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THE INFLUENCE OF ELECTROSPINNING PROCESS PARAMETERS ON THE MORPHOLOGY OF THE PVP NANOFIBERS

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Abstract:
The article describes the investigation of the effect of electrospinning process parameters wherein the distance between the electrodes was variable, the voltage and the flow rate of the polymer solution was constant for the obtained polymer nanofibers from the solution of PVP/EtOH with concentration of 10% by weight. The morphology of produced nanofibers was examined using a scanning electron microscope (SEM). Based on the SEM images a series of measurements of the nanofibers diameters for each sample were executed and then the results were averaged. The research shows that reducing the distance between the electrodes has a significant impact on the diameter of the PVP nanofibers.

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1. INTRODUCTION

People have been using fibers for centuries. In the year 5,000 BC, our ancestors used natural fibers such as wool or silk. Changes in the chemical, electronic and mechanical industry led to introduction of the new synthetic fibers such as nylon, polypropylene or polyester. Requirements and results in further progress led to producing high-functional fibers such as carbon fibers, viscose and aramid, high strength, high elastic modulus and good thermal resistance. Electrospun nanofibers can be divided into four categories: electro-nano composite nanofibers, metalloporabic nanofibers, electrophobic nanofibres for electrospun, and electrolyte nanofibres [1]. Traditional methods of making polymer fibers include spinning polymer melts, dry or wet silica gel. Those methods have provided possibility for obtaining fibers in the range of 5-500 micrometers. A typical process uses the electrical potential between the solution drop and the grounded collector. In the electrospinning process, polymeric nanofibres can be formed when the droplet of light elastic polymer is exposed to a high-voltage electrostatic field. The solvent droplets move in the air and evaporate leaving fibers that can settle on the substrate. Properties of untreated nanofibers, including crystallinity, mechanical properties and bio-functionality, are controlled and dependent on many parameters such as polymer solution, temperature, humidity and polymer type. Electrospinning is a technique that uses electrical power to process and produce fibers from a solution. Typical electrospinning devices consist of high-voltage power supply, syringe, metal nozzle and conductive collector (Fig. 1) [1-3].

Studies on electrically driven liquid streams were started in the XIX century. For example, John Francis Cooley filed the first patent for a device for electrical installation in 1900. Recent efforts to understand the physics of fluids in the electromagnetic field received a significant contribution from Johna Zeleny mathematics in 1914. Electrospinning of polymer fibers was first patented by Formhals in 1934. In 1960 Geoffrey

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Taylor, an eminent fluid dynamics scientist, described the cone formed by the droplet under the action of the electric field, which later was called the Taylor cone [2,16].

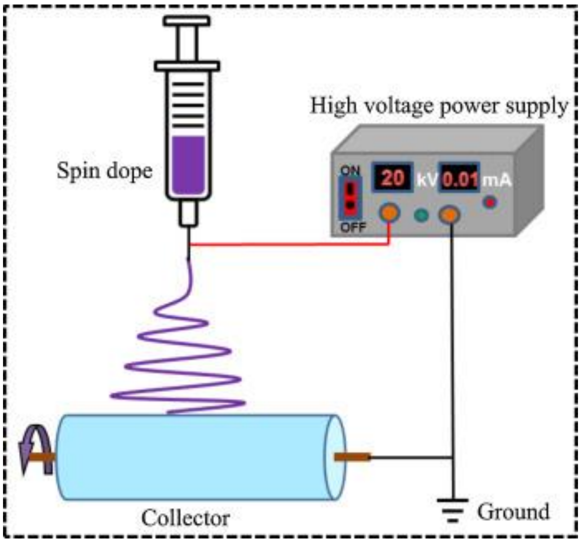

Fig. 1. Schematic illustration of an electrospinning process [18]

Nanofibers are used in many fields, such as medicine, pharmacy and automotive industry. They are applied in the production of organic-inorganic composites, absorbing layers, drug transporters and also in fuel cells, batteries, ionic or electronic sensors [4]. This is mainly caused by characteristic properties of nanofibers like very large surface to volume ratio, unique flexibility of physical or chemical changes. The physical and chemical properties of nanofibers are largely dependent on the shape, size and morphology of nanofibres [5].

