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Centrifugal Spinning: An Alternative Approach to Fabricate Nanofibers at High Speed and Low Cost

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Nanofibers are an important class of material that is useful in a variety of applications, including filtration, tissue engineering, protective clothing, battery separators, energy storage, etc. So far, electrospinning is the most used method for producing nanofibers. However, the wide-spread commercial use of electrospinning is limited mainly due to its low production rate. Most other nanofiber production methods, such as melt-blowing, bicomponent fiber spinning, phase separation, template synthesis, and self-assembly, are complex and can only be used to make nanofibers from limited types of polymers. Centrifugal spinning is an alternative method for producing nanofibers from various materials at high speed and low cost. In centrifugal spinning, the spinning fluid is placed in a rotating spinning head. When the rotating speed reaches a critical value, the centrifugal force overcomes the surface tension of the spinning fluid to eject a liquid jet from the nozzle tip of the spinning head. The jet then undergoes a stretching process and is eventually deposited on the collector, forming solidified nanofibers. Centrifugal spinning is simple and enables the rapid fabrication of nanofibers for various applications. This article gives an overview on the centrifugal spinning process, and compares it with conventional nanofiber production methods.

Keywords nanofibers, centrifugal spinning, electrospinning, processing-structure relationships

1. Introduction

Nanofibers are an important class of material that is useful in a variety of applications, including filtration, tissue engineering, protective clothing, composites, battery separators, energy storage, etc. Currently, the most commonly used method for producing nanofibers is electrospinning.^{1–4} A literature search using the Web of Science™ database shows that Year 2013 alone had publications of more than 2,000 peer-reviewed journal articles with respect to electrospinning of nanofibers. Among these publications, over 50% focused on the investigation of the electrospinning process and the characterization of the resultant nanofibers, and the others mainly addressed the innovative use of electrospun nanofibers for various applications. Although electrospinning is popular in academic research, the wide-spread industrial use of electrospinning is limited mainly due to its low production rate.^{5,6} Other

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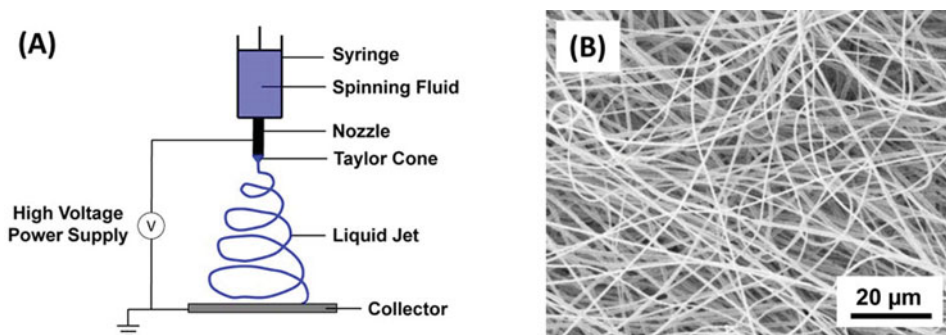


Figure 1. (A) Schematic of a single-nozzle electrospinning system, and (B) SEM image of PAN nanofibers produced by single-nozzle electrospinning.

nanofiber production methods have also been reported, such as melt blowing,⁷ bicomponent fiber spinning,^{8,9} phase separation,¹⁰ template synthesis,^{11–14} and self-assembly.^{15–18} However, the material choices for these complex methods are limited, which hinders their practical applications.^{10,11,15}

Therefore, the technological impact of establishing a new nanofiber production approach that can eliminate the limitations encountered by the above-mentioned methods would be significant. This article introduces an alternative process, centrifugal spinning, which can produce nanofibers from various materials at high speed and low cost. In centrifugal spinning, polymer solution or melt is placed in a rotating spinning head. When the rotating speed reaches a critical value, the centrifugal force overcomes the surface tension of the spinning solution or melt to eject a liquid jet from the nozzle of the spinning head. The jet then undergoes a stretching process and is eventually deposited on the collector surface forming dried nanofibers. As a simple and scalable process, centrifugal spinning avoids the use of high voltage and can be used to produce nanofibers from many different materials for various applications.

2. Conventional Nanofiber Production Methods

2.1. Electrospinning

Electrospinning is a simple, non-mechanical technique for fabricating nanofibers. Figure 1a shows a basic electrospinning setup. A typical single-nozzle electrospinning system contains a syringe, a metal nozzle, a power supply, and a collector. During electrospinning, a high voltage is applied between a syringe containing spinning fluid and a metallic collector. When the voltage reaches a critical value, the electrically-charged fluid generates a conical droplet (i.e., Taylor cone), from which a liquid jet is formed and elongated. The electrically-charged jet then undergoes a stretching-and-whipping process, during which the jet diameter decreases from hundreds of micrometers to as small as tens of nanometers. The as-spun nanofibers are accumulated on the surface of the grounded collector. Figure 1b shows an SEM image of polyacrylonitrile (PAN) nanofibers produced by electrospinning.

The morphology and diameter of electrospun nanofibers are dependent on a number of processing parameters that include: *i*) intrinsic properties of the polymer fluid such as rheological behavior, conductivity, surface tension, polymer molecular weight, and solution concentration; and *ii*) operational conditions such as electric field strength, solution

flow rate, nozzle diameter, nozzle-collector distance, and motion of the collector.^{3,19-24} Therefore, the electrospinning process can be adjusted to control the fiber diameter by varying the applied voltage, polymer solution concentration (or viscosity), or other processing parameters, while the duration of electrospinning controls the thickness of fiber deposition.^{2,23,24}

2.2. Melt Blowing

Melt blowing, first developed in the 1950s at the Naval Research Laboratory in Washington D.C., is currently one of the leading fiber and non-woven manufacturing processes. Typically, melt blown fibers have diameters larger than 1 μm ;²⁵⁻²⁷ however, it is possible to produce nanofibers from certain materials⁷ through careful control of operation conditions. The schematic of the melt-blowing process is shown in Fig. 2a. The polymer is first melted, and then polymer jets are extruded through multiple nozzles simultaneously, followed by attenuation by heated, high-velocity air streams, in which the diameters of the jets decrease drastically to obtain fine fibers. The fibers are eventually collected in random orientation to form a non-woven web. Figure 2b illustrates an SEM image of melt-blown polybutylene terephthalate (PBT) nanofibers. The structure of melt-blown fibers can be tuned by selectively adjusting parameters such as the flow rates of polymer melt and air, temperatures of polymer melt and air, intrinsic properties of polymer melt, nozzle geometry, etc.

2.3. Bicomponent Fiber Spinning

Nanofibers can be produced by splitting the different components or removing one of the components of bicomponent fibers that have two immiscible polymers arranged in the cross-section.^{8,9} In order to produce nanofibers, at least one of the components has a nanosized cross-section. Commonly-seen cross-sectional shapes of bicomponent fibers are islands-in-the-sea, segmented pie, and hollow segmented pie.²⁸ Figure 2c shows the cross-sectional shapes of these bicomponent fibers. Generally, when the islands-in-the-sea fibers are viewed in the cross-section, multiple individual fibrils (islands) made of one polymer component are surrounded by a matrix (sea) made of another polymer component. In the segmented pie fibers, two polymer components are arranged alternatively along the perimeter of the fiber cross-section, while the hollow segmented pie fibers also have a hollow structure in addition to the characteristics of the segmented pie fibers. These bicomponent fibers are formed by co-extruding two polymer melts through a spinneret with a desired cross-sectional arrangement.²⁹ After the bicomponent fibers are produced, splitting the components or removing one component is followed to make nanofibers. Figure 2d shows SEM images of CNT-filled PAN composite nanofibers synthesized by dissolving the sea (polymethyl methacrylate, PMMA) and sustaining the islands (CNT/PAN) from an islands-in-the-sea bicomponent fibers.

