RSC Advances



This is an *Accepted Manuscript*, which has been through the Royal Society of Chemistry peer review process and has been accepted for publication.

Accepted Manuscripts are published online shortly after acceptance, before technical editing, formatting and proof reading. Using this free service, authors can make their results available to the community, in citable form, before we publish the edited article. This Accepted Manuscript will be replaced by the edited, formatted and paginated article as soon as this is available.

You can find more information about *Accepted Manuscripts* in the **Information for Authors**.

Please note that technical editing may introduce minor changes to the text and/or graphics, which may alter content. The journal's standard <u>Terms & Conditions</u> and the <u>Ethical guidelines</u> still apply. In no event shall the Royal Society of Chemistry be held responsible for any errors or omissions in this *Accepted Manuscript* or any consequences arising from the use of any information it contains.



www.rsc.org/advances

RSC Advances

Journal Name

Cite this: DOI: 10.1039/x0xx00000x

Received 00th November 2014, Accepted 00th November 2014

DOI: 10.1039/x0xx00000x

www.rsc.org/

ARTICLE

RSCPublishing

Synthesis and performance evaluation of the aerogelfilled PET nanofiber assemblies prepared by electrospinning

Z. Mazrouei-Sebdani,^a A. Khoddami,^a H. Hadadzadeh^b and M. Zarrebini^a

This paper focuses on the potential use of sodium silicate based aerogels, instead of small precursor molecules, as a filler in the PET nanofibers (PNFs). For the first time, the aerogel micro-granules (AMG) were added to the electro-spinning solution of PET with successful embedding of them into the PET nanofibers. The evaluation of the samples was carried out by measuring 3M water repellency, sliding angle (SA), water contact angle (WCA), transmission spectroscopy, sound absorption, heat transfer, FTIR, BET, SEM, and AFM. The results indicate a successful synthesize of the AMG with a porosity of 91.8% and a surface area of 815 m^2/g . By addition of the AMG to the electro-spinning solution of PET, the bead-on-string aerogel-filled nanofibers were produced. This was accompanied by a significant improvement in hydrophobicity, dye-ability, and insulation properties. The most hydrophobic sample was prepared with the addition of 2% AMG. After the fluorocarbon treatment of this sample, the WCA was increased from 130.1 to 147.2°, the 3M water repellency was changed from 1 to 10, and the SA was reduced from 90 to 5°. In comparison with the primary PNF, the addition of AMG to the PNF was increased the dyeing rate and dye adsorption at equilibrium from 30% to 70% for Disperse Blue 56. This can be introduced as a low-temperature method for dyeing of PET fibers. Also, a decreasing trend in the heat transfer was observed from 21% (for the pure PNF) to 16.4% and 8.3% for 0.5AMG/PNF and 4AMG/PNF, respectively. The investigation of the acoustic properties was shown a direct relation between the amount of AMG in the pure PNF and the sound absorption coefficient (NAC). The comparison of the porous silica aerogel with a non-porous one has clearly shown a significant effect of the porosity on the different properties of the samples.

Keywords: SiO_2 aerogel micro-granules; PET nanofibers; Electro-spinning; Hydrophobicity; Dyeing and insulation properties.

1. Introduction

The term aerogel was first coined less than a century ago by Kistler. Aerogels are defined as gels in which the liquid is replaced by a gas, and the solid gel network remains intact [1, 2]. Aerogels are also known as frozen smoke or air-glass. These materials are composed of 10 nm or below nanoparticles and have nano-pores with diameters of less than 50 nm [3]. Silica aerogels have found extensive applications in end-uses such as buildings, automobile, pharmaceutical, biomedical, space & airborne, insulation, and art [2-5]. This can be attributed to their unique characteristics such as high surface area ($\approx 1000 \text{ m}^2/\text{g}$), low density ($\approx 0.030 \text{ g/cm}^3$), high optical transmission (≈90%), high porosity (≈99%), low thermal conductivity (≈ 0.020 W/mK), and low dielectric constant (≈ 2). Silica aerogels fabrication is initiated by a sol-gel process followed by aging [3] and supercritical drying (SCD) [6, 7] or ambient pressure drying (APD) [1, 2, 8, 9] of the formed gel. Conventionally, silica aerogels are generated by more energy consuming and expensive SCD method, using alkoxide precursors such as tetraethoxysilane (TEOS) or tetramethoxysilane (TMOS) [3]. The conventional method of aerogel formation due to high cost partly prohibits commercialization of the products. Therefore, for the aerogels

to be commercially viable, the use of an inexpensive precursor together with a rapid and low cost drying method is essential. Recent advances have pointed to enormous potential of the APD as an alternative fabrication route where sodium silicate (Na₂SiO₃) as a purely inorganic, available, and low-cost precursor is used. These aerogels are comparable to those obtained by the conventional methods [1-5, 8-9]. However, silica aerogels due to their nanoporous structure are generally very brittle with low strength [3, 10]. Mechanical properties of silica aerogels can be enhanced by compounding with fibers. This technique paves the way for the production of either organic or inorganic fiber-reinforced aerogel composites [10]. Researches on aerogel composites so far have focused on addition of micron and submicron inorganic or organic fibers to the silica sol as reinforcement [11-13]. It has been established that the monolith integrity and the mechanical stability of such a composite were improved if the reinforcing fibers are in nanoscale and the matrix particle are dimensionally more compatible with the fibers [14]. On the other hand, electro-spinning is the most logical technique for the production of superfine fibers with high specific surface area [14, 15].

Recently, addition of metal-containing precursor into electrospinning-used polymer solution has been used for preparation of metal oxide/nanofiber composites or metal oxide nanofibers. Many polymers have also been used as the matrices to host inorganic precursors [16]. For example, the crystallinity and morphology of the nanofibres produced by homogenously mixing of TiO₂ nanoparticles, with 10-20 nm particle sizes, in the electro-spinning solution of PET were investigated by Meng [17]. In the other research, SiO₂ nanoparticles were introduced to the PET solution, and then the crystallinity and thermal resistance of the resulted nanofibers were investigated [18].

Synthesis of nanofiber/SiO₂ aerogel involves impregnation of electro-spun fibers with the sol. This is followed by the solvent exchange, surface modification, and drying stages [15]. Excessive dusting of aerogels from the fiber assemblies is of important drawbacks [19]. This can be significantly reduced by addition of AMG to PET solution during the electro-spinning of fibers. Since, the addition of an aerogel powder to resins for improving their mechanical and thermal properties has been rarely investigated [20, 21], practically no information is available regarding the content of aerogel powders in polymeric fibers. For example, silica aerogel was chosen as threedimensional (3D) nanoporous filler for an epoxy resin to increase the stiffness and glass transition temperature of the final composites [20]. Also, a natural rubber-silica aerogel film was developed by latex compounding to form a thin film where the silica aerogel acts as a filler [22].

