DOI 10.31489/2023PH2/79-92

UDC 678.076

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The current state of electrospinning technology and its prospects for the future

With the development of nanotechnology and modern research methods, the use of biodegradable polymer materials in various sectors of human life is of interest not only to the scientific world community, but also is a way to solve one of the global problems related to resource conservation and environmental protection. Polyvinyl alcohol-based materials have been widely used in various fields due to their biological and physical properties, such as biocompatibility, biodegradability, antimicrobial ability, non-toxicity and the ability to easily form a film. One of the methods of obtaining polymer films that exist today is electrospinning, the advantages of which are the relative simplicity of the technological process and the possibility of obtaining continuous nanofibers from both synthetic and natural polymers. In this work, the influence of various process parameters on the formation of nanofiber mats from a biodegradable synthetic polymer by electrospinning was studied. The technology of wet spinning, melt spinning and dry spinning is discussed. A number of experimental studies have been carried out to identify optimal modes of obtaining nanofibers from polyvinyl alcohol with the most homogeneous structure without the formation of defects.

Keywords: electrospinning, technological parameters, nanofibers, polyvinyl alcohol (PVOH), nonwoven mats, SEM, nanofiber diameter, biodegradable polymer material.

Introduction

Over the past few decades, nanomaterials research has been one of the most popular topics in the field of nanotechnology. To date, nanomaterials have found their potential application in various fields of research and industry due to their unique properties. Petroleum-based polymers are widely used in the production of various polymer products for various commercial, machine-building and promising purposes. However, the depletion of natural resources and the increasing demand for polymer-based materials raises questions about the further production of synthetic polymers. Moreover, most of the petroleum-based polymers used are not biodegradable and are stored in the environment as non-degradable waste. To solve these issues, many studies have been directed at the production of biodegradable polymers from renewable resources [1] and the development of technologies for the production of nanostructured polymer fibers, one of which is electrospinning.

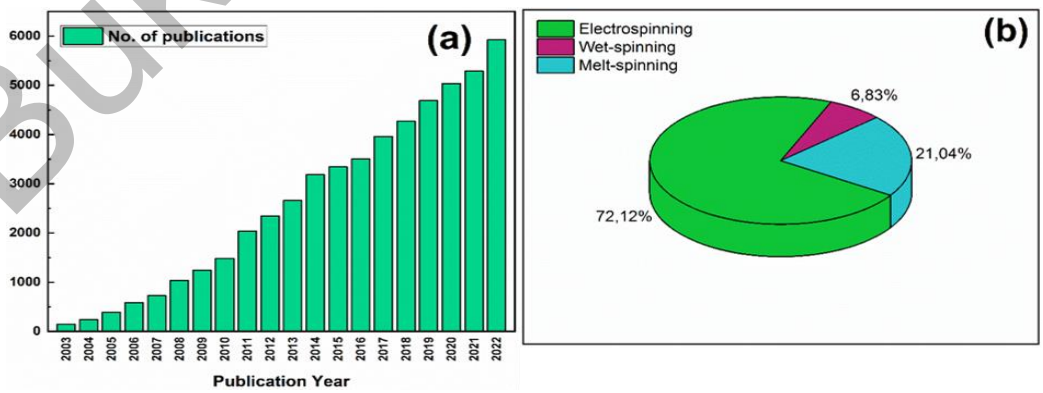


Figure 1. (a) Comparison of the annual number of scientific publications in the field of electrospinning over the past two decades and (b) the ratio of publications in the main areas of electrical engineering (the analysis of these publications was carried out using a single database of peer-reviewed scientific literature SCOPUS with the term "Electrospinning" as of February 15, 2023)

Due to the relative simplicity of the technological process that makes it possible to obtain multifunctional polymer mats from nanofibers, this technology is attracting increasing attention from the world scientific community, as evidenced by the increasing number of scientific publications in this field over the past two decades (Fig. 1).

Spinning is a technology for making fibers by pulling and twisting natural or synthetic materials, which is classified depending on the type of fiber as mechanical and chemical spinning. Mechanical spinning is a multi-step process in which fibers are physically twisted into yarn. Namely, rotary, annular, frictional or self-twisting rotation [2]. The production of threads from artificial fibers is also possible using chemical spinning. This is achieved by squeezing a viscous polymer solution through a die. There are three main types of production processes for the formation of synthetic fibers, namely wet spinning, melt spinning and dry spinning [3]. The wet spinning process is when the solidification of the dissolved polymer occurs after diffusion in the counterflow between the spinning solution and the coagulation bath. In this process, a very viscous polymer solution is squeezed out through small holes of a die immersed in a liquid bath. A diagram of the wet spinning process is shown in Figure 2. The technological analysis of the wet spinning process is much more complicated than dry spinning. Polymer solidification occurs as a result of diffusion exchange between freshly prepared fluid filaments and this bath. During this coagulation process, one or more components of the bath diffuse into the thread, and the solvent diffuses out of it.

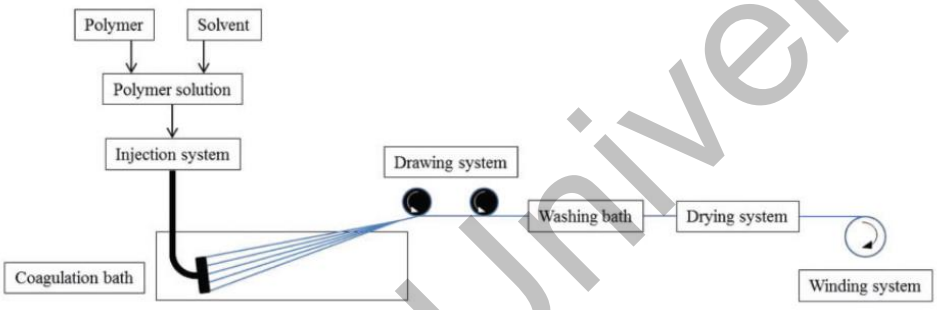


Figure 2: Diagram of the wet spinning process [4]

In melt spinning, phase transformations occur due to solidification of the molten mass. This type of spinning is the most economical process for the production of polymer fibers on an industrial scale [5]. The comparative ease of processing is an important advantage of melt molding. However, this method has some disadvantages, such as fiber rupture, non-uniform thread thickness, fiber fineness limit and die clogging (diagram of the process see in Figure 3).

