Geometrical Characterization of Polyethylene Oxide Nanofibers by Atom Force Microscope and Confocal Laser Scanning Microscope

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Motivation

The aim of our research is the development of a prototype of a bionanosensor which can detect pathogens in enclosed spaces, such as hospital, in real time [2]. We investigate the properties of polymers nanofibers as sensing elements of the biosensor. Hence, the geometrical, mechanical and electrical properties of the fibers are very important for the design of the sensor. The nanofibers are ideal as sensing element, because its high surface-to-volume radio, that assured a high sensitivity, and since the average size of a virus is approximately 100 nm [1].

Objective

The first goal is to find the optimal electrospinning parameters to produce polymer fibers under 100 nm, fig.1, and the geometrical characterization of it. Here we present the results of the electrospinning experiments, fig. 5 and 6, and a simple mathematical size-correction model for the width of the nanofiber shape, fig. 2. For this model we analyzed the geometry of the tip of the cantilever with CLSM to obtain a 2D image and 3D topography of it, fig. 3.

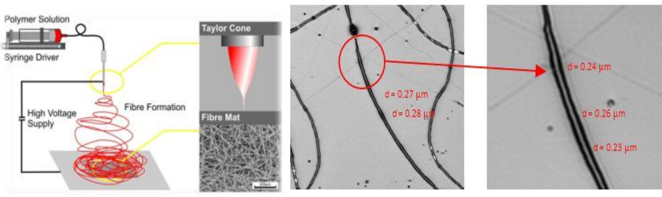

Figure 1. Electrospinning scheme and 2D pictures of polymer nanofibers.

Material & Method

We used a combination of an Atomic Force Microscope (AFM, JPK) with an inverse microscope Observer-A1 (C.Zeiss), and a microscope Imager-M2m with a Laser Scanning Microscope (LSM 700, C. Zeiss). The electrospinning setups: Syringe Pump Series-Apparatus Model `22´ (HARVARD), High voltage-PNC 30000-5 (Heinzinger), a needle with a 0.6 mm diameters and a PC to control the process. The polymer solution is a mixture of 4 g polyethylene oxide (PEO, 900000 MW) with 96 g of distilled water (4 wt% PEO). We deposited PEO nanofibers over a glass plate and used the AFM and the CLSM to measure the topography of the fibers, fig.1 and 4.

The AFM cantilever is an ACTA model (AppNano), with Silicon tip, radii of curvature of the tip, ROC < 6 nm, high between 14 and 16 µm.

The size-correction model is:

$$r_c = r\frac{1+\sin\theta}{\cos\theta+\tan\theta\tan\alpha} = r\cdot C_f \qquad (1)$$

where $2r_c$ is the appearance width of the object, $2r$ is real width, C_f is the correction factor, θ is the half cone angle and α is the angle to describe the distance between the tip and the substrate, fig. 2. When the tip reaches the substrate, fig. 2, we have $\alpha = 0$ and the equation for a asymmetric tip is:

$$r_c = r_{cl} + r_{cr} = r\frac{1+\sin\varphi}{\cos\varphi} + r\frac{1+\sin\theta}{\cos\theta} \qquad (2)$$

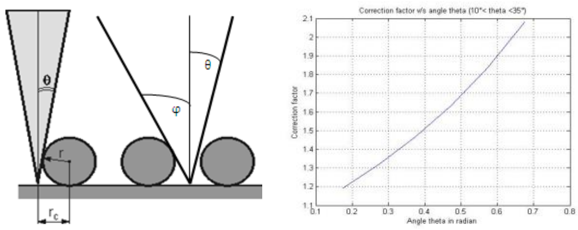

Figure 2. (right) Schematic of two kinds of tip, one symmetric (right) and another asymmetric (left) with two different angles, θ and φ. (left) Graphic of the maximal correction factor as function of theta, 10° < theta < 35°. The correction factor is between the values 1.19 and 1.92.

