

Electrospinning of Styrene-Ethyl Acrylate Emulsion Copolymers: Exploring the Impact of Polymer Polarity and Glass Transition Temperature on Fiber Formation and Hydrophobicity

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ABSTRACT: In this study the spinnability of styrene-ethyl acrylate copolymers prepared via emulsion polymerization was explored with the aim to establish the influence of glass transition temperature and polymer composition on morphological features of electrospun materials with regard to their hydrophobic/hydrophilic nature. Nonwoven mats from polymer solutions in *N,N'*-dimethylformamide were spun using the electrospinning method (NanospiderTM device). It was demonstrated that changing the copolymer polarity and glass transition temperature by copolymer composition or utilizing different supporting materials might lead to nanofiber webs with controlled hydrophobic/hydrophilic properties, which

predetermines them for a wide range of applications such as protective textiles, composites etc.

KEY WORDS: Electrospinning, Nanofiber, Latex, Emulsion Polymerization, Glass Transition Temperature

Introduction

Electrospinning method, in which a polymer solution or a polymer melt is extended by electrostatic force, is one of the techniques recently studied frequently. It has a long history, the first patent was applied in 1934 [1], and the production of ultra-fine fiber of less than 1 μm was reported in 1971 [2]. With the increase of ultra-fine fibers' demand, it attracts attention again as a technique of making ultrathin fibers from various materials. Recently, the nozzle-less technology opened the possibility to produce nanofiber layers in mass industrial scale. Nozzle-less electrospinning is featured as electrospinning of nanofibers directly from an open liquid surface. Numerous jets are formed simultaneously from the needleless fiber generator without the influence of capillary effect that is normally associated with needle-like nozzles. This method that introduces the formation of many Taylor cones from a free liquid surface was patented by Jirsak et al and the machines called Nanospider are developed by the ElMarco Company (Liberec, Czech Republic) [3].

Electrospun polymer nanofibers have a diameter in the order of a few nanometers to over 1 micrometer (more typically 50 ~ 500 nm) and possess unique characteristics, such as an extraordinarily high surface area to volume ratio coupled with excellent structural mechanical properties, high axial strength combined with extreme flexibility, low basic weight, and cost effectiveness, among others. Due to these characteristics, electrospun

nonwoven mats have potential applications in such areas as filters [4,5], sensor devices, protective cloths [6], composites [7] and for medical purposes [8,9].

Various polymers have been successfully electrospun into ultrafine fibers in recent years, mostly in solvent solution and some in melt form. Nevertheless, it becomes evident from the literature survey that no studies on the electrospinning of emulsion polymers have been published so far. Nowadays, the issues regarding environmental protection cause that technologies reducing or eliminating the application of organic solvents are appreciated. Therefore, waterborne polymers prepared via emulsion polymerization are of particular interest. Among the emulsion polymers, styrene/acrylic latexes play an important role due to their extraordinary properties including ease of modification, pigmentation and application as well as outstanding chemical resistance, resistance to atmospheric conditions and diversified mechanical features. By emulsion polymerization technique polymers can be prepared to meet the requirements of specific applications by variation of monomer composition, molar mass, cross-linking density, particle size or nature of surface charge. The most distinctive feature of emulsion polymerization refers to the fact that the radicals are distributed among different particles, thus losing the chance of terminating between them. This allows the simultaneous achievement of high polymerization rates and high molecular weights [10].

Most of the recent work on electrospinning has focused on trying to understand the fundamental aspects of the process in order to gain control of fiber formation, shape, texture and morphology. In fact, the principal parameters and variables that affect fiber diameter and structure are as follows [11,12,13,14,15]: 1) system parameters such as the molecular weight, molecular weight distribution and architecture (branched, linear, etc.) of the polymer and polymer solution properties (viscosity, electrical conductivity, dielectric constant, and surface tension, charge carried by the spinning jet) and 2) process parameters such as the applied voltage, flow rate, the distance between two electrodes, ambient parameters (temperature,

humidity and air velocity in the electrospinning chamber) and finally the motion of the target screen (of the grounded electrode) and type of the grounded electrode [16,17,18,19,20].

