УДК 538.975

# OBTAINING CARBON FIBERS BY THE METHOD OF ELECTROSPINNING

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**Abstract**
Electrospinning is a flexible method to process melts or solutions, generally of polymers, into continuous fibers with different diameters ranging from a few micrometers to a few nanometers. This method is applicable to effectively to many fusible or soluble polymers. Polymers can be chemically modified and can be tailored with additives vary from simple particles carbon black to complex species. Technique appears to be uncomplicated, but is a complex process that contingent on many molecular and technical parameters. Using electrospinning immediate access to completely new materials, which can have different chemical structures. Materials described here demonstrate potential of this technique in fundamental and application oriented research. However, more research is required to understand and accurately control the actual mechanics of the formation of various conductive fiber nodes. In this review, summarize data are presented based on the research of studies on the fabrication of carbon fibers by the method of electrospinning for their further application in industry.
**Keywords:** electrospinning, polymers, nanomaterials, carbon

## Introduction

With the discovery of nanomaterials, a new "epoch" of science was launched around the world. Carbon fibers occupy a separate position among a wide class of nanomaterials due to the uniqueness of their physicochemical properties and the further prospects of practical application. Studies of carbon fibers are relevant, which is confirmed by the large number of works carried out in this direction, and an increase in the number of publications on this topic.

Carbon fiber – a form of carbon, formed predominantly by carbon atoms, consisting of thin filaments with a diameter of 5 to 15 microns. Carbon atoms are combined into microscopic crystals aligned parallel to each other, which gives the fiber greater tensile strength.

Carbon fibers are characterized by high tension force, low specific gravity, low coefficient of temperature expansion and chemical inertness. It is expected that demand for carbon fibers will increase from 51,000 tons in 2015 to 89,000 tons in 2020 [1-3].

At present, telecommunications along with other methods is used to fabrication carbon fibers. Carbon fibers are actively used in the production of new types of composite materials and products from them, which could be applied into various industries.

## Research in the field of fibers

After a long period of development and use in specialized applications, the production of carbon fibers is now on the verge of widespread commercialization, facilitated by the decline in carbon fiber prices in the 1990s and the increase in their availability. Changes in the ratio of performance/price led to an increase in demand for composites. In addition, market conditions are more favorable to projects for commercial products that are lighter, stronger, faster and more economical than carbon fibers [4].

The history of fibrous materials begins in 1889 with the patents of Hughes and Chambers for the synthesis of filamentous carbon [5]. However, the true evaluation of fibers with a small diameter came much later, after studies by scientists Koyama and Endo from Japan [6], Tibbets [7], Terry, Baker from the USA, Bennisada [8] from France. Further attempts were followed to produce fibers by Formholtz, Simons, Baumgartner, who in one way or another perfected devices for electroforming and obtained fibers with a smaller diameter [9, 10]. Since 1980, Donaldson has been the world leader in the global market, which has been producing fibers for air filtration. Since then, and up to the present day, fibers have been used in such industries as filtration, transport, construction, agriculture, mining [11]. However, the full potential in the field of fibers has not been investigated.

Only since 2005, Elmarco has made a great breakthrough with the launch of the world's first production technology for fibers on an industrial scale [12].

Based on the statistical data of the invests of leading countries in the study of nanotechnology is more than 50%. The development of research in this field has achieved great success in less than 100 years [13]. Considering the world market and investments in nanotechnology in the period from 2004 to 2014, it can be noted that the investment curve is growing every year, which proves the interest and prospects for the development of this young research area. USA, Japan, China, Germany and France are the most active countries in the field of scientific research in the field of nanotechnology [14]. Composites have been widely used across industries like aerospace [15], wind energy [16], automotive [17], marine [18], oil and gas.

According to statistical data [19], materials based on carbon fiber are relatively more expensive than metal composites. The combination of costs and productivity are fundamental factors in the choice of composites. As a result, extra strong carbon composites made their impact on high-performance vehicles, such as spacecrafts, racing yachts, jet fighters and racecars. In 2014, the capitalization of composite materials market amounted to about 28 billion dollars, and today is increase approximately 15-20% per year. Thus, a lowering of cost of the composite materials will have a negative impact on the entire market. For a correctly, evaluation of the final cost of carbon composite materials, the following conditions must be considered: technology of production, storage life, price of raw materials, and overall profitability.

A typical cost comparison between various materials is shown in Fig. 1 presents the worldwide market estimates for carbon fiber.

