



# Polyacrylonitrile Nanofibers Containing Microencapsulated Phase Change Materials Produced for Thermal Energy Storage by Electrospinning Fabric

Ruhan Altun Anayurt<sup>1</sup> 0000-0002-7327-6871

Cemil Alkan<sup>2</sup> 0000-0002-1509-4789

<sup>1</sup> Çankırı Karatekin University / Department of Occupational Health and Safety / Çankırı, Türkiye

<sup>2</sup> Tokat Gaziosmanpaşa University / Department of Chemistry / Tokat, Türkiye

**Corresponding Author:** Ruhan Altun Anayurt, ruhanaltun@karatekin.edu.tr

## ABSTRACT

In this study, microencapsulated phase change materials (MEPCMs) were imparted into nanofibers using electrospinning and an original material composite systems, microparticles interconnected through nanofibers were produced as physically applicable fabric material. Poly(methyl methacrylate-co-ethylene glycol dimethacrylate-co-2-hydroxyethyl acrylate) and poly(methyl methacrylate-co-ethylene glycol dimethacrylate-co-2-hydroxyethyl methacrylate) shells, and n-hexadecane, and Rubitherm RT-21 core were formed for this study. Composites with low diameter nanofibers as compared to microparticles consisted of polyacrylonitrile (PAN) nanofibers and dispersed MEPCMs together. MEPCMs were produced through commonly used emulsion polymerization technique. They were dispersed into PAN solution as solid particles in dimethylformamide (DMF) for the first time and electrospun together. Thermophysical properties of MEPCMs dispersed nanofibers were determined using differential scanning calorimetry (DSC) as Fourier Transform Infrared (FTIR) Spectroscopy instrument was used to prove copresence of the cores, shells, and nanofiber materials. The energy storage capacity of the fabrics were found tunable with the used amount of the MEPCMs. The enthalpy value of microparticles changed from 70.8 to 117.3 J/g as the nanofibers have enthalpy of 12.6 and 20.8 J/g. The thermal transitions are reversible and without overcooling. Besides, Scanning Electron Microscopy (SEM) device was used to examine the unique morphology the MEPCMs in the nanofiber cross-section. The produced systems are unique fabric forms of microparticles for easy exploitation in the TES applications.

## ARTICLE HISTORY

Received: 14.09.2023

Accepted: 27.02.2025

## KEYWORDS

Nanofiber, Energy Storage, Phase change material, Electrospinning.

## 1. INTRODUCTION

Materials that can store large amounts of latent heat energy during the phase change period and release this stored energy are called phase change materials (PCMs) [1]. Among organic PCMs, n-alkanes are commonly used. n-alkanes, have several advantages such as high latent heat capacity, suitable phase change temperature, little or no super cooling, volumetric stability, good chemical and thermal stability, and nontoxicity [2-5]. PCMs have latent heat absorbing/releasing ability of a considerable high amount at nearly unvaried temperature [6,7]. With respect to the phase change type, they are generally separated to four subgroups as solid-gas, gas-liquid, solid-liquid, and solid-solid PCMs [8,9]. The great volume changes during solid-gas and gas-liquid phase changes restrict the application areas of PCMs. Solid-liquid PCMs have been widely investigated as promising latent heat thermal energy storage (LHTES) materials [10,11]. Microencapsulated phase change materials (MEPCMs) have

several advantages over PCM among existing methodologies, such as providing a large heat transfer area, reducing the reactivity of a PCM [12,13] towards the external environment, and being able to control changes in the volume of storage materials as phase change occurs [14]. Transforming PCM-containing microcapsules into nanofibers by electrospinning will allow them to be used in other application areas. In recent years, scientific and commercial interest in nanotechnology and nano-sized materials has increased, and parallel to this, studies on nanoscale fiber production are gaining momentum [15, 16]. Nanotechnology has enabled materials to acquire many different properties by processing them in nanometer ( $10^{-9}$  meters) dimensions [17]. Polymeric nanofibers; can be produced by different techniques such as drawing, mold synthesis, phase separation, self-assembly and electrospinning [18]. The most common method developed for nanofiber production is electrospinning [19, 20]. Electrospinning is based on providing nano-sized fiber formation from the polymer in

**To cite this article:** Anayurt R A, Alkan C. 2025. Polyacrylonitrile nanofibers containing microencapsulated phase change materials produced for thermal energy storage by electrospinning fabric. *Tekstil ve Konfeksiyon*, 35(2), 114-123.



liquid state with the help of electric field forces. With the electrospinning, the polymer liquid is charged electrically by using a high potential voltage in the fiber drawing from the polymer solution or melt, and the polymer jet coming out of the fine jet nozzle flows towards the grounded surface located opposite the nozzle. During this current, the polymer jet is scattered as very fine filaments and thus fibers with nano-sized diameter can be obtained [21].

Electrospinning leads to various advantages. It bears high encapsulation efficiency, ease of application, high loading capacity, the ability to work with synthetic and natural polymers, obtaining fibers in sizes ranging from a few nanometers to micrometers, obtaining mechanically durable and flexible fibers, no need for purification. In addition to these advantages, unstable jet formation, significant effect of many parameters on fiber formation and structure can be considered as the disadvantage of the method [22]. Nanofibers have a high molecular orientation. Due to their small size, they have fewer structural defects and thus have very good mechanical properties. Because of their small diameter, they have high surface / volume ratios, so they have high surface areas. The formation of structures with large surface area of nanofibers increases their capacity to hold or emit functional groups, ions and a wide variety of nano-level particles [23].

Nanofibers produced by electrospinning, due to their superior functional properties such as high coverage, porosity, etc., have usage areas such as filtration, composite, protective clothing and medical fields, and contribute to the development of new and high performance products [15].