Different from these works, this paper for the first time aims to fabricate AMG-filled PNFs using the micro-size nanostructured aerogel, instead of small precursor molecules, as a filler and the PET nanofibers as the substrate *via* electrospinning method and then, evaluate its performance. The effect of the AMG content on improvement of the hydrophobicity, dyeing, and insulation properties of the AMG-filled PNFs were also investigated. In addition, the reduction in the surface energy through the coating by a fluorocarbon derivative, Rucostar EEE, was studied.

2. Experimental

Materials

Strong-acid cationic exchange resin (Amberlite IR 120 H), waterglass (Na₂SiO₃), ammonia, propone-2-ol, n-hexane, electro-spinning solvents containing dichloromethane (DCM, CH₂Cl₂) and trifluoroacetic acid (TFA, CF₃COOH), and Disperse Blue 56 (M = 365.18 g/mol) colorant were of analytical grade from Merck, Germany. Trimethylchlorosilane (TMCS) was purchased from Daejung, Korea. Twice-distilled deionized water and PET chips were obtained from Havakesht and Tondgooyan companies, Iran, respectively. The selected fluorocarbon derivative, Rucostar EEE, was obtained from Rudolf, Germany. The dispersing agent in the dyeing bath was EKALIN F from Sandvz, Japan.

Preparation of the aerogel micro-granules, AMG

For the preparation of silica sols, a waterglass solution with a specific gravity of 1.39 was diluted to 1:4 (v/v) with water. A silicic acid solution with pH = 2.4-2.7 was prepared by removal of the sodium ions in the waterglass precursor, using Amberlite IR 120 H resin. The gelation of the prepared material was achieved by adjustment of the initial pH of the silica sol to 5, with diluted ammonia solution (1%). The mixture was then decanted into the plastic vessel to be gelled within approximately 10 min. For structural strengthening purposes of the gel, the prepared hydrogel was aged for 3 h. The hydrogel with water content in its internal pores was immersed in propone-2-ol. This was followed by immersion, first in n-

Hexane, and then in TMCS/n-hexane mixture (1:4 (v/v)) for 24 h at 50°C in a 150 rpm incubator. This sequence yields alcogel, organogel, and modified gel, respectively. The surface-modification of the gel was achieved by drying at room temperature for 24 h. The dried gels were then heat-treated at initial and final temperatures of 50 and 230°C for 1 h. A ball mill (Pm10, Retsch, Germany) with two zirconia balls at 150 rpm was used for grinding of the aerogel granules for 120 min where a suitable powder for successful electro-spinning was obtained. The grounded material was used in weight-wise proportions of 0.5, 1, 2, and 4% (w/w) in the electro-spinning process. For effective evaluation, the prepared nonporous SMG sample, without hydrophobic treatment using TMCS, was compared. Table 1 denotes selected abbreviation for the sample identification.

Preparation of the AMG-filled PET nanofibers

Prior to the electro-spinning process, the PET solution (15% (w/v)) was prepared by dissolving the PET chips in 1:1 (v/v) DCM and TFA. The mixture was first stirred using a magnetic stirrer for 12 h and then by an ultrasonic stirrer (UP200 H lab device (200 W, 24 kHz)) for 20 min. The prepared spinning solution was fed to a horizontal electro-spinning, STC-523 device, Japan, *via* a syringe. The electro-spinning process was carried out at a voltage of 13 kV and a solution injection rate of 0.2 mL/h. The distance of the syringe tip and a cylindrical collector was set at 15 cm. The nanofibers were collected on a screen with 49 mesh size. The formation of the PET/Aerogel composites and the effect of AMG at 0.5, 1, 2, and 4% (w/w) on the structure, hydrophobicity, dyeing and insulation properties were vividly established.

Table 1

The preparation of the hydrophobic silica aerogel and the AMG-filled PET nanofibers is shown in Figure 1.

Figure 1

Hydrophobic treatment

In order to reduce the surface energy of the nanofibers, a layer of a fluorocarbon derivative (Rucostar EEE) was coated on their surface. This was achieved by impregnation of the nanofibers in a treatment bath containing 40 g/L Rucostar EEE (at pH 5) for 10 min. The impregnated fibers were then dried and cured at 100 and 140°C for 10 and 4 min, respectively.

Disperse dyeing

The pure and composite PET nanofibers were dyed in a dyeing bath containing 0.5% Disperse Blue 56. A dispersing agent (10%) and diluted acetic acid (1%) were used for adjustment of pH to approximately 5.5. The operation was carried out at 90°C with L:R 100:1 for periods of 20, 60, 90, 120, 180, and 300 min using Polymat, AHIBA 1000, Data color Co., Swiss; a lab dyeing machine.

Evaluation methods

A Belsorp mini II apparatus built by BelJapan, Japan (ISO 9277) was used to obtain the parameters such as specific surface areas, total pore volume, and mean pore diameter based on the N_2 adsorption techniques or BET analysis [23]. This analysis aims to explain the physical adsorption of the gas molecules on a solid surface at different pressure of N_2 gas. The out gassing operation for the removal of water vapor, carbon

dioxide and other unwanted compounds in the voids was repeatedly performed for several hours at 120°C.

The particle size distribution of the powder was determined by static light scattering (Horiba, LA-930, Japan), equipped with 5 mW He/Ne (635 nm) laser beam, at room temperature (20 ± 2 °C). Before injection, a mixture of powder in water/propan-2-ol mixture was stirred for 5 h and then added to unit cell until the transmittance reached between 70-85 % of the water blank. The repellent properties of the electro-spun webs were evaluated by the WCA, SA, and 3M water repellency tests. The 3M water/alcohol drop test was employed with the test numbered liquids beginning from water to propan-2-ol in an ascending order of propan-2-ol percentage in the liquids. The ascribed (W-10) rating denotes a sample with most visible liquid remaining on the surface after 10 s. In general, the water repellency rating of 2 or greater is desirable [24]. The SA of the samples is defined as the critical angle upon which a water droplet of given weight begins to slide down the inclined plate. This was measured six times per sample using an instrument developed during course of previous research [24]. The location of a droplet on the surface of the hydrophobic treated samples allows evaluating the WCAs. The images were taken using a USB digital microscope, Micro View (magnification ratio 10-800X). The angles between the liquid/solid and liquid/vapor interfaces were measured using the Digimizer software. The absorbance of the dye solution [25, 26] for various dyeing periods was evaluated using Gretag Macbeth Colour Eye 7000A Spectrophotometer. The Beer-Lambert's law was used to measure the dye absorption:

$$A = \varepsilon cl \tag{1}$$

Where, A denotes the absorbance of the dye, ϵ represents the molar absorptivity, c is the concentration of the dye, and l corresponds to the path length. Based on equation 2, E the exhaustion (%) of the dyeing solution was calculated.

$$E\% = \frac{A_{\circ} - A}{A_{\circ}}$$
(2)

Where, A_{\circ} and A are the absorption at the time of t_{\circ} and t, respectively.