In the past, the effects of various parameters, particularly viscosity, surface tension and molecular weight, on fiber diameter have been investigated [21,22]. Polymer solution viscosity (controlled by changing the polymer concentration) has been found to be one of the biggest determiners of fiber size and morphology when spinning polymeric fibers [14,15,23,24,25]. Large viscosities are tied to the entanglement of the polymer chains. Obviously, longer chains of higher molecular weight and a higher concentration of chains favor more entanglement, and therefore larger viscosity. More viscous polymer solutions require larger electrostatic forces to be spun. Demir [15] has demonstrated that as the viscosity of polyurethaneurea increased via increased concentration, critical voltage increased proportionally. Increased viscosity has also a role in fiber diameter. Viscous solutions are cohesively strong, thus providing large resistance to the electrostatic forces drawing the fiber through the spinneret. As a result, larger viscosities result in larger fiber diameters [21].

The molecular weight of the polymer has a significant effect on rheological and electrical properties of the polymer solution, e.g. viscosity, surface tension, conductivity and dielectric strength. It has been observed that a too low molecular weight solution tends to form beads rather than fibers and a high molecular weight solution gives fibers with large average diameter. The molecular weight of the polymer reflects the number of entanglements of polymer chains in a solution, thus solution viscosity. Chain entanglement plays an important role in the processing of electrospinning [3,26]. Gupta et al. [27] have synthesized PMMA varying in molecular weight to investigate the effect of molecular weight of the polymer. They found that as the molecular weight increased, the number of beads and droplets decreased. However, the investigations of the dependence of fiber formation,

morphology and structure on polarity and glass transition temperature of the electrospun polymer are lacking.

The main goal of this work was to compare the spinnability of styrene-ethyl acrylate emulsion copolymers from the point of polymer polarity and glass transition temperature and to find the most preferable copolymer composition for the electrospinning procedure. Nonwoven ultra-fine fiber layers from polymer solutions in *N,N'*-dimethylformamide were collected on PP supporting material using the NanospiderTM device. Electrospinning of all polymer solutions was carried out at identical viscosity to neglect the effects of concentration, molecular weight, molecular weight distribution and topology. Electrospun fibers were characterized by measurements of fiber radius and water contact angle.

Experimental

MATERIALS

Emulsion copolymers investigated in this research work were synthesized of styrene, ethyl acrylate (EA), and acrylic acid (AA). All the monomers were purchased from Roehm (Germany). Disponil FES 993 IS (BASF, Czech Republic) was used as a surfactant and ammonium persulfate (Lach-Ner Company, Czech Republic) was utilized as an initiator of the polymerization reaction. *N,N'*-dimethylformamide (Sigma-Aldrich, Czech Republic) was used as the solvent for the preparation of polymer solutions. Ultra-fine fibers were collected on spunbond from polypropylene (PP) filaments ($Q = 30 \text{ g/m}^2$), a fine polyamide lattice ($Q = 80 \text{ g/m}^2$), and a rough polyamide lattice ($Q = 75 \text{ g/m}^2$), respectively. Macro-scale views of the supporting materials are provided in Fig. 1.

PREPARATION OF EMULSION COPOLYMERS

A series of styrene-ethyl acrylate emulsion copolymers was synthesized via emulsion polymerization. To improve the stability of water dispersions, carboxyl functionalities were introduced by copolymerization with a constant amount (2 wt. %) of acrylic acid into all the prepared copolymers. The contents of EA comonomer were 0, 10, 25, 50, 65, 75, 88, and 98 wt. %. That means the ethyl acrylate homopolymer containing only 2 wt. % of AA was prepared as well. Note: to simplify the sample notation, AA content is not mentioned in further text and all the percentages are % by weight.

All emulsion copolymers were prepared in a 2500 mL glass reactor by semi-continuous non-seeded emulsion polymerization under nitrogen atmosphere at 85 °C. This procedure should ensure nearly homogeneous statistical copolymers. The reactor charge (99 g water, 1.8 g Disponil FES 993 IS, and 0.5 g ammonium persulphate) was put into the reactor and heated to the polymerization temperature. Then the monomer emulsion (185 g water, 19 g Disponil FES 993 IS, 1 g ammonium persulphate, 200 g monomer mixture) was fed into the stirred reactor during 3 hours (at feeding rate of about 10 mL/min). After that, during 2 hours of hold period the polymerization was completed and the aqueous polymeric dispersion was cooled to room temperature.