Polyacrylonitrile (PAN) polymer, which is one of the polymers used in electrospinning, is one of the polymers that are frequently researched and studied because of its versatile applications and its superior properties [24, 25]. Electrospinning of PCMs is a topic attracting increasing interest from the scientists [26, 27, 28]. Baştürk et al. produced A series of Poly vinyl butyral–Poly (acrylic acid) (PVB-PAA) based form-stable phase change materials (PCMs) have been prepared for the use of thermal energy storage applications. Six types of formulations containing five different fatty alcohols were prepared by adding PVB to PAA [29]. Oktay et al. produced nanosilica reinforced nanofiber composite as PCMs. First, polyvinyl alcohol (PVA) and nanosilica was modified with mercaptopropionic acid and 3-(mercaptopropyl) trimethoxysilane there respectively and thiolated-PVA-nanosilica nanofibers were prepared by using electrospinning successfully [30]. Stearic acid was used for long chain fatty acid. Alay Aksoy et al. produced nanoencapsulated PCM-doped cotton/PAN nanofiber based composite yarns for thermoregulation and proved the performance as a textile material [31]. Akduman et al. produced knitted fabrics using phase change material incorporated thermoplastic polyurethane and cellulose acetate nanofibers by means of electrospinning [32].

Nanofibers produced by electrospinning have many usage areas such as cosmetic applications, tissue engineering,

filtration applications, agricultural applications, nanosensors, biomedical tools, protective clothing, reinforced composite making, and controlled active substance release [33, 34, 35].

Researchers have proposed various thermal response improvement techniques to improve the thermal conductivity of PCMs, which are mostly concentrated on incorporating metal and nonmetal fillers or their mixtures into PCMs [36-40], insertion of PCMs into finned tubes or plates of different configurations [19], dispersion of high thermal conductivity carbon nanotubes (CNTs) and carbon nanofibers (CNFs) [41-44], graphite [45], exfoliated graphite [46, 47], graphene [48], or boron nitride [49] into PCMs, or doping of PCM by in-situ reduction of metal salt solutions [50]. Extending the surface area of PCMs is another technique to improve their effectiveness because it improves the interaction area with the environment while decreasing the response time to heat stimuli. MEPCMs are little particles with large areas; nonetheless, they are particles, making application extremely challenging. Incorporating them into nanofiber architecture is promising because it does not reduce surface area but rather stabilizes the matrix as a whole. However, creating nano textiles combined with MEPCMs is problematic since the microparticles are not soluble in the probable solvents and their homogeneous distribution is difficult. Electrospun fibers are expected to respond rapidly to stimuli.

Microencapsulation is a well-known method to eliminate the handicaps in the applicability of solid-liquid PCMs [51-53]. However, since MEPCMs still do not have a body, they still have problems not fully solved at the point of application. Electrospinning has been used very little to evaluate PCM structures. MEPCMs have not been tried to distribute electrospun nanofiber matrices among the various types. If the electrospinning is evaluated, the fabric form of the structure can be obtained by creating connections between MEPCMs. In this study, microcapsules with latent heat storage properties have been turned into nanofibers using electrospinning to be used in a better suitable form. With the formed morphology, the study is unique in material science since previously electrospun PCMs were produced by coelectrospinning of PCM with the polymer. The achievements of extended surface area electrospun nanofiber with incorporated MEPCMs may be used in sophisticated applications like cooling electronics or cooling lithium batteries.

## 2. MATERIAL AND METHOD

### 2.1 Material

n-hexadecane (Fluka) and RT21 (Rubitherm) were used as received. Methyl methacrylate (MMA), ethylene glycol dimethacrylate (EGDM), 2-hydroxyethyl acrylate (HEA) and 2-hydroxyethyl methacrylate (HEMA) were obtained from Sigma Aldrich company. Triton X100 (Merck) were used as surfactant. Ferrous sulphate heptahydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ), ammonium persulphate ( $(\text{NH}_4)_2\text{S}_2\text{O}_8$ ), were all obtained from

Sigma Aldrich and used without further purification. Poly(acrylonitrile-co-vinylacetate) (PAN polymer with 92.8%AN and 7.2% vinylacetate w/w in composition) taken from Aksa Acrylic Chemistry Company. Dimethylformamide (DMF) was obtained from Carlo Erba.

## 2.2 Method

### 2.2.1. Preparation of PAN Nanofiber Composites of n-hexadecane@Poly(methyl methacrylate-co-hydroxyethyl acrylate) and RT21@Poly(methyl methacrylate-co-hydroxyethyl acrylate) Microcapsules

In this study, n-hexadecane (C16) and RT21 were used as PCMs. MMA, EGDM and HEA or HEMA in different concentrations formed the shell part of the microcapsule, as C16 and RT21 were used as core. PCM and a surfactant Triton X100 were added to deionized water and mixed by using a homogenizer for 30 minutes at a mixing speed of 10000 rpm at 50 °C. HEA and HEMA were used at concentrations of 1%, 20% and 30% and 40% in MMA-EGDM mixture. It is to show if the thermal energy storage property can be tuned or not. The monomers and crosslinker were purified with 1 M 50 ml NaOH solution prior to microencapsulation. The resulting homogeneous mixture was taken into the reaction vessel and its temperature was brought to 70 °C and initiators (ammonium persulfate, iron sulphate) were added. The system is maintained at the temperature for 3 more hours for completion. It is a semiemulsion polymerization generally called as miniemulsion process. Fig 1 depicted the footsteps of the synthesis of MEPCMs, while Fig 2 showed particle growth of the MEPCMs.

The procedure is just for the preparation of acrylic based MEPCMs that have been used by many different groups since its discovery [56-58]. Microencapsulation of phase change materials is still improving through novel shell materials [59,60]. Acrylic materials are assumed to be

compatible with the PAN in solution. In reality, polymers in solution are very much keen on adhering the surfaces and when MEPCMs are incorporated to the solutions, the potential surface for the PAN polymers dissolved is the surface of the microparticles. The compatibility of the counterparts is synergy for the solution to be electrospun [61]. PAN is very electroactive in the medium and driving force to take the MEPCMs to the other side of the needle during electrospinning. In other words, PAN in DMF was the driving force importing MEPCMs into nanofiber geometry.

