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Introductory Chapter: Electrospinning-smart Nanofiber Mats

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<http://dx.doi.org/10.5772/intechopen.77198>

1. Introduction

1.1. Electrospinning: the importance of the method

The available literature describes numerous ways to obtain nanofibers, nanowires or nanorods based on chemical or physical reactions. All of the materials mentioned, that is, nanofibers, nanowires and nanorods, belong to the same one-dimensional group of nanomaterials. Literature reports are not consistent in defining and differentiating these types of materials, yet it can be generally assumed that nanofibers are structures whose length is much larger than the fiber diameter (over 100 times).

They are formed in continuous processes (e.g., during the electrospinning process), as a result of which it is not possible to determine their exact length. Nanowires are considered to be structures of shorter length, in the order of a few nm to several μm .

Shorter than the length of nanowires, nanorods are the structures with the shortest length among the above-mentioned one-dimensional nanomaterials. The main methods of producing one-dimensional nanostructures include template-assisted synthesis, vapor-liquid-solid, physical vapor deposition, magnetron sputtering system, chemical vapor deposition, zol-gel method, molecular self-assembly, nanolithography, and **electrospinning**.

In 2017, over 6000 publications were released worldwide (almost 50% of all publications in the field of nanotechnology published that year), whose topics covered the scope of production methods and analysis of the physical properties (including mechanical, electrical and optical), chemical properties and application possibilities (e.g., in the military, medical or high technology sectors) of polymer nanofibers or composite materials produced with their participation (**Figure 1a**).

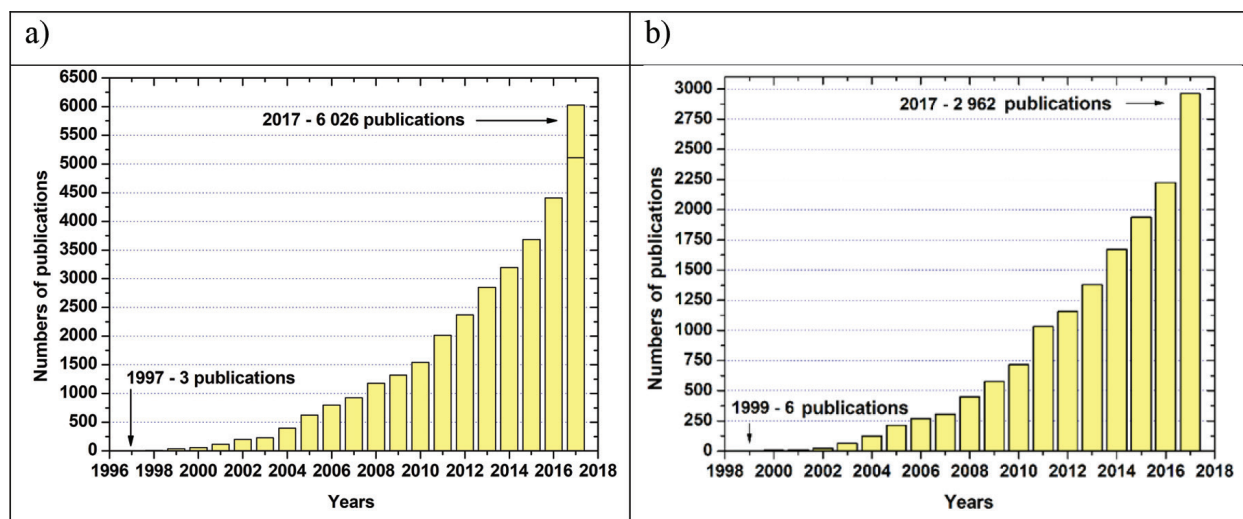


Figure 1. A chart presenting the number of publications in the field of: (a) production and/or testing of nanofibers in 1997–2017; via Scopus and ScienceDirect (keyword: nanofibers, December 2017); (b) electrospinning methods in the years 1998–2017; via ScienceDirect (keyword: electrospinning, December 2017).

A wide range of application possibilities of one-dimensional polymer and composite nanostructures affects the continuous growth of the value of the global market related to products based on nanofibers, which was worth USD 203.2 million in 2013, and a year later it already grew up to USD 276.8 million according to the BCC Research report of May 2016.

It is predicted that this market will increase from USD 383.7 million in 2015 to nearly USD 2 billion in 2020, which will correspond to the annual growth rate (CAGR) at a level of as much as 38.6% between 2015 and 2020.