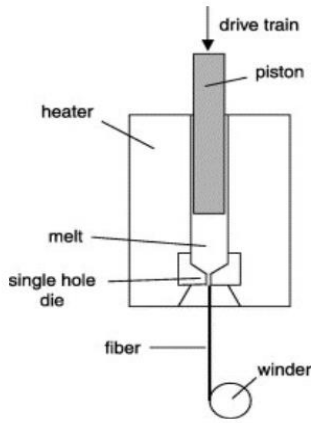


Figure 3. Diagram of the melt spinning process [6]

During dry spinning, the solvent contained in the spinning solution evaporates after the formation of a nanofiber mat, which leads to its drying. In the process of dry spinning, the fiber structure is formed by

squeezing the polymer solution through a thin nozzle and subsequent evaporation of the solvent (diagram of the process see in Figure 4).

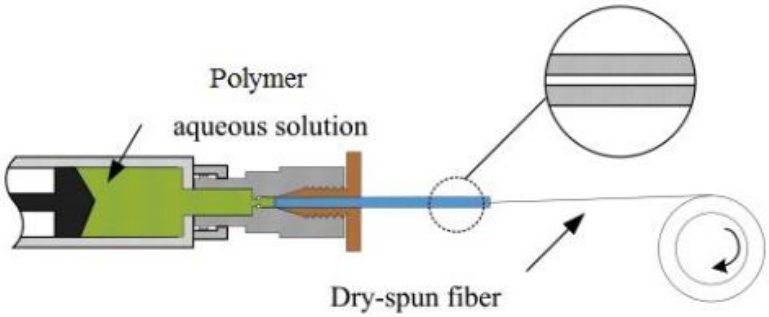


Figure 4. Diagram of the dry spinning process [7]

Electrospinning is a dry spinning method in which the solvent evaporates under high voltage [8]. This method makes it possible to produce ultrathin fibers for various polymers of submicron or nanometer size.

The principle of electrospinning is based on the deformation of a charged polymer in response to a strong applied electric field. The polymer solution is fed (e.g. by gravity) to a nozzle opposite an electrode. In the absence of an electric field the polymer solution surface forms a meniscus at the nozzle outlet. In an electric field, however, the meniscus deforms into a so-called Taylor cone and, if the field is strong enough, the electrostatic forces can overcome the surface tension and a jet is formed. The acceleration of this jet towards the electrode causes the thinning of the jet, and the evaporation of the solvent as the jet is accelerated towards the electrode leads to solid fibre formation at atmospheric pressure and ambient temperature [9].

Thus, the purpose of this work is to obtain nanofibers by dry electrospinning and to study the effect of voltage, the distance from the collector to the nozzle, and the formation time in the production of polymer fibers.

Experimental

This study was conducted at the laboratory of Lincoln University, Isaac Newton Building, UK. At the Electrospinz Nanofibre Engineering installation, nanofibers were obtained by electrospinning in an atmospheric environment on the surface of substrates (material: deltalab laboratory glass with dimensions of 26 x 76 mm^2) at room temperature of 20 °C and humidity of 33 % (Fig. 6). Evaporable material poly vinyl alcohol "PVOH" with added antiomicrobial preservatives (concentration: 8wt % PVOH Solution; 0.1wt % Sodium Benzoate; 0.1wt % Porassium Sorbate) is an environmentally friendly biodegradable synthetic polymer, which is widely studied due to its high film-forming and physical properties, as well as its high hydrophilicity, manufacturability, biocompatibility and chemical resistance [10-14]. Figure 5 shows a schematic molecular formula PVOH $(C_2H_4O)_x$.

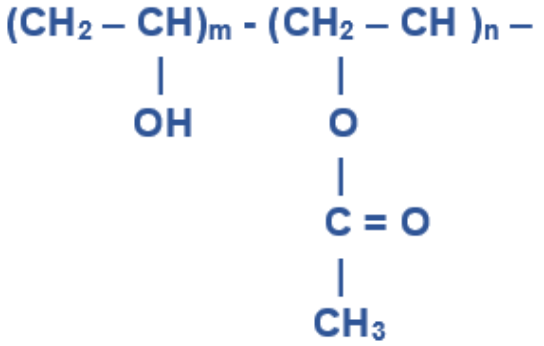
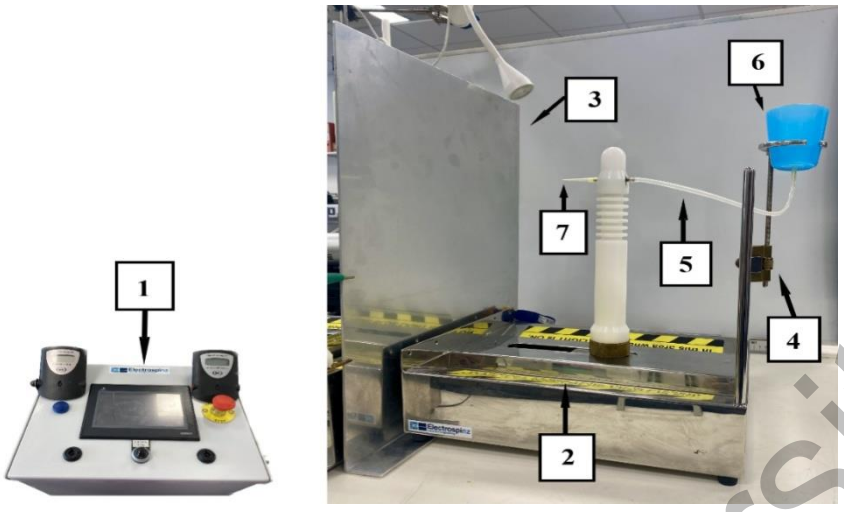


Figure 5. General structure of PVOH

During the electrospinning process, the applied electrostatic forces overcome the surface tension of the liquid, so the electrified liquid forms a jet from the tip of the capillary to the grounded filter on which the nanofiber material is formed. The appearance and scheme of the installation for electric spinning are shown

in Figures 6 and 7. The flow rate was controlled by gravity, depending on the height of the filled polymer in the container.



1 — High voltage and Control box, 2 — Spinning platform, 3 — Target plane, 4 — Constant head system, 5 — Hose, 6 — Header tank, 7– Nozzle.