Poly(vinylpyrrolidone) is a synthetic polymer exhibiting good compatibility and transparency. It is tested for antibacterial activity, biocompatibility and possible use in biomedical applications [6]. The described polymer is non-toxic, physiologically compatible, chemically neutral, temperature and pH resistant and exhibits high stability in such conditions. Moreover, the PVP is characterized by good solubility in water and other organic solvents [5, 6].

The PVP nanofibers were object of many researches, either for use as a crystallization inhibitor in medicines hydrophobic carrier cancer drugs or as dressings, ability to release the bioactive substances [7]. The test was either concentration effects or changes in high voltage on the diameter and length of the fibers, as well as the possibility of doping of additional fiber particles, silver or titanium oxide. [6-9]. The polymer is sensitive to ammonia and aliphatic amines, because it acts as a hydrogen bond acceptor groups on both the carbonyl and nitrogen, so that the PVP is used in sensors for detecting even the small concentration of hydrogen [10].

The process of electrospinning is one of the polymer making technique, whose aim is to obtain nanofibers. It is the cost-effective and versatile method of producing multifunctional fibrous mats with diameters from several tens of nanometers to microns [2, 4, 11, 12-17]. Some researches served an attempt to optimize the process of electrospinning and analyze the impact of the distance between the electrodes on the diameter of the resulting PVP nanofibers. The polymer fibers with small diameters are small frequency offset. This is due to the small surface area relative to the distance. This is particularly important because fibers are used in the PVP hydrogen sensors, smaller diameter fibers result in increased detection sensitivity of gas [9]. Nanofibers can be easily enriched with particles of other materials that affect their properties. It has been shown that nanoparticles built into nanofibers increase the thermal stability of the composite compared to pure polymer. Nanoparticles have an influence on the thermal stability of nanocomposites. By embedding different nanoparticles, unique physical properties were observed. The introduction of a metal nanotube or its compound (e.g. oxide) into a polymer matrix is primarily aimed at obtaining appropriate electrical properties, including conductive, semi conductive, antistatic. The most commonly used metals in polymer nanocomposites are silver, gold and copper. In contrast, metal oxides (Fe_2O_3, Fe_3O_4) with ferromagnetic properties or titanium dioxide (IV), whose presence in the nanocomposite results in a change in optical properties compared to the corresponding properties of the polymer, are among the metal compounds [14-16]. The SEM or AFM microscopes are used for imaging of nanofibers. They can also be investigated by the gel chromatography, elemental analysis, X-ray spectroscopy and Fourier spectroscopy - infrared spectroscopy [1, 9, 13, 19].

2. MATERIALS AND METHODS

2.1 MATERIALS

In the preparation of a spinning solution polymer poly(vinylpyrrolidone) (PVP, Mw = 1 300 000 g/mole, purity 99%, Sigma Aldrich) was used. Polymer powder was dissolved in solvent, which was ethanol (EtOH, purity 99,8%). The measured amount of PVP was added to ethanol and so obtained solution was mixed on magnetic stirrer for 24 h at room temperature. The final product, PVP/EtOH solution with 10% concentration by weight was placed in device pomp, which was a sterile syringe.

2.1 METHODS

The electro spinning process was conducted on the FLOW-Nanotechnology Solutions Electro spinner 2.2.0-500 using process parameters summarized in Table 1. The effect of the process was the thin PVP fibrous mats.

