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Chapter 1

Electrospinning: an advanced nanofiber production technology

Haitao Niu, Hua Zhou and Hongxia Wang

The electrospinning process has been regarded as one of the most facile and versatile techniques to prepare nanoscale fiber materials. Electrospinning technology has undergone enormous progress since its appearance in the 1930s. This chapter briefly reviews the recent improvements on electrospinning technologies and summarizes the state-of-the-art electrospinning setups. Thus, this chapter will be a valuable resource for scientists in the electrospinning field and engineers in related areas.

1.1 Introduction to electrospinning

Nanomaterials are materials that have at least one dimension below 100 nm, e.g. nanoparticles, nanorods, nanowires, nanotubes, and nanosheets. Nanomaterials have attracted considerable attention in the past decades owing to their excellent properties, outstanding performances, and wide applications. As the dimension of a material decreases, the percentage of atoms on the surface and the surface-to-volume ratio increase remarkably [1]. For a 100 nm nanoparticle, less than 0.2% of atoms are on the surface; meanwhile, 10% of the atoms are on the surface of a 10 nm nanoparticle and around 90% atoms distribute on the surface of a 2 nm nanoparticle [2]. The atoms on the nanomaterial surface have more dangling bonds, which make them very active and tend to bond with adjacent molecules. As a result, nanomaterials, in comparison with their bulk counterparts, often exhibit higher chemical activity, lower melting points, and higher phase transition pressure and solubility [3].

In the fiber and textile industry, fibers with a diameter less than 1 μm (1000 nm) are normally defined as nanofibers. In comparison with conventional fibers, these nanofibers have an enlarged surface area; for instance, the surface area of electrospun nanofibers are dozens of times larger than that of conventional fibers (figure 1.1). Accordingly, the pore size reduces and pore volume increases in nanofibrous materials. As a result, nanofibers show excellent performance in

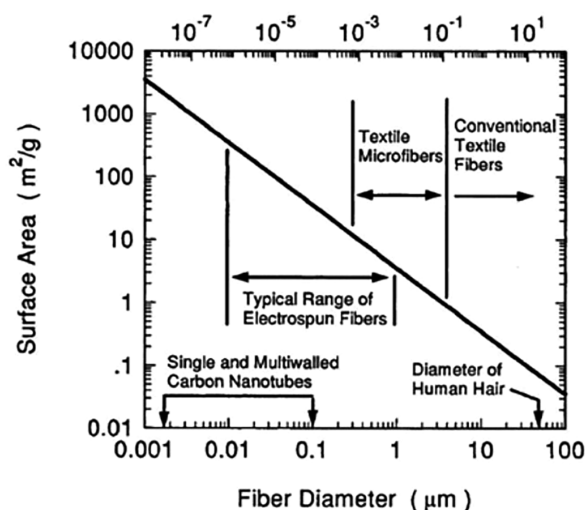


Figure 1.1. Relationship between specific surface area and diameter of different fibers. Reproduced with permission from Elsevier [11].

many different areas; for example, air/liquid filtration, energy generation and storage, biomedical and tissue engineering, sensors and catalysts, drug delivery, and nanocomposites [4–10].

Nanofibers can be fabricated by numerous approaches, such as phase separation [12], self-assembly [13, 14], template synthesis [15], melt-blowing [16], flash spinning [17], bicomponent spinning [18], and electrospinning [19, 20]. Among all the existing nanofiber-fabricating approaches, electrospinning technology is the most investigated and widely used because of its high efficiency, cost-effectiveness, and great adaptability. Especially, electrospinning technology is easy to scale up and has high commercialization potential.