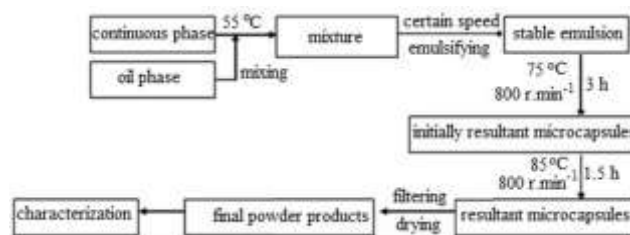


Figure 1. MEPCM production scheme block diagram [54]

Produced MEPCMs were put into a solution of 10% PAN in DMF by mass for the electrospun composite systems including randomly dispersed microparticles. The solution was mixed with a magnetic stirrer for one day. Electrospinning was applied and microparticles dispersed nanofibers were obtained. Electrospinning operation has been optimized in 18kV magnetic field conditions where the distance between syringe tip and collector panel is 20 cm. An Aluminium plate was used to collect the nanofiber composite incorporated with the microparticles. As a result of the synthesis studies, 1-1.5 mm thick membranes were obtained, and the thickness of the membranes was observed to increase with the solution concentrations. The time period is fixed as 2 hours for collecting the fabrics. The experiments were repeated at least three times to be sure from the homogeneity that is reproducibility. The experimental setup required for the electrospinning is shown schematically in Figure 3.

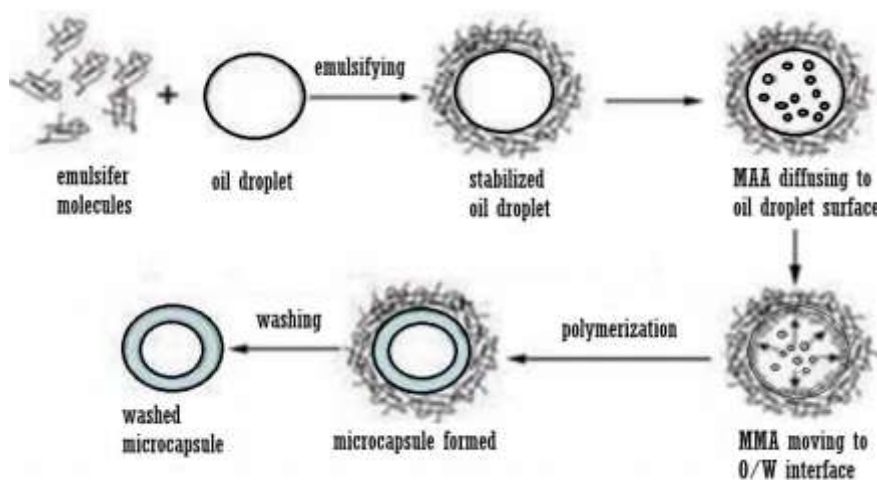


Figure 2. Scheme of formation process of miniemulsion polymerization [55]

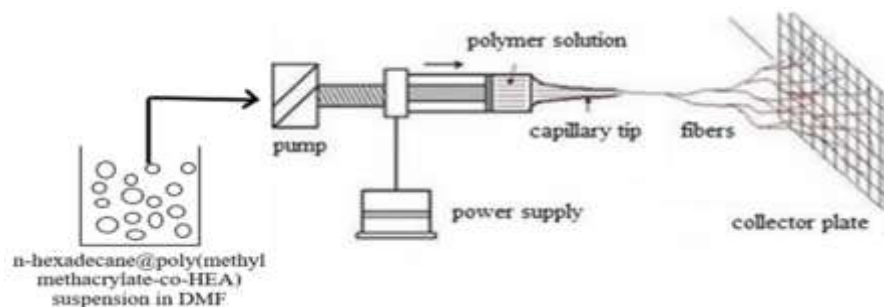


**Figure 3.** General view of an electrospinning apparatus with a difference of materials used in this work [62]

## 2.2.2 Chemical Characterizations

The MEPCMs are intentionally prepared as crosslinked to prevent dissolution or physical viscoelastic and thermal deformations [63-65]. Therefore, chemical characterization of electrospun nanofibers was generally performed using the Fourier Transform Infrared (FT-IR) spectroscopy technique. The content of the MEPCMs include different materials together and characteristic response peaks of those materials prove their existence in the medium. The technique is much dependable than NMR techniques due to the evaluation of NMR peaks for polymeric materials in complex systems (crosslinked matrices with PCMs). Furthermore, solid state NMR is not possible to be found when needed. Jasco FT-IR-4700 instrument with ATR accessory were exploited between 4000 and 400  $\text{cm}^{-1}$ . The resolution of the spectra was 4  $\text{cm}^{-1}$ , and the number of scans was 16 for the samples and background commonly in the FT-IR spectra.

## 2.2.3 Physicochemical Properties

Physicochemical properties of thermal energy storage materials are generally phase transition and application properties. For comparison MEPCMs and MEPCMs incorporated nanofibers are subjected to a differential scanning calorimetry (DSC, Netzsch DSC214 Polyma) instrument investigation. The data from the curves were averages of three measurements. The instrument was calibrated with bismuth, indium, zinc, tin, and cesium chloride before DSC measurements. The DSC measurements were performed at a heating-cooling rate of 5  $^{\circ}\text{C min}^{-1}$  in a temperature range of 0-80  $^{\circ}\text{C}$  under an inert  $\text{N}_2$  atmosphere at a 60 mL/min flow rate. Representations of nanofibers and abbreviations are given in Table 1.