Tab. 1. Selection of the electro spinning process parameters used during the sample preparation (U - voltage between the electrodes, V - solution flow rate)

Parameters	Sample 1	Sample 2	Sample 3
U [kV]	20	20	20
d [cm]	10	15	20
V [mL/h]	1.5	1.5	1.5

3. RESULTS AND DISCUSSION

The surface morphology investigation for sample 1 is shown in Fig. 2, which was made at the smallest distance between electrodes of 10 cm having the highest density. It was observed that applying the smallest distance between electrodes is causing production of nanofibers in the most common diameter value range of 600 to 800 nm (Fig. 2). Moreover, the average diameter of the nanofibers of this sample was 809 nm, which was the highest diameter value for obtained nanostructures (Fig. 5). Increasing the distance between the electrodes caused a decrease in the density and diameter of the nanofibers.

For sample 2, obtained by a distance of 15 cm between the nozzle and the collector, the largest amount of nanofibers formed in the diameters range of 200 to 400 nm (Fig. 3) and the average diameter of the obtained fibers is lower by 297 nm to the result obtained for sample 1 (Fig. 5).

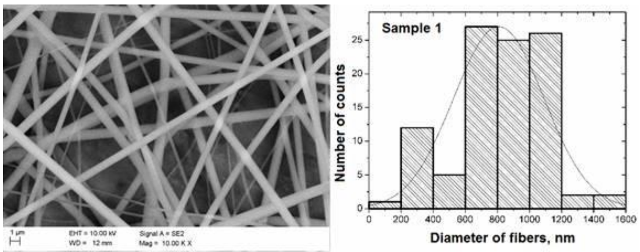

Fig. 2. The SEM image of the obtained PVP nanofibers for sample no 1 and histogram showing the values of nanofibers diameters

Using the greatest distance between the electrodes of 20 cm resulted in obtaining fiber mats with the smallest number of fibers per unit area (Fig. 4). Measurement of fiber diameters applied for this sample showed that the largest group of diameters values included nanofibers in the range up to 200 nm. Determined value of the average fiber diameter of the sample was the smallest 402 nm (Fig. 3) and shows a significant effect of the change in the parameter for the diameter of PVP nanofibers.

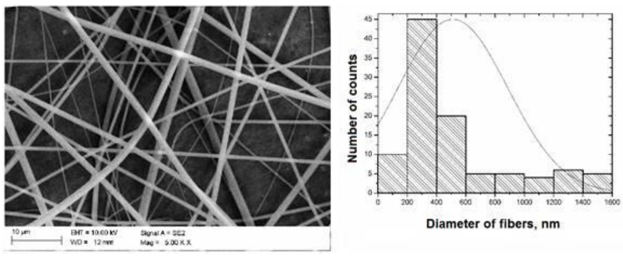

Fig. 3. The SEM image of the obtained PVP nanofibers for sample no 2 and histogram showing the values of nanofibers diameters

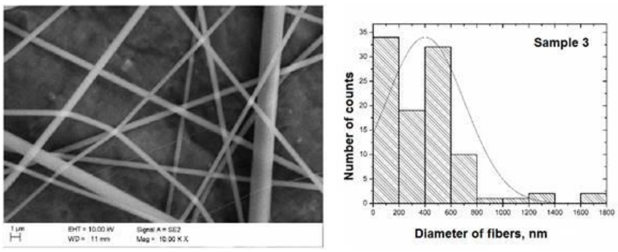

Fig. 4. The SEM image of the obtained PVP nanofibers for sample no 3 and histogram showing the values of nanofibers diameters

Furthermore, it is observed that for each sample, 1, 2, 3 electro spinning process resulted in making thin fibrous mats with randomly formed fibers. In addition, each fiber is characterized by absence of any structural defects, which means that the applied process parameters were properly used.

These observations show that process parameters have decisive effect on the morphology of obtaining the PVP nanofibers. Observed decrease in fiber diameter with increasing distance may result from an increase in the stream flight path resulting in larger stretching of the polymer solution along the way to the collector. This is due to the presence of electrostatic force (Coulomb force) operating in the induced charge on the surface of a drop of the solution coming out of the nozzle. With increasing distance between the electrodes, the Coulomb force acting on the drop of the solution coming out of the nozzle decreases, but prolonged duration of activity, which results in increased tension of the liquid polymer flow and the formation of fibers with smaller diameters [10-11,20].

