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Influence of the electrospinning parameters on the morphology of composite nanofibers

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ABSTRACT

Purpose: In the paper the fabrication of composite nanofibers using electrospinning technique was reported. That processing technique was used to synthesis composite nanofibers with various morphologies using a precursor composed of poly(vinyl) alcohol (PVA), copper acetate (CuAC) and acetic acid (C_2H_3OH). The morphology of formed nanofibers depends not only on the spinning parameters, but also on the composition of the polymer solution. The purpose of the study was to obtain results that allowed to determine the influence of parameters of the electrospinning process on morphology of the composite nanofibers.

Design/methodology/approach: The obtained nanofibers were characterized through high resolution scanning electron microscopy (SEM). It was noticed that the morphology of composite nanofibers depends on the applied voltage and nozzle-collector distance. The research was carried on a scanning electron microscope.

Findings: The influence of parameters of the electrospinning process on morphology of the composite nanofibers was determined.

Research limitations/implications: The research was carried out on samples, not on final elements.

Originality/value: The paper presents the influence of the electrospinning parameters on the morphology of composite nanofibers.

Keywords: Electrospinning technique; Nanofibers; Nanocomposites; Cu nanoparticles

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

The creation of nanomaterials aims to show completely new or improved physical, chemical and biological properties of already known materials. In fact, newer and better products are produced. Moreover, nanotechnology is a field which is rapidly growing and has enormous potential to affect many areas of science. It is very important to get to know the properties and the structure of newer and newer developing nanostructures which are used in materials production addressed directly to the consumers [1-3].

The most important methods to allow the production of polymeric nanofibers are: electrospinning, synthesis by template, pulling, phase separation, molecular self- organization [4].

Electrospinning is the most effective method for producing nanofibers from the polymer solution flowing from the nozzle-collector by the polymer drawing under the strong electric field. During electrospinning the polymer solution is pumped to the nozzle and is subjected to an electrostatic field forces that should create the fiber. It is important to know that minimum concentration for a given polymer, terming the critical entanglement concentration, below which a stable jet cannot be achieved and nanofibers cannot be formed exist. Shaped fiber moves in the direction of the collector with spiral motion along the electrostatic field lines reducing its diameter. Initially the polymer solution liquid spurt is evaporated under controlled conditions (temperature and humidity) what gives almost dry fiber with a diameter above 100 nm. The nanofiber is deposited on a grounded collector forming the nonwoven fabric [4-8].

The drawing speed of polymer nanofibers increased from 2 m/s to 200 m/s and depends on the physical properties of the solution and production conditions. By the observation the length, thickness, consistency and movement of the stream useful to predict the morphology of the nanofibers are formed. Inconsistent, oscillating stream is indicative of a variety of problems, for example: irregular nanofibers, clearly blurred shape, defects in the form of beads. The stream can be optimized by adjusting the composition of the solution and the configuration of the electrospinning device. The morphology of formed nanofibers depends not only on the spinning parameters, but also on the composition of the polymer solution [9,10].

The materials with composite nanofibers with Cu can be used to produce components such us filters, ventilation and air condition in case of their properties.

2. Materials and methodology

In order to produce polymer nanofibers the mixture of PVA solution in CuAC and C_2H_3OH mixture, in a weight ratio of 0.75 g : 1 g : 2 g respectively, was prepared. The solution was obtained using a magnetic stirrer.

To obtain the nanofibers the electrospinning process was applied using Yflow S.500 device.

The device for electrospinning consists of three major components: a high-voltage power supply, a metallic needle and a collector. A schematic illustration of the basic setup for electrospinning is show in Figure 1.

Electrospinning is a process in which a charged liquid jet is collected on a grounded collector. The polymer jet is formed when an applied electrostatic charge overcomes the surface tension of the solution There is a minimum concentration for a given polymer, terming the critical entanglement concentration, below which a stable jet cannot be achieved and nanofibers cannot be formed.