## 2.2.4 Morphology Analyses

Electrospun nanofibers with MEPCMs were investigated for surface morphology using an electron microscopy

instrument (Zeiss Sigma 300). Particle size investigations for the produced MEPCMs were performed in our previous studies [64]. The microcapsules' mean particle size and particle size distributions were analyzed using a particle sizer analyzer (PSD) instrument (Malvern MS2000E). They were found between 0.3 and 120  $\mu\text{m}$ .

## 3. RESULTS AND DISCUSSION

### 3.1 Determination of Thermal Properties of Nanofibers with MEPCMs

When it comes to the physical chemistry of MEPCMs, the first method that comes to mind is the DSC method. With this method, it is possible to determine the storage starting temperatures and the storage energies per gram of materials that store heat energy as latent heat [65]. DSC is also used to calculate many different parameters. The most important of these is the supercooling behavior. Supercooling is the difference between the melting and solidification temperatures. It is undesirable for phase change materials that are desired to work isothermally.

Melting and solidification temperatures and enthalpy values of PCM@P(MMA-co-EGDM-co-HEA) and PCM@P(MMA-co-EGDM-co-HEMA) microcapsules obtained from DSC curves were tabulated in Table 2. Besides, Table 3 shows the phase change temperatures and enthalpy values of nanofibers.

As seen in Table 1, no overcooling behavior was observed in the PCMs and their capsules used in this study. This is evidence that PCMs and capsules produced from them operate isothermally. At the same time, as a result of repeated DSC analyses, it was seen that the phase transitions were reversible, and the produced structures maintained their stability as a result of thermal cycles.

**Table 1.** Nanofibers with MPCMs and abbreviated names

| Representations of Nanofibers with MEPCM | Abbreviated name    |
|------------------------------------------|---------------------|
| C16@P(MMA-co-1%HEA)@PAN                  | 1% HEA Nanofiber    |
| RT21@P(MMA-co-20%HEA)@PAN                | 20% HEA Nanofiber   |
| RT21@P(MMA-co-30%HEA)@PAN                | 30% HEA Nanofiber   |
| RT21@P(MMA-co-20%HEMA)@PAN               | 20 % HEMA Nanofiber |
| RT21@P(MMA-co-30%HEMA)@PAN               | 30% HEMA Nanofiber  |
| RT21@P(MMA-co-40%HEMA)@PAN               | 40% HEMA Nanofiber  |

1% HEA Nanofiber is another PCM used in similar applications at slightly different conditions. Besides, RT21@P(MMA-co-40%HEMA)@PAN is to show that the tunability allows optimizing thermal energy storage property.



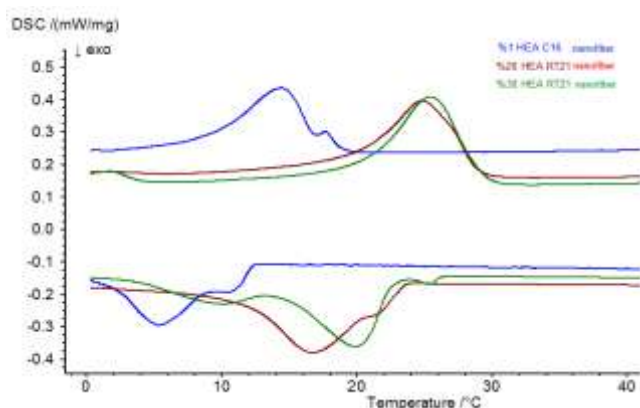
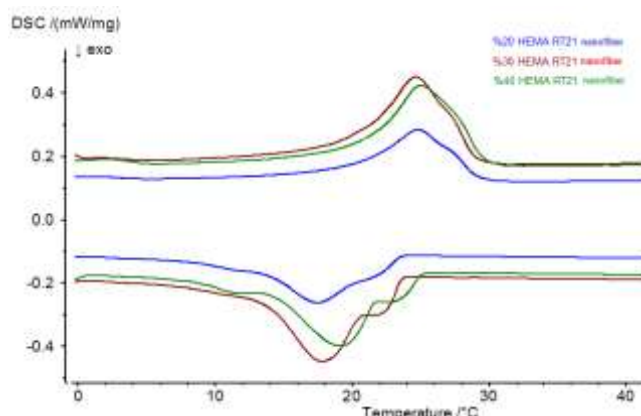
**Table 2.** DSC measurement results of PCM@P(MMA-co-EGDM-co-HEA) and PCM@P(MMA-co-EGDM-co-HEMA) MEPCMs

| Microcapsule Name      | Melting Enthalpy (J/g) | Melting temperature (°C) | Solidification Enthalpy (J/g) | Solidification temperature (°C) |
|------------------------|------------------------|--------------------------|-------------------------------|---------------------------------|
| C16                    | 202.5                  | 16.6                     | -201.7                        | 17.9                            |
| RT21                   | 120.0                  | 18.2                     | -115.8                        | 21.3                            |
| C16@P(MMA-co-1%HEA)    | 117.3                  | 15.8                     | -116.0                        | 14.2                            |
| RT21@P(MMA-co-20%HEA)  | 96.1                   | 17.9                     | -96.4                         | 21.6                            |
| RT21@P(MMA-co-30%HEA)  | 70.8                   | 18.5                     | -70.2                         | 21.7                            |
| RT21@P(MMA-co-20%HEMA) | 94.8                   | 19.8                     | -97.5                         | 22.8                            |
| RT21@P(MMA-co-30%HEMA) | 108.2                  | 18.8                     | -109.3                        | 22.3                            |
| RT21@P(MMA-co-40%HEMA) | 81.1                   | 18.3                     | -79.8                         | 21.9                            |

