# The effect of process parameters on the electrospun polystyrene fibers

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**REZUMAT – ABSTRACT**

**Efectul parametrilor de proces asupra fibrelor de polistiren electrofilate**

*Electrofilarea este una dintre metodele de obținere a nano/microfibrelor, utilizând soluții polimerice. Aceste membrane nanofibroase sunt foarte poroase, cu pori interconectați, o suprafață specifică ridicată și dimensiuni mici ale porilor, făcându-le un candidat adecvat pentru aplicațiile de filtrare. Proprietățile fibrelor electrofilate sunt influențate de soluția polimerică, solvent, concentrația soluției, viscozitate, conductivitatea electrică, tensiunea electrică, distanța dintre duza de filare și dispozitivul de colectare etc. Polistirenul expandat este un produs polimeric utilizat în mod obișnuit pentru izolare și ambalare. Reciclarea polistirenului expandat în nanofibre cu aplicații în filtrare ar putea fi utilă din punct de vedere economic. Scopul acestui studiu a fost investigarea influenței caracteristicilor soluției de polimer de polistiren expandat (concentrație, viscozitate) și a parametrilor de proces (tensiunea aplicată, distanța dintre vârful duzei și placa colectoare, debitul soluției polimerice) asupra morfologiei și proprietăților fibrelor electrofilate obținute. Trei soluții EPS cu o concentrație de 10, 15 și 20 procente masice au fost preparate și au fost electrofilate în condiții de procesare, cu o tensiune aplicată de 12, 15 și 18 kV, o distanță duza de filare-colector de 20 cm, un debit al soluției de 1,5 și 2 mL/oră, un diametru al duzei de filare de 0,8 mm și un substrat de cupru staționar. Morfologia fibrelor electrofilate a fost observată prin microscopia electronică de baleiaj. Proprietățile mecanice au fost evaluate prin teste de rezistență mecanică la tracțiune și alungire.*

*Cuvinte-cheie: polistiren expandat, electrofilare, fibre electrofilate, membrană, filtrare*

**The effect of process parameters on the electrospun polystyrene fibers**

*Electrospinning is one of the methods for obtaining nano/microfibers, using polymeric solutions. These nanofibrous membranes are highly porous with interconnected pores, having high specific surface area and small pore size, making them a suitable candidate for filtration applications. The properties of electrospun fibers are influenced by polymer solution, solvent, solution concentration, viscosity, electrical conductivity, electrical voltage, spinneret to collector distance etc. Expanded polystyrene is a polymeric product that is usually used for insulation and packaging. Recycling expanded polystyrene into nanofibers with applications in filtration could be useful from an economic point of view. The purpose of this study was to investigate the influence of expanded polystyrene polymer solution characteristics (concentration, viscosity) and the process parameters (applied voltage, distance between the tip and the collector plate, flow rate of the polymer solution) on the morphology and the properties of the obtained electrospun fibers. Therefore, three EPS solutions with 10, 15 and 20% wt. concentration were prepared and were electrospun under processing conditions with an applied voltage of 12, 15 and 18 kV, a spinneret-to-collector distance of 20 cm, a flow rate of solution of 1.5 and 2 mL/hour, a spinneret diameter of 0.8 mm and stationary copper substrate. The morphology of the electrospun fibers was observed by scanning electron microscopy. The mechanical properties were evaluated by tensile strength and elongation tests.*

*Keywords: expanded polystyrene, electrospinning, electrospun fibers, membrane, filtration*

## INTRODUCTION

Electrospinning is a process for developing non-woven membranes made of sub-micron to nano-scale fibers. These nano fibrous membranes are highly porous with interconnected pores, having high specific surface area and small pore size [1]. The structural properties of the Nano fibrous membranes make them a suitable candidate for filtration applications [2]. Electrospun nanofibers have many other applications in domains such as sensors, catalysis, drug delivery, tissue engineering, textiles, composite reinforcements, etc. [3–9].