In order to study the sample surface structure, the SEM image of the samples was obtained using EM3200, KYKY. The SEM image is analogous to a photograph except that features are much smaller to be seen with visible light. The samples were gold-coated with a sputter coater, SBC12, KYKY. The detailed topological features of the fiber surface can be determined using atomic force microscopy (AFM) (Bruker, Germany). A silicon probing tip was used for imaging in contact mode.

The density of the samples was calculated in terms of given mass and volume of the aerogel granule. The density and porosity were calculated using equations (3) and (4), respectively.

$$\rho_{\rm b} = \frac{\rm m}{\rm V} \tag{3}$$

$$Porosity = \left(1 - \frac{\rho_{\rm b}}{\rho_{\rm s}}\right) \times 100 \tag{4}$$

Where, ρ_b , ρ_s , m, and V represent the bulk density (g/cm³), the skeletal density of silica (g/cm³), mass (g) and the apparent

volume (cm³), respectively. The value of ρ s for silica was found to be approximately 2.2 g/cm³.

FTIR spectra of the samples were recorded by MB-Series 100, Hartman & Braun, Canada. The FTIR spectra were used to identify the aerogel particles in the nanofibers as well as the chemical bonding states within the material which help to elucidate changes in the chemical composition of the fiber surface.

For the evaluation of the acoustic performance of the samples according to ASTM E-1050, an impedance tube apparatus containing an external stainless steel tube with a smooth surface, a signal generator, a moveable microphone, and a speaker. The detailed structure of the device is shown in Figure 2. The size of the external tube was 100 cm in length and 2.5 cm in diameter. The internal tube which is connected to the microphone was 104 cm in length, 0.7 cm in external diameter, and 0.5 cm in internal diameter. A steel bracket supports the test samples at one end of the tube and also prevents transmission of the incident sound through the fabric/bracket assembly (Figure 2) [27]. As illustrated in Figure 2, the speaker is positioned on the other end of the tube. Sound which is sensed by the microphone is generated by the signal generator in the frequency range of 250-4000 Hz. An oscilloscope is used to display the sound waves. A digital voltmeter provides the data employed for calculating the NAC values of samples at each frequency. The acoustic behaviour of the absorptive material is expressed in terms of NAC. The NAC of the samples at sound frequencies as varied as 250, 500, 1000, 2000, and 4000 Hz were measured according to equation 5.

$$NAC = \frac{4S}{(1+S)^2}$$
(5)

Where, S is the ratio of the maximum to the minimum voltage as recorded by the voltmeter.

Figure 2

The dynamic heat transfer of the fabric samples was investigated using a previously method [28, 29] (Figure 3). This instrument has the ability to simulate the possible body condition comprising a controlled environmental chamber, a guarded hot plate, and a data acquisition system. A schematic of the instrument is shown in Figure 3. The guarded hot plate was placed in a chamber at ambient conditions of 25°C, and 65% relative humidity (RH). This guarded hot plate was comprised of different components including a 37°C hot plate as a heat source, a water container, a membrane for simulating human skin and the sensors for temperature determination. One side of the sample was faced on the membrane but not in contact, whereas the other side was exposed to the controlled environment. Two temperature sensors were located above and below the sample. A temperature-time curve was obtained from the data acquisition unit. The measurement was repeated twice for each sample and the average percentage of the heat transfer was calculated by equation (6).

Heat transfer (%) =
$$\frac{\Delta T_1}{\Delta T_2} \times 100$$
 (6)

Where, ΔT_1 and ΔT_2 are temperature differences of upper part the mat at the initial and end of the experiment and temperature differences of upper and lower part of the mat at the initial, respectively.

Figure 3

3. Results and discussion

Aerogel formation

The SiO₂ aerogel was synthesized in the presence of waterglass as an available and non-toxic precursor with drying at atmospheric pressure. The synthesis method and the aerogel characteristics are shown in Figure 1 and Table 2, respectively. As can be seen from Table 2, it is clear that the porous aerogel powder was successfully synthesized with a surface area of 815 m²/g and a porosity of 91.8%. The aerogel nanostructure is shown in Figure 4. Also, the graphs in Figure 4 (C) and (D) show the meso-porosity and nano-pores distribution of the resulted aerogel, respectively.

Table 2

Figure 4

A mild ball-milling helps to maintain porous structure of the resulted aerogel granules. Figure 4 (B) shows that the milling process leads to formation of rather irregular micro-granules. This can be attributed to the mechanical properties of the monolithic aerogels which is difficult to obtain the spherical micro-granules [30]. The size distribution of the powder granules is shown in Figure 4 (E) with an average size of 6.6 μ m. Although Figure 5 (A) shows that the aerogel granules exhibit WCA usually associated with that of high hydrophobic gels modified by the TMCS hydrophobic agent, beyond inclination of 180°, the detachment of the water droplet from the aerogel surface, as shown in Figure 5 (B), did not occur. This could be attributed to the existence of the nano-voids and subsequent high water absorbency of the aerogel surface that is resemblance of gecko toes. As for gecko toes, the millions of setae can trap air inside the nano-scale structures forming strong adhesion and WCA [31].

Figure 5

Electro-spinning of the AMG/PNF compounds

Brittleness nature and dust releasing from the base substrates are considered to be the key remaining challenges in the aerogels applications [3]. Therefore, in this work, the challenges were tackled by compounding the aerogel powders with the PET electro-spinning solution at various concentrations. Since the integrity of the aerogel porous structure is dependent upon the intensity of the granules grinding, thus, the grinding was carried out so as to maintain micro-size powder granules. The microscopic and FTIR analysis show the successful fabrication of the bead-on-string structured PET nanofibers with diameter from 401 to 182.7 nm as shown in Figures 6 and 7.

Figure 6

In Figure 6, the peaks at 1000, 800, and 500 cm⁻¹ can be attributed to the asymmetric, symmetric, and bending modes of O–Si–O, respectively [32]. The similar peaks at around 1260 and 840 cm⁻¹ and also, 3000 and 1450 cm⁻¹ can be related to Si–C and C–H bonds, respectively [33]. An overlapping of some peaks at the above wavenumbers with the peaks in polyester, allows comparison of the peak intensity near 453 cm⁻¹, related to O–Si–O, for the pure and AMG-filled PET nanofibers. Also, the broadening of the peak at 1100 cm⁻¹ from the pure to the AMG-filled polyester is due to the presence of O–Si–O [34].