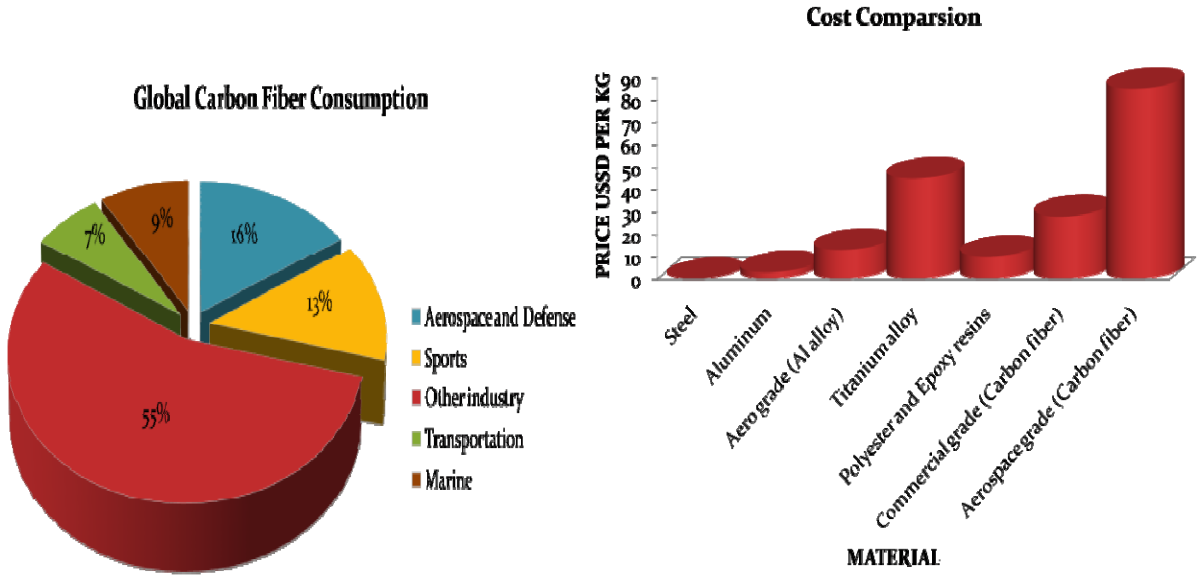



**Fig. 1.** a) Global carbon fiber consumption (2012);
b) cost comparison of materials [19].

The possibilities of using nanomaterials are inexhaustible; as are their interrelationships with such sciences as chemistry, physics, biology, but the physicochemical and technological aspects make a significant contribution to the world development of nanotechnology, since nanochemistry establishes the relationships between particle size and its properties [20]. Perfection of equipment allows to increase the number of methods and to obtain more and more functional and useful nanomaterials [21]. According to analysts, in the next decade the market of nanofibers will be the fastest growing and promising [22].

Thus, research in the field of fibers is closely related to their practical application. With their help, it is possible to solve the main problems in the development of human society. Demand for products containing fibers is expected to grow by as much as 40% [23].

**Comparative analysis of methods for forming fibrous materials**

It is known that, depending on the required functional properties and the field of application, it is possible to obtain polymer, ceramic, glass, metal, composite, synthetic and biodegradable fibers by selecting the appropriate technological method or combining several methods in one [24].

Methods for obtaining fibers are quite diverse and, in particular, include the drawing, blowing, die method, classical electroforming [25], laser [26], atomic-layer [27], using a hybrid membrane, double fiber.

*Phase separation*

According to the thermal induced liquid-liquid phase method, nanofibrous foam materials were obtained [28]. By the variation of manufacturing parameters (temperature, concentration) at the production of nanofibrous foams, makes possible to regulate the final morphological structure. Interconnected porous nanofiber networks have been formed from polymers with fiber diameters from 50 up to 500 nm, and porosities up to 98.5%.

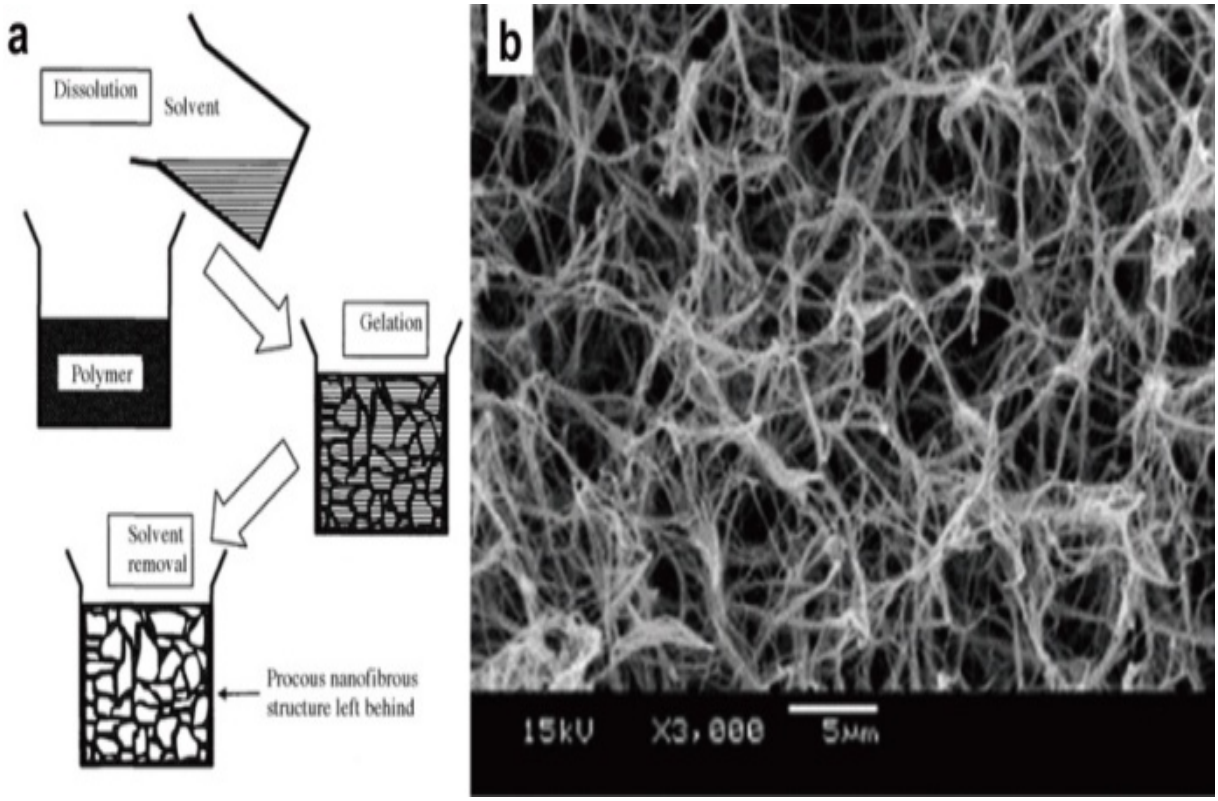



**Fig. 2.** A schematic (a) of nanofiber formation by phase separation [21] and an SEM image (b) of nanofibrous structure fabricated by this technique [29].