Figure 6. Appearance of the installation “ELECTROSPINZ NANOFIBRE ENGINEERING”

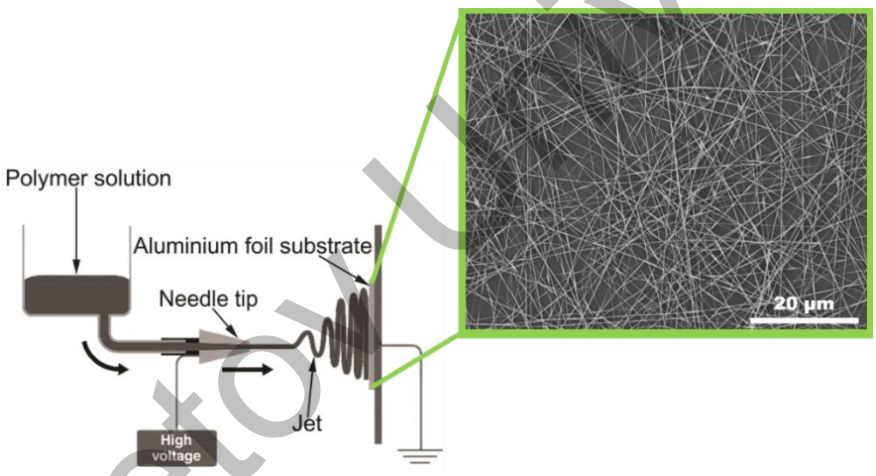


Figure 7. Installation diagram for electrospinning [15]

To provide conductive surfaces, nanofibers assembled on a collector (aluminum foil) by electrospinning were sprayed with a thin layer of gold on an “Emitech K550X” magnetron sputtering unit (Fig. 8) with an “Edwards RV8” vacuum pump, an argon pressure regulator. When using magnetron technology, the deposition process is quite “cold” and minimizes thermal damage to soft polymer materials.

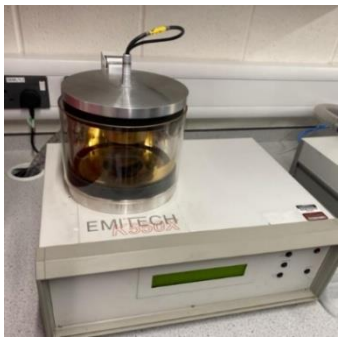


Figure 8. Appearance of the “Emitech K550X” installation

The morphology and size of PVOH nanofibers were observed and analyzed using a scanning electron microscope (SEM) “JeolNeoscope JCM 5000” at high vacuum with accelerating voltages of 10 and 15 kV, depending on the required magnification.

Results and Discussion

Some of the first results of the study obtained by the authors of this work were obtained of nanofibers by the electrospinning method are presented. Variables such as the applied electrical voltage, the duration of the process and the change in the distance from the collector to the needle were investigated. Their relationship with the microstructure of the obtained new fibers is briefly described below. Table 1 lists the process parameters used in the study for electrospinning at various voltages, the results of which are shown in Figure 9.

Table 1

Technological parameters of the electrospinning process

№	Voltage, ± 0.1 kV	Electrospinning time, ± 0.1 min	Distance, ± 2 mm
Sample 1	12.0	5.0	140
Sample 2	15.0	5.0	140
Sample 3	18.0	5.0	140
Sample 4	21.0	5.0	140
Sample 5	24.0	5.0	140
Sample 6	27.0	5.0	140
Sample 7	30.0	5.0	140

The applied voltage is an important factor in the process of electrospinning, since it controls the electric field strength between the tip of the nozzle and the collector and therefore the acceleration rate of the polymer jet. In principle, the higher the acceleration, the more stretching that occurs, leading to thinner fibres. However, this higher acceleration also means shorter flight duration between the nozzle and electrode, which may lead to thicker than expected fibres at high electrospinning voltages. It is not clear which of these factors is the more important in general, and results may differ depending on other process parameters.

The nanofiber diameter is measured by software-nano measurer using Start JCM-5000 open source image analysis software to determine the size and average diameter of nanofibers obtained by electrospun. Based on images obtained using a scanning electron microscope at constant magnification x8000 and a voltage of 10 kV, the diameters of 30 randomly selected PVOH nanofibers at each electrospinning voltage (12.0, 15.0, 18.0, 21.0, 24.0, 27.0 and 30.0 kV) were measured, and the average value calculated. The results are shown in Table 2 and shown in Figure 10.

Table 2

Average diameter of the PVOHfiber, depending on the voltage between the nozzle and the collector

Electrospinning voltage, ± 0.1 kV	12.0	15.0	18.0	21.0	24.0	27.0	30.0
Average diameter of the PVOH nanofiber (± 5 nm)	120	133	193	174	182	190	205

It was found that when a voltage is applied above the critical, charged jets are ejected from the Taylor cone. Indeed, when an electric field is applied to a drop of polymer solution at the tip of the nozzle, the surface of the drop is charged, and the electric force exceeds the surface tension force; as a result, an electrically charged jet is formed. A higher voltage increases the electrostatic repulsion force of the charged jet, which ensures relative uniformity of the diameters of electroformed polymer nanofibers [16].

However, after a certain value of the applied voltage, drops of liquid polymer appeared at the end of the nozzle, which subsequently caused the formation of defects in the form of beads or drops over the entire surface of the nanofibers shown in Figure 11.

Thus, for PVOH (8 wt %) polymer, the SEM study showed the importance of the applied voltage of 21 kV, the relative uniformity of the diameter of the electrospinning polymer nanofibers (Fig. 9).