PREPARATION OF COPOLYMER SOLUTIONS

The copolymers were isolated from aqueous dispersions by drying at ambient temperature in air for 10 days. The copolymer solutions were prepared by dissolving the isolated copolymers in *N,N'*-dimethylformamide (DMF) and mixing by a magnetic stirrer for 4 hours at ambient temperature. All the copolymer solutions were prepared at the identical viscosity of 200 ± 15 mPa.s to neglect the effects of concentration, molecular weight, molecular weight distribution, and topology during the electrospinning process. The viscosity

of the solutions was measured by a viscosimeter Brookfield LVDV-III+ (Brookfield Engineering Laboratories, Inc., US) at $25 \pm 0,1$ °C.

ELECTROSPINNING TECHNIQUE

The supporting materials were covered by a layer of styrene-ethyl acrylate ultra-fine fibers using a NanospiderTM NSLAB 500 (Elmarco, Czech Republic) laboratory machine [28]. A working scheme of the device is presented in Fig. 2. While rotating, the cylindrical electrode is covered by a film of polymer solution. By increasing the applied voltage, Taylor cones are formed. Only when an electrostatic force overcomes the surface tension of the polymer is a jet of polymer solution ejected from the Taylor cone. The jet moves toward the upper electrode and sets down on the substratum material. Meanwhile the nanofiber becomes thinner, the solvent evaporates and later solidifies [19]. During the experiments with styrene-ethyl acrylate copolymer solutions, the distance between electrodes was 20 cm, the applied voltage varied between 21 and 25 kV; the temperature of electrospinning environment was 25 ± 1 °C and the air humidity 45 ± 2 %.

CHARACTERIZATION METHODS

The glass transition temperatures (T_g) of the styrene-ethyl acrylate copolymers were determined by means of differential scanning calorimetry using a Pyris 1 DSC instrument (Perkin-Elmer, USA). The measurements were carried out on the isolated and vacuum-dried copolymers at the heating rate of 10 °C/min. Post-spinning characterization of the morphology of electrospun webs collected on various supporting materials was done using a Tescan MIRA 3 scanning electron microscope (Tescan, Czech Republic). The diameter of ultra-fine fibers was measured using ImageJ software [29]. The contact angles of the electrospun webs with water were measured by a Surface Energy Evaluation System optical

contact angle measuring device. All the fiber diameter and water contact angle measurements were carried out ten times for each type of electrospun web to check the reproducibility.

Results and Discussion

EFFECT OF POLYMER POLARITY AND GLASS TRANSITION TEMPERATURE

The spinnability of styrene-ethyl acrylate emulsion copolymers from the point of polymer polarity and glass transition temperature was compared and the most preferable copolymer composition for the electrospinning procedure performed under one set of conditions using the NanospiderTM device was investigated. The SEM micrographs of styrene-ethyl acrylate ultra-fine fiber nonwovens collected on PP supporting material are shown in Fig. 3. It can be seen clearly that the electrospun nonwoven webs composed predominantly of styrene (0 – 25 % EA) were formed by straight and uniform nanofibers with average radii around 400 nm. If the EA content in the copolymer reached 50 % the fibers still remained straight and uniform forming a regular nonwoven web, but their diameters increased slightly. With additional growth of EA amount in the copolymer, the diameters of fibers increased rapidly and their straightness, uniformity and web regularity decreased. Table I summarizes average fiber radii and water contact angles with standard deviations for all the prepared styrene-ethyl acrylate nonwovens collected on PP supporting material. For the sake of clarity, the effects of the EA content and glass transition temperature on fiber radius are demonstrated in Figs. 4 and 5. It was shown that the fiber diameter of electrospun nonwovens was strongly affected by the composition of the copolymer which is related to the glass transition temperature. In the case of performing the electrospinning procedure at the temperatures above or in the vicinity of polymer T_g , a significant gluing of fibers may occur resulting in formation of non-uniform fibers having increased diameters in the order of up to tens of micrometers. The solvent present in the polymer solution to be spun acts as a

plasticizer, thus lowering the frontier temperature of electrospinning environment behind which the formation of glued fiber structures occurs. Therefore, the nonwoven webs composed of nano-sized fibers were prepared in the case of styrene-ethyl acrylate copolymers having the glass transition temperatures sufficiently above the temperature of the electrospinning environment, whereas the copolymers comprising higher amounts of EA (88, 93 and 98 wt. %) were not spinnable.

