INFLUENCE OF THE SPINNING ELECTRODE MATERIAL ON THE FIBER STRUCTURE

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Abstract

The article focuses on the issue of the spinning process in AC electrospinning. The conducted study aimed to identify the possible influence of the material type of the spinning electrode on the fibres formed. As part of the work, studies were carried out on our own test spinning equipment intended for the production of nanofibers. The electrodes were made of three different materials: stainless steel, aluminium alloy and plastic. Productivity experiments and individual partial nanofibrous structures related to individual electrodes were performed on these electrodes. The mentioned structures were examined using an SEM microscope and also statistically evaluated. Other parts of the article are simulations of the electrostatic field for the used electrodes.

Keywords: Electrospinning, electrode, nanofibers, electrostatic field

1. INTRODUCTION

The issue of nanofibers and their use is currently among the sought-after directions of primary as well as applied research. They have high added value and broad application use. The most common applications of nanofibers are in the fields of filtering water [1], air [2] and other fluids [3], medical applications [4], etc. [5]. The production of nanofibers can be divided into two basic directions. The essential representative of the first group is production using melt-blown technology, which currently reaches fibre diameters of around 500 nm [6]. The second direction of production is processes that are directly focused on the production of nanofibers. A wide variety of processes are used to create nanofibers. The first and by far the most used process is the production of nanowires using direct current (DC). Another lesser-known method of creation is using alternating current (AC). A minor production method includes drawing [7] and force spinning [8]. Some of the methods can be combined with each other. For example, a combination of force spinning and electrospinning or electrospinning and melt-blowing.

The most widely used and well-known method of producing nanofibers is electrospinning. This process of creating nanofibers is a breeding ground for the scientific community, especially in the field of electrostatic field research [9]. Of the spinning methods listed above, DC-electrospinning is the most well-known and widespread method of producing nanofibrous structures. Another type of electrospinning is AC-electrospinning; compared to DC-electrospinning, it is a very productive method of creating nanofiber structures. To implement the method, it is necessary to use a high-voltage source that changes polarity during the formation of fibres. This phenomenon of polarity change creates a nanofibrous structure in which the positive/negative polarity alternates, and the effect of the mutual attraction of the fibres is achieved. It is necessary to point out that the use of high voltage is necessary for electrospinning. This fact entails several technological disadvantages and pests, especially in the area of safety and a limited range of materials for the preparation of the spinning solution [10].

In this study, AC electrospinning was used to perform experiments and create nanolayers using a conical electrode. Three basic materials from which the spinning heads of the electrodes were completed and tested during the experiment. Microscopic images and histograms related to the individual electrodes used are evaluated in the study. The obtained nanofibrous layers are comprehensively described in terms of different structures.

2. METHODS AND EXPERIMENTS

Equipment design

The main task in this part was the creation of a suitable technical background for implementing experiments. **Figure 1** shows schematically the equipment used during the experiment. The main part of the device is the spinning unit 1, which consists of the spinning electrode 2 placed on the dosing tube 3. This tube is used to place the spinning head in the required position, and secondly, with its help, the polymer solution is transported. The spinning tube is part of pumping device 4, which contains dosing device 5 and solution stock 6. The goal of the solution supply was to create a continuous layer of solution on the spinning head and tube, which flows around their outer shell. In this way, the liquid state of the solution is ensured, and there are no dry areas that cause product defects. An AC high-voltage source - Trek 50/12 high-voltage amplifier 7 - was connected to the dosing tube. The control of the dosing device and the drive of the collection rotary collector 8 was ensured by means of the connected PLC module (Programmable Logic Controller) 9. The resulting nanofibrous formation 10 was applied to some textile 11. During the experiment, the identical value of the effective voltage of 30 kV sinusoidal current with a frequency of 50 Hz was set on the high voltage source.