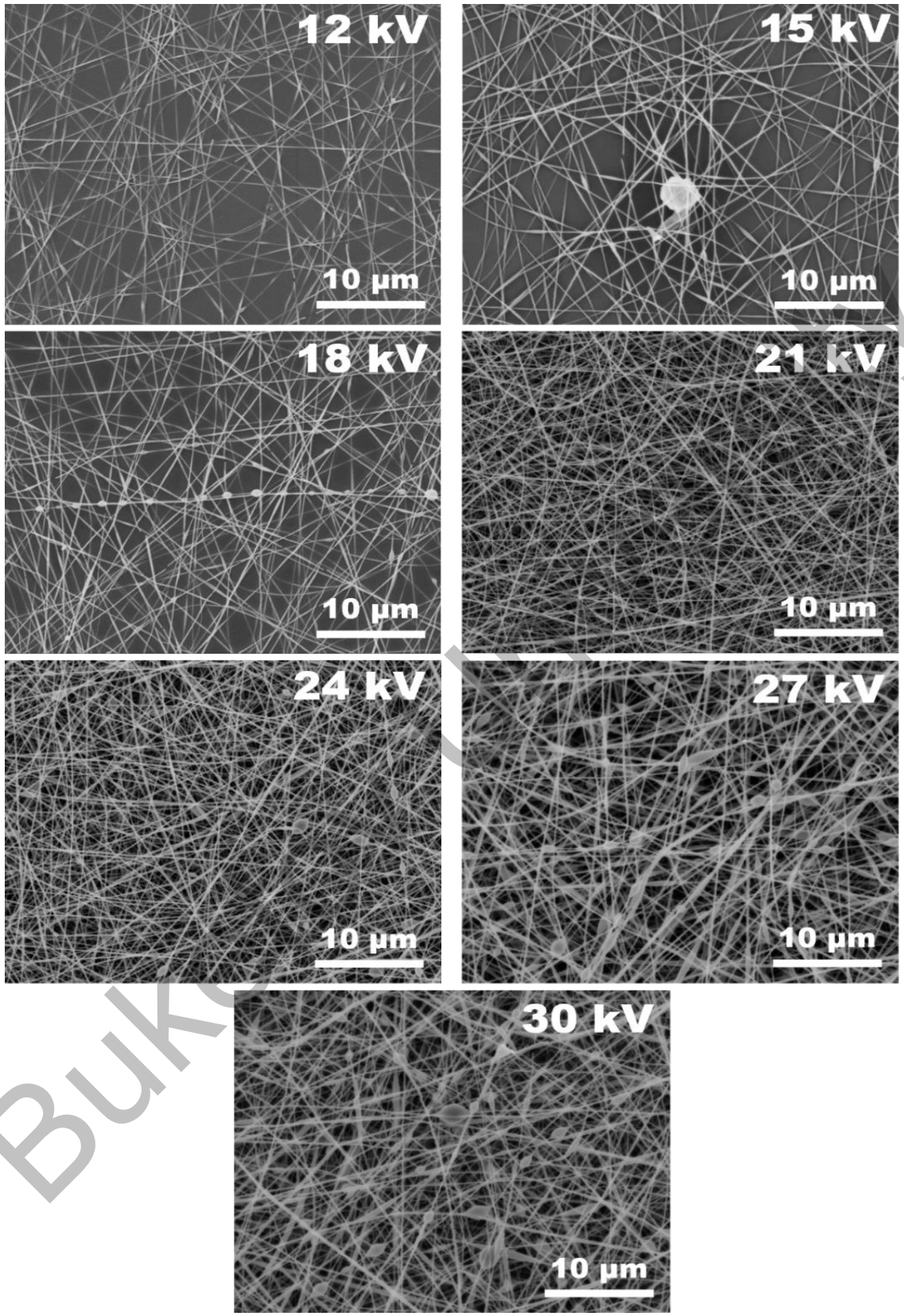


Figure 9. SEM image of the surface of the obtained nanofibers at different electrospinning voltage values