*Self assembly*

Self assembly is a spontaneous formation of ordered spatial or temporal structures in strongly nonequilibrium open systems (physical, chemical, bio, etc.). Continuous flows of energy or va, entering the system, support it in a state far from equilibrium. These peptide amphiphiles (PA), derived from a collagen ligand, allow for a self-assembling system that consists of a hydrophobic tail group and a hydrophilic head group [30]. Nanofibers with diameters around 5–25 nm can be formed by the self-assembly process.

*One-Step drawing technique*

In research [31] it is written that nanofibers can be obtained from viscous polymer liquids by mechanical drawing. Figure 3 shows the schematic illustration of the drawing process. The results demonstrate that the obtained polymer nanofibers exhibit high surface smoothness, length uniformity, high mechanical properties, and excellent flexibility.

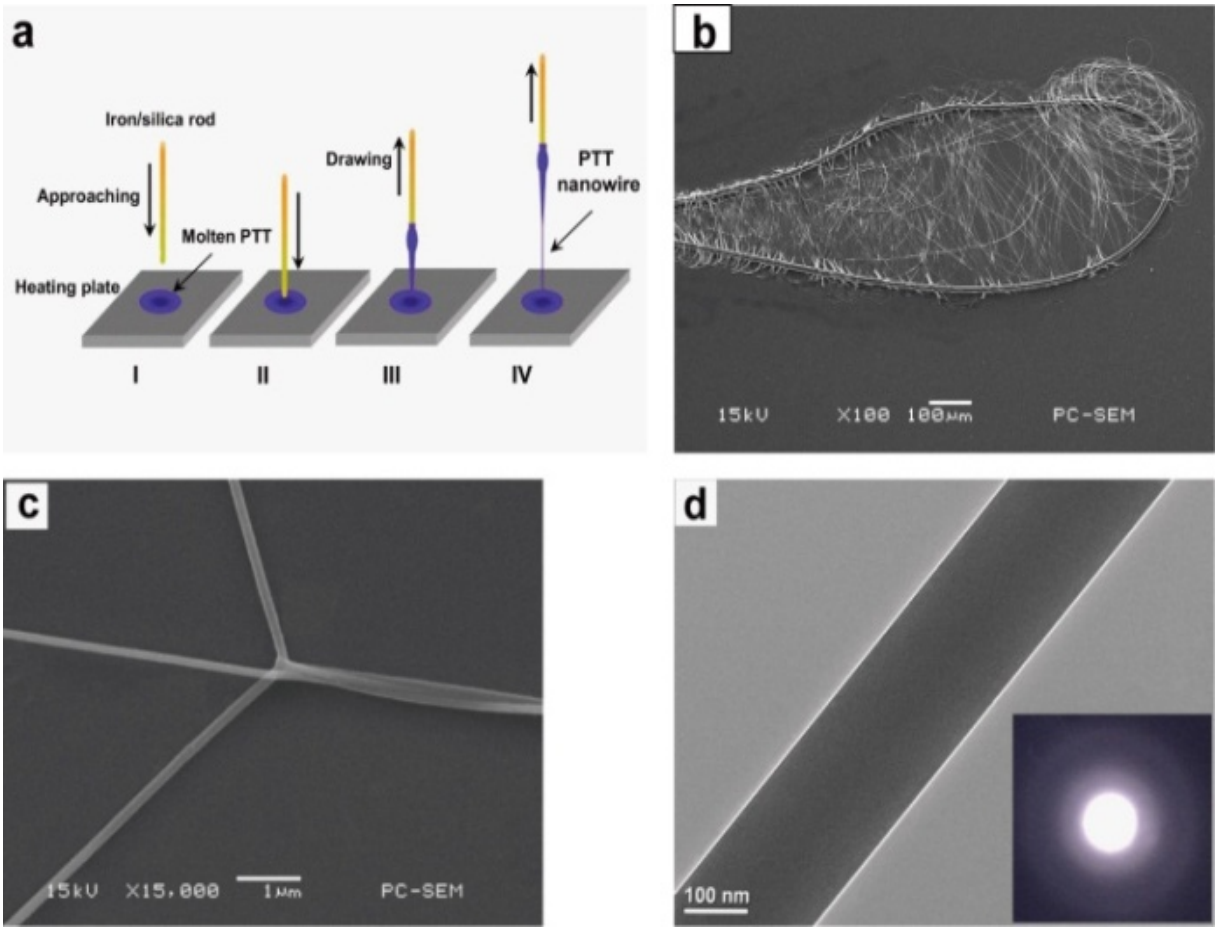



**Fig. 3.** a) Schematic of nanofiber fabrication by the drawing technique;
(b) SEM images of a nanofiber with average diameter of 280 nm coiled on a 12-μm-diameter PTT bending rod, the length of the nanofiber displayed is about 200 mm;
(c) Flexible and elastic enough nanofiber connection with diameters of 140 and 170 nm;
(d) Transmission electron microscope (TEM) image of a 190-nm-diameter fiber;
The inset shows its electron diffraction pattern [32, 33].