From the above-mentioned methods, the most effective method of producing polymer and composite nanofibers is the technology of producing fibers in the electrostatic field, which does not require the use of complicated procedures and expensive equipment. This type of process allows to produce one-dimensional polymer and composite nanostructures on an industrial scale in a relatively easy and quick way. Unlike other techniques for the production of nanofibers, the electrospinning method has a significant advantage that, in most cases, the process is carried out at room temperature, and atmospheric pressure. Furthermore, in order to produce nanofibers, only a properly prepared spinning solution, which is usually prepared using a solvent suitable for a given polymer and a simple magnetic stirrer, is required. As a result, it is possible to quickly and cheaply obtain fibrous nanostructures with strictly defined and controlled morphology, desired chemical composition, and structure. An additional advantage of this technology is the fact that in the electrospinning process, it is possible to use most of the polymers known to date in the world, which perfectly illustrates the application possibilities of the technique of electrospinning from the solution.

The method of electrospinning nanofibers shares the most features with classical technologies of obtaining synthetic fibers that enable forming and generating a stream of previously dissolved or melted polymer and its coaxial stretching, combined with the transition of the polymer from a liquid state to a solid state.

Although the technique itself is not new, due to the large application possibilities, it is enjoying a dynamically growing interest of scientists, which can be proved by the increasing trend of scientific publications on the electrospinning method published in the years 1997–2017 (**Figure 1b**).

The method of obtaining polymer fibers using the electrostatic field was patented for the first time by the American professional inventor and electrician, John Francis Cooley, in Great Britain in 1900. In his application, Cooley proposed four technological solutions, which included a standard nozzle, coaxial nozzles, a model in which the polymer bundle was blown through the air stream in addition to interaction with the electrostatic field, and a model with a rotating spinning solution distributor. The next two patent applications, “Apparatus for electrically dispersing fluids” and “Electrical method of dispersing fluids,” were registered successively at the US Patent Office in 1902 and 1903. Independently from Cooley, in 1902, an American physicist, William James Morton, registered a patent in which he described a method using a funnel as a kind of spinning nozzle, from which a solution based on nitrocellulose $[C_6H_7(NO_2)_3O_5]_n$ and diethyl ether $C_4H_{10}O$ freely flowed out. The spinning solution flowing out from the funnel was to interact electrostatically with the anode characterized by a spherical shape, and then a cobweb-like mass was to be collected onto the rotating reel. At that time, Morton had already noticed the industrial possibilities of the method of electrospinning from the solution, claiming that “it may be put to any industrial use.”

In 1920, another American physicist, John Zeleny, published a work in which he described the behavior of a drop of liquid escaping from the end of a metal capillary under the influence of an electrical voltage. This publication initiated mathematical attempts to analyze and models the behavior of a stream of liquid in an electrostatic field. So far, numerous mathematical models have been developed describing the behavior of the spinning solution bundle in the electrostatic field during the electrospinning process. However, none of the models described is accurate enough so that the theoretical results obtained overlap with experimental data.

In the period from 1964 to 1969, a British physicist, Sir Geoffrey Ingram Taylor, carried out scientific work on the conduct of conductive liquids in the electrostatic field. His work describing the mathematical modeling of the change in the shape of a spinning solution drop coming out of the nozzle due to the applied potential difference significantly contributed to the development of the method of obtaining polymer nanofibers by means of the technique of electrospinning from the solution. A characteristic conical shape that a spinning solution drop takes during the electrospinning process is now known as the so-called Taylor cone. In 1971, Peter K. Baumgarten conducted research on the production of polymer nanofibers by means of the method of electrospinning from the solution based on acrylic resin and dimethylformamide. The analysis of high-speed photography (images taken with very short exposure time) of a stream of spinning solution in the electrostatic field showed that although the electrospinning process is observed as a “hazy cloud” resembling a spinning solution bundle on the way between the nozzle and the collector, only one fiber is formed during the electrospinning process. Through interaction with the electrostatic field produced between the electrodes, this fiber moves in a spiral motion and then settles on the surface of the grounded collector in the form of a fibrous mat. In addition, calculations based on the photographs of the polymer bundle during the electrospinning process showed that the resulting fiber on the section between the nozzle and the collector probably moves at speed exceeding the speed of sound in the air.

Scientific works in the field of the process of electrospinning polymer nanofibers in the electrostatic field conducted in the 1990s of the twentieth century by research groups under the direction of an American physicist, Dr. Darrell H. Reneker, have shown that it is possible to produce fibers from numerous organic polymers. This fact has become the reason for the dynamic growth of interest in the electrospinning method observed to this day.

During the electrospinning process, an electrostatic field is generated under the influence of a high voltage of several to several dozens of kilovolts between the nozzle (a metal needle of a syringe to which the spinning solution is delivered at a constant speed) and a grounded collector (**Figure 2**).