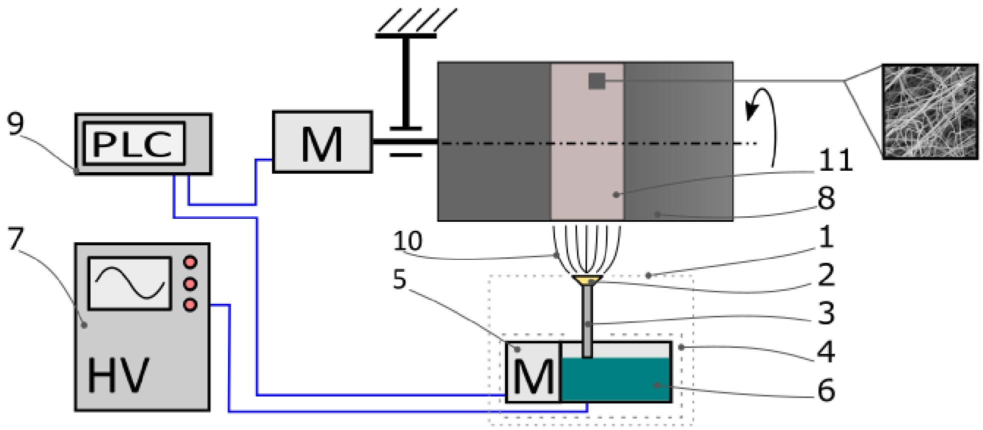

Figure 1 Scheme of the technical equipment used during the experiment

The next step was the design of a suitable shape of the electrode head or the spinning electrode head. A basic CAD geometry of the head was created in Creo 7.0.1.0 software. Furthermore, a mathematical model was created using geometry, and a series of analyses was performed with the aim of simulating a charge distribution in an electrostatic field. Based on a series of analyses of the distribution of the electrostatic field, the optimal geometry of the electrode head was determined. The analysis was performed for metal and plastic materials. The results of the analysis are shown in **Figure 2**. The Autodesk Simulation Multiphysics software was the primary tool for designing a suitable shape for the spinning head.

Simulation

Simulations were performed in FEM software Autodesk Simulation Mechanical 2015. The task was simulated as 2D axisymmetric, where the model's axis corresponds to the electrode's axis. **Figure 2 a)** shows the FEM model used for the simulations. The model consists of the following parts. A metal rod (1) with a relative permittivity of 109, a conical electrode (2) with a relative permittivity of 109 for duralumin and steel and 3.4 for PA6 material, a polymer solution (3) covering the electrode with a relative permittivity of 20, a disk (4) that simulates a lid and a container with a polymeric solution made of polyethene with a relative permittivity of 2.4 and the surrounding air (5) bounded by a spherical surface with a diameter of 2500 millimetres with a relative permittivity of 1. A boundary condition of a voltage value of 30 kV and an electrical stiffness of 109 A/V was applied to the bottom of the rod (6) and at the air boundary (7) with a voltage value of 0 V and an electrical stiffness of 109A/V.

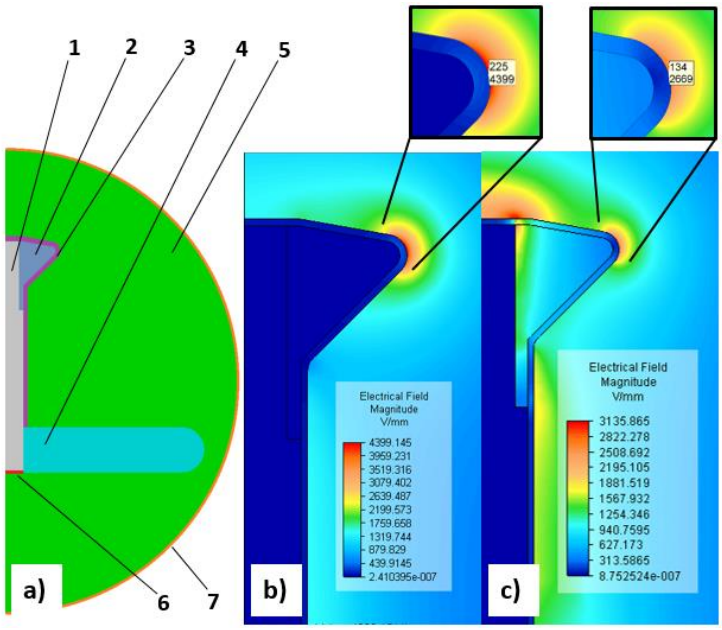

Figure 2 Distribution of electrical field

Experiment

Based on the analyses performed, real electrode models were produced using CAD models. The experiment was carried out using the methodology described in the previous paragraph with a set of spinning electrodes, see **Figure 3**. The assembly modification during the experiment consisted of replacing the spinning heads, which were geometrically identical and differed in the materials used. The relative humidity was 55 %, and the ambient temperature was 24 °C during all experiments.