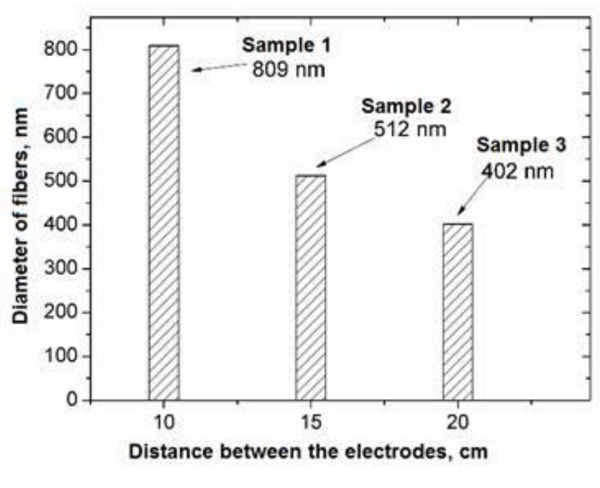

Fig. 5. Histogram showing the average nanofibers diameters for the obtained fibrous mats with various distance between the electrodes

4. CONCLUSION

The PVP nanofibers were successfully prepared from the liquid polymer solution with 10% concentration using electro spinning method. Parameters used in spinning in electrostatic field, constant flow rate and high voltage and variable distance between the electrodes resulted in making thin poly(vinylpyrrolidone) fibrous mats. The investigation of diameters values showed that with the increase in distance between the nozzle and the collector, the values of diameters decrease. It is mainly caused by the Coulomb forces, which occur in the polymer stream and increase the tension of polymer drop. The fibers obtained in the electrospinning process have a high density of applications because of their unique properties. The optimization process allows the formation of specific properties of nanofibers such as their diameter or arrangement. This allows fiber applications in areas such as electronics or medicine.

5. REFERENCES

[1] J. Zhu, Y. Ge, S. Jasper, X. Zhang, Physical characterization of electrospun nanofibers. Electrospun Nanofibers, A (-), 2017: pp.207-238. doi.org/10.1016/B978-0-08-100907-9.00009X

[2] W. Matysiak, T. Tański, M. Zaborowska, Analysis of the Optical Properties of PVP/ZnO Composite Nanofibers, Properties and characterization of modern materials. Eds.: Andreas Ochsner, Holm Altenbach, Springer, Singapore, 2017: p.43-49.

[3] D. Yu, K. White, J. Yang, X. Wang, W. Qian, Y. Li, PVP nanofibers prepared using co-axial electrospinning with salt solution as sheath fluid. Materials Letters, 67 (1), 2012: pp.78-80. doi.org/10.1016/j.matlet.2011.09.035

[4] T. Tański, W. Matysiak, Ł. Krzemiński, Analysis of optical properties of TiO_2 nanoparticles and PAN/TiO_2 composite nanofibers. Materials and Manufacturing Processes, 2017. (In press) dx.doi.org/10.1080/10426914.2016.1257129

[5] E. Ghafari, Y. Feng, Y. Liu, I. Ferguson, N. Lu, Investigating process-structure relations of ZnO nanofiber via electrospinning method. Composites Part B: Engineering, 116 (1) 2017: pp.40-45. doi.org/10.1016/j.compositesb.2017.02.026

[6] D. Archana Brijesh, K. Singh, J. Dutta, P. K. Dutta, Chitosan-PVP-nano silver oxide wound dressing: In vitro and in vivo evaluation. International Journal of

Biological Macromolecules, 73 (-), 2015: pp.49-57.