2.4. Phase Separation

The phase separation phenomenon can also be utilized to fabricate nanofibers. A typical phase separation process for making nanofibers involves polymer dissolution, gelation, phase separation, solvent removal, and drying.¹⁰ Figure 2e demonstrates the nanofiber formation using the phase separation. The polymer is first dissolved to form a homogeneous solution at room temperature or an elevated temperature. The solution is then maintained at the gelation temperature, where the polymer solution forms a gel and phase separates to

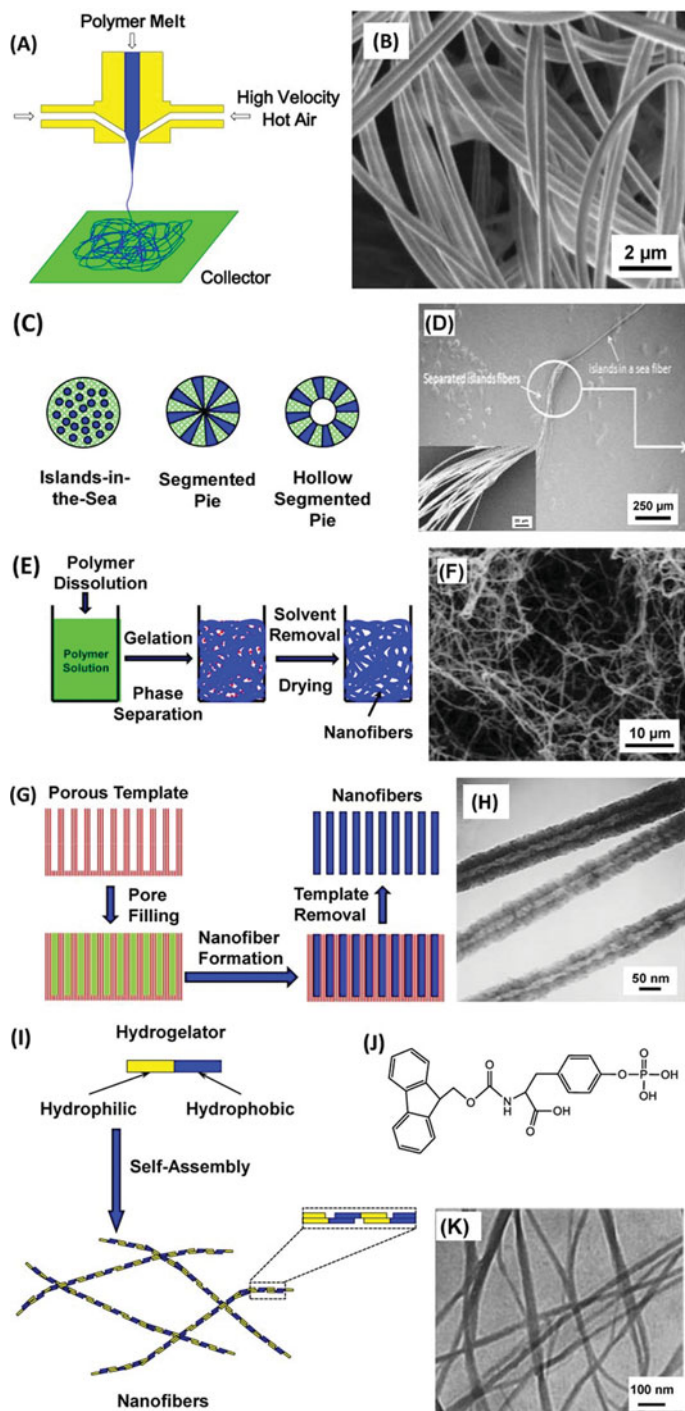


Figure 2. (Continued)

form a nanofibrous matrix. After removing the solvent and drying the matrix, nanofibers are produced. Figure 2f shows an SEM image of poly(L-lactic acid) (PLLA) nanofibers prepared by the phase separation method using a tetrahydrofuran solvent.

2.5. Template Synthesis

In the template synthesis method, nanofibers are produced in hollow channels of porous ceramic or polymer templates (Fig. 2g).^{11–14,30} The first step is to fill monomers in the porous template. Polymer nanofibers are then produced chemically or electrochemically from the monomers in the hollow channels of the porous template. Separated nanofibers are obtained after removing the template by dissolving or etching. Figure 2h shows an SEM image of polypyrrole (PPy) nanofibers prepared by the template synthesis method. Nanofibers produced by this method often have a hollow structure because the synthesized polymer is prone to precipitate onto the inner surface of the hollow channels. In addition to using monomers, nanofibers can also be produced directly from polymer solutions and in this method, polymer solutions, instead of monomers, are fed into the hollow channels and solidified into nanofibers after the removal of the solvent. Nanofibers produced from polymer solutions usually have larger diameters compared to those prepared from monomers because the high viscosity of polymer solutions makes it difficult to use the hollow channels with small diameters.

2.6. Self-Assembly

In the self-assembly approach, nanofibers are produced by holding small molecules together using intermolecular interactions. Different mechanisms can be employed to assemble nanofibers depending on the chemical structures of the small molecules. One of the commonly used mechanisms is based on the formation of hydrogel, which involves two interpenetrated phases, namely the liquid phase, which is typically water, and the solid phase, which is a network of nanofibers formed by the self-assembly of hydrogelator

Figure 2. (Continued) (A) Schematic of melt blowing process, (B) SEM image of PBT nanofibers produced by melt blowing.⁷ (C) Typical cross-sectional shapes of bicomponent fibers used for producing nanofibers, (D) SEM images of CNT/PAN composite nanofibers prepared from an islands-in-the-sea bicomponent fiber (island: CNT/PAN, sea: PMMA). The low magnification image shows the separation of CNT/PAN nanofibers from the islands-in-the-sea fibers after the removal of the sea component using nitromethane solvent, while the inset displays the separated CNT/PAN nanofibers.⁸ (E) Schematic of phase separation process, (F) SEM image of PLLA nanofibers produced by phase separation.¹⁰ (G) Schematic of template synthesis process, (H) SEM image of PPy nanofibers prepared by template synthesis.¹³ (I) Assembly of nanofibers by hydrogelator molecules, (J) chemical structure of a hydrogelator molecule, (K) SEM image of nanofibers formed by the self-assembly of hydrogelator molecules.¹⁷

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Table 1

Comparison of production rate, material choice, safety and cost for different nanofiber production methods.^{71–75}

Method	Production Rate	Material Choice	Safety	Cost
Centrifugal Spinning	High	Broad	High	Low for industrial production, medium for academic research
Electrospinning	Low	Broad	Relatively low due to the use of high voltage	Low for academic research, high for industrial production of nanofibers due to low production rate
Melt Blowing	High	Narrow	High	Medium
Bicomponent Fiber Spinning	Medium	Narrow	High	Medium
Phase Separation*	N/A	Narrow	High	N/A
Template Synthesis*	N/A	Narrow	High	N/A
Self-Assembly*	N/A	Narrow	High	N/A

*These three methods have only been used for laboratory research and their production rate and cost are not reported.

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molecules.^{15–18} Dried nanofibers are obtained after the removal of the liquid phase from the hydrogels. Figure 2i shows the formation of nanofibers by the self-assembly of hydrogelator molecules that have both hydrophilic and hydrophobic ends. Here, the self-assembly of nanofibers is achieved by weak intermolecular hydrogen bonding and hydrophobic interactions between the hydrogelator molecules. Figures 2j and 2k show the chemical structure of a hydrogelator and the nanofibers formed by the hydrogelator molecules, respectively.

2.7. Limitations of Conventional Nanofiber Production Methods

The comparison of production rate, material choice, safety, and cost between different nanofiber production methods is shown in Table 1. Electrospinning is the most studied nanofiber production method. However, the major obstacle for its wide-spread industrial application is its low production rate with the accompanied high cost per gram of produced nanofibers. The single-nozzle electrospinning process shown in Fig. 1a can only produce 1–100 mg nanofibers per hour.^{22,31–35} The production rate can be improved by multi-nozzle electrospinning or nozzle-less electrospinning; however, their production rates are still not sufficient for applications in which large amount of nanofibers are needed. The lab-scale electrospinning device is simple to set up, low cost, and is suitable for academic research. However, for scale-up production, the equipment becomes more complex and expensive.