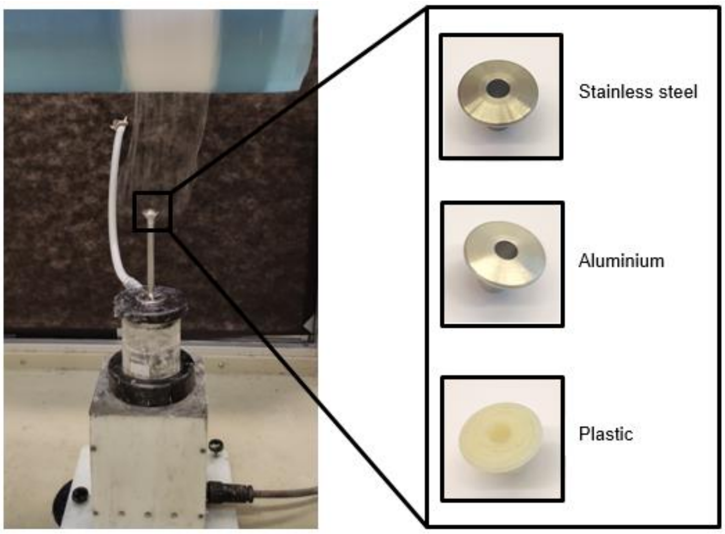

Figure 3 Nanofibrous layers

Preparation of solution

Fibrating solution concentration 10 wt. % was prepared by dissolving polyvinyl butyral (PVB – CAS 63148 65 2, Mowital B60H, Kuraray, Germany, Hattersheim) in 96% denatured ethanol (CAS 64 17 5; TechniSolv (1:1:1), VWR, Belgium, Leuven). Dissolution was carried out at room temperature for 24 hours using a magnetic stirrer.

Analysis of nanofibrous layers

Nanofibrous layers prepared by alternating electrospinning of a 10% PVB solution using three different types of electrodes were coated with a 10 nm layer of gold in a Quorum apparatus (Lewes, UK). Subsequently, images of individual samples were taken on a Vega 3 scanning electron microscope (SEM) (TESCAN, Brno, Czech Republic) at different magnifications. The applied voltage was 10 kV. Fibre diameters of the acquired layers were measured in ImageJ software (v. 3.3.1, Garching, Germany). In total, 250 diameters were measured for each analyzed sample.

3. RESULTS

In the first step, the spinning process was monitored, especially concerning the stability of the nanofibrous formation moving in the free environment between the place of creation and the place of collection of fibres. The tests showed that the compactness of the structure increases as the intensity of the electric field increases in the area where fibres are formed. Further, with the increase in intensity, there was a decrease in the misalignment from the desired trajectory of the movement of the fibres through the free environment.

The results of the SEM analysis are shown in **Figure 4**. By spinning the PVB solution, nanofibrous layers of good quality were prepared. Fibre branching and drop defects were noted, but their amount was minimal and did not affect the further usability of the product. The resulting fibre structure was fluffier and bulkier than that of fibres prepared by the direct spinning method. No differences were observed between samples prepared using electrodes of different materials. Neither the rate nor the representation of individual defects has changed. From the SEM images, it can be concluded that the material of the electrode has no direct effect on the electrospinning process.