Many polymers can be used in the electrospinning process, such as: polyacrylonitrile, polystyrene, polymethyl methacrylate, polyvinylchloride, polyamide, polyethylene terephthalate, cellulose acetate, polyvinyl alcohol, polyether imide, polyethylene glycol, nylon 6, polyethylene, polypropylene, etc. [9–12].

The polystyrene (PS) beads have been studied as a possible filter element in water treatment plants. In addition, the superhydrophobic PS nanofiber membrane was electrospun and it was found to be highly efficient at oil-water separation [13].

Expanded polystyrene (EPS) is a polymeric product that is usually used for insulation and packaging. Recycling EPS into nanofibers with applications in filtration could be useful from an economic point of view [14].

The properties of the obtained electrospun nanofibers are influenced by many parameters such as structural properties of polymers, polymer solution

parameters, processing conditions and the ambient parameters [15].
The purpose of this study was to investigate the influence of EPS polymer solution characteristics (concentration, viscosity) and the process parameters (applied voltage, distance between the tip and the collector plate, polymer solution flow rate) on the morphology and the properties of the obtained electrospun fibers.
The morphology of the electrospun fibers was observed by scanning electron microscopy (SEM). The mechanical properties were evaluated by tensile strength and elongation tests.

## EXPERIMENTAL WORK

### Materials

In this study, recycled expanded polystyrene (EPS) foam, waste from commercial insulating material used in constructions, was used as polymer source without further purification. The solvent used for dissolving EPS was dimethylformamide (DMF) with 0.94 g/cm$^3$ density, purchased from Alfa Aesar.

### Preparation of electrospinning solutions

Three EPS solutions in DMF with concentrations of 10, 15 and 20 % wt. were prepared by dissolving EPS foam in DMF.
A good dissolution of the EPS in DMF is very important in achieving good morphological properties of the electrospun fibers. Thus, EPS was dissolved in DMF by magnetic stirring at room temperature for 1 hour, at a rotational speed of 420 rpm (table 1). The solutions were electrospun immediately after preparation.

Table 1

| EPS/DMF solution | EPS10 | EPS15 | EPS20 |
|---|---|---|---|
| EPS concentration in DMF, % wt. | 10 | 15 | 20 |
| Rotational speed, rpm | 420 | | |
| Homogenization time, minutes | 60 | | |
| Temperature, °C | Room temperature | | |

The prepared EPS solutions were electrospun using NaBond unit under processing conditions with an applied voltage of 12, 15 and 18 kV, a spinneret-to-collector distance of 20 cm, a solution flow rate of 1.5 and 2 mL/h, a spinneret with a nozzle size of 0.8 mm and stationary substrate. Aluminium foil served as the substrate for fiber collecting.

### Characterization

Electrical conductivity of EPS solutions was measured with a VARIO COND portable conductivity meter model 340i with the cell constant K = 0.469 cm$^{-1}$.
The rheological behaviour of EPS solutions was studied using a rotational viscometer BROOKFIELD DV-II+ Pro, by measuring viscosity, shear rate and shear stress, at room temperature.
Morphological characterization of the EPS electrospun fibers by scanning electron microscopy (SEM) was performed by a FESEM/FIB/EDS Workstation Auriga produced by Carl Zeiss Germany, with an acceleration voltage of 2 kV, using the SESI detector.
Wettability testing of the polymeric membranes was carried out using the sessile drop method. The process of determining the contact angle of the polymeric membranes with distilled water was performed using an optical microscope equipped with a camera for images acquisition on the computer and the images were processed using the software Image J, Drop Analysis - Drop Snake.
The tensile strength and the elongation of the EPS electrospun fibers were determined by using a mechanical testing machine, model LFM 30 kN, Walter & Sai AG Switzerland.

## RESULTS AND DISCUSIONS

### EPS solutions electrical conductivity

The results regarding the electrical conductivity of EPS/DMF solutions (EPS10, EPS15 and EPS20) are presented in table 2.