**Table 3.** DSC data of MEPCMs dispersed nanofiber composites

| Nanofiber name    | Melting Enthalpy (J/g) | Melting temperature (°C) | Solidification Enthalpy (J/g) | Solidification temperature (°C) |
|-------------------|------------------------|--------------------------|-------------------------------|---------------------------------|
| 1% HEA Nanofiber  | 14.8                   | 9.5                      | -11.3                         | 12.5                            |
| 20% HEA Nanofiber | 19.3                   | 20.3                     | -19.9                         | 23.8                            |
| 30%HEA Nanofiber  | 20.1                   | 20.2                     | -21.2                         | 22.1                            |
| 20%HEMA Nanofiber | 12.6                   | 20.5                     | -13.4                         | 23.7                            |
| 30%HEMA Nanofiber | 19.1                   | 20.8                     | -20.9                         | 23.7                            |
| 40%HEMA Nanofiber | 20.8                   | 21.3                     | -20.2                         | 20.3                            |

The melting enthalpy values of (1%, 20%, 30%) HEA nanofibers were found to be 14.8, 19.3 and 20.1 J/gr, respectively. The melting enthalpy values of (20%, 30%, 40%) HEMA nanofibers were found to be 12.6, 19.1 and 20.8 J/gr, respectively. DSC graphics of nanofibers are given in Figures 4 and 5, respectively. When DSC results were considered, the enthalpy values of microcapsules were found quite high as they were lower than the ones theoretically expected in the nanofibers. It was assigned that the microcapsules were scattered in the environment outside the collection panel (collector plate Fig. 1) due to the magnetic field during electrospinning. That is, fewer microcapsules were confined within the fiber. In addition, it was also assigned that since the fiber drawing process took place for a long time, the microcapsules in the syringe had collapsed. Electrospinning recipes should mix well with the polymer solution in DMF for a homogeneous fiber. The recipe of nanofiber in the syringe was mixed from time to time to prevent the microparticles from collapsing and maintain homogeneous spraying.

**Figure 4.** DSC thermograms of PAN nanofiber composites with MEPCMs with segments from HEA comonomer**Figure 5.** DSC thermograms of PAN nanofiber composites with MEPCMs with segments from HEMA comonomer

### 3.2. Chemical characterization of nanofibers with MEPCMs

FT-IR spectroscopy method is not to reveal the chemical structures completely but it is sufficient to prove potential presence in a known media. Using all of the data from the FTIR spectra, the synthesis of the MEPCMs is proven. Methylmethacrylate monomer has carbonyl peaks between 1735  $\text{cm}^{-1}$  and 1750  $\text{cm}^{-1}$ . C-O stretching peaks of ester group between 1100-1300  $\text{cm}^{-1}$ . It has also C = C peak at 1639  $\text{cm}^{-1}$ . The monomers HEA and HEMA have -OH stretching peaks very close to each other at 3430 and 3426  $\text{cm}^{-1}$  respectively. Similarly they have carbonyl stretching peaks at 1720 and 1718  $\text{cm}^{-1}$  respectively. Vinyl peaks of them are seen at 1627 and 1637  $\text{cm}^{-1}$ . Peaks between 1300-1000  $\text{cm}^{-1}$  are ester-structured -C-O- stretching peaks in both spectra of HEA and HEMA.

The PCMs n-hexadecane and RT21 have similar structures. They are both aliphatic chains; RT21 is expected to be

mostly n-heptadecane because the physicochemical characteristics resembles it. FTIR spectra supports this fact since all the peaks are in the same places and characters. The peaks at wavelengths of  $2923\text{ cm}^{-1}$  and  $2859\text{ cm}^{-1}$  are the peaks of the C-H stretching peaks. Besides, the peaks at wavelengths of  $1457\text{ cm}^{-1}$  and  $1369\text{ cm}^{-1}$  are C-H bending peaks.

Determining the structure of the MEPCMs is somehow difficult. The  $3432\text{ cm}^{-1}$  band is the -OH stretch band from the HEA monomer. The  $3444\text{ cm}^{-1}$  band is the -OH stretch band from the HEMA monomer. The peak at  $2956\text{ cm}^{-1}$  is the C-H stress peak coming from the HEA monomer. The peak at  $2956\text{ cm}^{-1}$  is the C-H stretch peak from the HEMA monomer. The carbonyl peak at  $1741\text{ cm}^{-1}$  in the MMA monomer and  $1718\text{ cm}^{-1}$  in the HEA monomer appeared at  $1727\text{ cm}^{-1}$  in the microcapsule. This peak is the peak of the non-H-bonded carbonyl group in the MMA-co-HEA copolymer [66]. The carbonyl peak at  $1741\text{ cm}^{-1}$  in the MMA monomer and  $1720\text{ cm}^{-1}$  in the HEMA monomer appeared at  $1729\text{ cm}^{-1}$  in the microcapsule. This peak is the peak of the non-H-bonded carbonyl group in the MMA-co-HEMA copolymer. The peaks at  $2923\text{ cm}^{-1}$  and  $2852\text{ cm}^{-1}$  and the peaks at  $1457\text{ cm}^{-1}$  and  $1384\text{ cm}^{-1}$  wavelengths observed in the microcapsule structure are characteristic peaks of n-alkane. These peaks prove the presence of core in the capsule HEA structure. The peaks at  $2923\text{ cm}^{-1}$  and  $2854\text{ cm}^{-1}$  and the peaks at  $1455\text{ cm}^{-1}$  and  $1388\text{ cm}^{-1}$  wavelengths observed in the microcapsule structure are characteristic peaks of n-alkane [67]. These peaks prove the presence of paraffin in the HEMA capsule structure. The peaks at  $1627\text{ cm}^{-1}$  in the spectrum of the HEA monomer and  $1639\text{ cm}^{-1}$  in the MMA monomer are the C = C stretch peak in the structure of the monomers and are not seen in the spectra of the microcapsule. This explains the formation of the co-polymer wall structure of microcapsules. The peaks at a wavelength of  $1637\text{ cm}^{-1}$  in the spectrum of the HEMA monomer and  $1639\text{ cm}^{-1}$  in the MMA monomer are the C = C stretch peak in the structure of the monomers and are not seen in the spectra of the microcapsule [68]. This explains the formation of the co-polymer wall structure of microcapsules. The peaks at a wavelength of  $1243\text{ cm}^{-1}$  and  $1147\text{ cm}^{-1}$  are the peaks of the -C-O- stretch vibration of ester groups in HEA microparticles while the peaks at a wavelength of  $1243\text{ cm}^{-1}$  and  $1149\text{ cm}^{-1}$  are the peaks of the -C-O- stretch vibration of HEMA microparticles [69].