Results

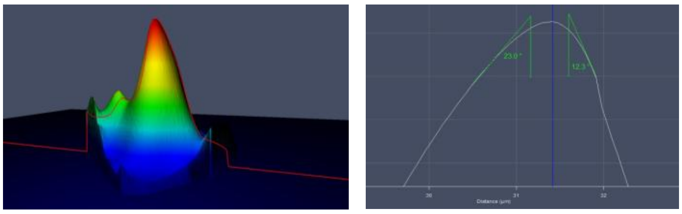

Figure 3. Measure of the cone half angle, θ =12.3° and φ =23°, of the tip by CLSM (right) and it 3D CLSM image (left).

Using the eq. (2) and the data of the measurement of the tip geometry, we obtain the following correction factor:

$$C_f = \frac{1+\sin 12.3}{\cos 12.3} + \frac{1+\sin 23}{\cos 23} = 1.24 + 1.51 = 2.75 \qquad (3)$$

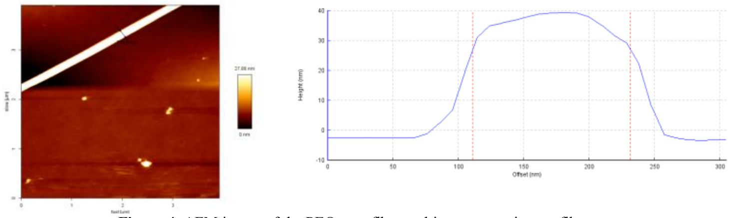

Figure 4. AFM image of the PEO nanofiber and its cross section profile.

From the fig.4 we have a high of 40 nm and $r_c = 150\ nm$. Applying the result of (3), we have: $r = \frac{r_c}{C_f} = \frac{150\ nm}{2.75} = 54.5\ nm$. Then width of the fiber is approximately $2 \cdot r = 109$ nm. The shape is like an ellipse with a width of 109 nm and a high of 40 nm.
The size-correction model is valid for a ROC << r. Where ROC is the radii of curvature of the tip and r is the radius of the fiber. In our case the ROC is smaller than the radius of the fiber and therefore we can apply the model. Yang et al., reported a similar model, [4].

Electrospinning: In this process, a strong electric field causes a viscous solution to form a Taylor cone, from which a thin fluid jet is formed. This fluid jet may harden by a variety of processes and become a continuous fiber, fig.1. During the experiment, two parameters were fixed and the effects of the third parameter in the geometry of the nanofiber have been studied. For example, the effect in the diameter of the nanofiber due the voltage, fig. 5. Heikkilä et al., [3] reported similar results as the fig. 5 and reported results of a curve with an inverse shape compared with the fig. 6.

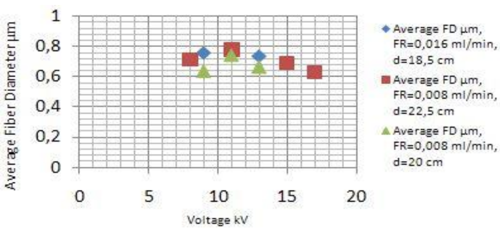

Figure 5. Fiber diameter v/s voltage for PEO nanofiber

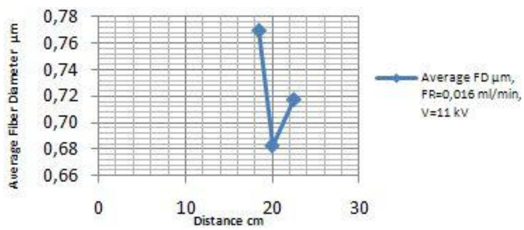

Figure 6. Fiber diameter v/s distance for PEO nanofiber.

Conclusion

It is possible to produce nanofibers under 600 nm diameters with the electrospinning method, fig. 3. We have found a few percentages of fibers with diameters between 40 and 180 nm, that means, fig.4, is possible to produce nanofibers under 100 nm diameters. The size-correction model is a good first approach to calculate the broadening due the complex interaction between the tip and the sample. To calculate the broadening effect is very important to know the shape (geometry) of the tip, because the tip can be symmetric or asymmetric and then the half cone angles are different. The nanofiber loses its cylindrical shape due to the collision with the glass plate, fig. 4.

References

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