By the observation the length, thickness, consistency and movement of the stream useful to predict the morphology of the nanofibers is formed. Inconsistent, oscillating stream is indicative of a variety of problems, for example: irregular nanofibers, clearly blurred shape,

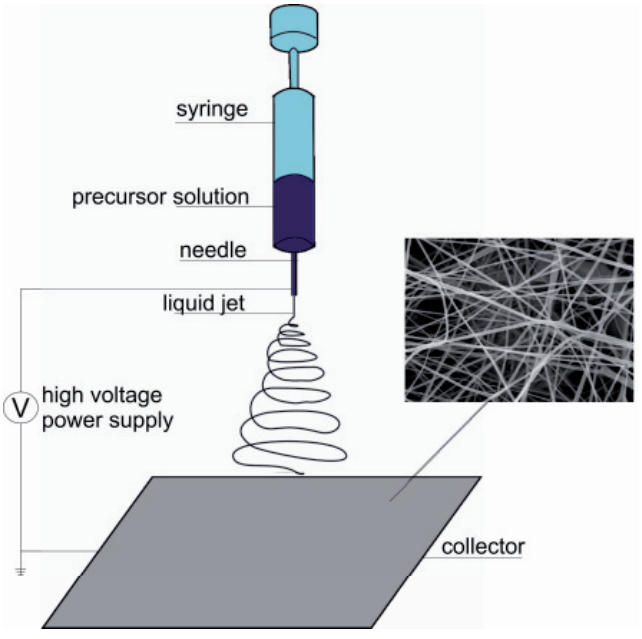

Fig. 1. Schematic illustration of the basic setup for electrospinning. The small photograph shows the SEM image composite nanofibers composed of poly(vinyl) alcohol (PVA), copper acetate (CuAC) and acetic acid (C_2H_3OH) [11]

defects in the form of beads. The stream can be optimized by adjusting the composition of the solution and the configuration of the electrospinning device.

The nanofibers were collected on aluminium foil during 20 minutes. The composite nanofibers were produced under different voltages (20, 25 and 30 kV), on nozzle-collector distances (10, 25 and 30 cm) and constant flow rate of 0.2 ml/h. Electrospinning parameters were presented in Table 1.

Table 1.
Electrospinning parameters for each sample: p – solution flow rate, d – distance between the electrodes, U – differential voltage at the nozzle and the collector

Sample	p, ml/h	d, cm	U, kV
Sample 1	0.2	10	20
Sample 2	0.2	10	25
Sample 3	0.2	10	30
Sample 4	0.2	15	20
Sample 5	0.2	20	20

3. Results

3.1. Morphology

The morphology of obtained nanofibers were characterized using high resolution scanning electron microscopy (SEM) SUPRA 35, Zeiss (Figs. 2-6).

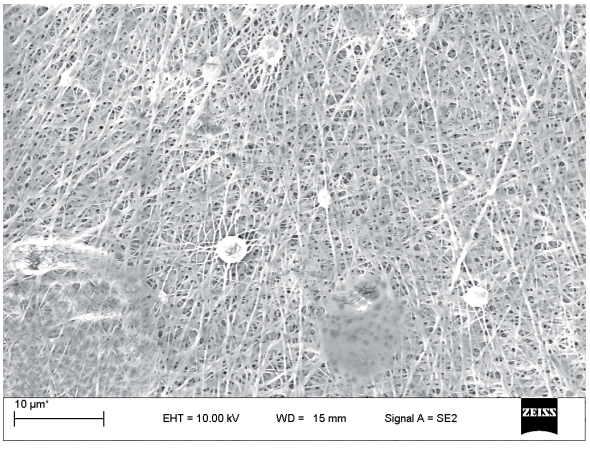

Fig. 2. SEM image of PVA nanofibers prepared at nozzle-collector distance equalling to 10 cm and a voltage of 20 kV