In conventional methods of fiber spinning, the material solution should be filtered. In order to prevent filament breakage during the fiber spinning, the pores of the filter media should be smaller than the diameter of a single fiber. These points to the fact that the particles added to the system must also have dimensions smaller than the diameter of the single fibers [35]. In this work, the micro-size beads were formed in the nanofibers due to the presence of AMG. The beads did not cause discontinuity and breakage up to concentration of 4%, as shown in Figure 7. Therefore, it can be deduced that the PET polymeric material coats the AMG with more diameter than the nanofibers. This can be regarded as a novel phenomenon in which not only porous regions are created in the nanofibers, but also beads formation provides a unique surface topography and new structural features in the AMG-filled PNFs which demands further research. The SEM micrographs in Figure 7, vividly confirm an acceptable distribution of the micro-granules in the AMG-filled PNFs. Figure 7 also points to existence of a direct relationship between the AMG concentration and the beads population.

Figure 7

The fibre diameter can be calculated from the SEM micrographs using the Digimizer software (Table 3). It was indicated that there is a decreasing trend in the diameter due to the presence of AMG. The diameter was reduced from 401 nm for PNF to 182.7 nm for 4AMG/PNF. To explain this effect, it is necessary to consider the physical aspects of the electrospinning in which, it can stated that during ejection of the drop from the syringe tip the micro-granules ejection lags behind the polymeric materials and as a result of strong electrical field on the drop, a tension is created between the micro-granules and the polymer which leads to thinning of the fibers without excessive breakage. The SPSS analysis showed no statistically significant differences in the diameters of PNF with 0.5AMG/PNF and 1AMG/PNF with 2AMG/PNF, all other factors were strongly significant.

Table 3

The histograms of diameter *versus* frequency, illustrated in Figure 8, show a normal distribution.

Figure 8

Also, the topological evaluation of the beads in the AMG-filled PNFs was considered. This is due to the fact that the aerogel micro-granules (AMG) are highly porous with vast number of nano-sized protuberances and cavities on their surface. Thus, the AFM images of the pure and AMG-filled samples ($5 \times 5 \mu$ m) were taken and are depicted with the respected roughness profiles in Figure 9.

Figure 9

Figure 9 (B) shows changes in the roughness from -20 to 20 nm on a randomly selected bead for the AMG-filled PNFs. The roughness change in the pure PNF, as shown in Figure 9 (A), was from -5 to 5 nm. Accordingly, it was revealed much higher surface roughness for the AMG-filled PNFs with Rms of 353, compared to the pure PNF with Rms of 179.9.

Following provides an explanation to the effect of the addition of the porous aerogel powder to the PET nanofibers in terms of surface structures, hydrophobicity, disperse dyeing, heat transfer, and sound absorption.

Hydrophobic properties

Use of aerogels on different substrates is greatly recognized for their insulation properties, but in this work, the effect of the aerogels addition on the polyester nanofibers with enhanced bulk and surface properties was investigated. The formation of beads as defect on the fiber surface is congenital. In contrast to

the previous researches, where the created beads had the same chemical composition as fibers, in the present work, the created beads are chemically different to the fibers and are formed by the inorganic and porous aerogel particles. These beaded nanofibers could be able to limit the aerogel dust-releasing because of the fact that the particles are embedded into the fibers. The successful embedding of AMG is extremely important for the industrial application with inhibition of the particles release and solving related health problems [36].

Beads can act as a secondary topological feature on the electrospun fibers surface which is able to create a proper surface roughness to enhance the hydrophobicity and superhydrophobic fibers [37]. Therefore, attempts have been made to create beads on the surface of the nanofibers by manipulation of electrospinning parameters [37-39].

The repellent characteristics of the untreated and fluoro-treated pure and AMG-filled PNFs are shown in Table 4.

Table 4

The untreated samples exhibited 3M water repellency of 1 *i.e.* these samples can just repel water. Irrespective of the amount of particles added to fibers, the treated samples due to low surface energy of the fluorochemicals have 3M water repellency of 9-10 [40, 41]. Therefore, the differences between both WCAs and SAs are due to the changes in the surface topology before and after the treatment with a fluorocarbon derivative [42, 43]. In addition, the AMG-filled samples showed higher values of WCA, compared to PNF. The WCA value was increased from 103.4° for PNF to 111.4° for the fluoro-treated PNF. Thus, in case of the pure PNF, the WCA parameter was not as large as the AMG-filled samples *i.e.* 123.8 and 135.4° as well as 130.1 and 147.2° for the untreated and the fluorocarbon-treated 0.5AMG/PNF and 2AMG/PNF samples, respectively.

Figure 10

It is noteworthy that prior to the treatment with a fluorocarbon derivative, the SAs remained unchanged at 90° for all pure and AMG-filled PNFs. The addition of AMG leads to an enhancement of the fiber surface porosity with strong adhesion similar to rose petals or gecko toes on which water droplets, due to air trapped inside the nano-scale structures, are firmly pinned to the surface but at the same time keep a large WCA [31]. This is important in cases like micro-liquid droplet transportation, protective coatings, nano patterning crystal fabrication, trace-liquid reactors, biochemical separation, and so on [44].

However, by increasing the AMG concentration over 1%, the SAs value of the fluoro-treated samples was sharply reduced due to the low surface energy couple with the created surface roughness. The maximum decrease in the SAs value occurred when the concentration of AMG was reached to 2%. This is in agreement with the previous study confirming that the SA is affected mainly by factors such as topography, chemical heterogeneity, and three phase contact line [43]. The superhydrophobic rough surfaces with low adhesion always exhibit a very low degree of contamination, which is what known as self-cleaning. Topography structure and chemical component results in these special adhesion states [31].

Therefore, the low adhesion superhydrophobic surface was obtained with the 2% concentration of AMG treated sample. This sample exhibited the highest WCA of 147.2° and 3M water repellency of 10 with the least SA equal to 5° *i.e.* less than 10°. These phenomena as shown in Figure 7 can be attributed firstly to congenital multi-scale surface roughness of the samples and also to the formation of the AMG beads.

Additionally, the AFM micrographs in Figure 9 point to roughness of the beads surface as the result of protrusions at nano-scale in comparison to the pure PET nanofibers. The importance of topological roughness hierarchy in relation to superhydrophobic properties and self-cleaning has also been reported in previous works [24].

A comparison of inherently smooth polyester film, PF, and PNF in terms of WCA can clarify the effect of electro-spun nanoweb structure on hydrophobicity. Table 4 shows while the SA value of the smooth PF, which is neither fibrous nor filled with AMG, remained unchanged, the WCA changes from 55.6 to 67.5° for the untreated and fluorocarbon-treated samples, respectively. Table 4 also shows the WCA values of 103.4 and 111.4° for the untreated and fluorocarbon-treated PNF, respectively. Considering the above mentioned results, it can be stated that the inherent topology of nanoweb is a key factor rendering the samples as hydrophobic. The SEM and AFM micrographs confirm that the proper surface irregularity occurs at concentration of 2%. This leads to an enhancement in the hydrophobicity of the AMG-filled PNFs, which is in agreement with the findings from previous studies [37-39, 45].

