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Investigation of Electrospun Polyacrylonitrile and Cellulose Acetate Smart Nanofibers Doped with Expanded Graphite for the Structure and Photothermal Effect

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ABSTRACT

In this study, photothermal effect by doping expanded graphite (EG) to smart nanofibers produced by electrospinning method was investigated. Fourier transform infrared (FT-IR) spectroscopy was exploited for chemical characterization. Thermal analysis experiments were carried out by heating and cooling curves. Surface morphology of the produced materials was investigated through scanning electron microscope (SEM). Contact angle was determined through contact angle measurement device. The appearance of the peak of the characteristic cyano group in the structure of Polyacrylonitrile (PAN) at 2237.02 cm^{-1} in the nanofibers having different percentages synthesized with EG and PAN was accepted as the evidence of PAN nanofibers formation. The temperature platforms in the heating/cooling curves exhibited that the temperature of the PAN and cellulose acetate (CA) nanofibers mixed with different EG percentage have higher than pristine nanofibers. The surfaces of the EG@PAN and EG@CA nanofibers were homogeneously distributed fibrous, excessive EG heterogeneously dispersed or electrosprayed in shape. The maximum contacts angles were measured as 67.96° and 52.88° for nanofibers synthesized with EG@CA and EG@PAN, respectively. As the result, the temperature of the nanofibers mixed EG at different percentages increased resulting from having the higher thermal conductivity of EG. Main goal of the study is both investigating photothermal effect in PAN and CA electrospun nanofibers doped with EG of activating heat accumulation property of the produced smart nanofibers for heat energy production from the solar. Thus, it will be possible to develop a new promising method in the production of the smart textile products that have the storage capacity of the solar energy.

Keywords: Solar energy, Photothermal effect, Electrospinning, Smart nanofibers, Expanded graphite.

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Introduction

In recent years, energy conservation is getting important as energy production. Studies on the efficient use of existing energy provide very important contributions to energy sector all over the world.

Energy requirement supplied by fossil fuels such as oil, coal, and natural gas. As a result of the oil crisis people faced in the 1970s and the detection of the ozone hole which caused by CFCs (chlorofluoracarbons), they turned to environmentally friendly, clean and renewable energy sources such as fuel cells, wind energy and solar energy [1-3]. In conventional energy production systems, energy is obtained in three stages. In the first stage, thermal energy is obtained as a result of the combustion of the fuel. The heat produced in the second stage is converted into mechanical energy. In the final stage, electrical energy is obtained from mechanical energy. All these conversion steps cause energy loss. Energy storage systems prevent this loss and ensure that the needed energy is used more efficiently [4, 5]. These renewable energy sources can be stored and used via thermal energy storage methods when energy is needed. With this method, two types of storage can be made as short-term (day and night) and long-term (summer-winter). Thus, by ensuring the sustainability of energy, both energy efficiency increased and the damage caused by fossil fuels to the environment is reduced [6].

Thermal energy is the sum of the kinetic and potential energies of the atoms and/or molecules that make up a substance. It is formed as a result of atomic or molecular vibration [7,8]. The transfer of thermal energy occurs with the heat flow caused by the temperature difference [9]. Thermal energy can be stored as sensible heat, latent heat, thermochemical heat or a combination of all these. The latent heat storage method is performed by storing the latent heat generated by phase change as a result of a significant change in the internal energy of the thermal energy storage (TES) material [7, 10]. Compared with other heat storage methods, the required storage volume for the latent heat storage method is smaller, the heat storage capacity of this method is high, and this method is suitable for constant temperature heat storage. The heat storage materials used in latent heat storage methods are called phase change material (PCM). In recent years, many applications have been carried out where solar energy is stored in PCMs in the form of latent heat storage and then

this stored heat is released by the PCM. Here, there is usually a solid-liquid phase change [7, 8, 11].

Moreover, PCMs have been used to produce thermostable textiles for garments that provide thermal comfort [12,13]. The use of PCM containing fibres or fabrics in home textile products such as bed linen, furniture fabrics, curtains is becoming increasingly common, besides garments. It is seen that PCM products are applied especially after microencapsulation into the polymer structure, in many textile studies [14-16]. Nowadays, the use of nanofibers produced by electrospinning method and that can store thermal energy are becoming increasingly common [17-19]. Production of bicomponent nanowebs, composed of Polyacrylonitrile (PAN) and PCM (polyethylene glycols (PEG), polyethylene glycol methyl ethers (PEGME)) and paraffin waxes (n-alkane) were carried out via coaxial electrospinning method. Özmen et al. (2020) produced nanofibers with heat storage/releasing properties composed of PCM (fatty acid) and polymethylmethacrylate (PMMA) by using a coaxial electrospinning method [20]. In the literature, it is seen that there are some studies thermo-active smart fibres are used in the applications such as drug release, separation processes, energy storage and conversion etc. [21]. Liu et al. (2023) reported to synthesize the flexible phase change nonwovens (GB-PCN) by wet-spinning hybrid graphene boron nitride (GB) fibre and subsequent impregnating paraffins such as eicosane and octadecane. [22]. A very small amount of the energy released from the sun reaches the earth's surface. Solar energy coming to the earth's surface, at various wavelengths consists of radiation. Recently, the studies were carried out to develop multifunctional hybrid polymeric materials that allow solar energy to be stored as thermal energy. [23-25].