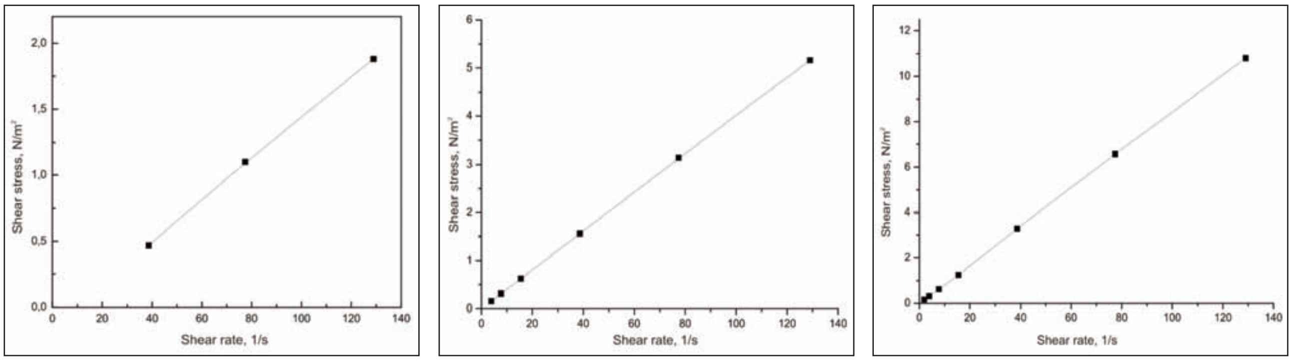
Table 2

| EPS/DMF solutions | Electrical conductivity [S/m] | Electrical resistivity [Ω × m] |
|---|---|---|
| EPS10 | $4.0\times10^{-4}$ | $2.50\times10^{3}$ |
| EPS15 | $4.8\times10^{-4}$ | $2.08\times10^{3}$ |
| EPS20 | $5.1\times10^{-4}$ | $1.96\times10^{2}$ |

From the data presented above, it can be seen that the electrical conductivity values of the EPS solutions are low, their electrical resistivity being in the semiconductor range ($10^{-5} \div 10^{8}$ Ω×m). Increasing the EPS concentration determines an increase in the electrical conductivity of corresponding solution.

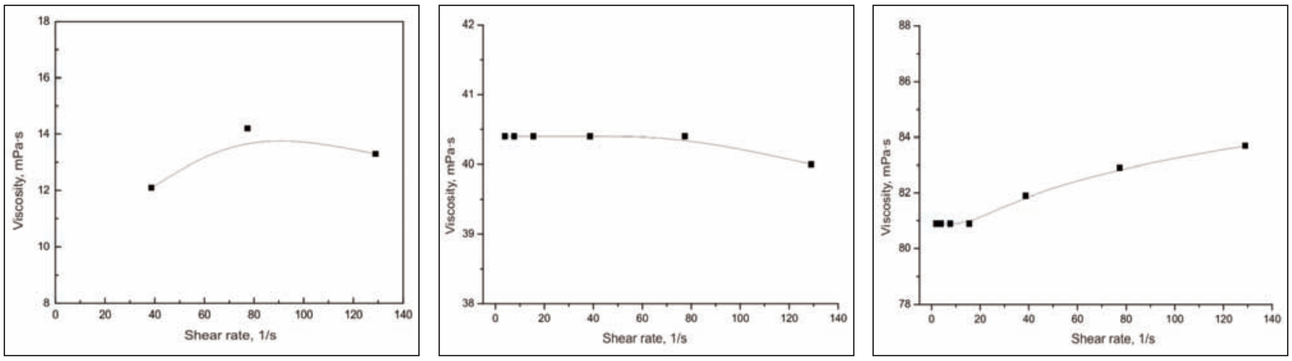
### EPS solutions rheological behaviour

The viscosity of polymer systems is one of the parameters that determine their behaviour under external electric fields applied. The rheology of polymeric systems is influenced by the molecular weight of the dissolved polymers, the shape and rearrangement of macromolecules, and polymer-solvent interactions. The viscosity of the solution increases monotonically with concentration up to a critical value for a given polymer molecular weight. These observations reflect the consequences of macromolecular associations and are valid for studying polymer solutions to a range of low to high shear rates.
The experimental results regarding the rheological behaviour of EPS/DMF solutions are presented in figures 1 and 2. We can see that the viscosity of EPS solutions increases with increasing shear rate, indicating that they have pseudoplastic non-Newtonian fluid characteristics. Also, the viscosity of EPS solutions increases with the increasing of the polymer concentration in DMF.