The hydrophobicity of the electrospun nonwovens is supposed to be supported by increasing the content of hydrophobic styrene monomer units in styrene-ethyl acrylate copolymers, whereas the more polar EA units are believed to introduce hydrophilic properties. This assumption was confirmed partially by water contact angle measurements (see Fig. 5.). Nanofibers composed of 25 and 50 wt. % of ethyl acrylate, respectively, were found to be hydrophilic; the more EA comonomer in the polymer, the more significant hydrophilic nature of nanofiber webs. Nevertheless, the effect of increased hydrophilic nature with rising EA content in the electrospun polymer was found to be affected by structural characteristics of the nonwovens (see Fig. 3). The micron sized fiber diameters leading to increased porosity of the electrospun web were shown to be responsible for the hydrophobic behaviour of nonwoven mats formed even by polar polymers (samples containing 65 and 75 wt. % of EA).

Based on the results mentioned above, it can be stated that styrene-ethyl acrylate emulsion copolymers comprising soft ethyl acrylate monomer units in the range between 10 to 50 wt. % are able to be spun via the NanospiderTM electrospinning device into sub-micron uniform fibers with varying hydrophobic/hydrophilic properties. For the purpose of being used in practical applications, such as protective textiles, the copolymer composition comprising 50 wt. % of ethyl acrylate comonomer was chosen as the most preferable sample

among all the investigated copolymer samples, providing the nonwoven web of high regularity and potential variability of hydrophobic/hydrophilic properties.

EFFECT OF SUPPORTING MATERIAL

The role of a supporting material type differing especially in roughness and porosity (see Fig. 1) on the structural characteristics and variation of hydrophobic/hydrophilic nature of nanofiber nonwovens based on the copolymer sample containing 50 wt. % of EA units was investigated. Sambaer et al. [30] refer that the type of supporting material has a very significant effect on the porosity of nanofiber nonwovens prepared by the electrospinning process, even if their fiber diameter distributions remain similar. The morphological structure and corresponding images of water contact angle measurements of nonwoven webs electrospun on different types of supporting materials are shown in Fig. 6. It can be observed that by increasing the porosity of the supporting material, nanofibers with decreased fiber diameters were formed and a hydrophobic character of nonwovens appeared, which may be attributed to increased nonwoven web porosity (see Fig. 7).

It was demonstrated that by using different supporting materials, the hydrophobic nature of the electrospun webs can be tailored easily and nonwoven structured layers of specific water permeability may be produced, which makes the styrene-ethyl acrylate copolymer nanofibers ideal for lot of applications, such as creating novel textile composite materials by combining them with usual nonwovens.

Conclusions

The spinnability of styrene-ethyl acrylate copolymers prepared by emulsion polymerization from the point of copolymer polarity and glass transition temperature was investigated. Nonwoven ultra-fine fibers from polymer solutions in *N,N'*-dimethylformamide

were collected on PP supporting material and were characterized by measurements of fiber diameter and water contact angle. It can be concluded that it was possible to produce straight and uniform nanofibers from styrene-ethyl acrylate copolymers having the glass transition temperatures sufficiently above the temperature of the electrospinning environment. In the case of performing the electrospinning procedure at the temperatures above or in the vicinity of the polymer glass transition temperature, gluing of fibers may occur resulting in formation of non-uniform fibers having increased diameters. Therefore, the nonwoven webs of high regularity composed of nano-sized fibers were prepared in the case of styrene-ethyl acrylate copolymers comprising ethyl acrylate monomer units in the range of 0 – 50 wt. %. It was demonstrated as well that the hydrophobic character of the electrospun mats was influenced by both the polarity of the copolymer and structural characteristics of the nonwoven webs in terms of fiber diameter. Changing the copolymer composition or utilizing different supporting materials might lead to nanofiber webs with tailor-made hydrophobic/hydrophilic properties, which predetermines them for a wide range of applications such as protective technical textiles, composites etc.

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Figure 1: Macro-scale images of utilized supporting materials: a) spunbond from PP filaments, b) fine polyamide lattice, c) rough polyamide lattice.

Figure 2: Scheme showing the electrospinning equipment Nanospider TM.

Figure 3: SEM micrographs of styrene-ethyl acrylate ultra-fine fiber nonwovens electrospun on PP spunbond support and differing in the styrene/ethyl acrylate (EA) content.

Figure 4: Variation of fiber radius and water contact angle with ethyl acrylate (EA) content for styrene-ethyl acrylate ultra-fine fibre nonwovens prepared by the electrospinning process and differing in the styrene/ethyl acrylate content.

Figure 5: Variation of fiber radius and water contact angle with glass transition temperature for styrene-ethyl acrylate ultra-fine fibre nonwovens prepared by the electrospinning process and differing in the styrene/ethyl acrylate content.