PAN is a thermoplastic polymer obtained as a result of polymerization of acrylonitrile monomers. This polymer, which has strong secondary interactions thanks to polar acrylonitrile groups, has superior properties such as good mechanical properties and thermal resistance. Thanks to these superior properties, PAN has become a highly demanded polymer by the fibre industry [26].

Cellulose acetate (CA) natural polymer with a wide range of properties and is used most of industrial applications such as membrane technologies, textiles and energy storage materials [27-29].

In this study, photothermal effect in PAN and CA electrospun nanofibers doped with EG was investigated, and heat accumulation property of the produced smart nanofibers were carried out. It will help to validate them for heat energy production from the solar. The produced structures and their energy harvesting properties were proven by FT-IR spectroscopy, photolytic heating measurements, SEM analysis and contact angle measurements.

Materials and Methods

Materials

CA was purchased from Across Organics. PAN was obtained AKSA Akrilik Kimya Sanayii A.Ş. Expanded Graphite (EG, thermal conductivity: 4.26 W/mK) was obtained from the Fluka Company. N, N-Dimethylformamide (DMF) (Merck) was used as a solvent. These chemicals were not further purified prior to use. These chemicals were not further purified prior to use due to their analytical purity percentages of 99 % and plus.

Synthesis of Nanofiber by Electrospinning

PAN and CA based smart nanofibers with EG were prepared by electrospinning of their DMF solutions. They are so called EG@PAN and EG@CA in this article. They are 8 different nanofibers as single matrix polymers and EG imparted composites at different weight ratios. The smart nanofibers produced using electrospinning instrument (Nano WEB electrospin 100 instrument) were having photothermal property. In Table 1, the produced smart nanofibers were shown. 5 ml syringe with a 21 G blunt tip needle was used for pumping the solution at a rate of 1.5 ml/hr. The syringe tip to collector distance was set to determine optimum distance between the needle and Al pad which was found as 18 cm and the optimum voltage between the needle tip to an aluminium foil covered collector was determined as 18 kV voltage. Application time to produce the smart nanofibers was found as 4 hours. The obtained nanofiber mats were collected and conserved at 25 °C temperature before characterization tests.

Table 1. The expanded graphite-polymer mixtures weight/weight (w/w) ratios in electrospinning solutions

Smart nanofiber sample	The expanded graphite-polymer (w/w) ratios (%)	
	EG (%)	Polymer, PAN, CA weight percentages (%)
PAN nanofiber	0	(PAN) 100
EG@PAN 10/90 nanofiber	10	(PAN) 90
EG@PAN 15/85 nanofiber	15	(PAN) 85
EG@PAN 20/80 nanofiber	20	(PAN) 80
EG@PAN 25/75 nanofiber	25	(PAN) 75
CA nanofiber	0	(CA) 100
EG@CA 10/90 nanofiber	10	(CA) 90
EG@CA 15/85 nanofiber	15	(CA) 85
EG@CA 20/80 nanofiber	20	(CA) 80
EG@CA 25/75 nanofiber	25	(CA) 75

Characterization

Chemical analysis

The chemical analysis of the smart nanofiber samples was carried out by using a Fourier transform infrared (FT-IR) spectrometry instrument (JASCO FT/IR-4700) with an attenuated total reflection accessory. FT-IR spectra were recorded between 4000 cm^{-1} and 400 cm^{-1} at a total of 16 scans.

Thermal analysis

Heating and cooling curves were drawn for the smart nanofibers samples with help of a 100 Watt lamp placed approximately 20 cm above as photothermal energy source (General Electric 100W E27 R95 INFRARED) in the closed system. Temperature data were recorded for constant periods of time during light on (heating) and during dark (cooling) by using a data-logger (Nova 5000) device.

Morphological analysis

Surface morphology investigation of the produced nanofibers samples were performed by using a scanning electron microscope (SEM) instrument (TESCAN MIRA3 XMU). The nanofibers samples surfaces were coated with a conducting paint including gold prior to the measurements.