a b c

Fig. 1. Rheological profile of solutions, shear stress–shear rate model: *a* – EPS10; *b* – EPS15; *c* – EPS20




a b c

Fig. 2. Rheological profile of solutions, viscosity–shear rate model: *a* – EPS10; *b* – EPS15; *c* – EPS20

## Morphological characterization of the electrospun EPS fibers by SEM

The figures below show the SEM micrographs of the EPS fibers. The values of the EPS fibers diameters determined from the SEM analysis are presented in table 3.

Analysing the SEM micrographs we find that the fibers morphology is influenced by the parameters of

Table 3

| Sample | Flow rate [ml/h] | Applied voltage [kV] | Fibers diameter [nm] | | |
|---|---|---|---|---|---|
| | | | average | min | max |
| EPS10 | 1.5 | 12 | 324.6 | 232.3 | 431.6 |
| | 2 | | 366.2 | 325.7 | 390.2 |
| | 1.5 | 15 | 398.9 | 275.1 | 485.2 |
| | 2 | | 487.0 | 289.5 | 664.1 |
| | 1.5 | 18 | 385.7 | 294.0 | 447.7 |
| | 2 | | 440.1 | 323.7 | 514.4 |
| EPS15 | 1.5 | 12 | 629.0 | 435.3 | 969.6 |
| | 2 | | 883.4 | 748.3 | 1091 |
| | 1.5 | 15 | 791.4 | 784.4 | 796.6 |
| | 2 | | 813.2 | 346.9 | 1276 |
| | 1.5 | 18 | 810.6 | 648.2 | 983.9 |
| | 2 | | 611.9 | 392.2 | 847.8 |
| EPS20 | 1.5 | 12 | 696.0 | 553.5 | 861.3 |
| | 2 | | 719.8 | 428.0 | 908.5 |
| | 1.5 | 15 | 795.4 | 699.6 | 852.2 |
| | 2 | | 699.6 | 563.3 | 791.5 |
| | 1.5 | 18 | 602.5 | 534.6 | 731.3 |
| | 2 | | 690.2 | 596.1 | 832.9 |