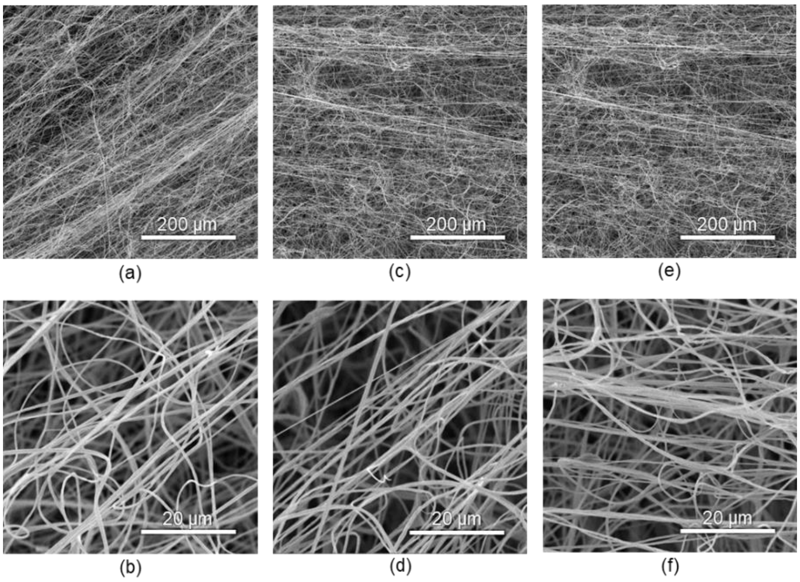

Figure 4 Images of nanofibrous layers (top) and their details (bottom) prepared using electrodes A (a, b), B (c, d) and C (e,f). It is clear from the images that the electrode material did not affect the resulting structure or quality of the nanofibers prepared by electrospinning.

Figure 5 shows the results of measuring the fibre diameters of samples 1, 2 and 3. Fibres with diameters in the range of 400 to 600 nm were most frequently represented. The smallest fibres reached approximately 200 nm, the largest then less than 1000 nm. The distribution of fibre diameters was almost the same for all three histograms. It can thus be judged that the material of the electrode did not particularly affect the diameters of the emerging fibres.

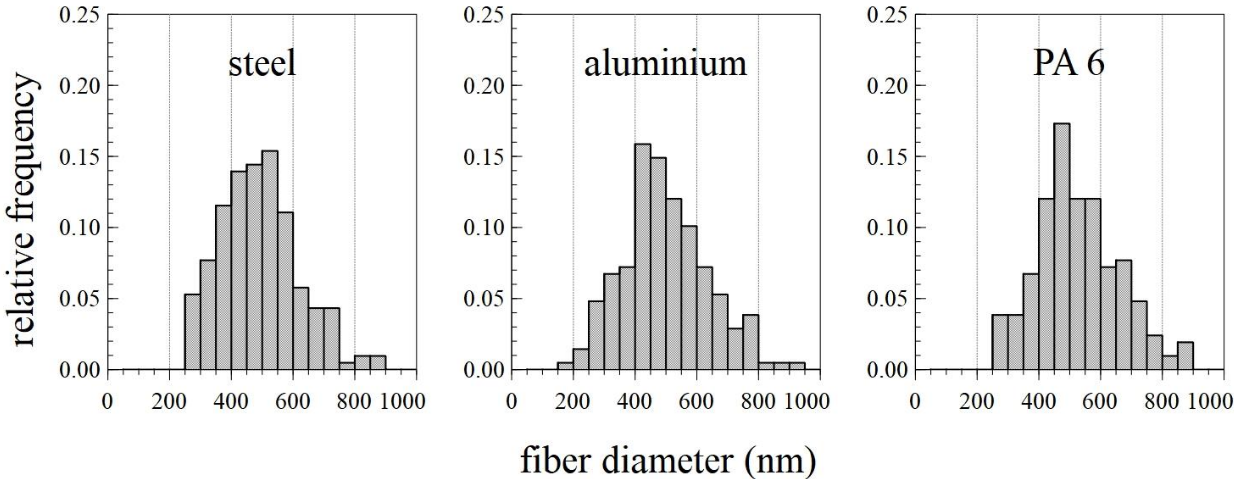

Figure 5 Histograms of fibre diameters of nanofibrous layers were prepared using electrodes of different materials. The graphs show that the material of the electrode did not fundamentally affect the diameters of the prepared nanofibers.

4. CONCLUSION

The work was focused on the possibilities of influencing the spinning process and the character of the nanofibrous structure by the type of spinning electrode material used. A device was assembled to allow the repeatability of the experiments during spinning. The only variable parameter was the type of electrode material. The geometry of the electrode was identical. Three basic materials resistant to the selected type of solution were selected, namely stainless steel (DIN 1.4401), aluminium (DIN 1725-1) and plastic (PA6). First, simulations of the distribution of the electric field intensity in the area of the electrodes were performed. For both metal materials (stainless steel and aluminium) the distribution of the electric field intensity was almost identical. The plastic electrode showed a lower level of intensity compared to the metal electrodes. The experiments proved the assumptions from the performed simulations and that with the increasing intensity of the electric field, higher stability of the moving nanofibrous formation is achieved from the place of its origin, i.e. from the electrode to the place of storage, i.e. the collector. From the results of the analysis of SEM images and created histograms, it was found that the selected base materials do not have a significant effect on the obtained structures. Research into the material type of spinning electrodes will continue with the aim of mapping a wider field of materials.

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