Contact angle analysis

The Contact angles of the nanofibers were determined through contact angle measurement instrument (Terra Lab Attension Theta Lite Optical Contact Angle Measurement Device).

Results and Discussion

In Table 2, the properties of the similar electrospun fibers used for thermocomfortable and thermoregulated textile productions were compared to the properties of the EG@PAN AND EG@CA nanofibers. It was seen that the surfaces of the nanofibers were smooth and homogeneously distributed fibrous in shape, when examined by SEM instrument.

Table 2. The properties of the electrospun fibers used for thermocomfortable textile products

Electrospun Fibers	Melting Temperature (°C)	Surface Morphology	Contact Angle	Reference
EG@PAN nanofiber	-	homogeneously distributed fibrous	52.88°	-
EG@CA nanofiber	-	homogeneously distributed fibrous	67.96°	-
Superhydrophobic–superoleophilic fibrous *PVDF membranes	-	smooth and homogeny surface	≥ 153°	[16]
*PEG 1000-PVA nanofiber	28°C	homogenic surface	-	[17]
*PMMA16-KA-GA nanofiber	35.1°C	smooth and homogeny surface	-	[20]
*PMMA16-LA-GA nanofiber	49.7°C	smooth and homogeny surface	-	[20]
*PMMA16-MA-GA nanofiber	63.4°C	smooth and homogeny surface	-	[20]

*PVDF:
*PEG 1000-PVA:
*PMMA-KA-GA: polymethylmethacrylate-capric acid-graphene
*PMMA-LA-GA: polymethylmethacrylate-lauric acid-graphene
*PMMA-MA-GA: polymethylmethacrylate- myristic acid-graphene

Chemical Properties of the EG@PAN and EG@CA Nanofibers

The FT-IR spectra of the EG@PAN nanofibers and the EG@CA nanofibers were showed in Figures 1 and 2, respectively.

In the FT-IR spectrum of PAN nanofiber the peak appeared at 2237.02 cm^{-1} attributed from the characteristic -C≡N group in the structure of PAN. The peak observed at 2925.48 cm^{-1} is due to the aliphatic methylene (CH_2) group [26, 30]. The peak appeared at 1232.29 cm^{-1} is attributed by the C-O-C vibrations of vinyl acetate in the PAN nanofiber structure in Figure 1 [26, 31].

In the FT-IR spectrum of CA nanofiber, the peaks appeared at 1734.66 cm^{-1} related to C=O stretching and 1366.32 cm^{-1} related to C-H bending. In addition, the peaks seen in 1216.86 cm^{-1} and 1034.62 cm^{-1} wave numbers are associated with C-O stretching in Figure 2 [32, 33].