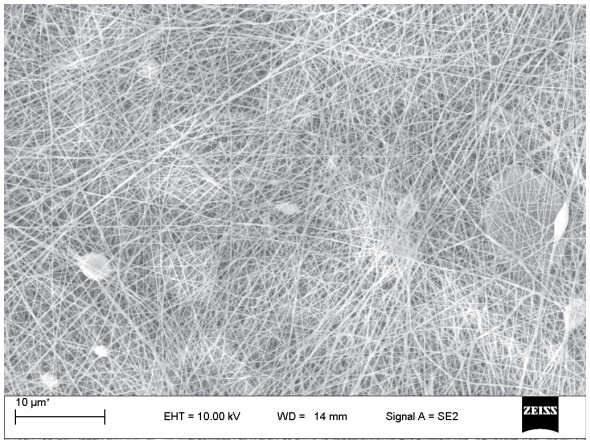

Fig. 3. SEM image of PVA nanofibers prepared at nozzle-collector distance equalling to 10 cm and a voltage of 25 kV

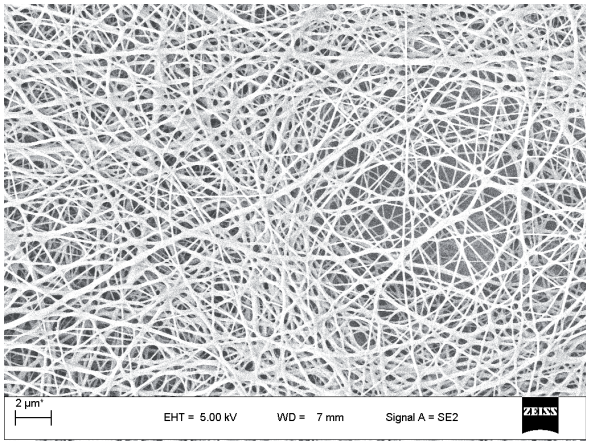

Fig. 4. SEM image of PVA nanofibers prepared at nozzle-collector distance equalling to 10 cm and a voltage of 30 kV

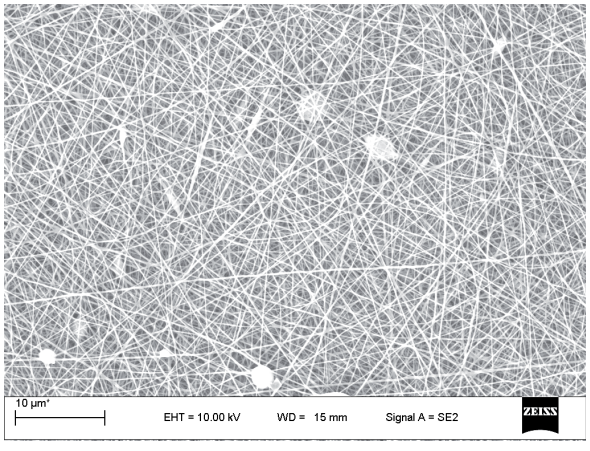

Fig. 5. SEM image of PVA nanofibers prepared at nozzle-collector distance equalling to 15 cm and a voltage of 20 kV

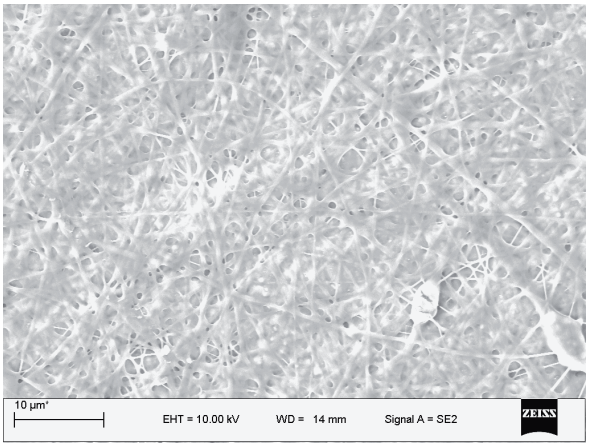

Fig. 6. SEM image of PVA nanofibers prepared at nozzle-collector distance equalling to 20 cm and a voltage of 20 kV

3.2. The chemical composition

The composition of composite nanofibers obtained by electrospinning as determined by EDS analysis is given in Fig. 7.