The presence of a potential difference between the electrodes due to electrostatic interactions causes the electric charges to be induced on the surface of the spinning solution drop emerging from the nozzle. A negatively charged collector located under the nozzle causes the repulsion of negative charges of the solution drop toward the soul, which additionally attracts them with Coulomb forces due to its positive resultant charge, as a result of which positive charges are accumulated on the surface of the spinning solution drop emerging from the nozzle. Under the influence of electrostatic field forces caused by a correspondingly large potential difference between the electrodes, the drop of solution at the mouth of the nozzle opening becomes distorted and adopts a conical shape (the so-called Taylor cone), which is accompanied by the movement of charges carried by a stream of the spinning solution toward the grounded collector. The outflow of spinning fluid toward the collector is initiated by exceeding the critical intensity of the electrostatic field, and then a drop of the polymer solution is stretched to a thin fiber under the influence of field forces. When the diameter of

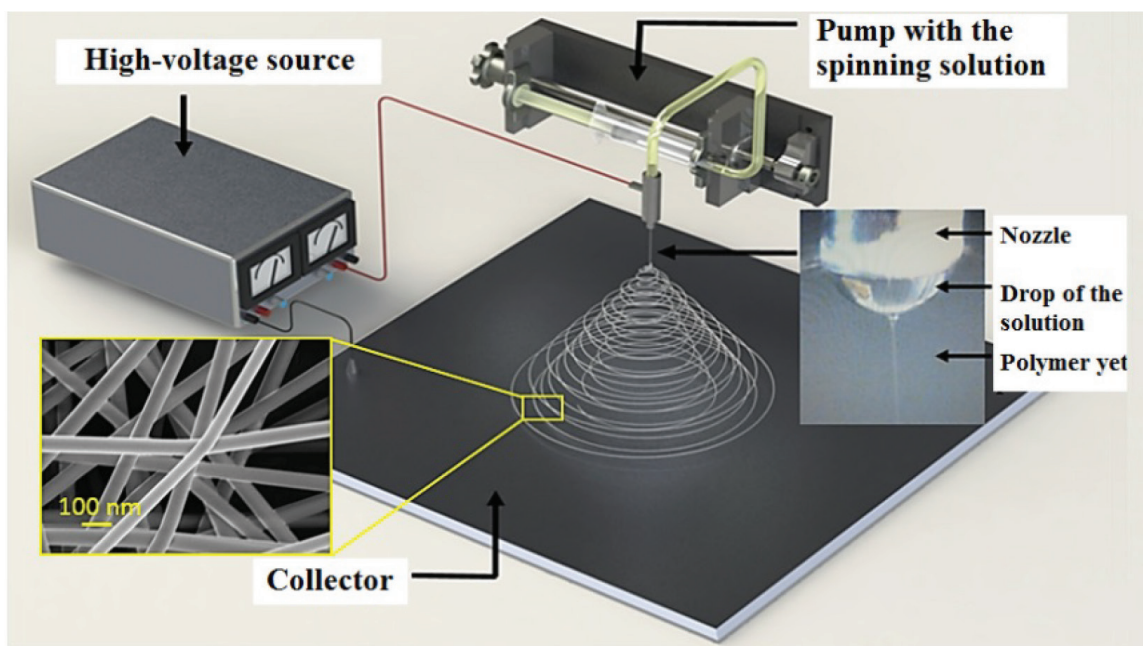


Figure 2. A diagram of a stand for the production of nanofibers containing a picture showing the mouth of the nozzle and a drop of the polymer solution that forms under the influence of high voltage in the so-called Taylor cone from the apex, which is followed by a shot of a thin stream of the solution.

the stream decreases, the ratio of the stream surface to its volume increases. The solvent evaporates, and the spinning solution solidifies to a form of a polymer fiber, which then settles on the surface of the conductive collector.

The morphology and properties of polymer nanofibers obtained by means of the electrospinning method are influenced by many factors that can be divided into three main groups: spinning solution parameters, apparatus parameters, and environmental parameters.

The main parameters resulting from the type of spinning solution used include: solution viscosity, polymer mass concentration relative to the solvent used, the molecular weight of the polymer used, type of solvent, electrical conductivity, and surface tension of the produced solution. In addition, the key parameters that have a significant impact on both the morphology and physical properties of the fibers produced are process parameters used during electrospinning. These parameters include the speed of feeding the spinning solution to the mouth of the nozzle, the difference in potentials, and the distance between the electrodes, that is, the nozzle and the collector. These process parameters can be changed during the electrospinning process, which makes it possible to control their values in order to obtain a stable Taylor cone formed from a spinning solution drop at the mouth of the nozzle. In addition, the process parameters also include apparatus parameters, taking into account the inner diameter, and the length of the nozzle used as well as the type of collector. We can distinguish two basic types of collectors used during the electrospinning process of nanofibers. They include flat plate collectors and drum collectors, which rotate at a precisely defined angular speed during the electrospinning process, thus winding the produced fibers as on a reel. As a result, nanofibers with a chaotic arrangement are obtained using a flat plate collector, while the use of a drum collector allows for obtaining nanofibers with a strictly defined orientation and a parallel arrangement of individual fibers relative to each other. The environmental parameters include ambient conditions in which nanofibers are produced, that is, temperature, air humidity, and atmospheric pressure.

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