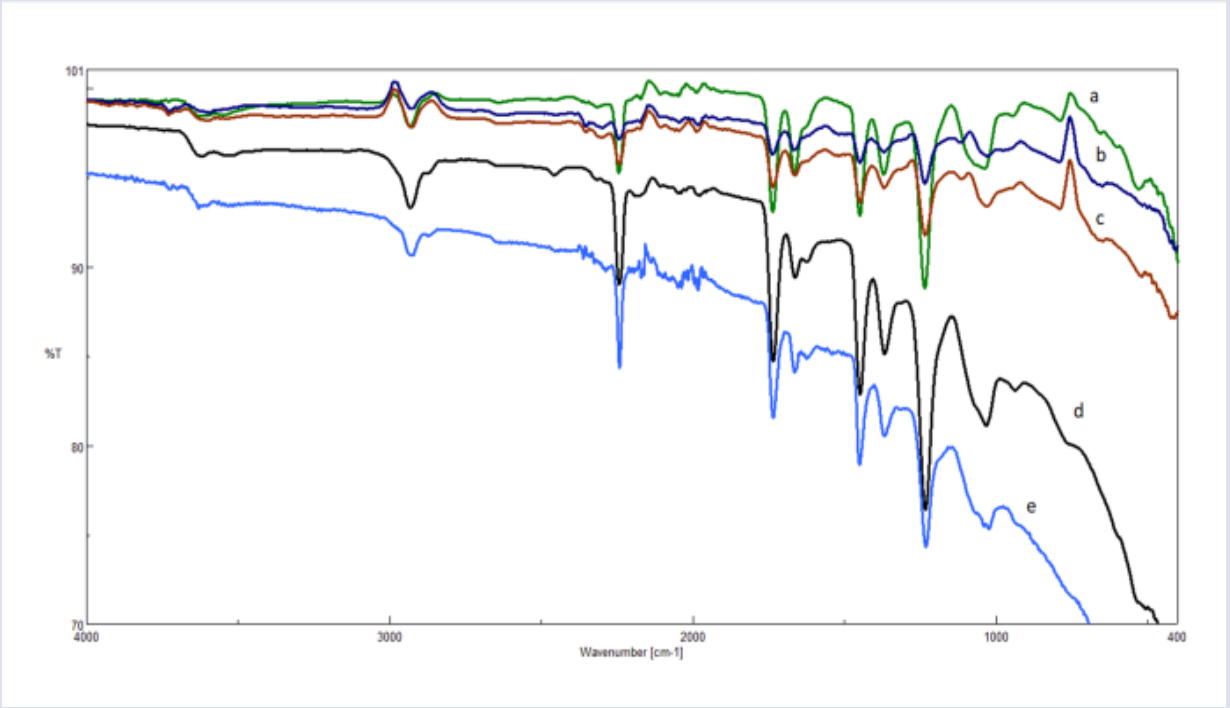

Figure 1. The FT-IR spectra of the EG@PAN nanofibers at different EG weight percentages (a: Pristine PAN nanofiber; b: 10 % EG + 90 % PAN; c: 15 % EG + 85 % PAN; d: 20 % EG + 80 % PAN; e: 25 % EG + 75 % PAN)

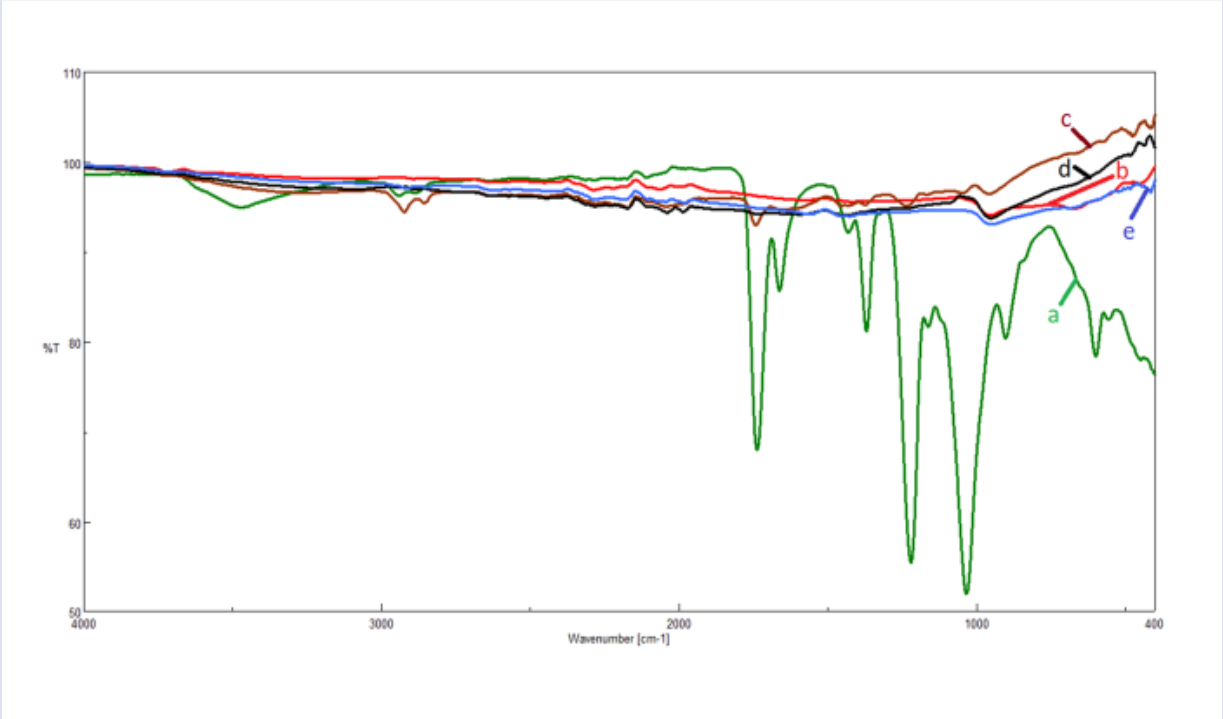

Figure 2. The FT-IR spectra of the EG@CA nanofibers at different EG weight percentages (a: Pristine CA nanofiber; b: 10 % EG + 90 % CA; c: 15 % EG + 85 % CA; d: 20 % EG + 80 % CA; e: 25 % EG + 75 % CA)

Thermal Properties of the EG@PAN and EG@CA Nanofibers

The thermal properties of the nanofibers were investigated by determining the heating and cooling curves. The photothermal energy source was used in the closed system. The heating and cooling curves of the EG@PAN and EG@CA smart nanofibers having different percentages were showed in Figures 3 and 4, respectively. The temperature platforms in the heating/cooling curves exhibit heat storage/release process in these figures. The temperature converts slowly during the heating/cooling process. As seen in Figures 3 and 4, the temperature of the nanofibers mixed EG at different percentages is always highest. The reason is that EG has high thermal conductivity which is helpful for the heating/cooling process.