[7] S. Liparoti, R. Adami, E. Revechon, Super critical Assisted Atomization: effect of operative conditions on PVP micro particle size and morphology. The Journal of Supercritical Fluids, 97 (-) , 2015, pp.31-35. doi.org/10.1016/j.supflu.2014.10.028

[8] T. Tański, W. Matysiak, B. Hajduk, Manufacturing and investigation of physical properties of polyacrylonitrile nanofibre composites with SiO_2, TiO_2 and Bi_2O_3 nanoparticles, Beilstein Journal of Nanotechnology, 7 (-), 2016: pp.1141-1155. doi:10.3762/bjnano.7.106

[9] X. He, R. Arsat, A.Z. Sadek, W. Wlodarski, K. Kalantar-Zedeh, J. Li, Electrospun PVP fibers and gas sensing properties of PVP/36° YXLiTaO3 SAW device. Sensors and Actuators B: Chemical, 145 (2), 2010: pp.674-679. dx.doi.org/10.1016/j.snb.2010.01.013

[10] S. Chuangchote, T. Sagawa, S. Yoshikawa, Electrospun PVP fibers and gas sensing properties of PVP/36° YXLiTaO3 SAW device. Journal of Applied Polymer Science, 112 (1), 2009: pp.277-279.

[11] D. Yu, K. White, J. Yang, X. Wang, W. , Ying Li, PVP nanofibers prepared using co-axial electro spinning with salt solution as sheath fluid. Materials Letters, 67 (1), 2012: pp.78-80. doi.org/10.1016/j.matlet.2011.09.035

[12] Q.Q. Ni, X.F. Jin, H. Xia, F. Liu, 7 - Electrospinning, processing and characterization of polymer-based nano-composite fibers. Advances in Filament Yarn Spinning of Textiles and Polymers, Woodhead Publishing, 2014: p.128-148.

[13] D. Archana B.K. Singh, J. Dutta, P.K. Dutta, Chitosan-PVP-nano silver oxide wound dressing: In vitro and in vivo evaluation. International Journal of Biological Macro molecules, 73 (-), 2015: pp.49-57.

[14] M. Rasekh, C. Karavasili, Y.L. Soong, N. Bouropoulos, M. Morris, D. Armiitage, X. Li, D.G. Fatouros, Z. Ahmad, Electrospun PVP–indomethacin constituents for transdermal dressings and drug delivery devices. International Journal of Pharmaceutics, 473 (1-2), 2014: pp.95-104. dx.doi.org/10.1016/j.ijpharm.2014.06.059

[15] A. Sionkowska, M. Wisniewski, H. Kaczmarek, J. Skopinska, P. Chevallier, D. Mantonovan, S. Lazare, V. Tokarev, The influence of UV irradiation on surface composition of collagen/PVP blended films, Applied Surfaces Science, 253 (4), 2006: pp.1970-1977. doi.org/10.1016/j.apsusc.2006.03.048

[16] L.A. Mercante, V.P. Scagion, F.L. Migliorini, L.H.C. Mattoso, D.S. Correa, Electrospinning-based (bio)sensors for food and agricultural applications: A review. TrAC Trends in Analytical Chemistry, 91 (-), 2017: pp.91-103. doi.org/10.1016/j.trac.2017.04.004

[17] Y. Qian, Y. Su, X. Li, H. Wang, C. He, Electrospinning of Polymethyl Methacrylate Nanofibers in Different Solvents. Iranian Polymer Journal, 19 (2), 2010: pp.123-129.

[18] Y. Dinga, H. Houa, Y. Zhaob, Z. Zhub, H. Fong, Electrospun polyimide nanofibers and their applications. Progress in Polymer Science, 61 (-), 2016: pp.67-103. doi.org/10.1016/j.progpolymsci.2016.06.006

[19] H. Albetran, Y. Dong, I. M. Low, Characterization and optimalization of electrospun TiO2/PVP nanofiber using Taguchi design of experiment method. Journal of Asian Ceramic Societies, 3 (3), 2015: pp.292-300. doi.org/10.1016/j.jascer.2015.05.001

[20] M. M. Aria, F. Razi Astaraei, S. Sharait, R. Shariat, Ethanol sending properties of PVP electrospun membranes studies by quartz crystal microbalance. Measurement, 78 (-), 2016: pp.283-288. doi.org/10.1016/j.measurement.2015.10.018

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