Figure 6: SEM micrographs and corresponding images of water contact angle measurements of styrene-ethyl acrylate nanofibres (EA content in the copolymer 50 wt. %) electrospun on different types of supporting materials: spunbond from PP filaments, a fine polyamide lattice, and a rough polyamide lattice.

Figure 7: Comparison of average fiber radii and water contact angles with standard deviations for styrene-ethyl acrylate copolymers (EA content in the copolymer 50 wt. %) electrospun on different types of supporting materials: spunbond from PP filaments, a fine polyamide lattice, and a rough polyamide lattice.

Table 1: Fiber Radii and Water Contact Angles of Styrene-Ethyl Acrylate Electrospun Fibres Differing in the Content of Ethyl Acrylate and Glass Transition Temperature

Figure 1

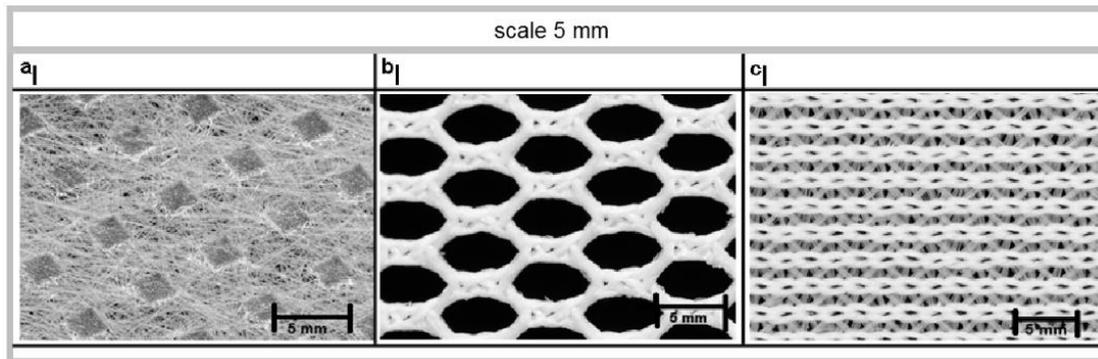


Figure 2