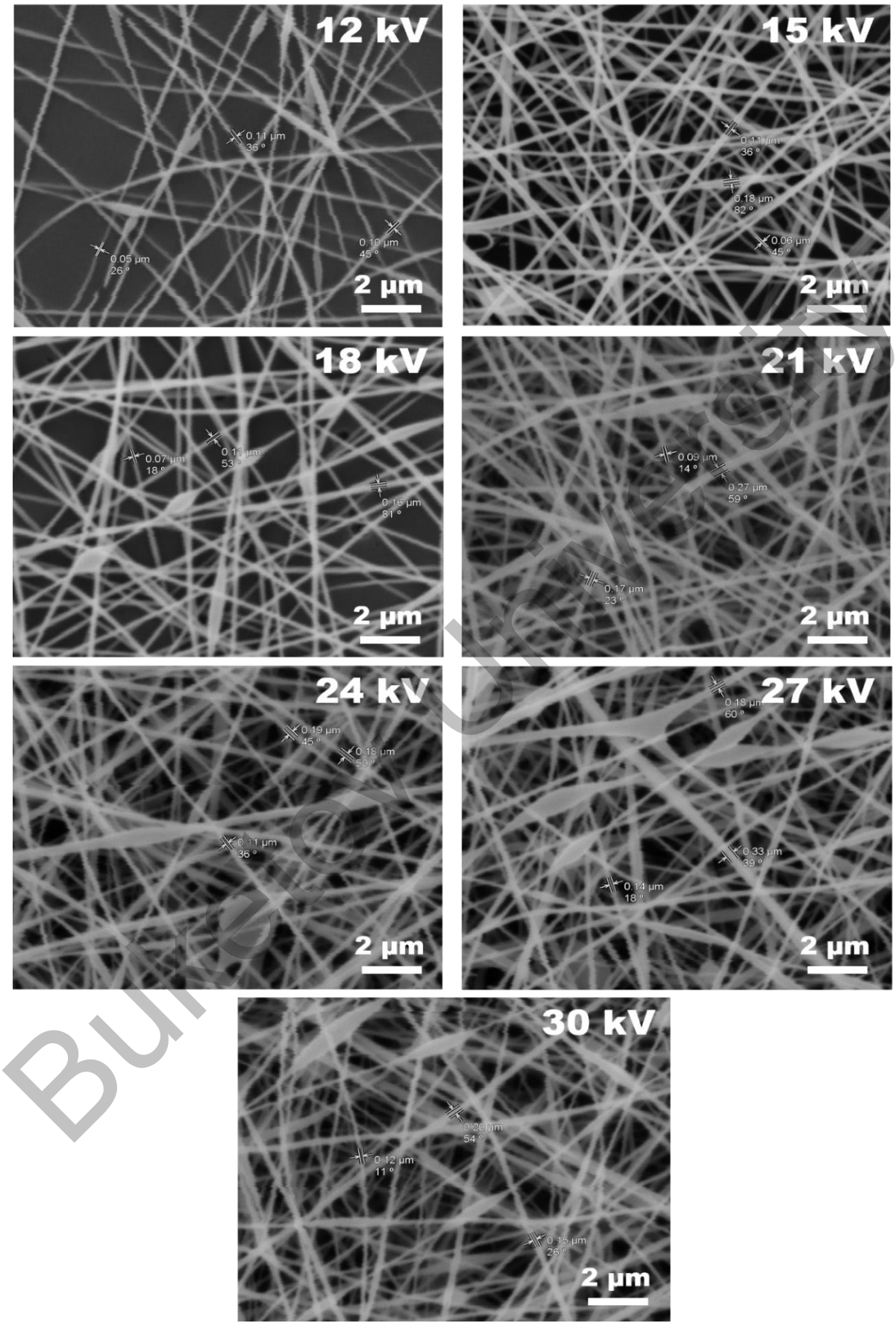


Figure 10. SEM images of PVOH nanofibers

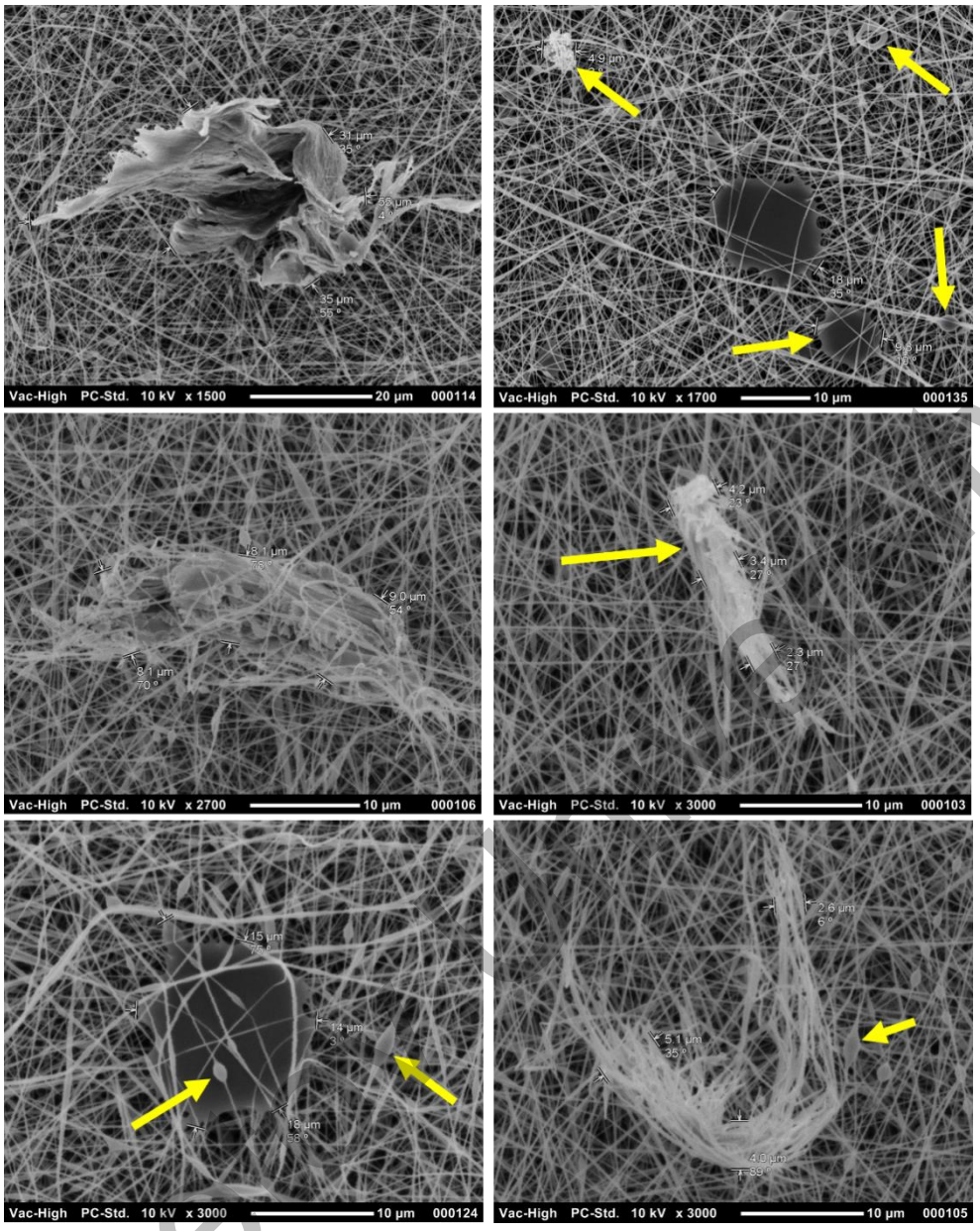


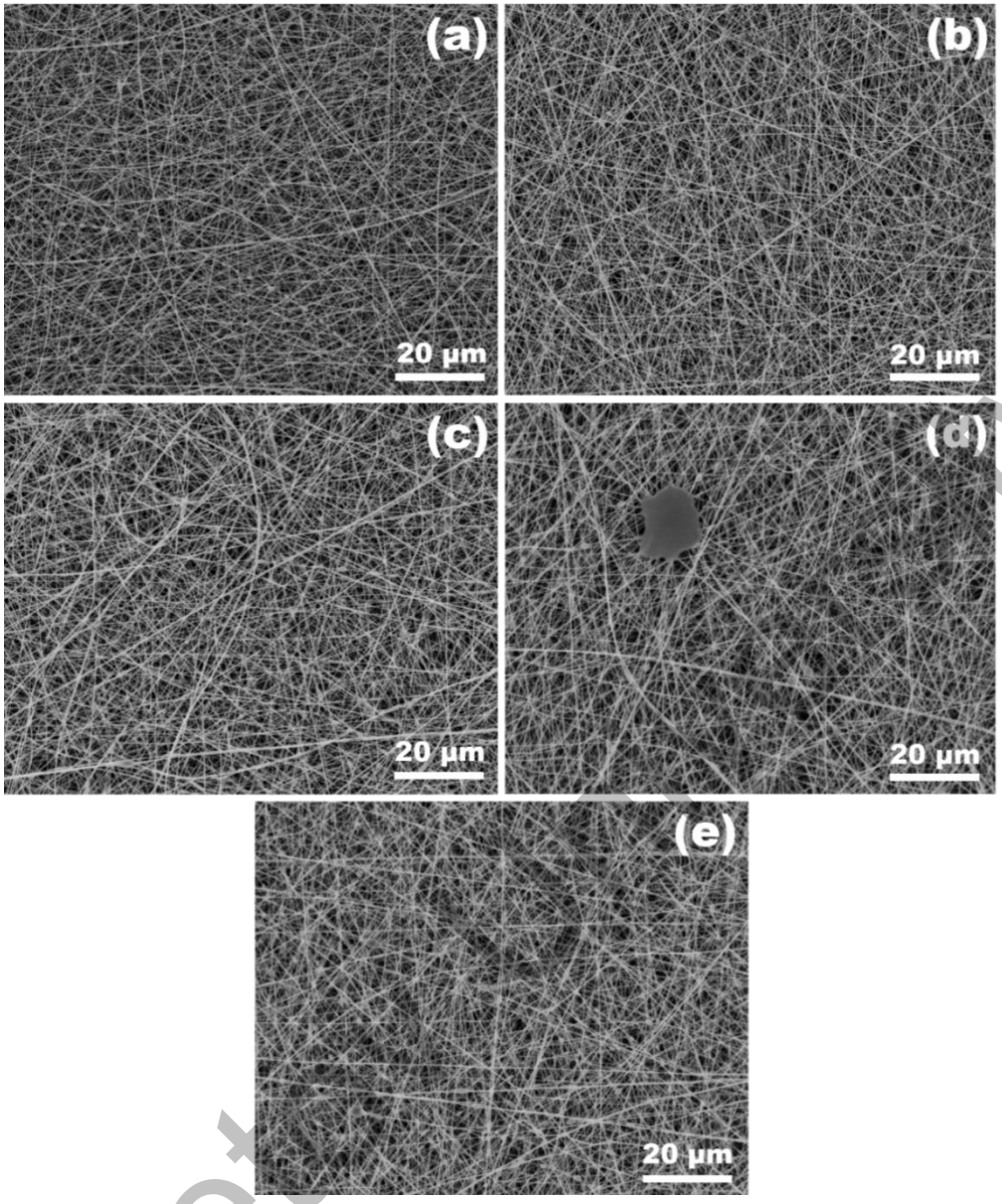
Figure 11. SEM image of defects on the surface of nanofibers at high electrospinning voltage values