Polyacrylonitrile has characteristic peaks of cyanogroup around  $2260\text{--}2150\text{ cm}^{-1}$  and C-C at around  $1300\text{--}800\text{ cm}^{-1}$ . Dimethylformamide (DMF) was used as the solvent. Its characteristic peaks are carbonyl stretching peak at  $1673\text{ cm}^{-1}$  wavelength. C-N stretching peak at around  $1389\text{ cm}^{-1}$  and C-H stretching peak at around  $3280\text{--}3240\text{ cm}^{-1}$ . PAN is the matrice of the nanofabrics and the characteristic peaks of this polymer is persistent in all the nanofiber spectra. The solvent spectrum is used to show solvent purity of the MEPCMs.

FT-IR spectra of HEA and HEMA nanofibers are given in Figures 6 and 7. When FT-IR spectra were examined, it was seen that the peaks of C-H stretching at wavelengths of PCMs were observed at  $2923\text{ cm}^{-1}$  and  $2859\text{ cm}^{-1}$  as C-H bending peaks appeared at wavelengths of  $1457\text{ cm}^{-1}$ ,  $1369\text{ cm}^{-1}$ . They were at  $2854\text{--}2953\text{ cm}^{-1}$  in nanofibers. The peaks of the bond C=N stretching were observed between the wavelengths of  $2260$  and  $2150\text{ cm}^{-1}$  due to PAN matrix in the nanofiber structures. The stretching peak of DMF for carbonyl group was seen at a wavelength of  $1673\text{ cm}^{-1}$  as it appeared at  $1724\text{ cm}^{-1}$  in the polymers and nanofibers.

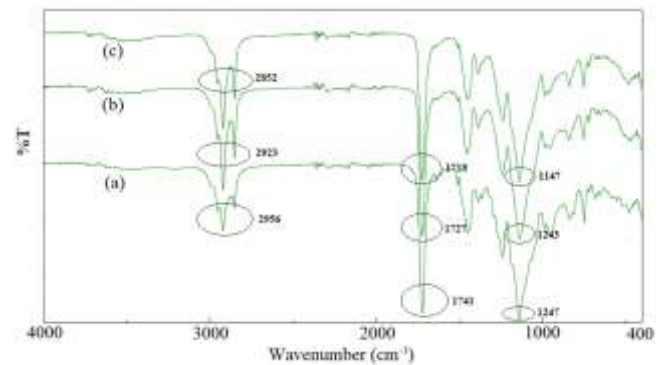


**Figure 6.** FT-IR spectra of nanofibers with PMMA based microparticles (a) with segments from 1%HEA comonomer, (b) with segments from 20%HEA comonomer, and (c) with segments from 30% HEA comonomer

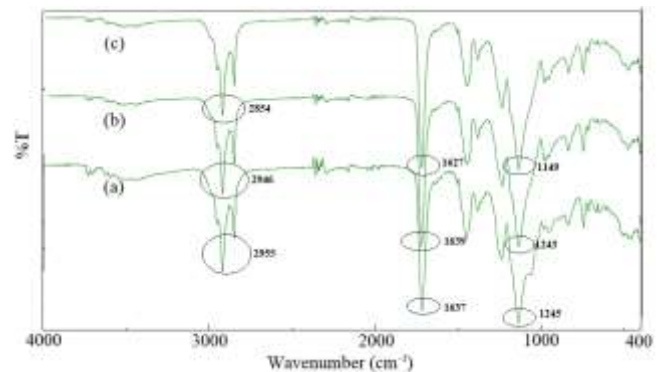


**Figure 7.** FT-IR spectra of nanofibers with MEPCMs with segments from 40%HEMA comonomer (a), 30%HEMA comonomer (b), 20%HEMA comonomer (c)

### 3.3. Morphology Investigation by SEM

MEPCMs particle morphology have been studied many times in our group previously using polarised optical microscopy, particle size distribution analysis and SEM investigations [64, 65]. Thus they are not given here. SEM images of the produced nanofibers are given in the Figures of 9, 10, 11, 12, 13, and 14. When looking at the SEM images, the presence of both capsules and fibers is clearly visible. Intermittently microcapsules clogged the syringe tip and the fibers were ruptured. Pilling occurred in some places due to this problem. In spite of that, nanofibers have been successfully produced and the average diameter of the fibrillar geometry was found as 200 nm. Fig. 8 shows that microparticles of C16@P(MMA-

co-1%HEA) were distributed in a nanofiber structure with PAN interconnections. PAN is present in the wires and onto the particles.

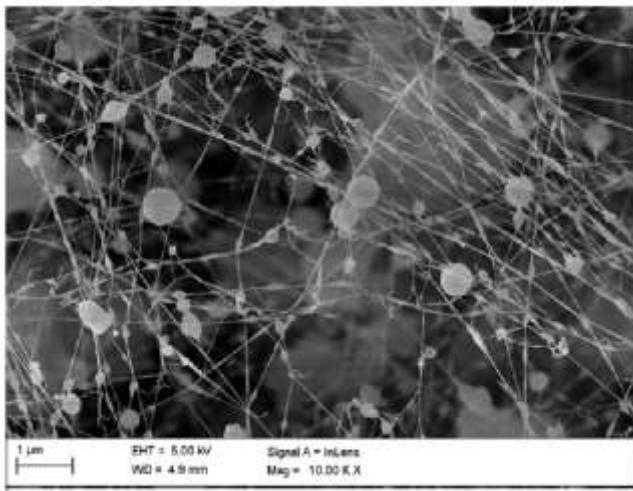


**Figure 8.** SEM images of PAN nanofiber composite system with C16@P(MMA-co-1%HEA) particles

Fig. 9 shows that microparticles of RT21@P(MMA-co-20%HEA) were distributed in a nanofiber structure with PAN interconnections. PAN is present in the wires and onto the RT21@P(MMA-co-20%HEA) particles.