Electrospinning can be used to produce nanofibers from various materials. However, the material choices for other nanofiber production methods, including melt blowing,⁷ bicomponent fiber spinning,^{8,9} phase separation,¹⁰ template synthesis,^{11–14} and self-assembly,^{15–18}

are limited. For example, melt blowing, a highly productive route, has been used widely in industry for producing fibers with diameters greater than $1\ \mu\text{m}$. When used for producing nanofibers, melt blowing is limited to a few polymers, such as PBT, polypropylene (PP), and polystyrene (PS).⁷ In bicomponent fiber spinning, it is critically important to distribute two different polymers on the fiber cross-section at the nanoscale, which requires the matching of their rheological properties, thereby limiting the material choice. Compared with electrospinning, the costs of melt blowing and bicomponent fiber spinning are moderate. The phase separation method involves multi-steps: gelation, phase separation, solvent exchange, and freezing-drying. So far, only a few polymers, such as polylactide (PLA) and polyglycolide, have been made into nanofibers using the phase separation method.¹⁰ In template synthesis, nanofibers are prepared by directly filling polymers in hollow channels of porous templates, followed by a tedious template removal process.^{12–14} This method cannot produce nanofibers with long fiber length. The self-assembly method produces nanofibers by forming supramolecular hydrogels through the self-assembly of small molecules via weak interactions such as hydrogen bonding and hydrophobic interactions.^{15–17} Currently this method is limited to prepare nanofibers from small active molecules (e.g., vancomycin, dipeptides, and glucosamine) that can self-assemble by themselves or under an external stimulus.¹⁵

Therefore, the technological impact of establishing a new nanofiber production approach that can eliminate the limitations encountered by the above-mentioned methods would be significant. Recently, centrifugal spinning, also called rotary spinning or rotational jet spinning, gained attention due to its capability and feasibility in generating large quantities of nanofibers with well-defined structures at high speed and low cost. The following sections present the history, fiber formation process, spinning systems, material types, and processing-structure relationships of centrifugal spinning.

3. A Brief History of Centrifugal Spinning

Centrifugal spinning is not new to the industry. For example, centrifugal spinning has been extensively used for producing glass fibers (also called fiberglass or glass wool) with diameters greater than $1\ \mu\text{m}$ for thermal insulation and filtration applications for over half a century. In the fiberglass industry, centrifugal spinning is known as the glass wool process.³⁶ Figure 3 shows the schematic of a typical centrifugal spinning process that is used in the fiberglass industry. A stream of molten glass is fed into a spinning head, which is basically a metal container with several thousand holes uniformly distributed around on the sidewall. To keep the glass at its liquid state, the temperature in the spinner is typically maintained at $900\text{--}1100^\circ\text{C}$. The spinning head rotates at a high speed, ranging from 2000 rpm to more than 3000 rpm. The centrifugal force created at the high rotating speed causes molten glass to flow through the small holes in the sidewall of the spinning head to form fine streams of glass. The glass streams coming out of the side wall are attenuated by high velocity air (or other gas or steam) and are broken into fine ($>1\ \mu\text{m}$) glass fibers which are several centimeters in length. A binder is sprayed on the glass fibers when they are formed. Broken glass fibers move through a “forming hood” placed under the spinning head, and they are subsequently collected on a conveyor belt in the form of a mat. The function of the forming hood is to distribute the broken fibers evenly to form a random alignment across the width and length of the collected glass fiber mat. The conveyor belt delivers the glass fiber mat through a curing oven which dries and cures the binder. The glass fiber mat can then be cut in desired lengths and widths for different uses. The centrifugal spinning of glass fibers is mainly conducted in industry, and detailed processing parameters such as rotor

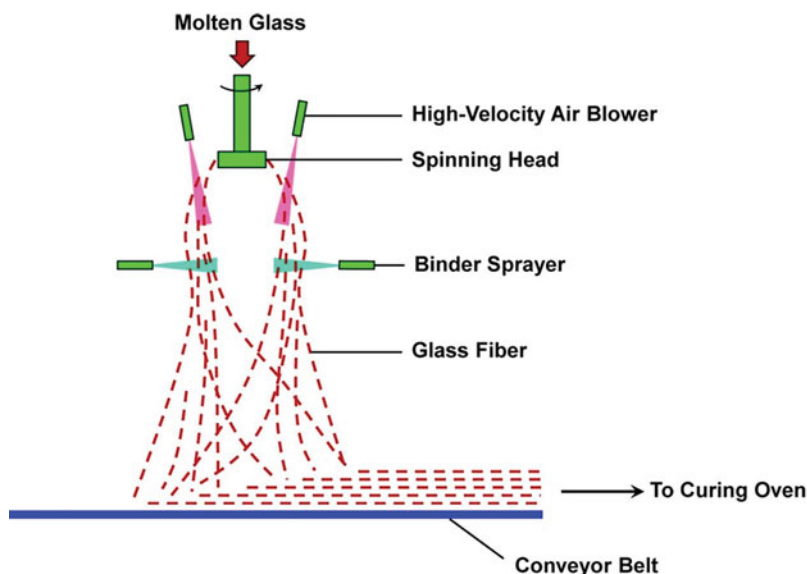


Figure 3. Schematic of a typical centrifugal spinning process used in the fiberglass industry. Forming hood is not shown for the purpose of simplicity.

diameter, spinning head radius, glass viscosity, flow rate, and external air temperature are not available in literature. However, it is seen that the concept of using centrifugal spinning to produce various fibers has seeded in this industrial practice.

Although centrifugal spinning has been widely used in the fiberglass industry for making micrometer-scale glass fibers, the use of centrifugal spinning for producing polymer fibers, especially polymer nanofibers is relatively new. In the 1990s, several companies, including BASF Aktiengesellschaft, Owens Corning Fiberglas Technologu, and Akzo Nobel NV have attempted to use centrifugal spinning to produce polymer fibers and have patents on spinning heads that can process polymers.^{37–39} More recently, FibeRio Technology Corporation commercialized large-scale centrifugal spinning machines (Cyclone FE 1.1 M/S and Cyclone FS 1.1) for mass production of polymer nanofibers. FibeRio's centrifugal spinning machines are based on a patented technology called Forcespinning® - The Force For Nanofibers®, which was developed by Lozano and Sarkar at the University of Texas at Pan American.⁴⁰ The Forcespinning® technology utilizes high-speed rotating spinning heads and can produce nanofibers from various polymers.

The centrifugal spinning of nanofibers has also attracted attention in academia. In 2008, Weitz and coworkers⁴¹ reported a so-called “unexpected finding of nanoscale fibers with a diameter down to 25 nm that emerge from a polymer solution during a standard spin-coating process.” Figure 4 shows the spinning process and an SEM image of the resultant PMMA nanofibers. During spinning, a 5 wt.% PMMA solution in chlorobenzene was first dripped onto the center of a rotating chuck of a spin-coater and nanofibers with average diameter of 25 nm were obtained at a rotating speed of 3,000 rpm. The fiber formation process relies upon the competition between the centrifugal force and the solution surface tension.⁴¹

In 2010, Lozano and coworkers⁴² reported a three-plate spinning head that allows the spinning of nanofibers at higher rotating speeds (Fig. 5a). Using such a spinning head system, they were able to obtain polyethylene oxide (PEO) nanofibers with diameters up to

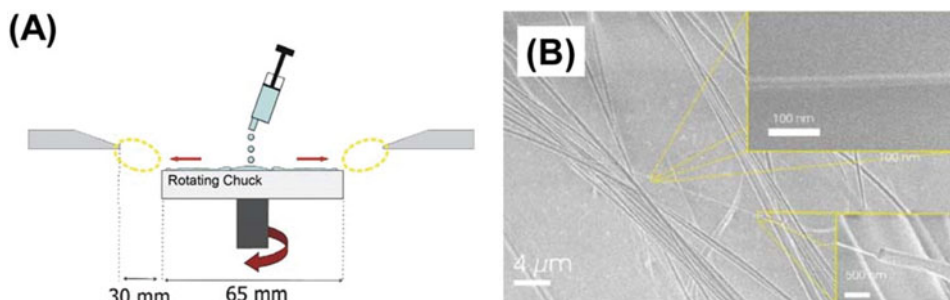


Figure 4. (A) Schematic of the spinning process used by Weitz et al., (B) SEM image of the resultant PMMA nanofibers.⁴¹

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300 nm (Fig. 5b), and the rotating speeds used were in the range of 3,000–5,000 rpm. This spinning head design eventually led to the Forcespinning[®] technology used by FibeRio Technology Corporation, as discussed above. Since 2010, there have been more academic researchers reporting their work on the centrifugal spinning of nanofibers, with a total of around 20 journal articles so far.^{43–59}

