength significantly (from 3.7 to 8.8 MPa); however, it decreased slightly the strain of the PVDF/PEG 1000 composite nonwoven mat (from 350 to 290%). This may be due to the fact that the inclusion of silica, especially fumed silica, was known as reinforcement.²⁸

CONCLUSIONS

In this study, PEG 1000 was chosen as PCM because of its low melting point, high heat of fusion, and low viscosity that facilitated the electrospinning of PVDF/PEG 1000 blends. Nonwoven mats were prepared from the e-spun fibers of PVDF/PEG 1000 blends with different PEG 1000 contents (wt %). The higher the PEG 1000 content of the blend, the larger was the diameter of the PVDF/PEG 1000 e-spun fibers. The shape of these e-spun fibers changed significantly after heat treatment, as observed in the FE-SEM images. Addition of fumed silica to the PVDF/PEG 1000 blend helped retain the shape of the fiber and prevent the leakage of the molten PEG 1000 after heat treatment; hence, a form-stable PCM was obtained. This composite fiber had high latent heats of melting and crystallization (59.2 and 55.4 J/g, respectively). The PVDF/PEG 1000 nonwoven mat containing SiO₂ shows remarkable mechanical performance, indicating that it has promising applications in the manufacture of smart textiles and solar energy storage materials.

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