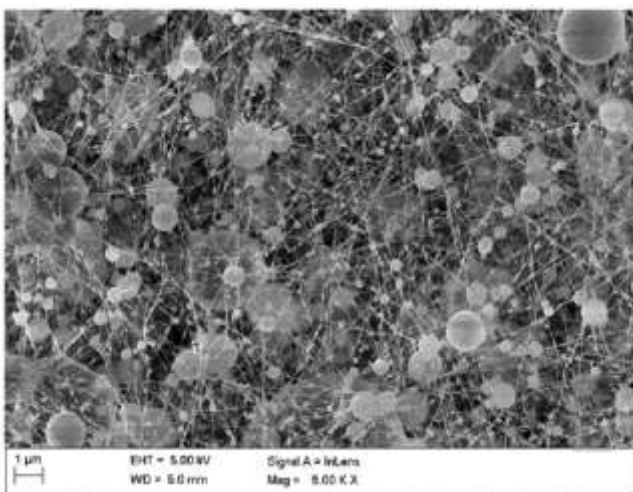


**Figure 9.** SEM images of PAN nanofiber composite system with RT21@P(MMA-co-20%HEA) particles

Fig. 10 shows that microparticles of RT21@P(MMA-co-30%HEA) were distributed in a nanofiber structure with PAN interconnections. PAN is present in the wires and onto the RT21@P(MMA-co-30%HEA) particles.

Fig. 11 shows that microparticles of RT21@P(MMA-co-20%HEMA) were distributed in a nanofiber structure with PAN interconnections. PAN is present in the wires and onto the RT21@P(MMA-co-20%HEMA) particles.

Fig. 12 shows that microparticles of RT21@P(MMA-co-30%HEMA) were distributed in a nanofiber structure with

PAN interconnections. PAN is present in the wires and onto the RT21@P(MMA-co-30%HEMA) particles.

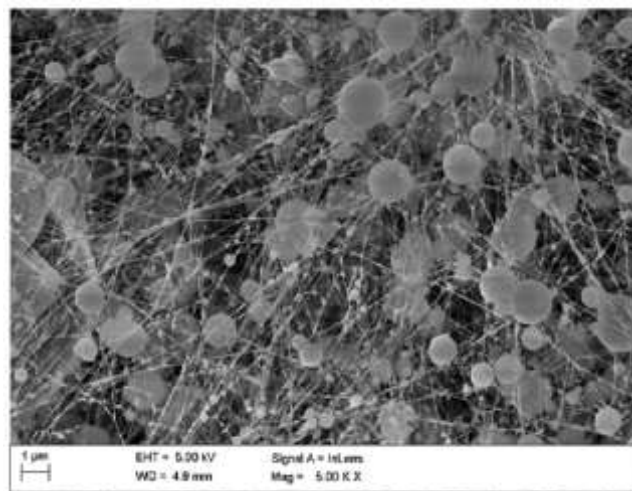


**Figure 10.** SEM images of PAN nanofiber composite system with RT21@P(MMA-co-30%HEA) particles

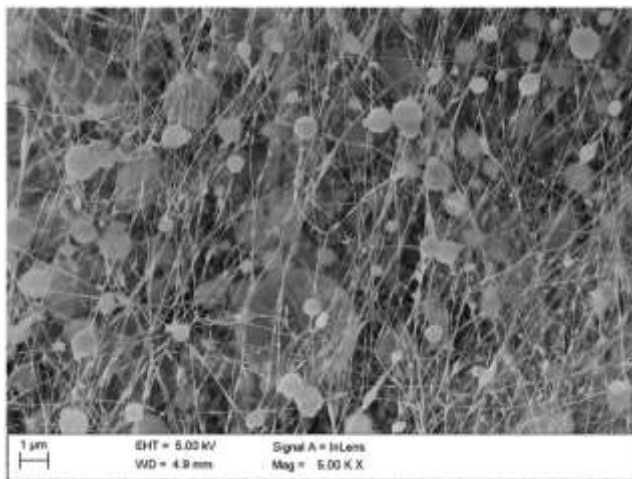


**Figure 11.** SEM images of PAN nanofiber composite system with RT21@P(MMA-co-20%HEMA) particles

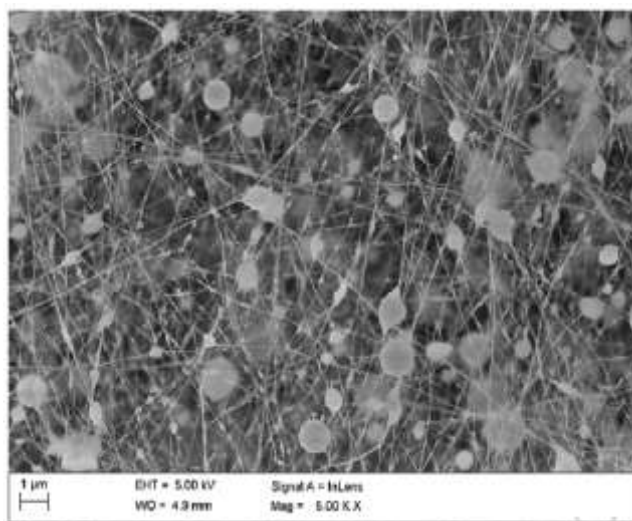


**Figure 12.** SEM images of PAN nanofiber composite system with RT21@P(MMA-co-30%HEMA) particles

Fig. 13 shows that microparticles of RT21@P(MMA-co-40%HEMA) were distributed in a nanofiber structure with PAN interconnections. PAN is present in the wires and onto the RT21@P(MMA-co-40%HEMA) particles. It is seen from the figure that the concentration of the microparticles is increasing.