In order to determine the solid/air and water/air fractions of the hydrophobic sample at 2% concentration, the Cassie-Baxter equation (7) was used. The fractions in turn are indicative of the surface topology that affects the surface hydrophobicity. The Cassie-Baxter equation is developed based on assumption that, the droplet just remains on the top of surface protrusions and cannot reach the surface *i.e.* air packets are formed below water droplet [34, 42].

 $\cos \theta' = f_1 \cos \theta + f_2 \cos 180 = f_1 \cos \theta + f_1 - 1$ (7) Since

$$f_1 + f_2 = 1$$

.

(8)

Where, f_1 and f_2 are the solid/water and air/water fractions, $\hat{\theta}$ and θ represent the WCA of rough and smooth surfaces, respectively. The air/water fraction of 2AMG/PNF sample according to the values of the fluorocarbon-treated PF and 2AMG/PNF WCAs was found to be 88.6%. The air/water fraction of 88.6% clearly indicates the entrapment of considerable amount of air in the surface of the AMG-filled PNFs which increases the sample hydrophobicity and facilitates the water droplets movement.

In order to justify the effect of porosity of AMG on the hydrophobicity of the AMG-filled fibers, the non-porous particles, SMG, at 4% concentration were added to the PET nanofibers. The obtained results (Table 3) indicate the superior hydrophobicity of the AMG-filled samples to the SMG one.

Dyeing properties

In order to evaluate the dyeing behavior of the AMG-filled PNFs, the dyeing process of the pure and AMG-filled nanofiber samples were carried out in 300 min at 90°C. The absorbance of the wastewater samples was determined at 650 nm after 0, 20, 60, 90, 120, 180, and 300 min. The absorbance of the disperse dye of the screen composed of the conventional PET fibers was measured separately under the same condition. This screen was used to prevent shrinkage of the nanofibers during the dyeing process. Figure 11 (A) shows the light staining of the screen with a grey scale of less than 1/2 which indicates that its dye adsorption is practically negligible. Figure 11 (B) confirms a high level of dyeing evenness of the AMG-filled PNFs.

Figure 11

The absorbance of the wastewater samples and the exhaustion percentages, from 0 to 300 min of dyeing, are shown in Figure 12. The compounding of the electro-spinning solution has resulted in an increase in the nanofibers dye up-take and subsequent reduction of the wastewater absorbance. The formation of the porous beads due to compounding has caused changes in the morphology, chemical and physical properties of the nanofibers. The enhanced porosity provides more available sites for the adsorption of the dye molecules. It was reported that a high specific pore volume of the aerogels renders them advantageous agent to entrap, adsorb or release a large variety of molecules [46, 47]. Additionally, the O-CH₃ functional groups in the embedded AMG in the nanofibers can be responsible for more interactions between the dye molecules and the porous area which is vividly confirmed by the AFM micrographs shown in Figure 9. The increase in the dye up-take can also be due to increase in the specific surface area of the AMG-filled fibers due to the reduction of the nanofibers diameter (Table 3). After the initial impregnation of the nanofibers by the dye molecules, the penetration of the dye molecule into the fibers occurs. This followed by a fixation stage at a higher temperature [48].

Figure 12 also shows approximately two-folded increase in the dyeing rate of the AMG-filled sample for the first 20 min of the dyeing process as compared with the pure PNF.

Figure 12

Furthermore, it can be stated that the compounding of the PET nanofibers not only reduces the dyeing time, but also helps in achieving better equilibrium exhaustion. A distinctive dyeing performance was observed at the AMG concentration of 4%. This was achieved within the first 20 min of the dyeing operation with 35% exhaustion yield and increased to about 70% at the equilibrium point, approximately twofold better than the pure PNF.

Statistically, no significant difference was observed between 4AMG/PNF with 2AMG/PNF, and 2AMG/PNF with 1AMG/PNF. However, a significant statistical difference between 0.5AMG/PNF and 4AMG/PNF was observed. This finding may assist development of the conventional PET fibers with similar dveing behaviour as the AMG-filled PNFs. Because there have been many attempts to modify the dyeing process of the PET fibers like easy disperse dyeability under normal pressure as well as cationic and pigment dyeability [49]. In order to justify the effect of porosity on the dyeing behavior of the AMG-filled fibers, the non-porous particles, SMG, were used to compound PET nanofibers at 4% concentration. The SMG-filled nanofibers were dyed in the exactly same manner as described above for the AMG-filled fibers. As shown in Figure 12, the results clearly indicate a remarkable decrease in the dye up-take of the nonporous SMG.

Insulation properties

Insulations are tribute of modern structures such as buildings, machines, and industrial processes. Thus variety of materials can be incorporated in these structures to achieve the desired level of insulations. Insulation is among salient properties of aerogels [47,50]. Global CO_2 emission concern together with scarcity of conventional energy resources and space saving restrictions have emphasized the necessity of research on insulation in general and thermal insulators in particular [3]. Fibrous materials such as the AMG-filled samples of this work are textile structures that provide various insulations. The heat transfer and sound insulation of the AMG-filled PNFs at 0.5

and 4% concentrations together with the pure and 4SMG/PNF with 4% silica micro-granules are illustrated in Table 5. As shown, the calculated 20.9% heat transfer for PNF is significantly more than the heat transfer of 16.4% and 8.3% calculated for 0.5AMG/PNF and 4AMG/PNF, respectively. Therefore, the results indicate the existence of an indirect relation between the heat transfer coefficient and the concentration of AMG in the PET solution. The SPSS statistical analysis of the data is significant at the 0.05 level. The reduction in the heat transfer of the samples can be attributed to embedding of the porous AMG inside the nanofibers [51]. In term of PNF and 0.5 AMG/PNF, since, their physical properties more or less are similar, lower heat transfer of 0.5AMG/PNF strongly points to existence of the porous particles in these nanofibers, because low thermal conductivity is an important character for silica aerogels. This together with nonflammability renders silica aerogels among the most preferred thermal insulating materials [52].

Table 5

Table 5 shows that despite of an insignificant difference in the thickness of the AMG-filled samples with 0.5 and 4.0% AMG, the latter has slightly lower mass/unit area. This can be due to thinning of the nanofibers as the result of the addition of AMG leading to the reduction of the AMG-filled PNFs density. Thermal conductivity of less dense materials is lower than their dense counterparts [53]. Heat flow in porous materials obeys conduction mechanism within the solid part, convection through gases phase, and radiation mechanism [3, 51 and 52]. Due to the high fineness of the fibers which leads to formation of smaller voids (Figure 7), the heat transfer of 4AMG/PNF is low [3].