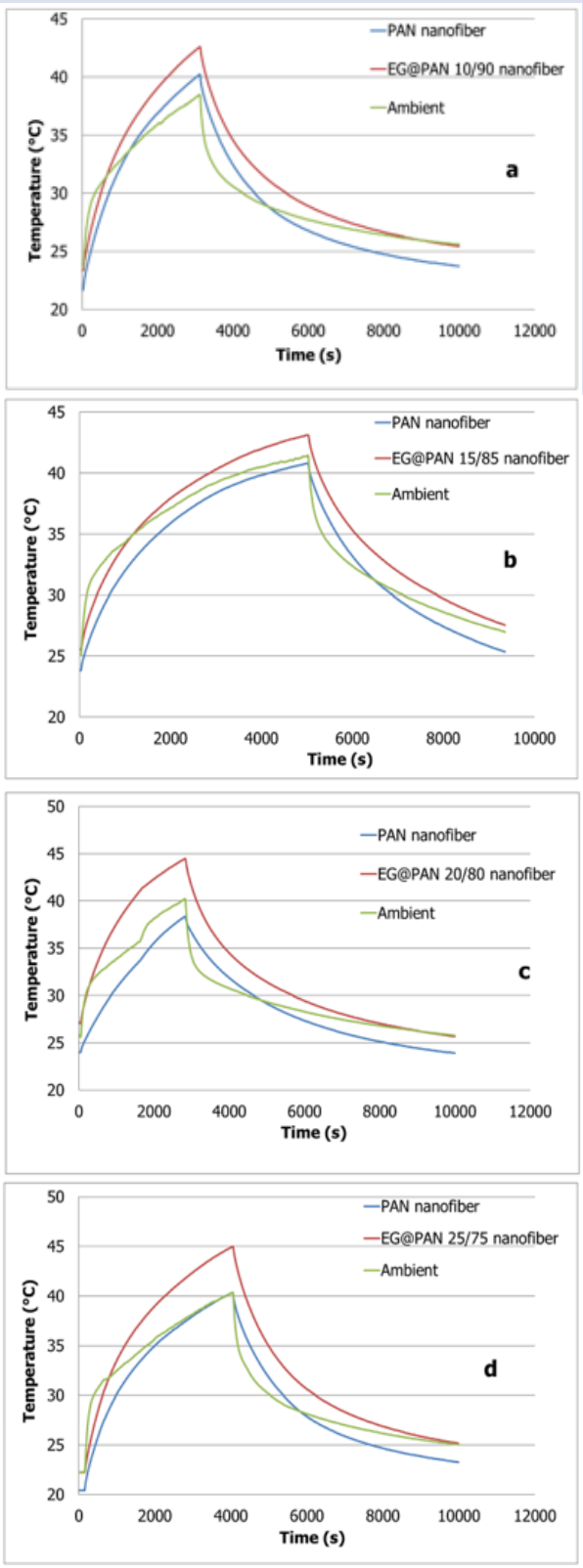

Figure 3. The Heating and cooling curves of the EG@PAN nanofibers at different EG weight percentages (a: 10 % EG + 90 % PAN; b: 15 % EG + 85 % PAN; c: 20 % EG + 80 % PAN; d: 25 % EG + 75 % PAN)