*Templating*

Polymer fibers can be fabricated using templates such as self-ordered porous alumina. Alumina network templates with pore diameters from 25 to 400 nm, and pore depths from around 100 nm to several 100 μm have been be fabricated. Polymer fiber arrays can be released from these molds by destruction of the molds or mechanical detachment (Fig. 4) [34, 35]. The length of fibers fabricated from alumina templates can be controlled as a function of parameters, such as melt time and temperature.

*Extraction*

Fibers can be extracted from natural materials using chemical and mechanical treatments. Cellulose fibrils can be disintegrated from plant cell walls. In one example, cellulose nanofibers were extracted from wheat straw and soy hull with diameters ranging from 10 to 120 nm and lengths up to a few thousand nanometers [36].

**The essence of the method of electrospinning**

Electrospinning, also known as electrostatic spinning, has its basis in early studies. In 1745, Bose described aerosols generated by the application of high electric potentials to drops of fluids. In 1882, Lord Rayleigh investigated the question of how many charges are needed to overcome the surface tension of a drop. Later, the first devices to spray liquids through the application of an electrical charge were patented by Cooley and Morton, in 1902 and 1903. In 1929, Hagiwaba et al. described the fabrication of artificial silk through the use of electrical charge. The crucial patent, in which the electrospinning of plastics was described for the first time, appeared in 1934 with

Anton Formhals from Mainz as the author (and can be traced back to a German patent filing in 1929). Despite these early discoveries, the procedure was not utilized commercially. In the 1970s, Simm et al. patented the production of fibers with diameters of less than 1 mm.

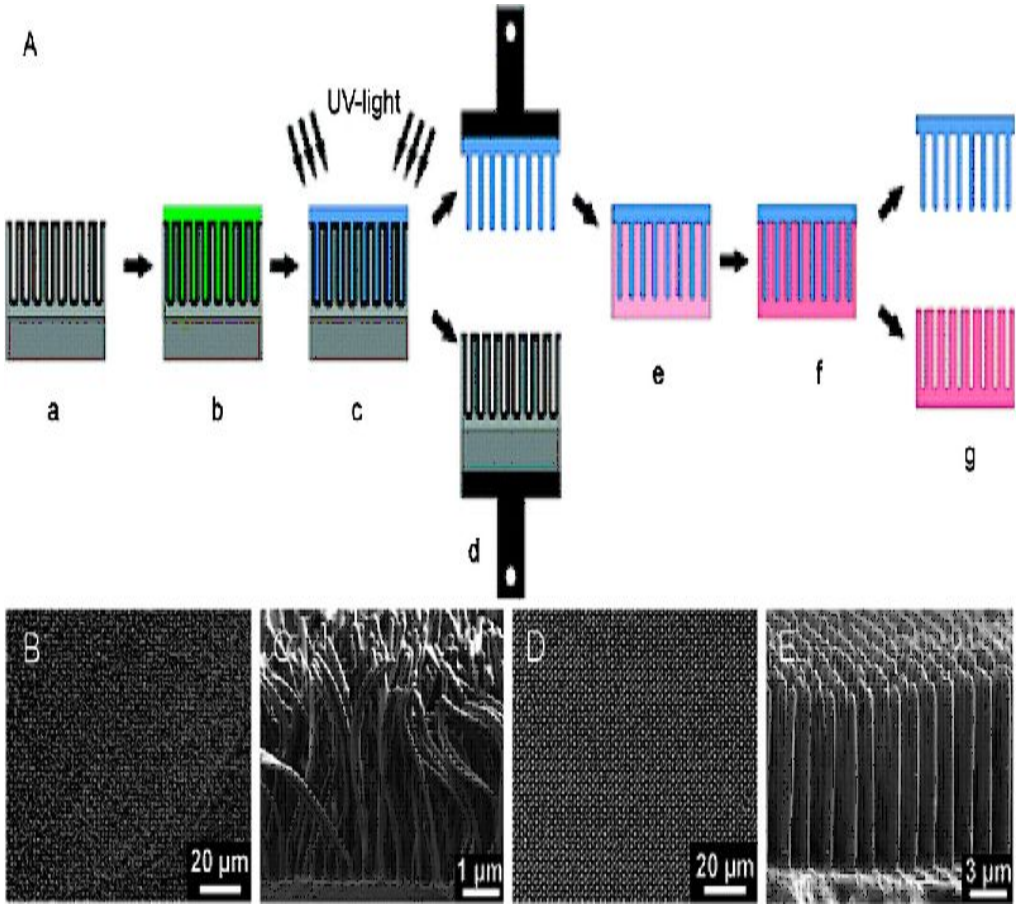



**Fig. 4.** a) Schematic of the fabrication of polymer nanofibers using a nondestructive templating technique (grey: alumina template, green: resin, blue: polymer nanofibers, pink: silica replica template. (b-e) SEM images of 120 nm (b&c) and 1μm (d&e) polymer fibers fabricated by the above technique [34].

Electrospinning gained substantial academic attention in the 1990s, which was partially initiated by the activities of the Reneker group. One reason for the fascination with the subject is the combination of both fundamental and application oriented research from different science and engineering disciplines. These research efforts usually target complex and highly functional systems, which could certainly be applied on a commercial level. Fiber systems in which the macroscopic properties (that is, specific chemical, physical or biological combinations of properties) can be targeted through modifications on the molecular level are of particular interest.