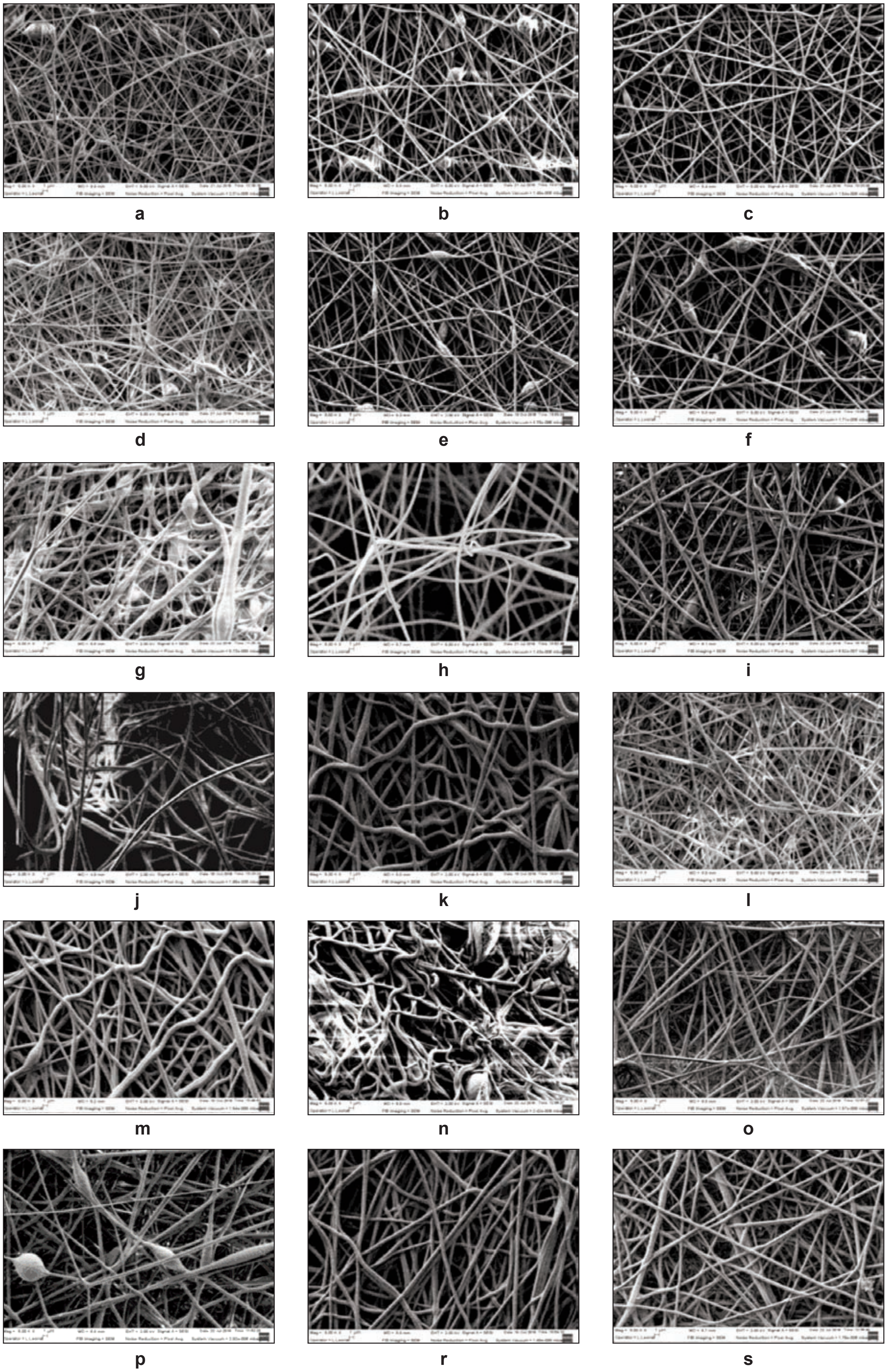



Fig. 3. SEM micrographs of samples electrospun on aluminum foil substrate:
EPS10: *a* – 1.5 ml/h, 12 kV; *b* – 1.5 ml/h, 15 kV; *c* – 1.5 ml/h, 18 kV; *d* – 2 ml/h, 12 kV; *e* – 2 ml/h, 15 kV; *f* – 2 ml/h, 18 kV;
EPS15: *g* – 1.5 ml/h, 12 kV; *h* – 1.5 ml/h, 15 kV; *i* – 1.5 ml/h, 18 kV; *j* – 2 ml/h, 12 kV; *k* – 2 ml/h, 15 kV; *l* – 2 ml/h, 18 kV;
EPS20: *m* – 1.5 ml/h, 12 kV; *n* – 1.5 ml/h, 15 kV; *o* – 1.5 ml/h, 18 kV; *p* – 2 ml/h, 12 kV; *r* – 2 ml/h, 15 kV; *s* – 2 ml/h, 18 kV

electrospinning process (concentration of polymer solution, flow rate, voltage).

Increasing the concentration of EPS in the solution from 10% to 20% leads to uniform fibers without defects (beads). At the same time, there is an increase in the average diameter of the electrospun fibers from 300 nm to 900 nm.

Increasing the flow rate of the polymer solution from 1.5 to 2 mL/h leads to an increase in the average diameter of the electrospun fibers by approximately 50–100 nm.