A comparison of the 4AMG/PNF and 4SMG/PNF samples simply demonstrates the effect of the particle porosity on the heat flow through these samples. The microparticles added to the latter had no porosity while the added microparticles to the former as already mentioned were quite porous. This structural difference is responsible for the lower heat transfer of 4AMG/PNF over 4SMG/PNF samples. No statistical difference between 4SMG/PNF and 0.5AMG/PNF reveals that the same heat transfer can be achieved with a lower concentration of the porous AMG, instead of a higher concentration of the nonporous particles. Due to their high porosity, fibrous structures have been extensively used as acoustic barriers with various functions in miscellaneous applications. Fibrous structures simultaneously can absorb, reflect and transmit the incident sound waves. This ability together with low-cost and ease of use has made textiles as one of the most effective sound proofing materials, Moreover, the physical properties of the constituent fibers of a fibrous structure significantly affect the sound absorption properties of the fibrous material [54].

As shown in Table 5, due to the addition of AMG to the PET solution, the sound absorption coefficient of the samples was generally enhanced in frequency range of 250 to 4000 Hz. The increase in NAC for 0.5AMG/PNF at frequencies of 250 and 500 Hz is more than NAC of 4AMG/PNF sample at the same frequencies. The opposite trend was observed at frequencies of 1000 and 2000 Hz with higher NAC for 4AMG/PNF, as compared with 0.5AMG/PNF. At frequency of 4000 Hz, NAC of both samples are statistically similar. These phenomena can be related to changes in the nanofiber structure as a result of the AMG addition. Because the AMG-filled fibers vibrate due to impinging of sound waves, void geometry, surface topology, fineness, and density [55].

Figure 13 (A) shows larger voids in 0.5AMG/PNF structure. At low frequencies, the sound waves are reflected by the voids, while at high frequencies, the sound waves are transmitted through the voids. Due to the smaller voids of 4AMG/PNF, this sample reduces the high-frequency sound. These findings are in agreement with other studies [54 and 56].

A quantitative measurement of the formed pores between the nanofibers was done using image processing by the Matlab software. The images were turned into a binary (black and white) image based on a lighting threshold (T) value. The T value was achieved from lighting histogram of the samples in a loop in which T firstly is concerned the half of the lighting histogram [57]. According to the results, the mean value of the pores size for the pure fibrous material was 4154.7 nm, while the samples containing 0.5 and 4% aerogel showed the mean values of 8625.3 and 3598.46 nm, respectively. Also, as shown in micrographs 7 and 9, the changes in the surface topology of the fibers can be one of the reasons behind the improved sound absorption of the AMG-filled samples. Additionally, the increase in the fineness and subsequent increase in the specific surface area of the fibers in 4AMG/PNF sample may be responsible for the enhanced acoustic performance of this sample [58, 59].

4. Conclusion

Demands for advanced aerogel-based materials with precise functional performance materials have steadily been expanded. Thus, in this research, the application potential of novel AMGfilled PET fibers was investigated. The AMG-filled PNFs have low dust-releasing behaviour, high hydrophobicity, high dyeability, and high insulation properties. A successful synthesis of the bead-on-string AMG-filled PET nanofibers using aerogel content up to 4% concentration was achieved. The AMG-filled PNFs were subjected to the fluorocarbon treatment. The treated fibers not only had a low surface energy but also due to changes in their surface topology were highly superhydrophobic. For the 2% AMG-filled PNFs, the values of different factors such as WCA, SA, and 3M water repellency were found to be 147.2°, 10 and 5°, respectively. For the first time, the effect of the aerogels on the fibers dying process was evaluated. It was also found that the embedding of the AMG into the PET nanofibers leads to a novel low-temperature disperse dyeing with shorter dyeing process time and increase of the equilibrium up-take using disperse colorants. The highest level of equilibrium exhaustion approximately 70% was obtained with the nanofibers containing 4% AMG. A decreasing trend in the heat transfer percentage in proportion to the concentration of AMG in the samples was observed. The sample at 4% concentration exhibited the lowest heat transfer. This trend was followed by the sample at 0.5% AMG concentration and then the pure PET nanofibers. It was concluded that the addition of AMG to the spinning solution of PET increases the sound absorption coefficient NAC of the nanofibers within frequency range of 250 to 4000 Hz. In comparison to 4AMG/PNF samples, the nanofibers spun with 0.5AMG/PNF solution exhibited higher NAC at low frequencies of 250 and 500 Hz. On the other hand, the NACs at frequencies of 1000 and 2000 Hz for the 4AMG/PNF were found to be higher than those of the 0.5AMG/PNF. The importance of the porosity on the examined factors was established by running the same experiments on the non-porous silica micro-granules. Therefore, the low-weight aerogel embedded nanofiber materials with absorbing and hydrophobic characteristics can be one of the most important materials for

sound insulation and absorption. Finally, it was concluded that the findings of this research potentially can be extended to conventional polyester fibers with added properties such as enhanced porosity, ease of dyeing, superhydrophobicity, and insulation abilities. Thus, future studies with more experimental data are needed to assess the effects of aerogel embedded fibers on their varying properties.

5. Acknowledgement

Financial support of the Isfahan University of Technology (IUT) is gratefully appreciated. Mr Ali Karimi is most sincerely acknowledged for his exceptional skill in spinning of the nanofibers. Assistance of Mr. Digambar Nadargi of EMPA is highly appreciated during aerogel preparation.

Notes and references

^a Department of Textile Engineering, Isfahan University of Technology, Isfahan 84156-83111, Iran

^b Department of Chemistry, Isfahan University of Technology, Isfahan 84156-83111, Iran