Electrospinning is currently the only technique that allows the fabrication of continuous fibers with diameters down to a few nanometers [37]. Electrospinning is an electrostatically driven method of fabricating polymer fibers. Fibers are formed from a liquid polymer solution or melt that is feed through a capillary tube into a region of high electric field [38]. The electric field is most commonly generated by connecting a high voltage power source in the kilovolt range to the capillary tip (Fig. 5). As electrostatic forces overcome the surface tension of the liquid, a Taylor cone is formed and a thin jet is rapidly accelerated to a grounded or oppositely charged collecting target. Instabilities in this jet cause violent whipping motions that elongate and thin the jet allowing the evaporation of some of the solvent or cooling of melts to form solid nanofibers on the target site. Nanofiber size and microstructure can be controlled by several processing parameters including: solution viscosity, voltage, feed rate, solution conductivity, capillary to collector distance, and

orifice size [39]. The electrospinning technique is very versatile and a wide range of polymer and copolymer materials with a wide range of fiber diameters (several nanometers to several microns) can be obtained using this technique. Many different types of molecules can be easily incorporated during the electrospinning fabrication process to produce functionalized fibers. Electrospun fibers are collected on the surface of the substrate in a chaotic or ordered form from an electrospinning jet.

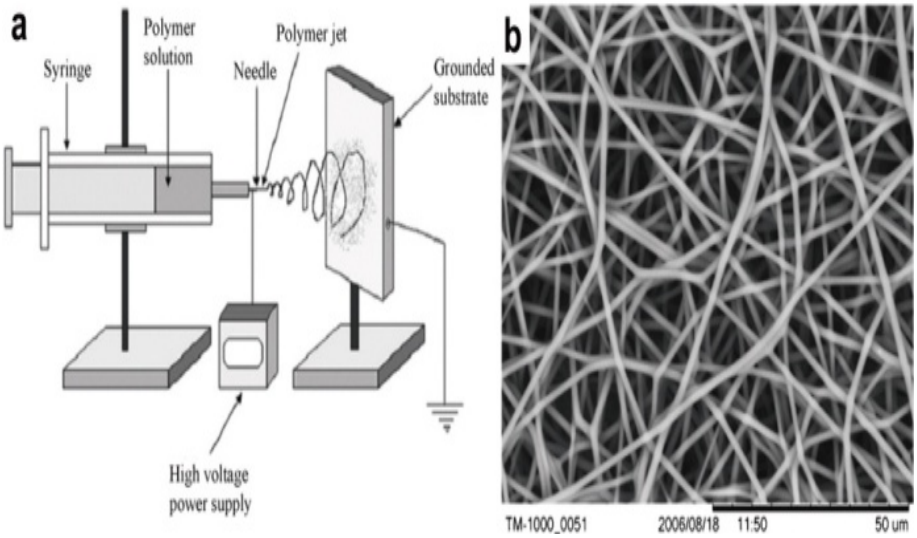



**Fig. 5.** a) Schematic of a standard electrospinning setup [38] and a scanning electron microscope (SEM) image (b) of electrospun polyurethane nanofibers.

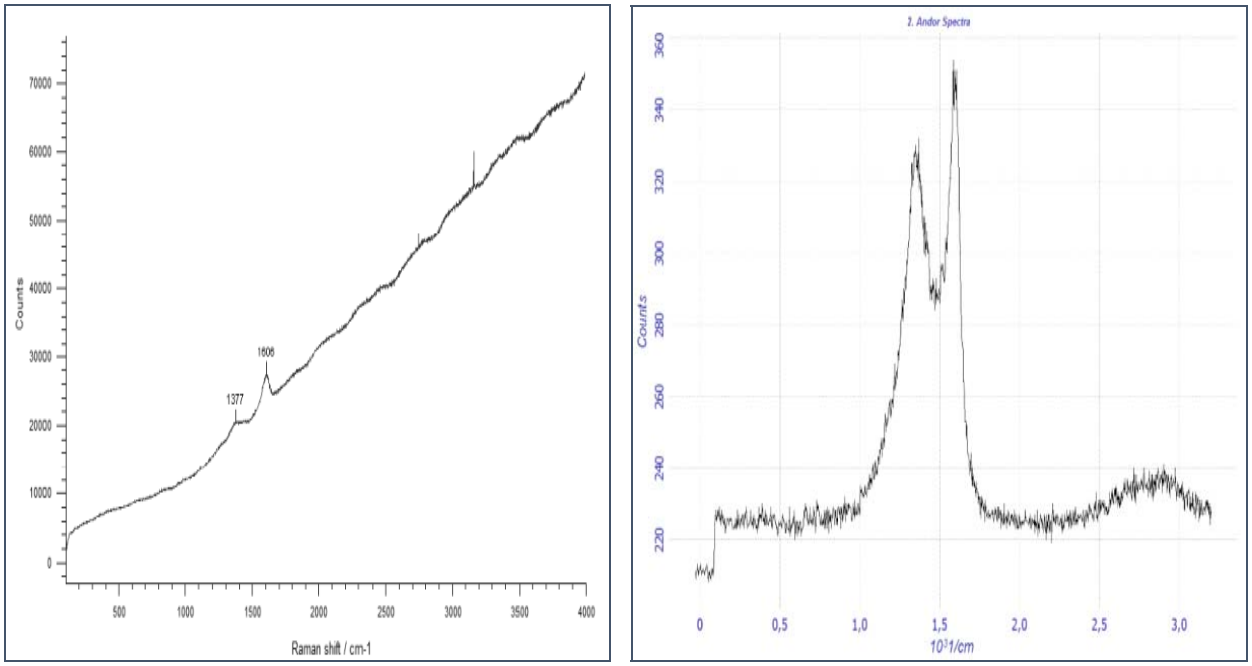
Parameters influencing the process of electrospinning. By the variation of the parameters of electrospinning has a direct effect on the final morphological structure of the fibers. It is much easier and more possible to obtain desired fiber diameters and morphologies through control of these parameters. The ideal targets in electrospinning of a polymer into fiber are:

1. The diameters of the fibers must be consistent and controllable;
2. The fiber surface must be defect-free or defect-controllable;
3. Continuous single fibers must be collectable.

Fiber diameter is among the most important quantities in electrospinning. Another challenge is the uniformity of the fiber diameters. A serious problem was identified [40], in the form of the appearance of defects such as beads and pores.

Electrospinning can produce composite fibers, as well as modified composite fibers from various polymers. When forming the fibers, the choice of solvents, for composite polymers, and for the modifying compound is important. Modifying additives can have a different nature and can be introduced into the structure of the resulting fiber differently. In Figs. 6 and 7 showed results of obtained carbon fibers with a diameter up to 1 μm by scientists from the Institute of Combustion Problems [41].

Spinnable pitches were prepared from cheap petroleum and coal-based resources, PFO, slurry oil, and coal tar, via two-stage distillation without any additives. Carbon fibers with various diameters were successfully produced by the meltspinning of spinnable pitches produced from the PFO and the coal tar. Although the spinnable pitches and carbon fibers were prepared from each precursor under the same processing conditions, their properties and morphologies were obviously different, depending on the raw materials. A fine carbon fiber could not be obtained from the slurry oil due to the development of a partial mesophase form. Even though excellent carbon fibers were stably produced from the PFO and the coal tar, the average diameters of the carbon fibers produced at various winding speeds were different, being about 7.5 μm for the PFO and 10.0 μm for the coal tar. The inherent properties of the raw materials seemed to be reflected in the carbon fibers produced through the simple distillation, and they should be fully considered when producing a carbon fiber with target properties. In Fig. 8 showed results of obtained carbon fibers with a various diameter by scientists from the South Korea and Japan [46].




a) b)

**Fig. 6.** a) RAMAN-spectrum of bitumen/PAN fibers after pre-carbonization at 500 °C,
b) RAMAN-spectrum of bitumen/PAN fibers after carbonization at 800 °C.

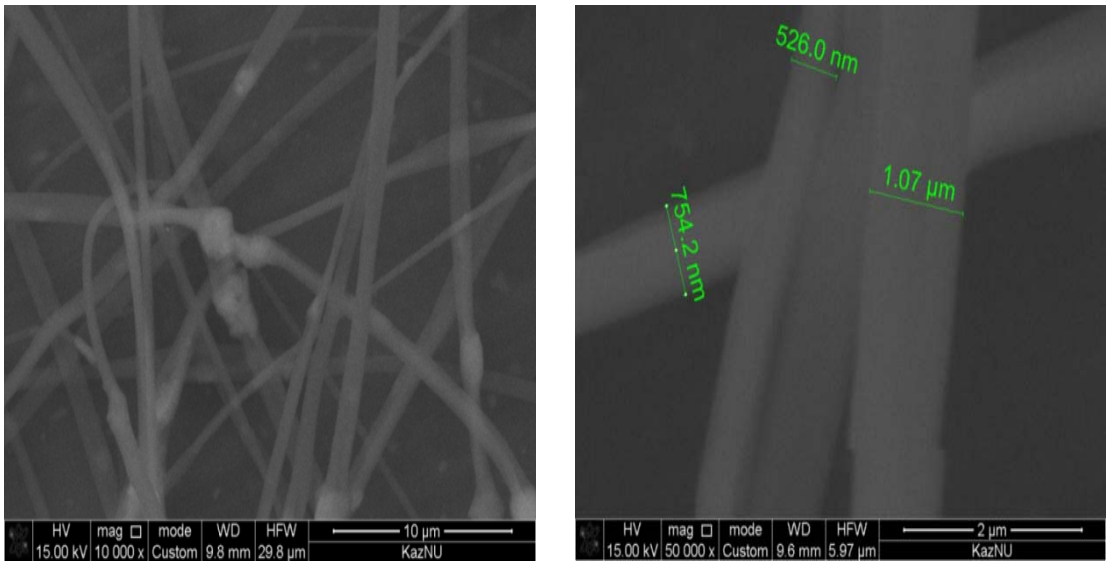
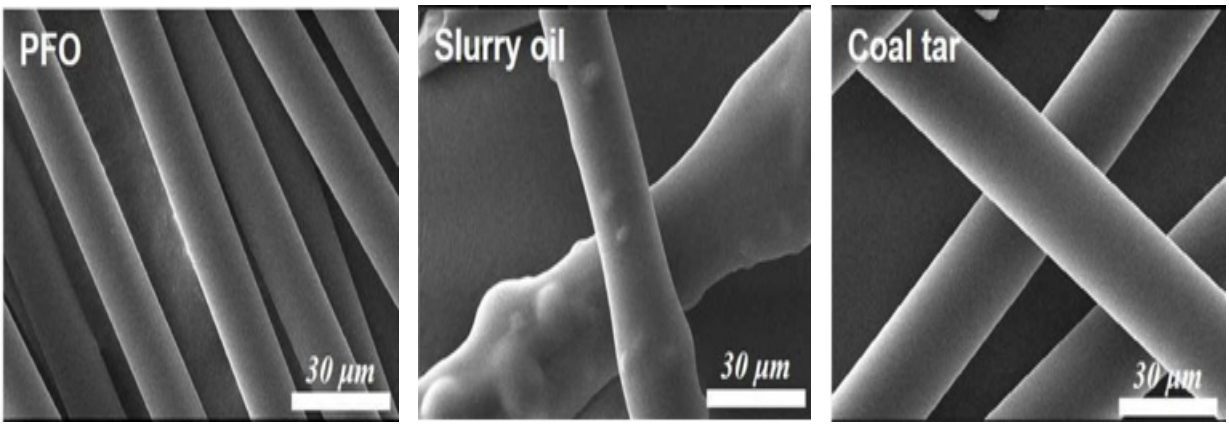