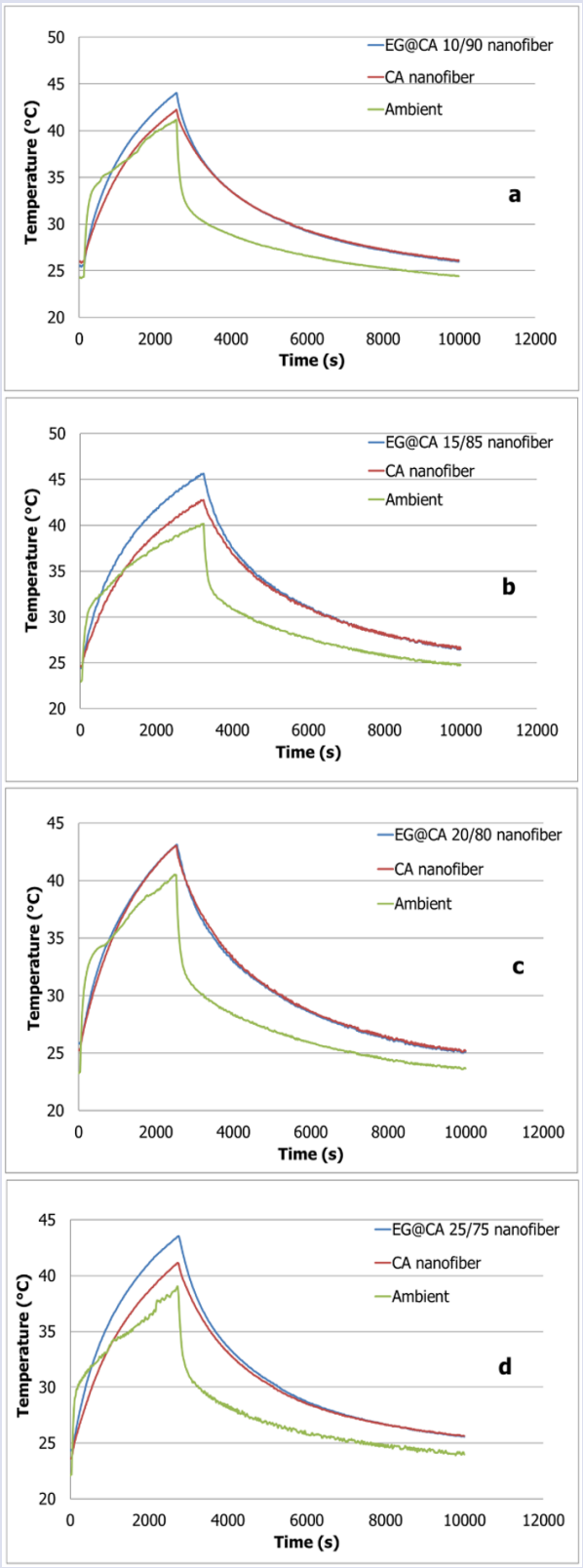

Figure 4. The Heating and cooling curves of EG@CA nanofibers at different EG weight percentages (a: 10 % EG + 90 % CA; b: 15 % EG + 85 % CA; c: 20 % EG + 80 % CA; d: 25 % EG + 75 % CA)

Morphological Properties of the EG@PAN and EG@CA Nanofibers

The SEM images of the matrices PAN and CA nanofibers, and EG@PAN and EG@CA composite nanofibers at different percentages of EG were given in Figures 5 and 6, respectively. According to the SEM images, the surfaces of the EG@PAN and EG@CA nanofibers were homogeneously distributed fibrous, excessive EG heterogeneously dispersed or electrosprayed in shape. The surface morphology of the nanofibers was a coated by the polymer structure, and light reached through the host polymer to bear photothermal effect [34]. EG has a better dispersion in nanofibers synthesized with PAN and, consequently, it greatly improves the overall performance of the nanofibers.

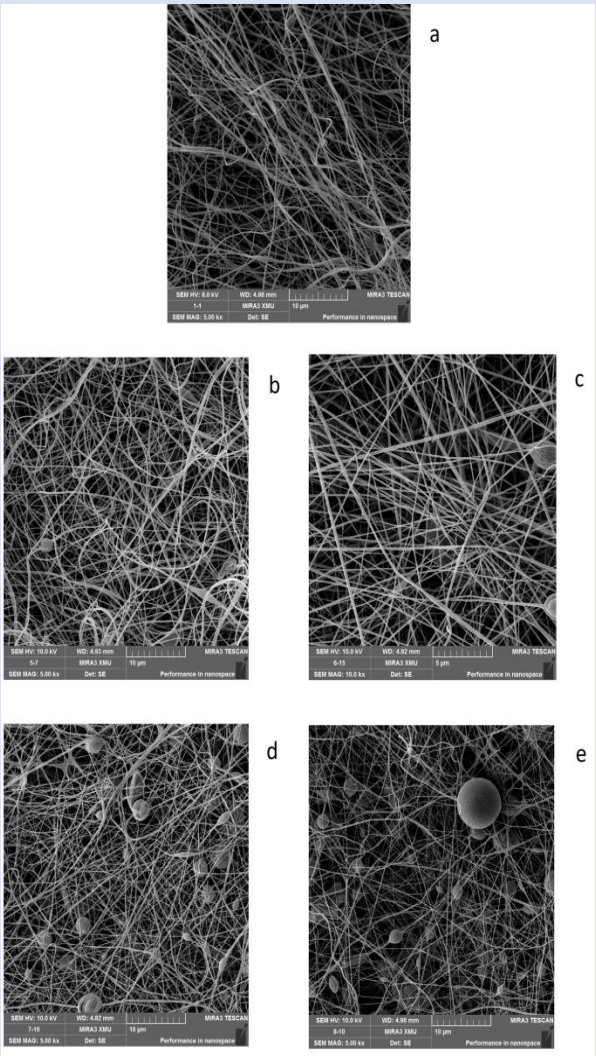

Figure 5. The SEM images of the EG@PAN nanofibers at different EG weight percentages (a: Pristine PAN nanofiber; b: 10 % EG + 90 % PAN; c: 15 % + 85 % PAN; d: 20 % + 80 % PAN; e: 25 % + 75 % PAN)