4. Nanofiber Formation During Centrifugal Spinning

Figure 6a shows a basic bench-top centrifugal spinning setup for the production of polymer nanofibers. During fiber formation, a spinning fluid is placed in a rotating spinning head, which is perforated with multiple nozzles around the sidewall. When the rotating speed reaches a critical value, the centrifugal force overcomes the surface tension of the spinning fluid, and liquid jets are formed from the nozzle tips of the spinning head. Figure 6b shows the path of a liquid jet after its ejection from the nozzle tip. The centrifugal force, together with the air frictional force, elongates the jets and leads to the formation of nanofibers. In addition to the centrifugal force and air friction force, other forces, such as rheological force, surface tension, and gravitational force, might also influence the nanofiber formation

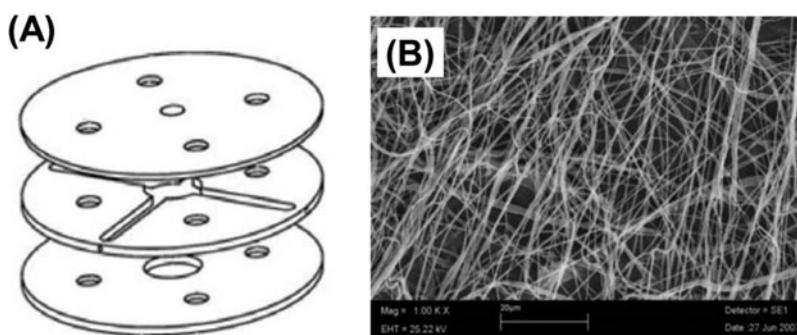


Figure 5. (A) Schematic of a three-plate spinning head system, and (B) SEM image of PEO nanofibers prepared using the spinning head system shown in (A).⁴²

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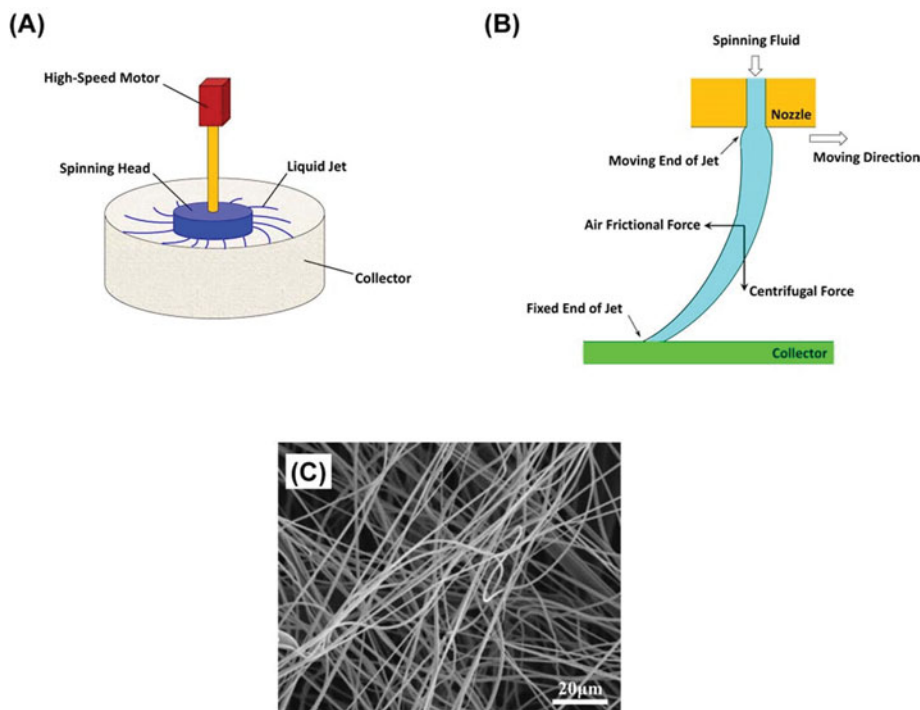


Figure 6. (A) Schematic of a basic bench-top centrifugal spinning setup, (B) the path of a liquid jet ejected from the nozzle tip during centrifugal spinning, and (C) SEM image of PAN nanofibers prepared by centrifugal spinning.

process. The stretched jets are deposited on the surface of the collector forming a nonwoven mat of nanofibers.

Figure 6c shows an SEM image of PAN nanofibers produced from this centrifugal spinning setup. One important feature of the centrifugal spinning process is its high production rate. With only two side-wall nozzles, the average production rate of a simple centrifugal spinning setup is around 50 g/hour which is at least two orders of magnitude higher than that of a typical lab-scale electrospinning process. Similar production rates have also been demonstrated by Lozano and coworkers⁵³ in their Forcespinning[®] system. High production rate is the most significant advantage of centrifugal spinning. While such high production rate is not often necessary for academic purposes, it can be a game changer in industry. The high production rate of centrifugal spinning suggests that it can be a low-cost, large-scale approach for mass production of nanofibers.

In order to thoroughly understand the nanofiber formation process in centrifugal spinning, researchers have conducted simulation and modeling work and brought valuable insights. For example, Badrossamay and coworkers⁵⁰ reported a scaling framework complemented by a semi-analytic and numerical method to characterize the regimes of nanofiber formation and proposed a theoretical model for fiber radius as a function of process parameters including viscosity, angular speed, radius of orifice, and distance to the collector, which is meaningful for controlling the morphological quality of centrifugally-spun nanofibers. Hlod and coworkers⁶⁰ described centrifugal spinning as a thin jet of viscous Newtonian fluid flowing between the rotor and coagulator under the influence of the two inertial body

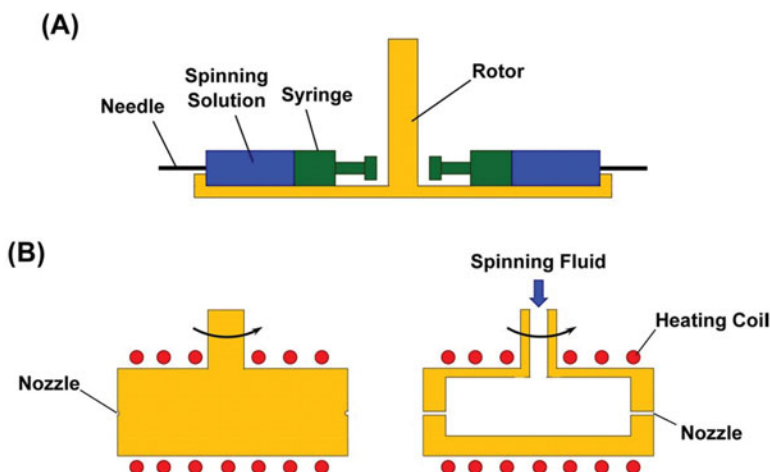


Figure 7. Schematics of (A) syringe-based spinning head, and (B) cylinder-shape spinning head.

forces (Coriolis and centrifugal forces) and forming fibers after hitting the collector. In the model, the vertical motion of the jet due to gravity was neglected and the thin jet flew in two dimensions in the horizontal frame. The researchers studied on the specific boundary conditions depending on the balance between the inertia and viscosity in the momentum transfer through the jet cross-section and found two possible regimes in the spinning process, namely inertial, where inertia dominates in the momentum transfer through the jet cross-section, and viscous-inertial, where viscosity dominates near the nozzle and inertia dominates near the collector. Two situations where the centrifugal spinning process is not possible were also described, either because the jet wrapped the rotor or due to the lack of a steady-jet solution. In this modeling work, the effects of inertia, longitudinal viscosity, and centrifugal forces on the fibers structure and mechanical properties were not given.

5. Centrifugal Spinning Systems

The key components for practical centrifugal spinning systems are: *i*) rotating spinning head, and *ii*) nanofiber collecting system.