**Fig. 7.** SEM images of carbon fibers after carbonization, obtained by electrospinning from bitumen [41].

**Precursors for the obtaining carbon fibers**

*Acrylic precursors*

Various acrylic precursors have been utilized to produce carbon fibers for use in carbon fiber based composite applications owing to certain desirable physical properties [42]. The acrylic precursors for the carbon fiber industry originated from the companies that were established commercial-scale producers of textile-grade acrylic fibers. This was because the carbon fibers were produced through the pyrolysis of the acrylic fibers. Therefore, the manufacturers of carbon fibers could most readily adapt the existing technology for precursors to manufacture carbon fibers. In particular, the resultant carbon fibers from acrylic precursors such as PAN-based carbon fibers have been widely used as reinforcing materials in automobile, aerospace, recreational, and various other industries [43].




a) b) c)

**Fig. 8.** Scanning electron microscopy images of the carbon fibers spun at 63 m/min (200 rpm). PFO, slurry oil, coal tar (a-c) [46].

*PAN precursors*

PAN-based polymer precursors for carbon fibers could be primarily classified into pure homopolymer and comonomers. Generally, the comonomers are widely used in PAN-based polymer precursors to manufacture carbon fibers.

The homopolymer PAN product is slightly difficult to process into carbon fibers because the initial oxidation stage of the process cannot be easily controlled owing to the sudden and rapid evolution of heat, coupled with a relatively high initiation temperature. Such heat could result in poor properties of carbon fibers owing to the chain scission from the thermal shock. It is known that the homopolymer PAN product has never been used as a precursor for manufacturing carbon fibers. To overcome the resultant poor properties in the carbon fibers due to the rapid evolution of heat, the exothermic reaction should be adequately controlled using suitable comonomers such as itaconic acid [43].

*Cellulosic precursors*

Cellulose as a precursor for making carbon fibers was first used by Thomas Edison in the 1880s for his revolutionary electric lamp filament. Almost 80 years later, in 1959, the National Carbon Company (a division of Union Carbide) produced a carbon cloth from a rayon precursor, and two years later, in 1961, a carbon yarn was introduced [44].

Tang and Bacon [45] proposed the four simplified stages mentioned below for the conversion of cellulose to carbon:

1. Stage I: Physical desorption of approximately 12 % absorbed water (25–150 °C) with a small degree of change in the lateral order.

2. Stage II: Dehydration from the –H and –OH fragments present in the cellulose unit (150–240 °C). IR shows that –C = O and –C–C– are involved, and hence, dehydration is essentially intramolecular.

3. Stage III: Thermal cleavage of the glycosidic linkage and scission of other C = O and some C–C bonds via a free radical reaction (240–400 °C) which leads to the formation of large amounts of tar, $H_2O$, CO and $CO_2$.

4. Stage IV: Aromatization (400 °C and above), wherein each cellulose unit breaks down into a residue containing four C atoms, which then polymerize through condensation reactions involving the removal of –H above 400 °C into a C-polymer with a graphite-like structure.

*Petroleum pitch and coal tar pitch precursors*

Petroleum pitch can be obtained from various sources such as heavy residue obtained from a catalytic cracking process and steam cracker tar – a by-product of the steam cracking of naphtha or gas oils to produce ethylene or any residues from crude oil distillation or refining. Many methods can be used for the production of pitch and are based on an initial refining process, which can include either one method or combination of several treatment methods listed below:

1. Prolonged heat treatment to advance the molecular weight of the components

2. Air bowing at approximately 250 °C

3. Steam stripping and application of vacuum to remove low boiling components

4. Distillation

In common with the coal tar pitch, the chemical and physical characteristics of petroleum pitch are dependent on the process and conditions em-

ployed especially the process temperature and heat treatment time. Generally, longer times and higher temperatures produce pitches with increased aromaticity and higher anisotropic contents. The petroleum pitches are usually less aromatic compared to the coal tar pitch. A technological scheme of obtaining carbon fibers from petroleum bitumen and coal tar is shown in Fig. 9:

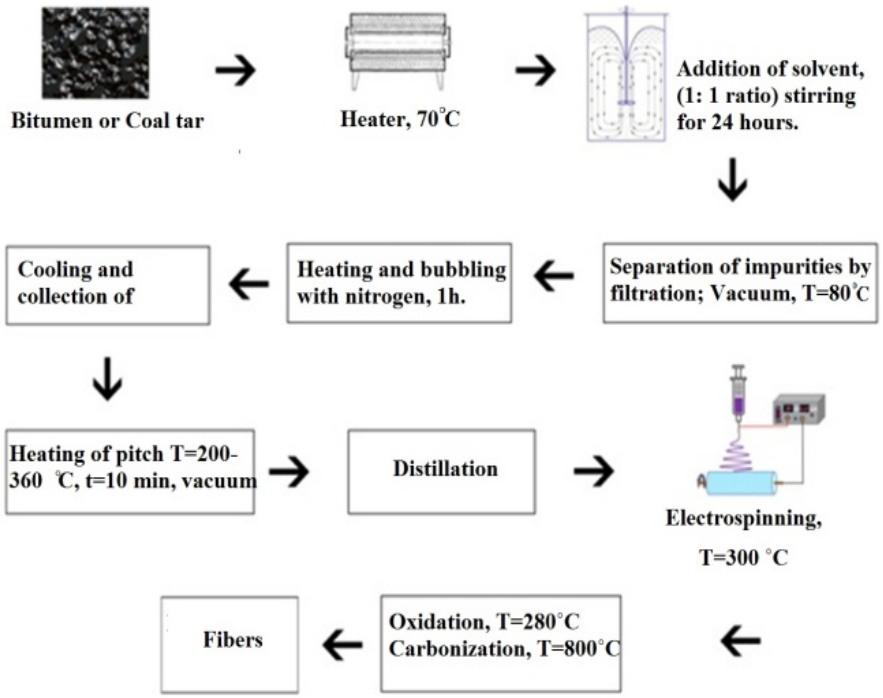



**Fig. 9.** Schematic of carbon fibers fabrication by the electrospinning [41].

Coal tar is a by-product of the coking of bituminous coals to produce cokes. The metallurgical cokes are produced at high temperatures (between 900 and 1100 °C), but it produces smokeless fuels at lower temperatures (approximately 600 °C). The low-temperature process affords a smaller amount of tar compared to the high-temperature process. Coal pitches are produced by distillation and heat treatment from coal tar. The pitch is the residue, which follows the removal of the heavy (creosote or anthracene) oil fractions. The pitches are complex mixtures containing many different individual organic compounds, and the precise compositions and properties vary according to the source of the tar and method of removal of low molecular weight fractions. Smith and Guigon reported that roughly two-thirds of the compounds isolated hitherto from the coal tar pitch are aromatic, and the rest are heterocyclic. Most of the compounds of coal tar pitches have boiling points in the ranges 340-550 °C [43].

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## ПОЛУЧЕНИЕ УГЛЕРОДНЫХ ВОЛОКОН МЕТОДОМ ЭЛЕКТРОСПИННИНГ

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**Аннотация**

Электроспиннинг – универсальный метод получения непрерывных волокон диаметром от нескольких нанометров до микрометров из растворов или расплавов полимеров. Этот метод применим практически к любому растворимому или плавкому полимеру. Полимеры могут быть химически модифицированы различными добавками, начиная от простых частиц сажи, до комплексных соединений. Электроспиннинг представляется простым, но довольно сложным процессом, который зависит от множества молекулярных, технологических и технических параметров. Данный метод обеспечивает доступ к совершенно новым материалам, которые могут иметь сложные химические структуры. Результаты, описанные в данном обзоре, демонстрируют большой потенциал электроспиннинга в фундаментальных и прикладных исследованиях. Тем не менее, требуется больше исследований, чтобы точно контролировать все процессы при образовании различных волокон. В этом обзоре представлены обобщенные данные на основе анализа исследований по получению углеродных волокон методом электроспиннинга для их дальнейшего применения в промышленности.

**Ключевые слова:** электроспиннинг, полимеры, наноматериалы, углерод

# ЭЛЕКТРОСПИННИНГ ӘДІСІМЕН КӨМІРТЕК ТАЛШЫҚТАРЫН ӨНДІРУ

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**Аннотация**
Электроспиннинг – бұл диаметрі бірнеше нанометрден микрометрге дейінгі полимерлердің ерітінділерінен немесе балқымаларынан тұратын үздіксіз талшықтарды алудың әмбебап әдісі. Бұл әдіс кез-келген еритін полимерге қолданылады. Полимерлер әртүрлі қоспалармен химиялық түрде модифицирленген болуы мүмкін, олар қарапайым ұсақ бөлшектерден күрделі қосылыстарға дейін болады. Электроспининг – қарапайым, бірақ көптеген молекулалық, технологиялық және техникалық параметрлерге байланысты күрделі процесс болып табылады. Бұл әдіс күрделі химиялық құрылымға ие жаңа материалдарға қол жетімділікті қамтамасыз етеді. Осы шолуда келтірілген нәтижелер фундаменталды және қолданбалы зерттеулерде электроспиннингтің үлкен әлеуетін көрсетеді. Алайда, әртүрлі талшықтарды қалыптастыру кезінде барлық үдерістерді дәл бақылау үшін көбірек зерттеулер жүргізу қажет. Бұл шолу көміртегі талшықтарының өндірісте оларды одан әрі пайдалану үшін электроспиннинг әдісімен жүргізілген зерттеулерге негізделген жалпыланған деректерді ұсынады.
**Түйінді сөздер:** электроспиннинг, полимерлер, наноматериалдар, көміртек