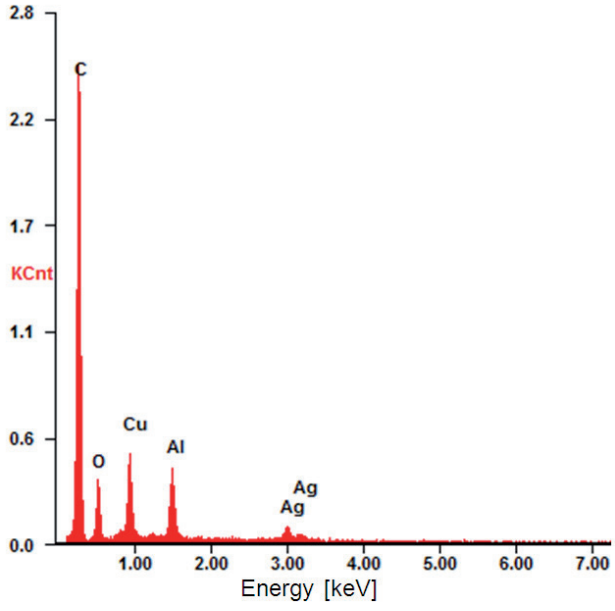

Fig. 7. EDS analysis of example sample – nanofibers collected on aluminium substrate

Al signals represents the substrate on which the samples were collected. Ag signals originate from the conductive layer that was deposited on the sample prior to a scanning electron microscope (SEM) investigation.

3.3. Determination of nanofibers diameter

Determination of nanofibers diameter was achieved using DigitalMicrograph programme. For each sample measurements of five random selected nanofibers were taken and then results were averaged. Obtained results were presented in Fig. 8.

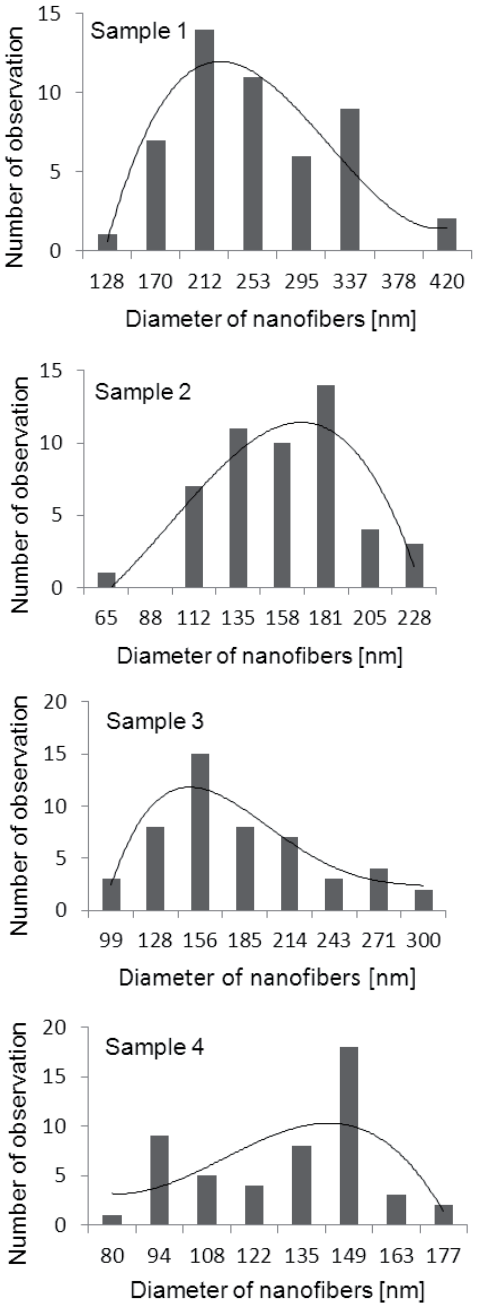

Fig. 8. Comparison of measurements of the diameter of the composite nanofibers

In the case of sample 5 measurements of the diameter of the fiber was not taken due to excessive blurring of their shape. Table 2 shows the average sizes of the diameters of the composite fibers.