The dependence of the nanofiber formation process on the deposition time, the parameters of which are presented in Table 3, was investigated. A decrease in the rate of electrospinning over time was observed, since the deposited fibre acted as an insulator allowing charge to accumulate on the target, leading to a charge screening effect between the target electrode and nozzle. The structure and thickness of the obtained fibres with varying deposition time is shown in Figure 12.

Table 3

The mode of formation of nanofibers with variable values of the duration of the process

№	Voltage, ± 0.1 kV	Electrospinning time, ± 0.1 min	Distance, ± 2 mm
Sample 1	21.0	3.0	140
Sample 2	21.0	6.0	140
Sample 3	21.0	9.0	140
Sample 4	21.0	12.0	140
Sample 5	21.0	15.0	140



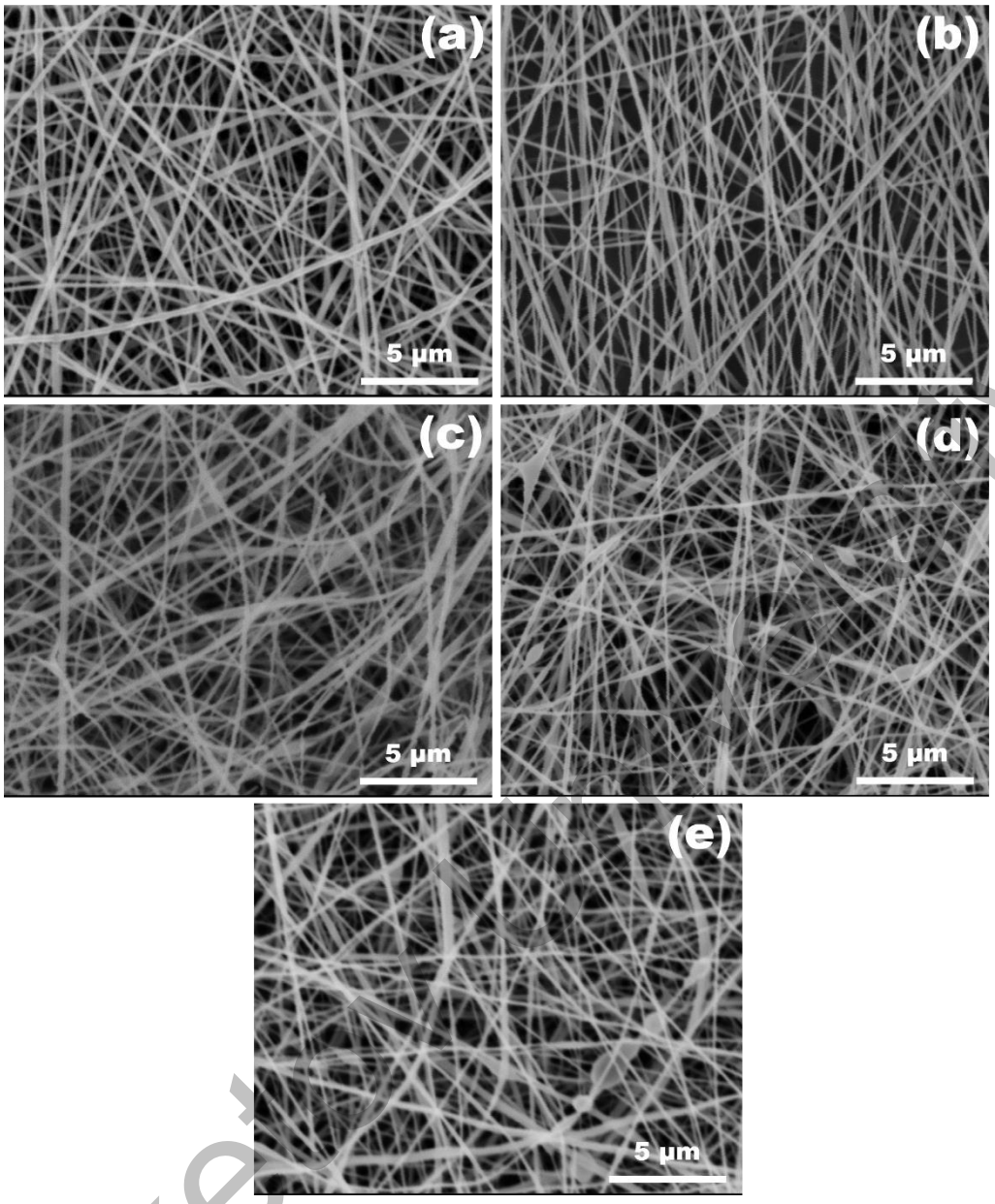
a) 3 min; b) 6 min; c) 9 min; d) 12 min; e) 15min.
Figure 12. SEM image of the surface of PVOH nanofibers

A study was conducted to identify the dependence of the nanofiber formation process on the distance between the needle tip and the collector, the values of which are presented in Table 4.