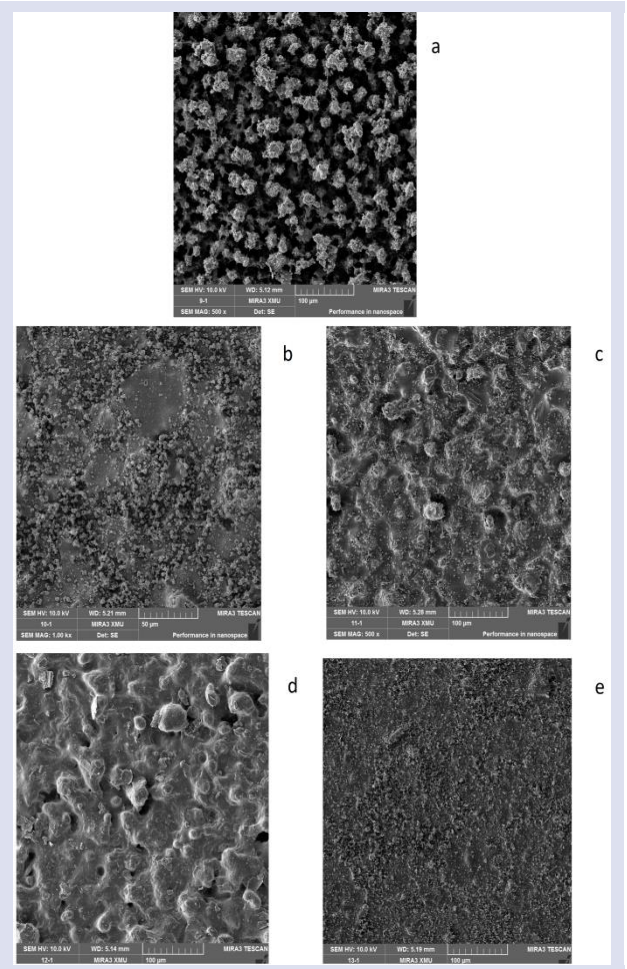

Figure 6. The SEM images of the EG@CA nanofibers at different EG weight percentages (a: Pristine CA nanofiber; b: 10 % EG +90 % CA; c: 15 % EG + 85 % CA; d: 20 % EG + 80 % CA; e: 25 % EG + 75 % CA)

Contact Angle Measurements of the EG@PAN and EG@CA Nanofibers with Water

The contact angle measurements images of the EG@PAN and EG@CA nanofibers which have different percentages were given in Figures 7 and 8, respectively. As seen in Figures 7 and 8, while the water contacts angle of the nanofibers synthesized with EG and CA at different percentages increase, the water contacts angle of the EG@PAN nanofibers at different percentages decrease. The increase in the contact angles of the EG@CA nanofibers indicated the wettability decreased. The maximum contacts angles were measured as 67.96° and 52.88° for EG@CA nanofibers and EG@PAN nanofibers, respectively. The surface morphology of the EG@PAN and EG@CA nanofibers has played a crucial role in the hydrophobicity of the resulting ultrathin fibrous nanofibers. In the experimental study it has been seen that the water contact angles of electrospun EG@PAN nanofibers are lower than those of EG@CA nanofibers.