Table 2.
The average of the diameters of the composite nanofibers

Sample	Sample 1	Sample 2	Sample 3	Sample 4
Diameter of nanofibers, nm	235	145	167	124

3.4. The influence of the voltage on the nanofibers morphology

A graph (Fig. 9) shows the comparison of diameters of the nanofibers for samples taken at differential voltages between the nozzle and the collector, with a constant flow rate of the solution and for a constant distance between the nozzle and the collector.

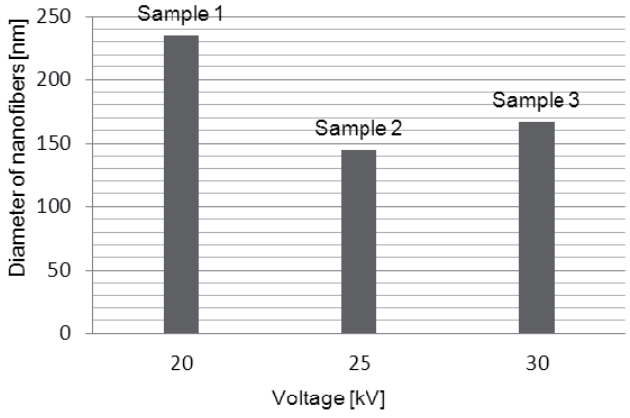

Fig. 9. The influence between the diameters of the nanofibers and the voltage during the electrospinning process

By the observation the length, thickness, consistency and movement of the stream useful to predict the morphology of the nanofibers is formed. Inconsistent, oscillating stream is indicative of a variety of problems, for example: irregular nanofibers, clearly blurred shape, defects in the form of beads.

In the case of samples 1 and 2 with the increase of the voltage between electrodes from 20 kV to 25 kV, flow rate 0.2 ml/h and constant nozzle-collector distance (10 cm) diameter of the nanofibers decrease. The same is observed for the samples 1 and 3, when the voltage change from 20 kV to 30 kV. However, in the case of increasing the voltage from 25 kV to 30 kV the diameter of the composite nanofiber increases. Nanofibers with a clearly blurred shape and a lot of defects in the form of beads are observed for both samples 1 and 2. The greatest tendency to merge the nanofibers and defects is in a sample 1, in the case of sample 2 the fibers are clearer with lower number of defects. The sample made at the voltage 30 kV (sample 3) has clear nanofibers without defects in the form of beads.

3.5. The influence of the nozzle-collector distance on nanofibers diameter

A graph (Fig. 10) shows the comparison of the diameter of the nanofibers for samples taken at different distances between nozzle-collector distance, with a constant flow rate of the solution and for a constant voltage.

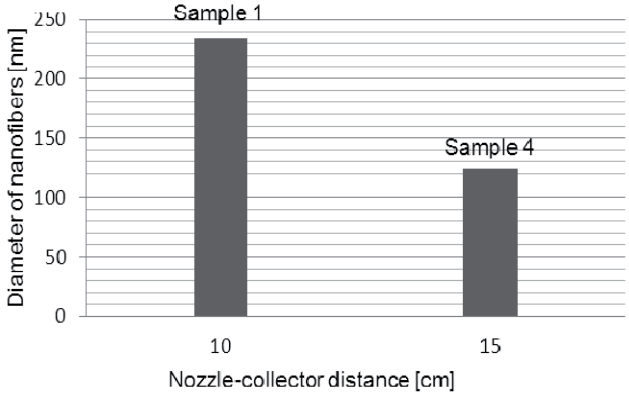

Fig. 10. The influence between the diameters of the nanofibers and the distance between the nozzle and the collector

With the increase of distance between electrodes from 10 cm to 15 cm, a flow rate of 0.2 ml/h, a constant voltage of 20 kV, in the case of samples 1 and 4, the diameter of the composite nanofiber is clearly reduced.