Table 4

Values of the distance between the nozzle tip and the collector during electrofspinning

№	Voltage, ± 0.1 kV	Electrospinning time, ± 0.1 min	Average distance, ± 2 mm
Sample 1	21.0	3.0	80
Sample 2	21.0	3.0	100
Sample 3	21.0	3.0	120
Sample 4	21.0	3.0	140
Sample 5	21.0	3.0	160



a) 80 mm; b) 100 mm; c)120 mm; d) 140 mm; e) 160 mm.
Figure 13. SEM image of the surface of PVOH nanofibers

Figure 13 shows that when the distance between the collector and the nozzle was 80 mm, the distribution of fibers was loose and heterogeneous. At a distance of 100-140 mm, the fibers were located closer to each other, and the pores between the fibers were relatively small. When the distance was 160 mm, the fibers were located just as close to each other, but defects in the form of beads formed over the entire surface.

Thus, reducing the distance between the electrodes to a certain value leads to a decrease in the diameter of the nanofibers. The difference in the distance between the tip of the nozzle and the collector has a direct effect on the electric field strength. The smaller the distance between the collector and the nozzle, the greater the electric field strength. As a result, the acceleration and elongation of the jet increase during the electric spinning process, and the diameter of the electric-spun polymer nanofibers decreases. In order to determine the thermal stability of the obtained nanofiber mats, annealing was carried out, the conditions of which are shown in Table 5. To minimize the ingress of various impurities on the surface of nanofibers during heating, a high-vacuum installation “NanoPVD-S10A” was used.

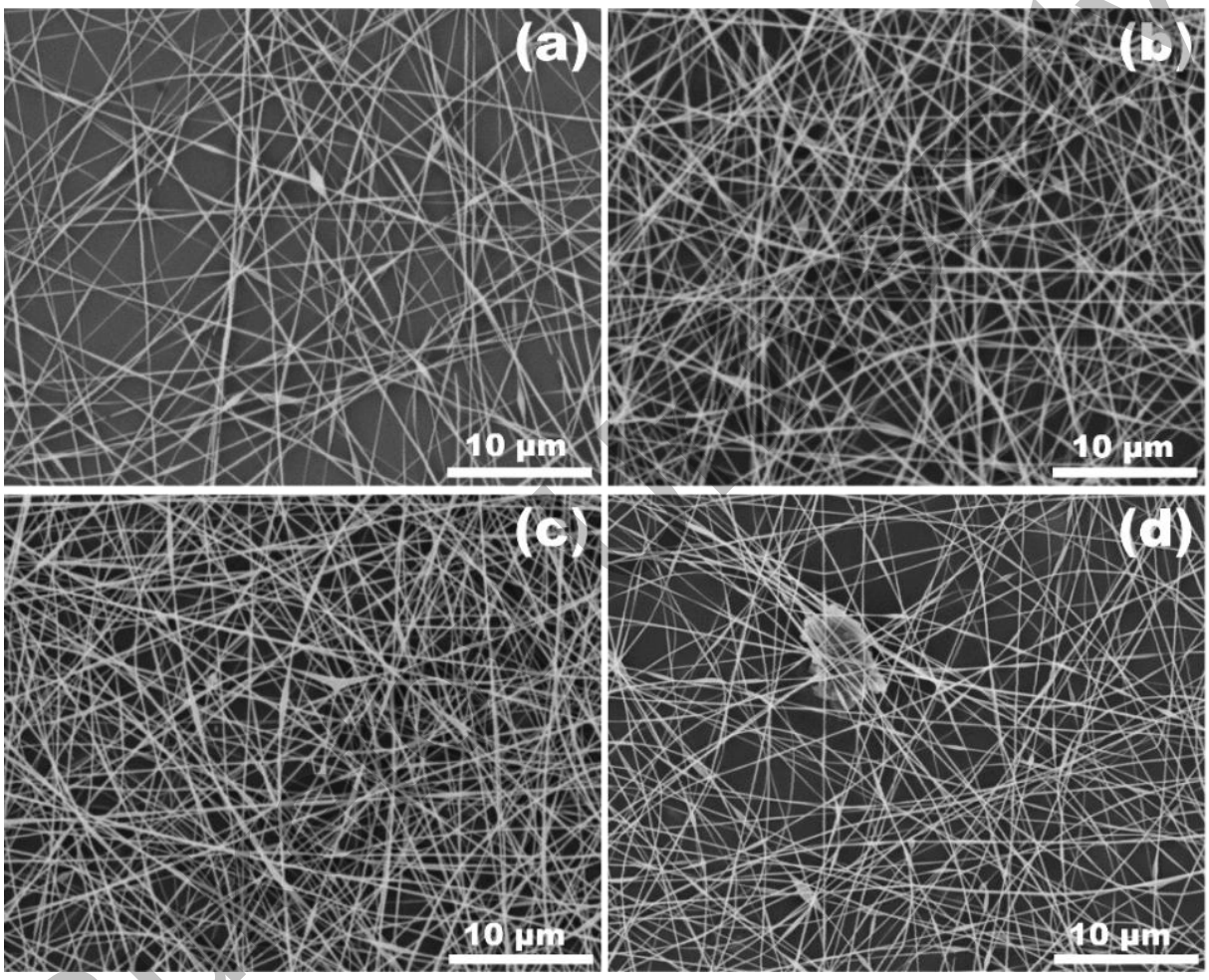
After the annealing, no significant changes in the fiber samples were observed (Fig. 14), which are associated with insufficient temperature for melting and a short duration of exposure time. To determine the melting point of T_3, a number of experimental work is required at the “identiPol QA2” installation. Matrices

of nanofibers were obtained at various voltages listed in Table 1. The paper presents the results of one of the experiments performed at an applied voltage of 12 kV (Table 5).

Table 5

Annealing conditions of nanofiber samples obtained by electroforming

№	Voltage, ± 0.1 kV	Electrospinning time, ± 0.1 min	Distance, ± 2 mm	Heat, ± 1 °C	Time to heat, ± 0.1 min
Sample 1	12.0	5.0	140	90	30.0
Sample 2	12.0	5.0	140	100	30.0
Sample 3	12.0	5.0	140	110	30.0
Sample 4	12.0	5.0	140	120	30.0



a) 90 °C; b) 100 °C; c) 110 °C; d) 120 °C.