- 1. R. A. Venkateswara, R. A. Parvathy and M. M. Kulkarni, J. Non-Cryst. Solids, 2004, 350, 224–229.
- S.W. Hwang, H. H. Jung, S. H. Hyun and Y. S. Ahn, J. Sol-Gel Sci. Technol., 41, 2007, 139–146.
- 3. M. A. Aegerter, N. Leventis, M. M. Koebel, Aerogels Handbook, Springer, New York, 2012.
- F. J. Heiligtag, M. J. I. Airaghi Leccardi, D. Erdem, M. J. Süess and M. Niederberger, *Nanoscale*, 2014,6, 13213-13221
- 5. A. P. Rao, A. V. Rao, G. Pajonk and P. M. Shewale, *J. Mater. Sci.*, 2007, 42(20), 8418–8425.
- P. H. Tewari, A. J. Hunt and K. Lofftus, *Mater. Lett.*, 1985, 3(9), 363–367.
- A. M. Anderson, C. W. Wattley and M. K. Carroll, J. Non-Cryst. Solids, 2009, 355(2), 101–108.
- A. P. Rao, A. V. Rao and U. K. H. Bangi, J. Sol-Gel Sci. Technol., 2008, 47, 85–94.
- S. W. Hwang, T. Y. Kim and S. H. Hyun, *Micropor. Mesopor. Mat.*, 2010, 130:295–302.
- Y. Liao, H. Wu, Y. Ding, S. Yin, M. Wang, A. Cao, J. Sol-Gel Sci. Technol., 2012, 63, 3, 445-456
- 11. J. Chandradass, S. Kang and D. S. Bae, J. Non-Cryst. Solids, 2008, 354, 4115–4119.
- 12. Y. WO Pat. 2007146945, 2007.
- 13. R. Trifu, N. Bhobho US Pat. 2007173157R, 2007.
- Zh. M. Huang, Y. Z. Zhang, M. Kotaki and S. Ramakrishna, Compos. Sci. Technol., 2003, 63, 2223–2253.
- 15. H. Wu, X. Zhou, Y. Chen, Q. Chen, V. Ding and H. Gao, J. Nanomater., 2013, 2013, 1-8.
- 16. D. Li, Y. Xia, Adv. Mater., 2004, 16(14), 1151-1170.
- 17. X. Meng, J. Polym. Sci., 2006, 44, 2468-2474.
- 18. M. Qian, B. Mao, P. Cebe, J. Therm. Anal. Calorim., 2012, 1-7.
- 19. V. Prevolnik, P. K. Zrim, T. Rijavec, Contemporary Materials, 2014, 1, 117-123
- A. Du, B. Zhou, Y. Li, X. Li, J. Ye, L. Li, Z. Zhang, G. Gao J. Shen, J. Reinf. Plast. Compos., 2011, 30(11), 912–921.
- 21. A. Du, B. Zhou, Z. Zhang, J. Shen, *Materials*, 2013, 6, 941-968.
- N. K. On, A. A. Rashid, M. M. M. Nazlan, H. Hamdan, J. Appl. Polym. Sci., 2012, 124, 3108–3116.
- 23. T. Lours, J. Zarzycki, A. Craiewich, D. Dos Santos and M. Aegerter, J. Non-Cryst. Solids, 1987, 95&96, 1151–1158
- 24. Z. Mazrouei-Sebdani, A. Khoddami, J. Prog. Org. Coat., 2011, 72, 638-646.
- 25. M. Ferus-Comelo, *Color. Technol.*, 2009, 125(6), 352–356.
- M. F. Comelo, M. Clark and P. Parker, Color. Technol., 2005, 121, 5, 255–257.
- 27. S. Hassanzadeha, H. Hasani and M. Zarrebini, J. Text. I., 2014, 105, 3, 256–263.

Page 8 of 28

- S. Borhani, S. Seirafianpour and S. A. Ravandi, J. Eng. Fiber Fabr., 2010, 5, 42-8.
- G. Song, P. Chitrphiromsri and D. Ding, *JOSE*, 2008, 14(1), 89– 106.
- C. Prahsarn, R. L. Barker, B. S. Gupta, *Text. Res. J.*, 2005, 75, 346–351.
- 31. M. Alnaief and I. Smirnova, TU Hamburg Harburg, Institut für Thermische Verfahrenstechnik.
- Y. K. Lai1, Z. Chen and Ch. J. Lin, *JNAN*, 2011, 1, 18–34.
 A. V. Rao, R. R. Kalesh, G. M. Pajonk, *J. Mater. Sci.*, 2003, 38,
- 4407–4413. 34. A. P. Rao, A. V. Rao and G. M. Pajonk , *Appl. Surf. Sci.*, 2007,
- 253, 6032–6040.
 35. J. Lin, Y. Cai, X. Wang, B. Ding, J. Yu and M. Wang, *Nanoscale*, 2011, 3, 1258–1262
- F. Fourne, Synthetic Fibers: Machines and Equipment, Manufacture, Properties, Hanser Publishers, Munich, 1999.
- 37. Y. Liu, X. Chen and J. H. Xin, Bioinspir. Biomim., 2008, 3, 1.
- N. Nuraje, W. S. Khan, Y. Lei, M. Ceylanb and R. Asmatulu, J. Mater. Chem. A, 2013, 1, 1929-1946.
- 39. J. Fang, H. Wang, X. Wang and T. Lin, J. Text. I., 2012, 103(9), 937–944.
- X. Wanga, B. Dinga, J. Yub and M. Wang, *Nano Today*, 2011, 6, 510—530.
- 41. A. J., Hall, London, Heywood Books, 1966.
- T. Nishino, M. Meguro, K. Nakamae, M. Matsushita and Y. Ueda, Langmuir, 1999, 15, 4321-4323.
- 43. M. Wolfs, T. Darmanin and F. Guittard, *Polym. Rev.*, 2013, 53, 460–505.
- 44. W. Barthlott, and C. neinhuis, planta, 1997, 202(1), 1-8.
- 45. Q. Yu, Z. Zeng, W. Zhao, M. Li, X. Wu, Q. Xue, Colloid. Surf. A: Physicochem. Eng. Aspects, 2013, 427, 1–6.
- 46. Z. Guo, W. Liu and B. L. Su, J. Colloid. Interf. Sci., 2011, 353, 335–355.

- 47. M. Schmidt, F. Schwertfeger, J Non-Cryst. Solids, 1998, 225, 364–368.
- J. Fricke and A. Emmerling, J. Sol-Gel Sci. Tech., 1998, 13, 299– 303.
- 49. J. R. Aspland, Text. Chem. Col. 1992, 24(12), 18-23.
- W. U. Rong-rui, Development of Technology of PET Fiber Modification in China, Chinese Polymer Bulletin, chaina, 2008, 08.
- M. Venkataraman, R. Mishra, J. Wiener, J. Militky, T.M. Kotresh and M. Vaclavik, J. Text. I., 2014, DOI: 10.1080/00405000.2014.939808.
- 52. L. W. Hrubesh and R. W. Pekala, J. Mater. Res., 1994, 9(3), 731-738.
- 53. B. E. Yoldas, M. J. Annen and J. Bostaph, *Chem. Mater.*, 2000, 12, 2475–2484.
- 54. T. Y. Wei, S. Y. Lu and Y. Ch. Chang, J. Phys. Chem. C, 2009, 113, 7424–7428.
- 55. M. Tascan, Ph.D. thesis, Clemson University, 2005.
- H. Bahrambeygi, N. Sabetzadeh, A. Rabbi, K. Nasouri, A. Mousavi Shoushtari and M. R. Babaei, *J. Polym. Res.*, 2013, 20, 72-82.
- 57. Parvane Sadeghi, Master of Science thesis, Isfahan University of Technology, 2013.
- K. C. Vengala, Bachelor thesis, Jawaharlal Nehru Technology University, 2007.
- J. Gross, J. Fricke, L. W. Hrubesh, J. Acoust .Soc. Am., 1992, 91, 2004–2006.

Page 9 of 28

Journal Name

RSC Advances

Caption for figures

Figure1. Aerogel production and compounding with the PET nanofibers

Figure 2 Schematic design for the impedance tube

Figure 3 Schematic design for the instrument to measure heat transfer

Figure 4 (A and B) SEM micrographs: (A) aerogel nanostructure with nanovoids, (B) aerogel powder particle size and shape, (C) N2 adsorption/desorption isotherm, and (D) pore size distribution of the aerogel

Figure 5 Sessile water droplets on: (A) hydrophobic aerogel, and (B) inverted aerogel at 180°.