5.1. Rotating Spinning Head

Figure 7a shows one simple spinning head design. In this design, two identical syringes containing the same amount of spinning fluids are connected to the rotor. At a high rotating speed, the centrifugal force generates two liquid jets simultaneously from the needles connected to the syringes. The challenge of this spinning head design is to constantly balance the weights of the two fluid-containing syringes during the entire centrifugal spinning process. The three-plate spinning head designed by Lozano and coworkers contains one single reservoir for the spinning fluid and can maintain balanced rotating during the spinning process (Fig. 5). Most recently reported work used cylinder-shape spinning head designs. Figure 7b shows one cylinder-shape spinning head design, where two nozzles are imbedded on the opposite sides of the cylinder wall. The diameter and length of the nozzle are important for controlling the fiber diameter. This cylinder-shape spinning head can spin nanofibers directly from a polymer solution. To spin nanofibers from a polymer

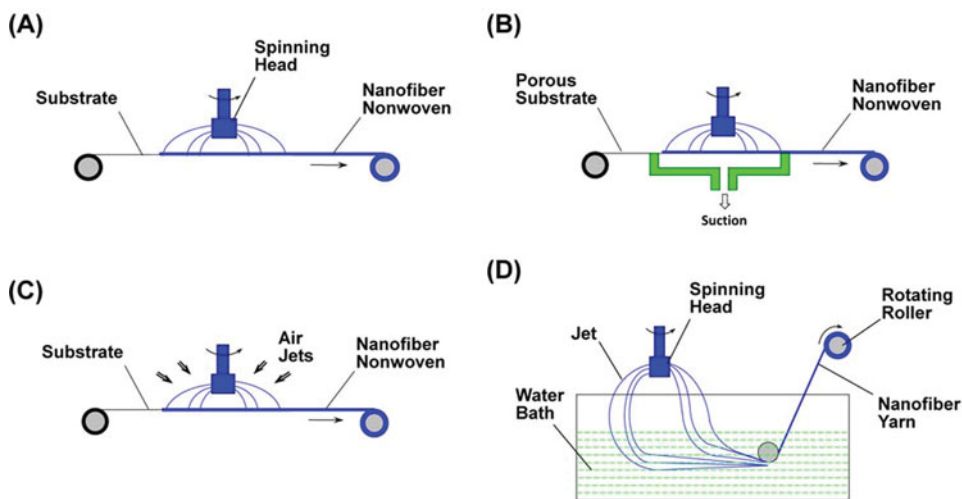


Figure 8. Schematics of (A) gravity-assisted nanofiber nonwoven collector, (B) suction force-assisted nanofiber nonwoven collector, (C) air jet-assisted nanofiber nonwoven collector, and (D) water bath-assisted nanofiber yarn collector.

melt, inductive heating coils or other heating elements need to be installed to melt the polymer. Multiple nozzles can be introduced to increase the production rate. In addition to cylinder spinning heads, other types of spinning heads, with spheroid, oblate spheroid, or trapezoidal shapes, can also be used.

5.2. Nanofiber Collecting System

To collect nanofibers in different forms, various types of collectors are employed. In the centrifugal spinning setup displayed in Fig. 6a, nanofibers are collected on the surface of the inner wall of a circular metal or plastic collector. The diameter of the circular collector, which determines the nozzle-collector distance, is an important parameter for controlling the structure of resultant nanofibers. The circular collector is suitable for batch production of nanofibers.

The continuous production of nanofibers can be realized by using a moving substrate placed under the spinning head (Fig. 8a). The gravity of nanofibers helps deposit nanofibers onto the substrate, forming a continuous nonwoven. The continuous collection of nanofiber nonwovens can be assisted by suction force (Fig. 8b) or air jets (Fig. 8c), which help adjust the distribution and packing density of nanofiber nonwovens. When suction force is used, porous substrates, such as textile fabric, paper, or other porous membranes, are needed for the collection of nanofiber nonwovens. Figure 8d shows a yarn collector. In this case, nanofibers are collected in a water bath and the rotating roller allows the collection of continuous yarns of nanofibers.

6. Types of Centrifugally-Spun Nanofibers

So far, centrifugal spinning is mainly used to produce nanofibers from polymer materials. However, nanofibers of other materials, such as carbon and ceramic, can also be obtained by the centrifugal spinning of their polymer precursors, followed by thermal treatments.

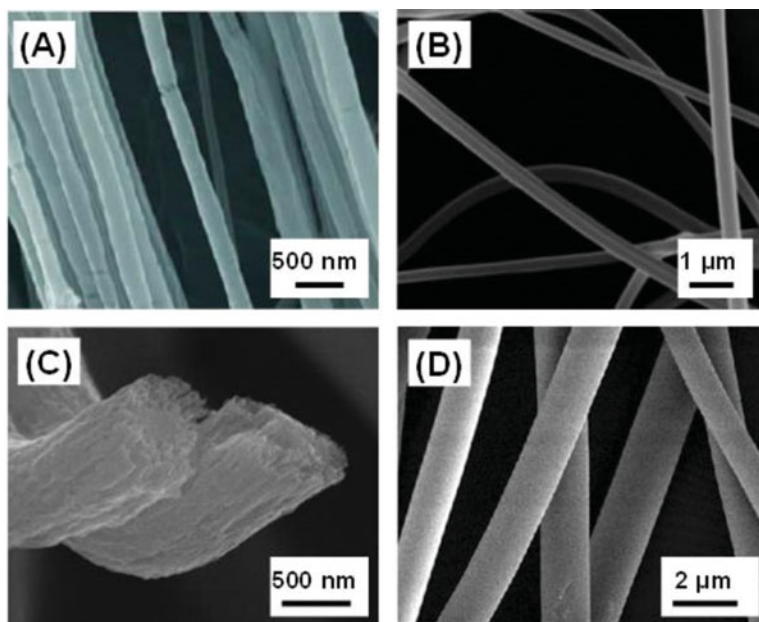


Figure 9. SEM images of (A) PEO nanofibers produced by centrifugal spinning of aqueous solution,⁴⁸ (B) PP nanofibers produced by centrifugal spinning of polymer melt,⁵⁴ (C) carbon nanofibers produced by carbonization of centrifugally-spun PAN/PMMA precursor, and (D) TiO₂ nanofibers produced by calcination of centrifugally-spun titanium tetrachloride precursor.⁴⁷

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6.1. Polymer Nanofibers

Polymer nanofibers can be produced by the centrifugal spinning of polymer solutions or melts. Several studies have demonstrated successful fabrication of micro/nanofibers through centrifugal spinning from solutions of various polymers, including PEO,^{48,51,52,61} PLA,^{46,48} PS,⁶² polyacrylic acid (PAA),⁴⁸ PVDF,⁴⁵ PMMA,⁶³ and polycaprolactone (PCL).⁴⁴ Figure 9a shows an SEM image of nanofibers prepared from an aqueous solution of 5 wt.% PEO by using a rotating speed of 12,000 rpm. It is seen that these PEO nanofibers with a smooth surface have an average diameter of about 500 nm. Using the centrifugal spinning approach, nanofibers could be aligned along the radial direction in respect to the spinning head, possibly resulting in reasonable strength of the resultant nanofiber mat, which might be required in certain applications.

Although most centrifugal spinning research focuses on the preparation of nanofibers from polymer solutions, it is feasible to produce nanofibers directly from polymer melts using the centrifugal spinning technology. For example, in 2011, Huttunen and Kellomaki⁵⁵ used a cotton-candy machine to produce fibers directly from polylactide melts. The cotton-candy machine works in a fashion similar to the centrifugal spinning process. However, the spinning head of a cotton-candy machine has relatively large nozzles and it rotates at a relatively low speed during operation. Hence, the polylactide fibers produced by Huttunen and Kellomaki⁵⁵ have diameters greater than 1 μm since cotton candy machines

do not have the appropriate operational parameters for nanofiber formation. More recently, Lozano's group⁵⁴ and Ellison's group⁵⁶ produced PP and PBT nanofibers, respectively, from their melts using appropriate operation parameters with the Forcespinning[®] system. Figure 9b shows the nanofibers produced from polypropylene melt using a rotating speed of 12,000 rpm and a processing temperature of 225°C. Producing nanofibers directly from polymer melts avoids the use of harmful solvents, and helps protect the production personnel and the environment as a whole. The absence of solvent during nanofiber formation also makes the centrifugal spinning technology more attractive since structures created by the solvent-free process are much easier to register for many commercial products, especially for medical applications.

6.2. Carbon Nanofibers

Carbon nanofibers (CNFs) are often prepared by the spinning of precursor polymers, such as PAN, polyimide, and pitch, etc., followed by subsequent heat-treatment of the resultant precursor fibers. Currently, a well-established approach to prepare CNFs involves the fabrication of electrospun carbon precursor nanofibers, followed by a two-step thermal treatment process including stabilization in air and carbonization in inert atmosphere.⁶⁴ However, the carbon precursor fibers can also be prepared via centrifugal spinning process. For example, we have prepared porous carbon nanofibers by the carbonization of centrifugally-spun PAN/PMMA precursor nanofibers at 800°C in argon gas (Fig. 9c). During the carbonization process, the PAN polymer was converted to carbon while PMMA degraded to generate a porous structure. These porous carbon nanofibers are lightweight, have a large surface area, good electrical conductivity, excellent adsorptive capacity, and a strong affinity for bacteria and other microorganisms. Hence, they can be used in applications for aerospace, semiconductors, metal processing, and energy conversion and storage.