For sample 5 made at nozzle-collector distance equalling to 20 cm, a constant flow rate of 0.2 ml/h and a voltage of 20 kV, measuring of the nanofibers diameter is impossible. For a distance of 10 cm (sample 1) obtained nanofibers are of a clearly blurred shape and possess a number of defects in the form of beads. In the case of sample 4 obtained at a distance between the electrodes of 15 cm fibers are much more clearer comparing to samples

1 and 5 and are significantly less defected. Sample 5 has the highest number of defects and the nanofibers have a blurry shape.

4. Conclusions

The work presented the way to understand the morphological properties of composite nanofibers and how it is influenced by process parameters, for example: differential voltage at the nozzle and the collector, distance between the electrodes and solution flow rate.

Other parameters which also can be varied while environmental parameters such as temperature and moisture and all those variables can influence the electrospun nanofibers. The reason of the nanofibers merging in the sample 1 is probably too small distance between the electrodes. The solution had evaporated on the road between the nozzle and the collector. Although the increase of distance during preparation of sample 5, and using the same voltage during electrospinning, as at the sample 1, the nanofibers are much more blurry, which may be caused by applying too low voltage. For samples 2 and 4 nanofibers are clear, but have defects in the form of beads. With the increase of distance between the nozzle and the collector of 10 cm, and while reducing the voltage of 5 kV diameter of the obtained nanofiber is reduced by approx. 20 nm.

PVA matrix composite nanofibers reinforced with the Cu nanoparticles without visible defects in the form of beads and blur was achieved only in the case of sample 3.

During the electrospinning process, the liquid spurt was stable only in the case of sample 1. During the producing of sample 2 and 5 liquid spurt was unstable while in the case of samples 3 and 4 occurred multispurt. The obtained results have the influence of applied process parameters on the morphology of the nanofibers.

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References

[1] Applied Nanotechnology, The Conversion of Research Results to Products, A volume in Micro and Nano Technologies (2014) 49-60.

[2] A. Khalil, B. Singh Lalia, R. Hashaikeh, M. Khraisheh, Electrospun metallic nanowires: Synthesis, characterization, and Applications, Journal of Applied Physics 114 (2013) 171301.

[3] J. Doshi, D.H. Reneker, Electrospinning process and application of electrospun fibers., Journal of Electrostatics 35 (1995) 151-160.

[4] S. Ramakrishna, K. Fujihara, W.E. Teo, T.Ch. Lim, Z. Ma, An introduction to electrospinning and nanofibers. Word Scientific Publ., Singapore, 2005.

[5] L.A. Hoover, J.D. Schiffman, M. Elimelech, Nanofibers in thin-film composite membrane support layers: Enabling expanded application of forward and pressure retarded osmosis, Desalination 308 (2013) 73-81.

[6] K. Kurzydłowski, M. Lewandowska, Structural and functional engineering nanomaterials, Scientific Publishing PWN, Warsaw, 2011 (in Polish).

[7] G.S. Lotey, S. Kumar, N.K. Verma, Fabrication and electrical characterization of highly ordered copper nanowires, Applied Nanoscience 2/1 (2012) 7-13.

[8] X. Li, M.A. Kanjwal, L. Lin, I.S. Chronakis, Electrospun polyvinyl-alcohol nanofibers as oral fast-dissolving delivery system of caffeine and riboflavin, Colloids and Surfaces B: Biointerfaces 103 (2013) 182-188.

[9] S. Sundarrajan, D. Pliszka, A. Jaworek, A. Krupa, M. Lackowski, S. Ramakrishna, A novel process for the fabrication of nanocomposites membranes, Journal of Nanoscience and Nanotechnology 9 (2009) 4442-4447.

[10] F. Pavanello, S. Giordano, How imperfect interfaces affect the nonlinear transport properties in composite nanomaterials, Journal of Applied Physics 113/15 (2013) 154310.

[11] D. Li, Y. Xia, Electrospinning of Nanofibers: Reinventing the Wheel?, Advanced Materials 16/14 (2004) 1151-1170.