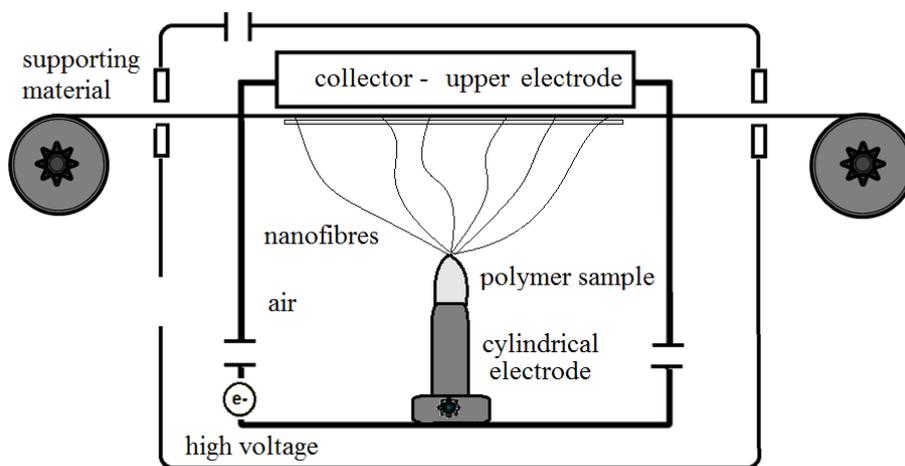


Figure 3

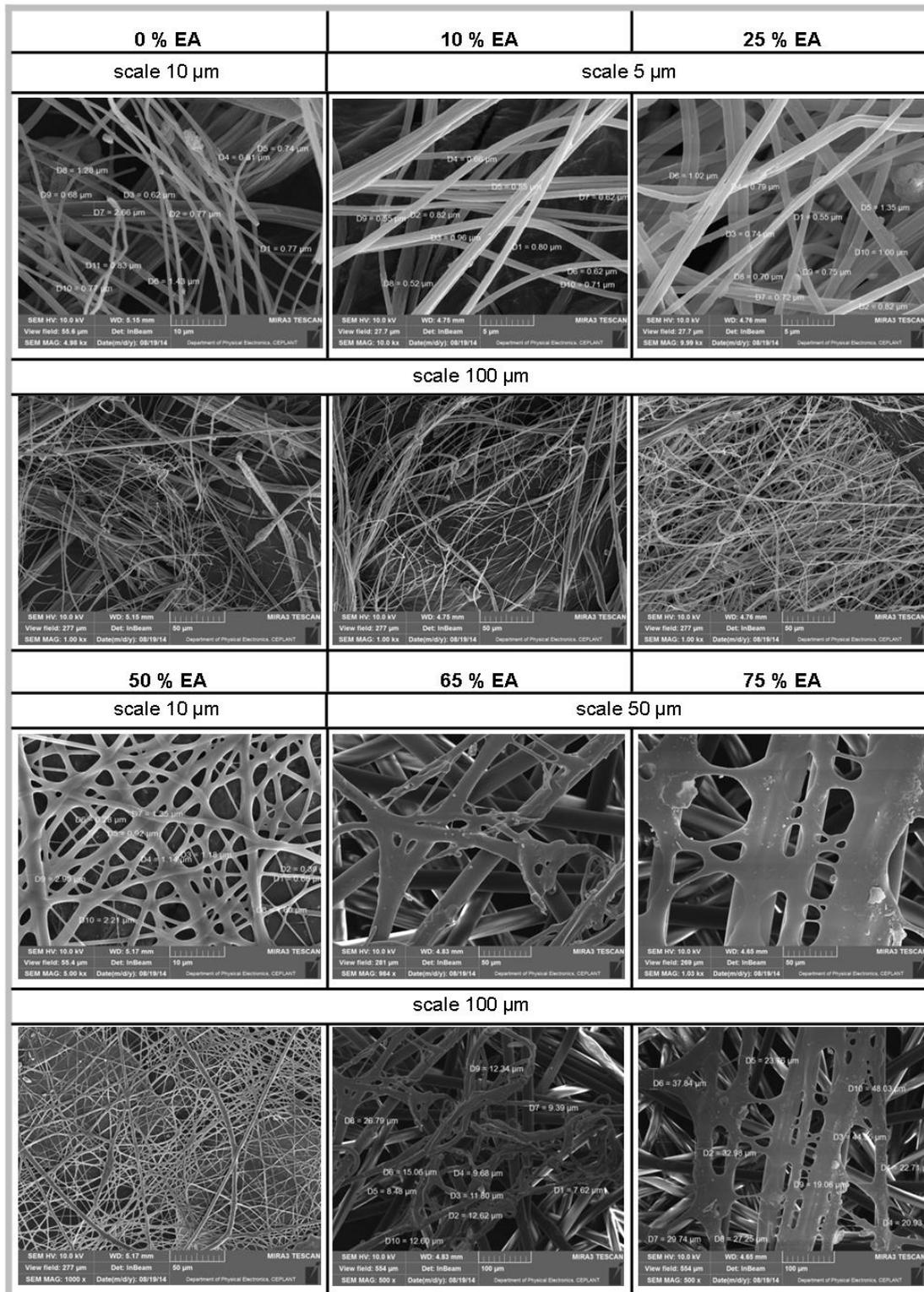


Figure 4

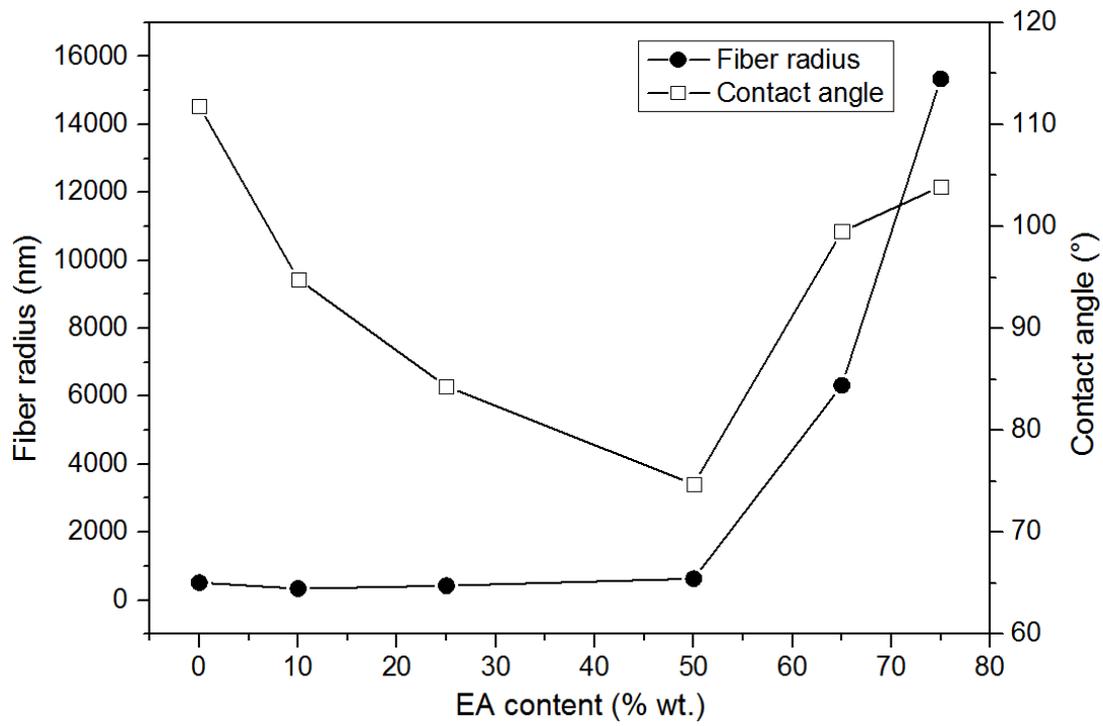


Figure 5

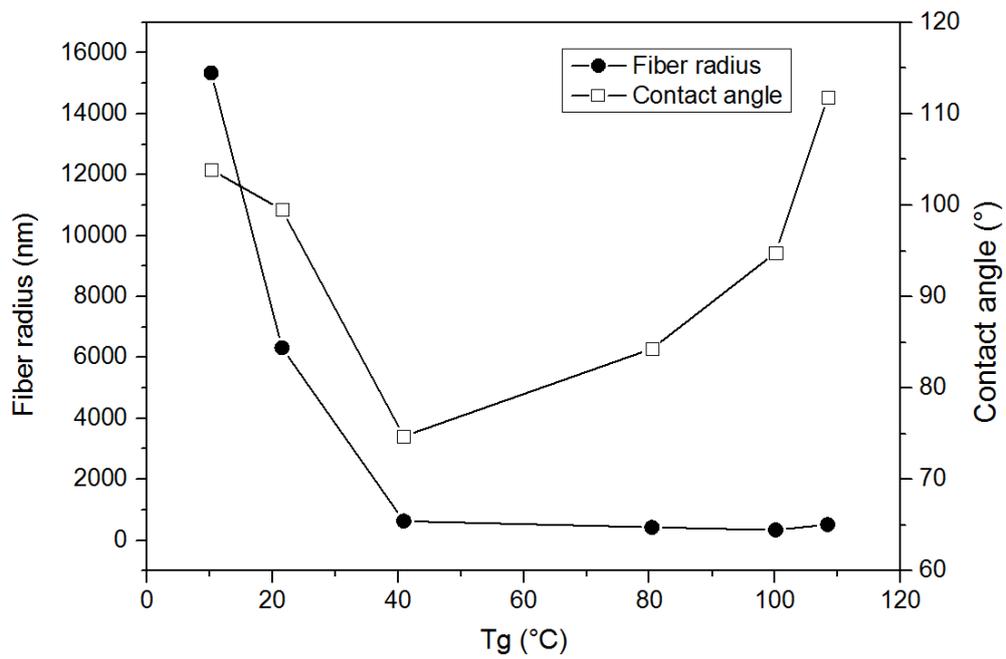


Figure 6

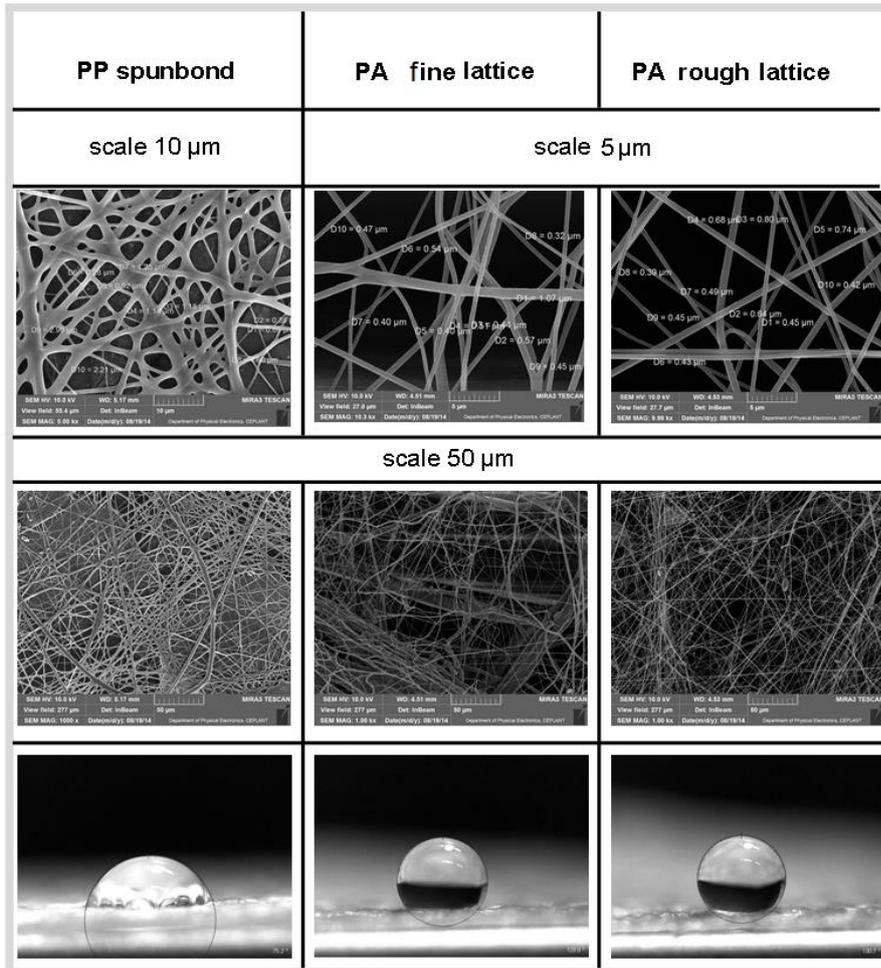


Figure 7

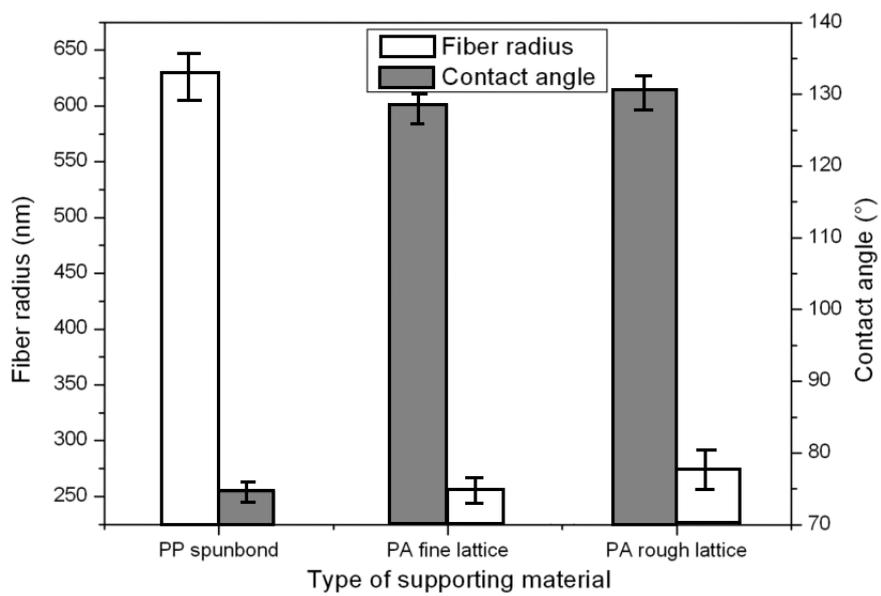


Table 1: Fiber Radii and Water Contact Angles of Styrene-Ethyl Acrylate Electrospun Fibres Differing in the Content of Ethyl Acrylate and Glass Transition Temperature

EA content (% wt)	T_g (°C)	Fiber radius (nm)	Contact angle (°)	Interaction with water
0	108.4	515 ± 65	111.8 ± 3.9	Hydrophobic
10	100.1	340 ± 50	94.8 ± 2.2	Hydrophobic
25	80.4	420 ± 90	84.3 ± 3.0	Hydrophilic
50	40.9	630 ± 145	74.7 ± 2.7	Hydrophilic
65	21.5	6 320 ± 960	99.5 ± 4.1	Hydrophobic
75	10.2	15 345 ± 1890	103.9 ± 3.8	Hydrophobic
88	1.5		Not spinnable	
93	-2.2		Not spinnable	
98	-5.8		Not spinnable	