6.3. Ceramic Nanofibers

Ceramic nanofibers can also be synthesized by the calcination of centrifugally-spun precursor nanofibers. In 2013, Liu and coworkers⁴⁷ prepared TiO₂ nanofibers from titanium tetrachloride precursor. Before centrifugal spinning, acetylacetone was used as a chelating agent to convert titanium tetrachloride into spinnable polyacetylacetonatotitanium. After centrifugal spinning, the precursor nanofibers were then calcinated at 700°C to form TiO₂ nanofibers. Figure 9d shows an SEM image of the TiO₂ nanofibers. Using a similar approach, Liu and coworkers⁵⁹ have also prepared ZrO₂ nanofibers by the calcination of centrifugally-spun zirconyl chloride precursor.

7. Processing-Structure Relationships of Centrifugal Spinning

In centrifugal spinning, the morphology of nanofibers is dependent on a number of processing parameters: *i*) intrinsic properties of the spinning fluid such as viscosity, surface tension, molecular structure, molecular weight, solution concentration, solvent structure, and additive; and *ii*) operational conditions such as rotating speed, spinning head diameter, nozzle diameter, and nozzle-collector distance.⁶⁵

7.1. Fluid Properties

There are many fluid properties that can affect the structure of nanofibers. When a polymer solution is used to produce nanofibers, the most important solution properties include

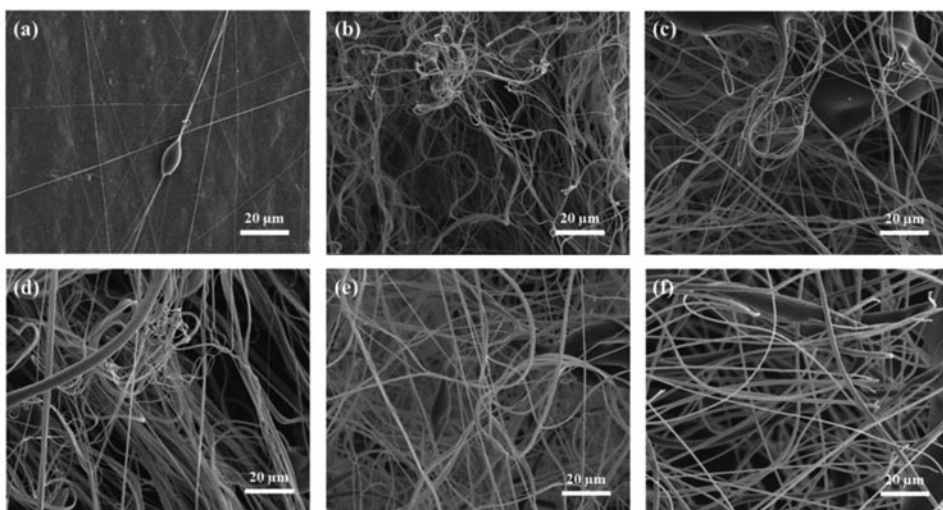


Figure 10. SEM images of PAN nanofibers centrifugally-spun from solutions with various Berry numbers: (a) 7.2, (b) 9.0, (c) 10.8, (d) 11.7, (e) 12.6 and (f) 13.5.⁶⁵

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viscosity, surface tension, molecular structure, molecular weight, solution concentration, solvent structure, and additive. However, not all of these properties are equally important in deciding nanofiber structure: Viscosity and surface tension dominate fiber formation, whereas the other factors influence the process by altering these two solution properties. Analogously, when the spinning fluid used is a polymer melt, melt properties including viscosity, surface tension, molecular structure, molecular weight, and additive synergistically influence the fiber formation process with the first two as key dominating factors. Therefore, this section focuses the discussion on how to manipulate the nanofiber structure by controlling the viscosity and surface tension of spinning fluids.

Viscosity.—The viscosity of a polymer melt is typically controlled by selectively adjusting the molecular structure, molecular weight, and processing temperature. However, the most rational and convenient way to control polymer solution viscosity is to adjust the concentration of the polymer in the solution. In addition to directly adjusting concentration, it is also reasonable to use Berry number (Be) to relate the viscosity and the diameter of nanofibers. Be , a dimensionless term, is the product of intrinsic viscosity and solution concentration and is widely used in electrospinning for controlling the fiber morphology.^{66,67} Similarly, in the centrifugal spinning process, Be can also be used to describe the influence of polymer chain entanglements on the diameters of the resultant nanofibers. In general, to produce nanofibers using centrifugal spinning, Be should surpass a critical value, Be^* , at which concentration and viscosity are adequately high and polymer chains are sufficiently entangled. On the contrary, if Be is lower than Be^* , inadequate chain overlap will result in difficulty in fiber formation.

Figure 10 shows SEM images of PAN nanofibers centrifugally-spun from solutions with Be values ranging from 7.2 to 13.5. At a low Be of 7.2, the polymer chains in the solution are rarely overlapped, and hence few fibers are produced and loosely aligned with large beads on them, forming a bead-on-string structure. If Be reaches 9.0 or

Table 2

Fiber diameter statistics of centrifugally-spun PAN nanofibers demonstrating the effects of solution concentration and operational conditions. Experiments were carried out with a spinning head diameter of 3 cm and nozzle length-to-diameter ratio of 7.5 at ambient temperature

	Solution Concentration (wt. %)	Berry Number	Rotational Speed (rpm)	Nozzle Diameter (mm)	Nozzle-Collector Distance (cm)	Fiber Diameter (nm)
Effect of Concentration	8	7.2	4,000	0.4	10	N/A*
	10	9.0	4,000	0.4	10	406 ± 108
	12	10.8	4,000	0.4	10	458 ± 135
	13	11.7	4,000	0.4	10	440 ± 118
	14	12.6	4,000	0.4	10	665 ± 114
	15	13.5	4,000	0.4	10	1077 ± 302
Effect of Rotating Speed	13	11.7	2,000	0.4	10	663 ± 232
	13	11.7	3,000	0.4	10	541 ± 153
	13	11.7	4,000	0.4	10	440 ± 118
Effect of Nozzle Diameter	14	12.6	4,000	0.4	10	665 ± 114
	14	12.6	4,000	0.8	10	807 ± 190
	14	12.6	4,000	1	10	895 ± 256
Effect of Nozzle- Collector Distance	14	12.6	4,000	0.4	10	665 ± 114
	14	12.6	4,000	0.4	20	658 ± 149
	14	12.6	4,000	0.4	30	647 ± 146

*The amount of fibers produced at this solution concentration is inadequate for statistics.

greater, the polymer is completely converted into fibers without the formation of beads, indicating significant chain entanglement is present in the solution. Given the different morphology and fiber yield in Figs. 10a and 10b, it is reasonable to infer that Be^* is somewhere between 7.2 and 9.0. Through the fiber diameter statistics for nanofibers produced from various solution concentrations (Table 2), it can be concluded that the fibers become larger in diameter with higher Be , suggesting the importance of selecting a proper Be value so that large amount of bead-free, small-diameter nanofibers can be obtained.

Surface Tension.—Surface tension also plays an important role in nanofiber formation. Surface tension, a driving force for bead formation in conventional spinning techniques, is prone to transform the liquid jet to spheres by restricting the surface area.⁶⁸⁻⁷⁰ During centrifugal spinning, three forces, namely surface tension, centrifugal force, and rheological force interplay and co-determine the morphology of the resultant product. Specifically speaking, centrifugal force tends to draw the liquid jet and enlarge the surface area, while rheological force resists rapid changes in shape and hence assists the formation of smooth fibers. In practice, adjusting molecular structure, molecular weight, and solvent type are feasible approaches to control surface tension. It is also possible to adopt other easier ways, such as adding additives and mixing solvents with different surface tensions.

7.2. Operational Conditions

Operational conditions that can influence the structure of centrifugally-spun nanofibers include rotating speed, spinning head diameter, nozzle diameter, nozzle-collector distance, etc.