Increasing the applied voltage from 12 to 18 kV also causes an increase in the average diameter of the electrospun fibers. Uniform and faultless fibers are obtained at an applied voltage of 18 kV and solution concentrations of 15% and 20%.

### Wettability testing of the electrospun EPS fibers

For static contact-angle measurements, small strips of the samples were cut and placed onto a planar stage to ensure a flat viewing surface. A drop of water was dropped on the polymeric layer's surface from a micro syringe at room temperature (~28°C). The drop was allowed to reach equilibrium before the measurement was recorded and before evaporation occurred. Figure 4 displays the contact angle of distilled water with the EPS fibers.

The data presented above shows that the membranes obtained from EPS exhibit hydrophobic behaviour, due to the fact that the contact angle has high values of about 125–133° for all the polymer concentrations used.

### Mechanical properties of the electrospun EPS fibers

In order to conduct tensile strength and elongation tests, the EPS fibers were deposited on a textile substrate (gauze fabric) for 6 hours. The samples were prepared with the following dimensions: length of 100 mm and width of 20 mm. The tests were carried out with a drawing speed of 50 mm/minute. Five tests were carried out for each type of material and then an average value of the parameters was calculated. Figures 5 (a–d) display the tensile strength curves of the EPS15 and EPS20 fibers. Table 4 contains the mechanical properties of the EPS fibers electrospun on the gauze fabric.

From the data presented above, it is found that the tensile strength has values in the range of 3.73-6.11 MPa, the average elongation is between 1.45 and