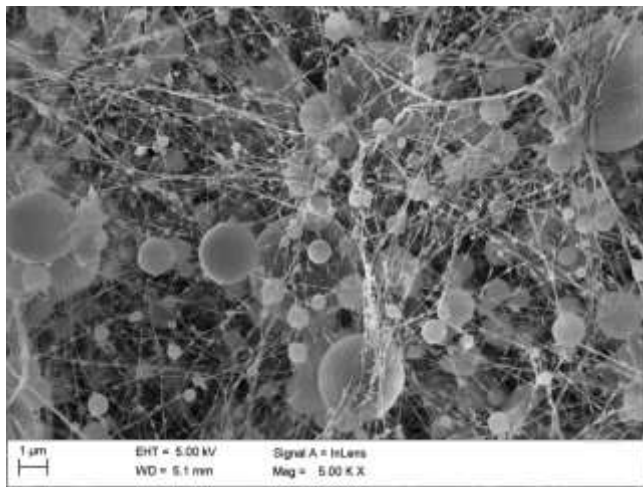


**Figure 13.** SEM images of PAN nanofiber composite system with RT21@P(MMA-co-40%HEMA) particles

In these SEM pictures it is clear that although the MEPCMs have larger diameters, nanofibers hold them in the system providing connections between them. While this structure gives MEPCMs a body, it also points to an expanded surface area. As a result, the existing structure has become usable especially in compact systems such as cooling electronic devices. Even it can be stated that the technology readiness level of the nanofiber materials could reach up to a level prior to market demonstrations. Besides, the transport of the particles are due to PAN in DMF and the bigger the particles more difficult to impart them into nanofiber cross-section. Therefore the yielded particles are mostly small ones.

The electrospinning method, including MEPCMs in the nanofiber section, has not been tried anywhere else yet. Literature generally includes studies on the electrospinning of PCMs lost in the fiber section from a solution where both PCMs and the polymer forming the fiber are dissolved [69]. In these studies, the main focus is the nanofiber formation, which generally coincides with the literature. However, high molecular weight electrospinnable PCMs like PEGs have generally been evaluated in structures. Because low molecular weight PCMs create irregularities in the structure outside the usual fiber form. In this study, MEPCMs in solid form have been included in the nanofiber cross-section. The study's expected feature is the irregular structure due to higher particle size than nanofiber cross-section. It means that these fibers, which aren't desired to have mechanical strength, are replaced with the powder form, which is sometimes dangerous in terms of health in passive applications. Therefore, there is no comparable

study in the literature. In addition, this method has paved the way for electrospinning PCMs operating at low temperatures and turning them into nanofabric form. On the other hand, the energy storage ability of the fabrics produced in the study is significantly higher than the compounds produced by different existing electrospinning methods. This study provided a serious innovation and made it possible to incorporate some PCMs, which are impossible to electrospin under normal conditions, into the nanofiber structure. On the other hand, in previously produced electrospun nanofiber structures with thermal energy storage properties, the PCM was used as a basis to place the PCM completely into the nanofiber body to prevent flow behavior. While this is the real reason the fiber image was obtained precisely here.

#### 4. CONCLUSION

The segments from the HEA and HEMA comonomers in the shell were helpful for the microparticles dispersed into the nanofiber matrix in the potential difference area since the distributions were very good according to the DSC and SEM measurements. The highest enthalpy values were observed in nanofibers with the highest percentages of comonomers. While the melting enthalpy value of 30% HEA nanofiber was 20.1 J/g, the lowest enthalpy value belonged to 1% HEA nanofiber (14.8 J/g). When we look at HEMA nanofibers, the highest enthalpy value belongs to MEPCMS, with 40% of HEMA nanofibers (20.8 J/g). The reason for the disproportion in enthalpy values can be explained by that some MEPCM particles were not effectively surrounded by the PAN in solution. This is attributed to the big particles having PAN at low ratios on their surfaces. Looking at FTIR spectra, the characteristic peaks of paraffin in the spectra of the nanofibers indicated the presence of microcapsules. Characteristic peaks of PAN and shell copolymer supported the presence of the microparticles in the nanofiber structure. In addition, when SEM images were considered, it was clear that nanofibers and microcapsules were present. Because the microcapsules in the polymer solution blocked the tip of the syringe and did not spray properly sometimes, rupture and thinning of the fiber occurred in places. Still, the dispersed particles were not due to pilling in the fiber structure because they were from insoluble MEPCMs in the PAN solution in DMF. The fibrous structure was fruitful in holding microparticles in discrete nanofibers with a physically easy-to-apply form. The nanofiber form increased the surface area and decreased response time to thermal stimuli.

The electrospun nanofibers produced with MEPCMs are further in thermal energy storage than those produced by co-electrospinning. In the presented materials, particles need not be inside a smooth surface of nanofibers. Besides, leakage is no longer a problem due to the double shell of microcapsules and nanofibers. The presented preparation

method solved microparticle utilization problems by fixing them in a constant place. The produced nanofibers were found to be potential materials for different energy storage applications due to their easy-to-apply character.

## Acknowledgement

This study was supported by Çankırı Karatekin University Scientific Research Projects Funding Office (the project no

YMO150219B09). The experimental work of this study was carried out in Polymer Research Laboratory at Chemistry Department of Tokat Gaziosmanpaşa University. The authors are also thankful to Tuğba Özdemir from Molecular Biology and Genetic Department of Tokat Gaziosmanpaşa University of Türkiye since the electrospinning instrument was belonged to her.

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