Rotating Speed.—The rotating speed of the spinning head, which directly affects the centrifugal force and air frictional force, is one of the most important operational conditions. During centrifugal spinning, the centrifugal force along with the air frictional force elongates the liquid jets into nanofibers. When the spinning fluid is placed in a rotating nozzle tip, the centrifugal force (F_{centri}) exerted on the liquid can be described by:

$$F_{centri} = m\omega^2 D/2 \quad (1)$$

where m is the mass of the fluid, ω the rotating speed of the spinning head, and D the diameter of the spinning head. To eject the liquid jet from the nozzle tip, the rotating speed must surpass a critical value, at which sufficient centrifugal force is generated to overcome the surface tension of the spinning fluid. Thus, to produce nanofibers, it is crucial to determine the critical rotating speed of the spinning fluid. Different spinning fluids have different surface tensions and viscosities, and hence the rotating speed needed to generate sufficient centrifugal force is also different. In addition, the spinning head design, especially the diameter, affects the rotating speed needed for centrifugal spinning. In literature, the rotating speed used for centrifugal spinning of polymer nanofibers varies from 3,000 to 12,000 rpm.^{44,48}

After the liquid jet ejects from the nozzle tip, the frictional force applied onto the jet can still be calculated by Eq. (1); however, ω should be changed to the rotating speed of the jet and D becomes the diameter of the path of the jet. Meanwhile, the air frictional force (F_{fri}) can be calculated by:

$$F_{fri} = \pi C \rho A \omega^2 D^2 / 2 \quad (2)$$

where C is a numerical drag coefficient, ρ the air density, A the cross-sectional area of the jet, ω the rotating speed of the jet, and D the diameter of the path of the jet.

As the liquid jet travels from the nozzle tip towards the collector, its rotating speed decreases gradually. However, a higher rotating speed of the spinning head always gives a higher rotating speed of the jet after a certain distance of travel. Hence, the centrifugal force and air frictional force applied on the liquid jet increase as the rotating speed of the spinning head increases, leading to greater elongation of the liquid jet and the reduction of fiber diameter. Figures 11a to 11c show SEM images of PAN nanofibers centrifugally-spun with different rotating speeds, and the average fiber diameters of these fibers are displayed in Table 2. It can be seen that with rotating speed increasing from 2,000 rpm to 4,000 rpm, the average fiber diameter decreases from 663 nm to 440 nm.

However, it must be noted that when the rotating speed is too high, thick fibers might be obtained since the flight time for the liquid jet to reach the collector is shorter, and the reduced flight time is not sufficient for the jet to stretch and elongate. Moreover, higher rotating speed also results in larger mass throughput of the jet, which also contributes to larger fiber diameter. Thus, it is critical to determine the optimal rotating speed that can bring about the smallest fiber diameter.

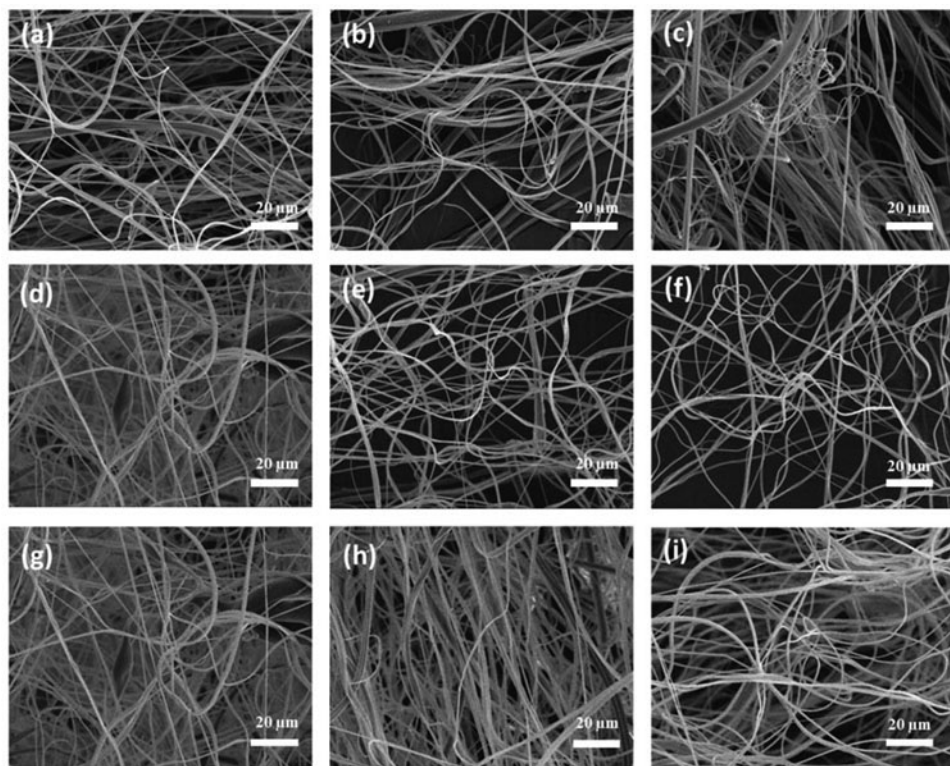


Figure 11. SEM images of centrifugally-spun PAN nanofibers demonstrating the effects of rotational speed: (a) 2,000 rpm, (b) 3,000 rpm, and (c) 4,000 rpm; nozzle diameter: (d) 0.4 mm, (e) 0.8 mm and (f) 1.0 mm; and nozzle-collector distance: (g) 10 cm, (h) 20 cm, and (i) 30 cm.⁶⁵

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Spinning Head Diameter.—The diameter of the spinning head is another crucial parameter to determine the structure of nanofibers. According to Eq. (1), when the rotating speed is constant, the centrifugal force increases as the spinning head diameter increases. Consequently, it is easier to eject the liquid jet from a spinning head that has a larger diameter. In addition, a larger spinning head diameter is also beneficial for forming thinner fibers by exerting greater stretching and elongation of the liquid jet. However, in practice, the maximum diameter of the spinning head is often limited by the capability of the high-speed motor. When the spinning head diameter is too large, it is difficult for the high-speed motor to maintain balanced rotating.

Nozzle Diameter.—Adjusting the nozzle diameter is another means to control nanofiber structure. In this approach, the mass throughput of liquid jet is adjusted to alter the nanofiber structure. Using a nozzle with smaller diameter basically restrains the mass throughput and thus yields finer nanofibers. Figures 11d to 11f display SEM images of centrifugally-spun PAN nanofibers using different nozzle diameters, and their average fiber diameters are shown in Table 2. When changing the nozzle diameter from 1.0 mm to 0.4 mm, the average fiber diameter decreases from 895 nm to 665 nm, suggesting the significance of choosing a small nozzle diameter when thinner fibers are desired.

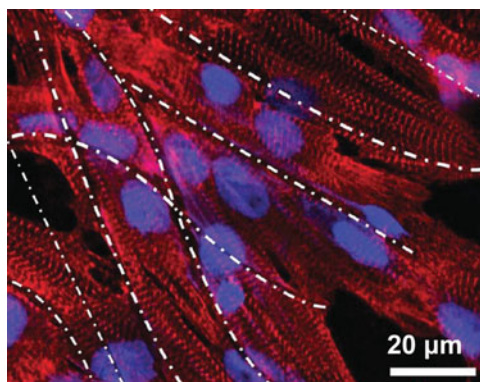


Figure 12. Laser scanning confocal image of anisotropic cardiac muscle on a centrifugally-spun PLA scaffold. PLA fibers are marked with dashed white lines, nuclear DNA of the myocyte is stained in blue, and R-actinin of the myocyte is red.⁴⁸

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However, if the nozzle diameter is too small, it becomes impossible to eject the liquid jet and therefore nanofibers cannot be formed.

Nozzle-Collector Distance.—The nozzle-collector distance has a direct influence on the liquid jet's flight time. When a solution is used as the spinning fluid, a minimum distance between the nozzle and the collector is required so that the liquid jet can have sufficient time to evaporate most of the solvent before reaching the collector. In addition, given an increased nozzle-collector distance, the liquid jet must travel a lengthened course, which is favorable for reducing fiber diameter. However, compared with other operational conditions, the nozzle-collector's distances influence on fiber diameter is less significant. Figures 11g to 11i demonstrate SEM images of PAN nanofibers centrifugally-spun using different nozzle-collector distances, and their average fiber diameters are shown in Table 2. It is seen that when the nozzle-collector distance increases from 10 cm to 30 cm, the average fiber diameter exhibits insignificant change (from 665 nm to 647 nm). This result reveals that 10 cm is a sufficiently long distance for solvent evaporation and the nozzle-collector distance greater than 10 cm has negligible influence on the fiber diameter.