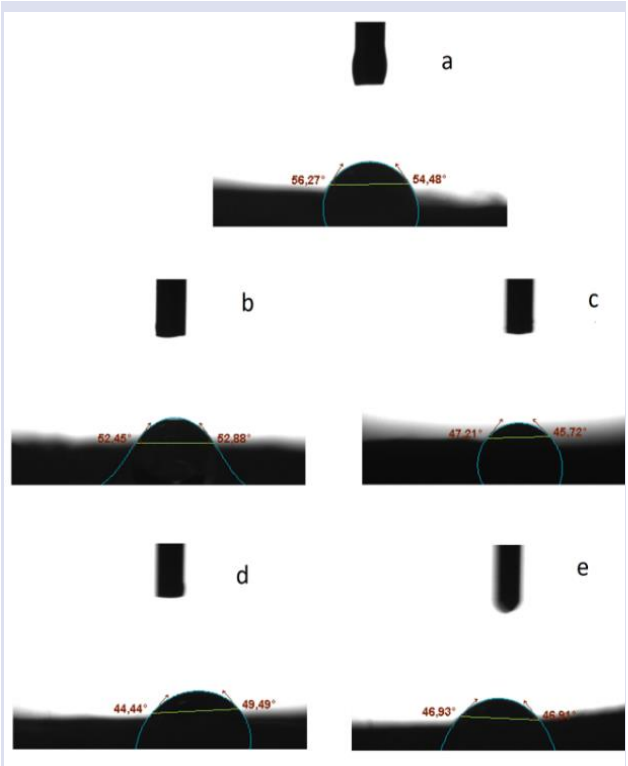

Figure 7. The contact angle images of the EG@PAN nanofibers at different EG weight percentages (a: Pristine PAN nanofiber; b: 10 % EG + 90 % PAN; c: 15 % EG + 85 % PAN; d: 20 % EG + 80 % PAN; e: 25 % EG + 75 % PAN)

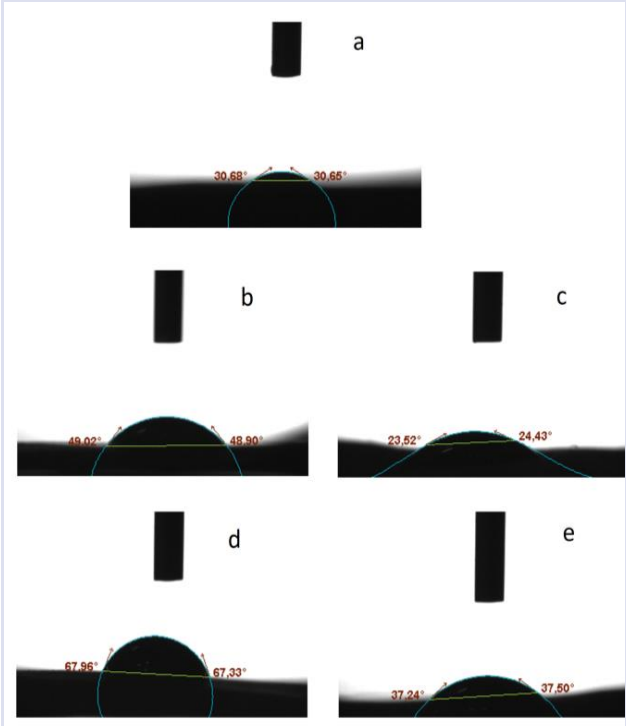

Figure 8. The contact angle images of the EG@CA nanofibers at different EG weight percentages (a: Pristine CA nanofiber; b: 10 % EG + 90 % CA; c: 15 % EG + 85 % CA; d: 20 % EG + 80 % CA; e: 25 % EG + 75 % CA)

Conclusions

EG as a photothermal effect agent was embedded into nanofiber matrices of CA and PAN polymers by electrospinning in DMF solution in order to have smart fabrics with energy harvesting property.

Characterization by FT-IR spectroscopy revealed the copresence of the polymers and expanded graphite together in the smart fabric structures. The appearance of the peak of the characteristic -C≡N group in the structure of PAN at 2237.02 cm^{-1} in the nanofibers synthesized with EG and PAN at different percentages was accepted as the evidence of PAN nanofibers formation. At 1734.66 cm-1 related to C=O stretching in the nanofibers synthesized with EG and CA at different percentages was appeared. The temperature platforms in the heating/cooling curves exhibit that the temperature of the PAN and CA nanofibers mixed with different EG percentage have higher than pristine PAN and CA nanofibers. The surfaces of the EG@PAN and EG@CA nanofibers were homogeneously distributed fibrous, excessive EG heterogeneously dispersed or electrosprayed in shape. This is evidence of the increase in surface area which is very important for energy harvesting property of the smart fabrics. Therefore, they will interact to light much more. It was determined that the temperature of the nanofibers including EG in the matrices at different weight percentages was increasing when subjected to light. Thermal conductivity increment by the expanded graphite content was also helpful for energy harvesting property at the proposed level. When examined by SEM instrument, PAN nanofibers with expanded graphite showed some seepage at high expanded graphite contents whereas CA nanofibers resulted in compatible but not fibrous structures applicable for energy harvesting applications. The maximum contacts angles were measured as 67.96° and 52.88° for nanofibers synthesized with EG and CA and nanofibers synthesized with EG and PAN, respectively. It has been seen that the water contact angles of electrospun EG@CA nanofibers are higher than those of EG@PAN nanofibers and have higher hydrophobicity as a result of the measurements of the contact angles.

Conflicts of interest

There are no conflicts of interest in this work.

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