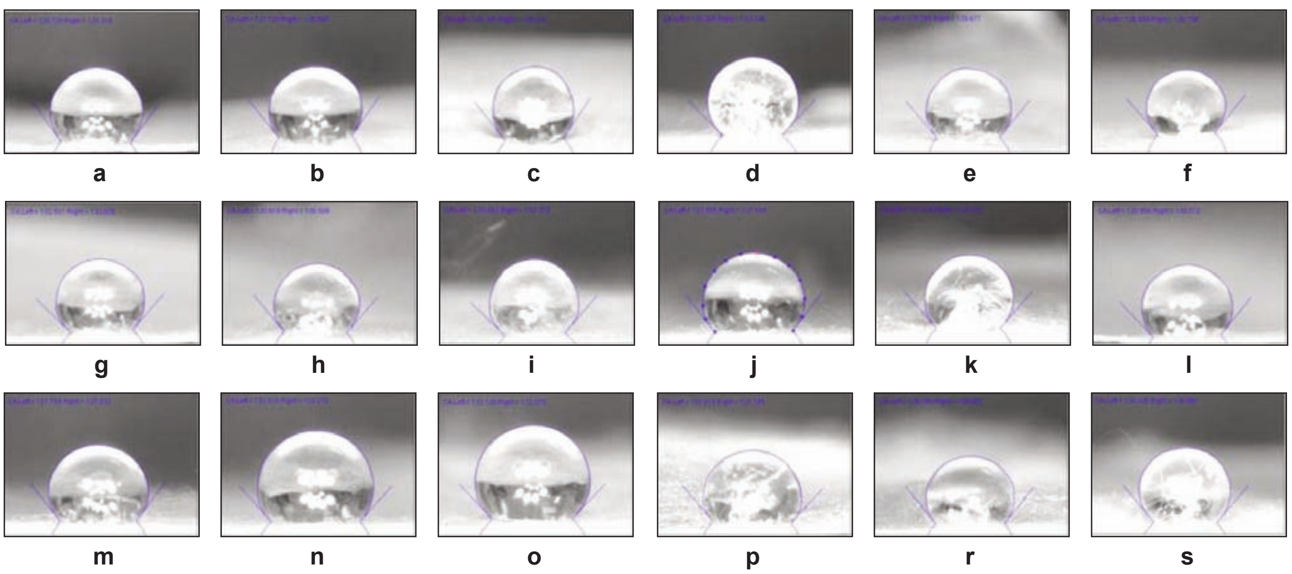



Fig. 4. Images of the water contact angle with the fibers surface:
EPS10: *a* – 1.5 ml/h, 12 kV; *b* – 1.5 ml/h, 15 kV; *c* – 1.5 ml/h, 18 kV; *d* – 2 ml/h, 12 kV; *e* – 2 ml/h, 15 kV; *f* – 2 ml/h, 18 kV;
EPS15: *g* – 1.5 ml/h, 12 kV; *h* – 1.5 ml/h, 15 kV; *i* – 1.5 ml/h, 18 kV; *j* – 2 ml/h, 12 kV; *k* – 2 ml/h, 15 kV; *l* – 2 ml/h, 18 kV;
EPS20: *m* – 1.5 ml/h, 12 kV; *n* – 1.5 ml/h, 15 kV; *o* – 1.5 ml/h, 18 kV; *p* – 2 ml/h, 12 kV; *r* – 2 ml/h, 15 kV; *s* – 2 ml/h, 18 kV

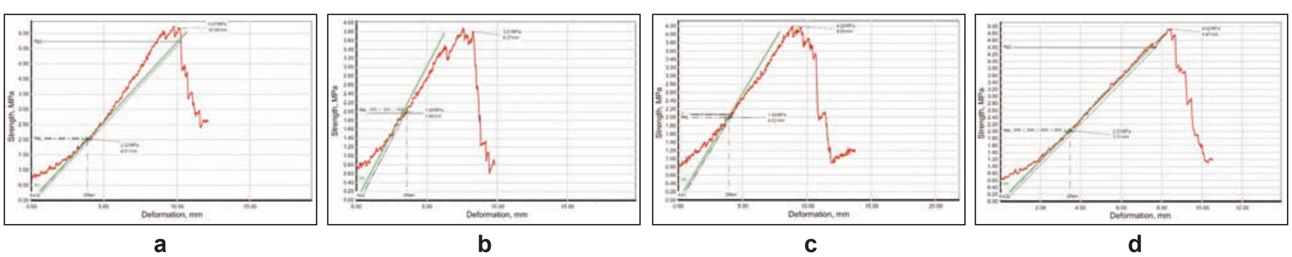



Fig. 5. Tensile stregth curves of the samples: *a* – EPS15 1.5 ml/h, 15 kV; *b* – EPS15 1.5 ml/h, 18 kV;
*c* – EPS20 1.5 ml/h, 15 kV; *d* – EPS20 1.5 ml/h, 18 kV

Table 4

| Sample | Membrane thikness [µm] | Average tensile strength [MPa] | Average elongation [%] | Average elastic modulus [GPa] |
|---|---|---|---|---|
| EPS15 1.5 mL/h 15 kV | 280 | 5.41 | 2.94 | 0.06 |
| EPS15 1.5 mL/h 18 kV | 320 | 3.84 | 1.45 | 0.07 |
| EPS20 1.5 mL/h 15 kV | 340 | 4.16 | 1.70 | 0.06 |
| EPS20 1.5 mL/h 18 kV | 400 | 4.86 | 2.69 | 0.08 |

2.94%, and the average elastic modulus is between 0.06 and 0.08 GPa for all analysed samples. EPS20 samples exhibit a slight increase of the mechanical characteristics with the increase of the thickness of the electrospun layer.

## CONCLUSIONS

In the present work, polymeric membranes of EPS were prepared under various conditions through an electrospinning process. Experiments were conducted to identify the influence of the process parameters on the morphology and properties of the electrospun EPS fibers.

Increasing the concentration of EPS in the solution from 10% to 20% and the applied voltage from 12 to 18 kV, leads to the obtaining of uniform fibers without defects (beads).

Increasing the concentration of EPS and increasing the flow rate of the polymer solution from 1.5 to 2 mL/h leads to an increase in the average diameter of the electrospun EPS fibers.

The EPS fibers exhibit a hydrophobic behaviour, the contact angle having values of about 125–133°.

EPS20 fibers, compared to EPS15 fibers, exhibit a slight increase of the mechanical characteristics with the increase of the thickness of the electrospun layer.

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