8. Application of Centrifugally-Spun Nanofibers

Centrifugally-spun nanofibers generally can be used in the same applications as those fabricated by electrospinning and other conventional methods. So far, the most studied application of centrifugally-spun polymer nanofibers is tissue engineering. Badrossamay and coworkers⁴⁸ used centrifugal spinning to prepare PLA nanofiber scaffold. To evaluate the feasibility of using centrifugally-spun PLA nanofibers as tissue engineering scaffolds, they seeded chemically dissociated neonatal rat ventricular myocytes on the as-prepared fibrous constructs. It was found that the myocytes were spontaneously bound to and aligned with the fibers. In addition, the multicellular constructs self-organized along the fibers and formed anisotropic muscle with aligned and elongated myocytes and ordered myofibrils (Fig. 12). Their work demonstrated that centrifugal spinning is a simple, facile means to produce anisotropic, biodegradable nanofiber scaffolds.

In another study, Ren and coworkers⁴⁶ investigated the capability of centrifugal spinning in preparing tissue engineering scaffold from polyvinylpyrrolidone (PVP) and PLLA composite fibers. During the centrifugal spinning, PVP and PLLA were phase-separated and after the composite fibers were synthesized, PVP was etched away from the binary system to adjust the hydrophilicity of the fibrous constructs and create more pores and surface roughness, which are favorable features for successful cell attachment and proliferation. Using human dermal fibroblasts to simulate partial skin graft, this study demonstrated that centrifugally-spun porous PLLA nanofiber constructs are promising tissue engineering scaffolds with enhanced cell attachment and growth. Similarly, Wang's team assessed the use of centrifugally-spun poly-lactic-co-glycolic acid and PS nanofibers as cell culture scaffolds.⁶² On the other hand, Amalorpava Mary and coworkers⁴⁹ evaluated the feasibility of using centrifugally-spun PCL and PVP nanofiber blends as drug delivery vehicle.

Although current research focuses on the application of centrifugally-spun nanofibers in tissue engineering, it can be expected that centrifugally-spun nanofibers—similar to their electrospun counterparts—are favorable candidates in applications such as filtration, protective clothing, composites, aerospace, semiconductors, and energy conversion and storage. For instance, we prepared porous carbon nanofibers by centrifugal spinning of PAN/PMMA precursor nanofibers and subsequent heat-treatment at 800°C in argon gas. During the heat-treatment, PAN was converted to carbon while PMMA acted as pore generator since it can thermally degrade at elevated temperatures. The resultant porous carbon nanofibers exhibit good electrical conductivity and large surface area, and thus can be used as the electrode material in energy storage systems such as supercapacitors. Figure 13 displays the electrochemical performance of the porous carbon nanofibers as electrodes in supercapacitors. The highest specific capacitance achieved for this material is 170 F/g and more than 80% of the capacitance is retained after 1,000 cycles.

9. Summary and Outlook

Nanofibers are an important class of material that is useful in a variety of applications. So far, electrospinning is the mostly used method for producing nanofibers. However, the wide-spread commercial use of electrospinning is limited mainly due to its low production rate. Most other nanofiber production methods, such as melt-blowing, bicomponent fiber

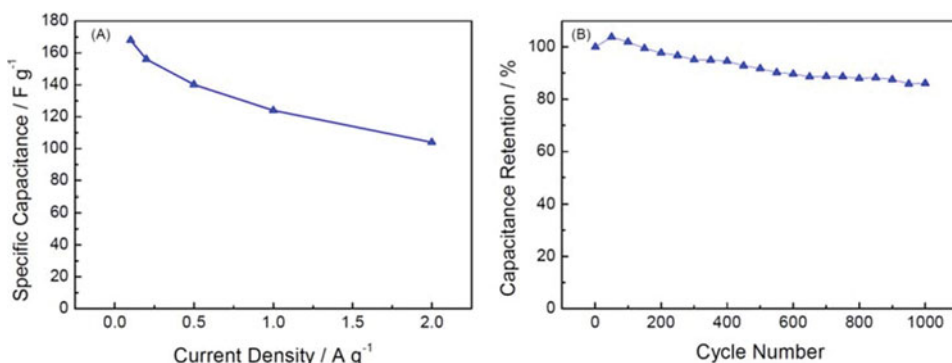


Figure 13. Capacitive performance of porous carbon nanofiber electrode prepared from centrifugally-spun PAN/PMMA precursor fibers with the weight ratio of PAN: PMMA = 1:1: (A) rate capability at different current densities, and (B) capacitance retention at 0.5 A/g for 1,000 cycles.

spinning, phase separation, template synthesis, and self-assembly, are complex and can only be used to make nanofibers from limited types of polymers. Centrifugal spinning is an alternative process to produce nanofibers. In centrifugal spinning, the spinning fluid is placed in a rotating spinning head. When the rotating speed reaches a critical value, the centrifugal force overcomes the surface tension of the spinning fluid to eject a liquid jet from the nozzle of the spinning head. The jet then undergoes a stretching process and is eventually deposited on the collector surface forming dried nanofibers.

Compared with conventional nanofiber manufacturing approaches, centrifugal spinning has several remarkable advantages: the approach is easy to deploy, it avoids the use of high electric fields, it can work with concentrated polymer solutions or viscous polymer melts, and the technology has high production rate and low cost. Consequently, centrifugal spinning can be used to produce polymer nanofibers, carbon nanofibers, ceramic nanofibers, etc. The morphology and diameter of centrifugally-spun nanofibers are controlled by selectively adjusting the intrinsic properties of the spinning fluid such as viscosity, surface tension, molecular structure, molecular weight, solution concentration, solvent type, and additive type and concentration, and the operational conditions such as rotating speed, spinning head diameter, nozzle diameter, and nozzle-collector distance. To date, the exploration in potential applications of centrifugally-spun nanofibers is mainly concentrated on tissue engineering scaffolds. However, nanofibers produced by centrifugal spinning can also be potentially employed in protective clothing, filtration, aerospace, composites, semiconductors, and energy conversion and storage.

The above-mentioned advantages of centrifugal spinning make it a promising manufacturing candidate for producing nanofibers in a low-cost, large-scale fashion. However, up to now, centrifugal spinning is still less popular than electrospinning. First, from the viewpoint of academic research, the centrifugal spinning device is complex to set up, especially in terms of selecting appropriate motor and spinning head geometry. However, such complexity is less an issue when centrifugal spinning is being scaled up for industrial production. For the mass production of nanofibers by centrifugal spinning, the increase of production rate can be realized by simply increasing the number of spinning heads and the number of nozzles on each spinning head. Second, compared to electrospinning, centrifugal spinning usually uses polymer solutions that have higher concentrations, leading to slightly thicker fibers. This can be addressed by using motors with higher rotational speed. Third, the fiber mats collected in a lab-scale centrifugal spinning device are loosely packed, and compared to the densely-packed fiber mats prepared by electrospinning, such loosely-packed fiber mats might not be desirable in some applications. This issue can be addressed by post-treatments, such as hot pressing and calendaring, after fiber collection. Fourth, most currently-reported nanofibers by centrifugal spinning are based on a simple solid structure. However, it is possible to prepare core-shell nanofibers by centrifugal spinning of two solutions coaxially with carefully-designed spinning head. Hollow nanofibers may also be prepared by centrifugal spinning of islands-in-the-sea bicomponent fibers, followed by the removal of the "islands" component. The preparation of core-shell and hollow nanofibers can greatly broaden the application of centrifugally-spun nanofibers. Last but not least, most current studies on centrifugal spinning is focused on preparing various materials and their applications; however, more detailed work such as computational modeling on various forces involved in the process of centrifugal spinning and their impact on the mechanical properties of fibers, and studies on the influence of different temperatures on fiber structures can be done to help thoroughly understand the process and the processing-structure-property relationship. If the device set-up and collection issues can be addressed and the process can be better understood, centrifugal spinning will soon

become a preferable option of high-speed, low-cost nanofiber production. This will open up opportunities for the research and development in both academia and industry.

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