Figure 14. SEM image of the surface of PVOH nanofibers after exposure to vacuum PVD 30 min

Manufacture of electrospun polymer materials for tissue engineering frameworks [17], wound dressings [18], drug shells [19] and other areas of biomedicine, the use of electroformed polymer nanofibers as a medium for filtration of solid particles from air, water or any target liquid at different filtration levels [20], for the absorption of toxic gases from industrial exhaust gases or for separating water from oil [21] as well as the use (Fig. 15) of electroformed nanofiber materials in separators [22], electrodes for lithium-ion batteries and supercapacitors, sensitized solar cells, electrolytes [23], nanogenerators [24] for energy conversion and storage show a high demand for these materials.

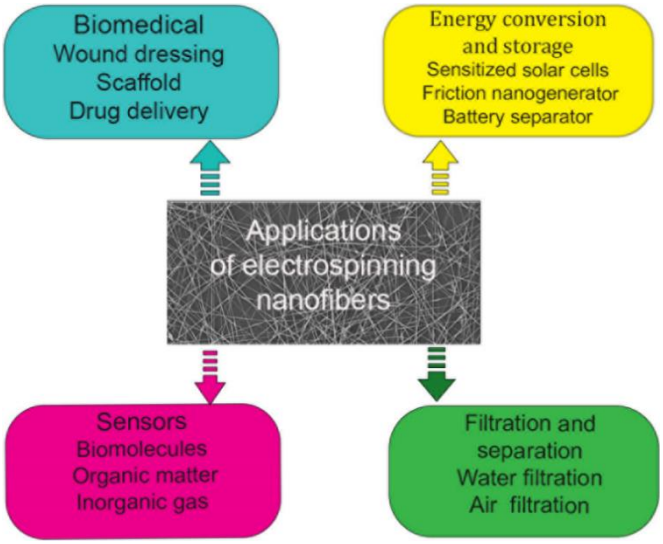


Figure 15. Potential applications of electroformed polymer nanofibers

Polymer nanofibers obtained by electrospinning provide unlimited possibilities for the formation of substrates and materials with different properties, controlled by different additional materials depending on the field of application. Showing high practical significance and having an undiscovered potential that is of interest to the world scientific community, electrospun polymer materials are one of the valuable achievements in the field of nanotechnology.

Conclusions

In this paper, the main processes for the formation of synthetic fibers such as wet spinning, melt spinning and dry spinning were discussed.

The investigated polymer fibers were obtained by dry electrospinning from a solution of PVOH polymer. The effect of the electrospinning voltage, the deposition time and the distance from the collector to the nozzle were all investigated using an Electrospinz electrospinning machine.

It was found that the small diameter of nanofibers is determined by the optimal combination of the applied voltage level and the distance from the tip of the nozzle to the collector.

The parameters of the electrospinning process required to obtain uniform defect-free nanofibers with a diameter from 120 to 174 nm were as follows: the distance between the nozzle and the collector was 140 mm and the voltage between the needle and the collector was 12-21 kV.

The above process parameters of electrospinning, are not independent of the concentration and type of polymer solution used. The effect of these additional details on the formation of nanofibers will be considered in the future.

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Электрспиннинг технологиясының қазіргі жағдайы және оның болашағы

Нанотехнологиялар мен зерттеудің заманауи әдістерінің дамуымен биологиялық ыдырайтын полимерлі материалдарды адамзат өмірінің әртүрлі салаларында қолдану ғылыми әлемдік қауымдастықтың қызығушылығын тудырып қана қоймайды, сонымен қатар ресурстарды үнемдеу мен қоршаған ортаны қорғауға байланысты жаһандық мәселелердің бірін шешуге жол болып табылады. Поливинил спирті негізіндегі материалдар биоүйлесімділік, биологиялық ыдырау қабілеті, микробқақарсы қабілеті, уытсыз үлдірді оңай түзу қабілеті сияқты биологиялық және физикалық қасиеттеріне байланысты әртүрлі салаларда кеңінен қолданылады. Полимерлі үлдірлерді алудың қазіргі кездегі әдістерінің бірі — электроспининг, оның артықшылығы технологиялық процестің салыстырмалы қарапайымдылығы мен синтетикалық және табиғи полимерлерден үздіксіз наноталшықтарды алу мүмкіндігі болып табылады. Бұл жұмыста биологиялық ыдырайтын синтетикалық полимерден электроспиннинг әдісімен наноталшықты төсеніштердің түзілуіне технологиялық үрдіс параметрлерінің әсері зерттелді. Ылғал-

ды спиннинг, балқыма спиннинг және құрғақ спиннинг технологиясы талқыланған. Поливинил спиртінен құрылымы біртекті, ақаусыз наноталшықтарды алудың оңтайлы режимдерін анықтау үшін бірқатар тәжірибелік зерттеулер жүргізілді.

Кілт сөздер: электрспиннинг, технологиялық параметрлер, наноталшықтар, поливинил спирті (PVOH), тоқылмаған төсеніштер, СЭМ, наноталшық диаметрі, биологиялық ыдырайтын полимер материалы.

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Современное состояние технологии электроспиннинга и ее перспективы

С развитием нанотехнологий и современных методов исследования применение биоразлагаемых полимерных материалов в разных отраслях жизнедеятельности человечества вызывает интерес не только научного мирового сообщества, но и является путем к решению одной из глобальных проблем, связанных с ресурсосбережением и охраной окружающей среды. Материалы на основе поливинилового спирта получили широкое применение в различных областях благодаря своим биологическим и физическим свойствам, таким как биосовместимость, способность к биологическому разложению, антимикробная способность, нетоксичность и способность легко образовывать пленку. Одним из методов получения полимерных пленок существующих на сегодняшний день является электроспиннинг, преимуществом которого является относительная простота технологического процесса и возможность получения непрерывных нановолокон как из синтетических, так и натуральных полимеров. В статье было изучено влияние переменных характеристик технологических параметров процесса на формирование нановолоконных матов из биоразлагаемого синтетического полимера методом электроспиннинга. Обсуждены технологии мокрого прядения, прядения из расплава и сухого прядения. Проведен ряд экспериментальных исследований для выявления оптимальных режимов получения нановолокон из поливинилового спирта с наиболее однородной структурой без образования дефектов.

Ключевые слова: электроспиннинг, технологические параметры, нановолокна, поливиниловый спирт, нетканые матрицы, СЭМ, диаметр нановолокон, биоразлагаемый полимерный материал.