Figure 6 FTIR spectra: (A) PNF, (B) 2AMG/PNF, and (C) 4AMG/PNF

Figure 7 SEM micrographs: (A) PNF, (B) 0.5AMG/PNF, (C) 1AMG/PNF, (D) 2AMG/PNF, (E) 4AMG/PNF, and (F) 4SMG/PNF (inset: higher magnification).

Figure 8 Histograms of diameter versus frequencies: (A) PNF, and (B) 4AMG/PNF

Figure9 AFM micrographs of the surface topology and roughness profiles along the marked line: (A) PNF, (B and C) 4AMG/PNF.

Figure 10 Sessile water droplet on (A) fluoro-treated PF, (B) fluoro-treated PNF, (C) 2AMG/PNF, and (D) fluoro-treated 2AMG/PNF.

Figure 11 (A) Light staining of the screen, and (B) high dyeing evenness

Figure 12 (A) The absorbance of the wastewater samples at 650 nm, and (B) the exhaustion (%) of the pure and AMG-filled nanofibers

Caption for tables Table 1 Name and abbreviation of the samples

Table 2 Aerogel characteristics

Table 3 Mean diameter of the pure and AMG-filled PET nanofibers

Table 4 Repellency of the untreated and the fluorocarbon-treated pure and AMG-filled PET nanofibers

Table 5 Thermal and acoustic behaviour of the pure and AMG-filled PET nanofibers



Graphical abstract



Figure1. Aerogel production and compounding with the PET nanofibers



Figure 2 Schematic design for the impedance tube



Figure 3 Schematic design for the instrument to measure heat transfer

ARTICLE



Figure 4 (A and B) SEM micrographs: (A) aerogel nanostructure with nanovoids, (B) aerogel powder particle size and shape, (C) N₂ adsorption/desorption isotherm, (D) pore size distribution of the aerogel, and (E) size distribution of the powder granules



Figure 5 Sessile water droplets on: (A) hydrophobic aerogel, and (B) inverted aerogel at 180°.



Figure 6 FTIR spectra: (A) PNF, (B) 2AMG/PNF, and (C) 4AMG/PNF



Figure 7 SEM micrographs: (A) PNF, (B) 0.5AMG/PNF, (C) 1AMG/PNF, (D) 2AMG/PNF, (E) 4AMG/PNF, and (F) 4SMG/PNF (inset: higher magnification).



Figure 8 Histograms of diameter versus frequencies: (A) PNF, and (B) 4AMG/PNF



Figure9 AFM micrographs of the surface topology and roughness profiles along the marked line: (A) PNF, and (B) 4AMG/PNF.



Figure 10 Sessile water droplets on (A) fluoro-treated PF, (B) fluoro-treated PNF, (C) 2AMG/PNF, and (D) fluoro-treated 2AMG/PNF.





Figure 11 (A) Light staining of the screen, and (B) high dyeing evenness

ARTICLE





Figure 12 (A) The absorbance of the wastewater samples at 650 nm, and (B) the exhaustion (%) of the pure and AMG-filled nanofibers

Table 1 Name and abbreviation of the samples

Sample	Concentration	Abbreviation		
	SMG	AMG		
PET film	-	-	PF	
	-	-	PNF	
	4	-	4SMG/PNF	
PET nanofibers	-	0.5	0.5AMG/PNF	
	-	1	1AMG/PNF	
	-	2	2AMG/PNF	
	-	4	4AMG/PNF	

Table 2 Aerogel	characteristics
-----------------	-----------------

Density (g/cm ³)	Total Pore volume (m ³ /g)	Mean poreSurface areadiameter (nm)(m²/g)		Porosity (%)	WCA (°)	
0.18 (0.02)*	3.5310	23.928	815.14	91.8 (0.9)	146.4 (0.8)	

*The number in parenthesis is the standard deviation

Table 3 Mean diameter of the pure and AMG-filled PET nanofibers

sample	PNF	4SMG/PNF	0.5AMG/PNF	1AMG/PNF	2AMG/PNF	4AMG/PNF
Diameter (nm)	401.0 (84.3)*	405.9 (98.7)	392.5 (104.1)	268.6 (79.9)	263.9 (73.2)	182.7 (55.7)

*The number in parenthesis is the standard deviation

Table 4 Repellency of the untreated and the fluorocarbon-treated pure and AMG-filled PET nanofibers

	WC	'A (°)	SA	. (°)	3M water repellency		
Sample	BFC^{*}	AFC**	BFC	AFC	BFC	AFC	
PF	55.6 (0.1)***	67.5 (0.1)	>90	>90	1	9-10	
PNF	103.4 (0.2)	111.4 (0.6)	>90	>90	1	9-10	
4SMG/PNF	110.9 (2.9)	127.1 (0.8)	>90	>90	1	10	
0.5AMG/PNF	123.8 (0.9)	135.4 (0.9)	>90	>90	1	10	
1AMG/PNF	121.9 (1.2)	135.6 (0.5)	>90	55	1	10	
2AMG/PNF	130.1 (1.0)	147.2 (0.3)	>90	5	1	10	
4AMG/PNF	126.1 (0.8)	132.0 (0.4)	>90	10	1	10	

*Before the fluorocarbon coating: BFC and **after the fluorocarbon coating: AFC

*** The number in parenthesis is the standard deviation

Table 5 Thermal and acoustic behaviour of the pure and AMG-filled PET nanofibers

sample	Heat transfer (%)	Frequency (Hz)				Thickness	Mass per unit area [*]	Density	
		250	500	1000	2000	4000	(mm)	(g/cm^2)	(g/cm)
PNF	20.9 (0.14)**	0.3225 (0.0001)	0.2883 (0.0002)	0.4329 (0.0006)	0.3906 (0.0021)	0.5724 (0.0010)	0.18 (0.012)	0.0089 (0.001)	0.047 (0.03)
0.5AMG/PNF	16.4 (0.9)	0.5258 (0.0012)	0.4909 (0.0005)	0.4700 (0.0005)	0.6995 (0.0003)	0.6805 (0.0001)	0.17 (0.010)	0.0097 (0.003)	0.052 (0.04)
4AMG/PNF	8.3 (0.14)	0.3527 (0.0008)	0.4018 (0.0002)	0.5116 (0.0012)	0.7575 (0.0003)	0.6505 (0.0002)	0.18 (0.010)	0.0062 (0.002)	0.036 (0.03)
4SMG/PNF	15.0 (1.83)	0.2226 (0.0031)	0.1718 (0.0022)	0.3950 (0.0001)	0.3711 (0.0012)	0.4773 (0.0011)	0.24 (0.014)	0.0094 (0.002)	0.041 (0.02)

The mass per cubic centimeter of the screen was 0.007 ± 0.001 g/cm² and its thickness was $0.11 \pm .01$ mm

**The number in parenthesis is the standard deviation