1.1.1 Electrospinning history

In as early as 1600, William Gilbert first reported the observation about a spherical water drop on a dry surface being drawn and deformed into a cone under the influence of electrostatic force [21]. Nearly 300 years after Gilbert's observation, Formhal patented in the 1930s a setup to produce continuous fine fibers [22, 23]; this is regarded as the real beginning of electrospinning technology. In the 1960s, Geoffrey Taylor investigated the shape of a cone formed by a fluid droplet under the action of an electric field, and reported the existence of a conical angle of 49.3° [24]. This angle was later named the 'Taylor cone' and it has been widely used to explain electrospinning and electrospraying. In addition, Taylor [25] proposed that a jet in a parallel electric field experiences two critical instabilities: Rayleigh instability and bending instability. His works greatly improved the understandings of electrospinning and promoted its development.

Henceforth, there were no noteworthy improvements on electrospinning technology, until Doshi and Reneker [19, 26] reported their work on using this technique to

fabricate nanostructured materials. Since then, electrospinning technology and electrospun nanofibers have begun to draw more and more interest from both academic and industrial fields. Tremendous efforts have been devoted to the development of electrospinning technology, finding the fiber-making mechanism, characterization of nanofibers, and exploring novel applications. Large-scale production and commercialization of electrospun nanofibers have been accomplished. Electrospinning technology and processes have made fast advancement in recent years to meet the ever-increasing demands on nanofibers for various applications.

1.1.2 Basic apparatus

The conventional electrospinning setup (figure 1.2) comprises a capillary nozzle connected to a high-voltage DC power supply, a grounded collector, and a solution reservoir to supply solution [27]. This type of electrospinning setup works based on the capillary effect: the solution is transported to the tip of a thin nozzle and jet initiation happens at the nozzle tip. In the past two decades, many advancements have been made to control nanofiber collection, and various fiber-generating designs including needleless, near-field, melt electrospinning, yarn electrospinning, and multicomponent electrospinning designs, have been developed. An overview of this advanced fiber-making technology and state-of-the-art progress are reviewed in this chapter.

An electrospinning process can be briefly described as follows. Spinning fluid is fed to the capillary tube (usually a syringe needle) from a fluid reservoir. When a high voltage is applied to the needle (typically around 10 kV–20 kV), a high electric field is formed between the needle and collector (grounded or oppositely charged), which applies electrostatic force on the fluid droplet. Electrospinning fluid is described as a ‘leaky dielectric’ that has sufficient conductivity for the induced charges to quickly accumulate on the free surface in a short time scale or acts as a dielectric [28]. The repulsion between charges on the free surface of fluid droplet works against surface tension and fluid viscosity to deform the droplet into a cone

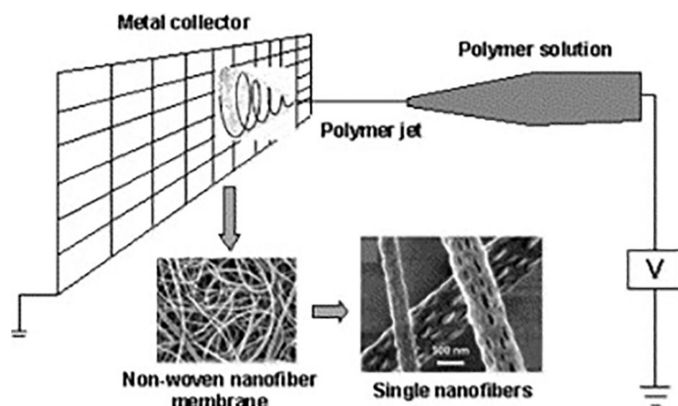


Figure 1.2. Illustration of typical electrospinning process. Reproduced with permission from Elsevier [27].

shape (Taylor cone) with increasing applied voltage (higher electric field intensity) [24]. When the applied voltage exceeds a critical value, the electrostatic force can overcome fluid surface tension, and jet initiation proceeds from the vertex of the Taylor cone. The generated fluid jets fly to the grounded collector under the action of electrostatic force. During the flying process, solvent evaporation from the fluid jet results in dry nanofibers depositing on the collector. Although the electrospinning process is relatively easy to implement, it is very complex considering the co-existence and combined action of Coulombic force, gravity, fluid surface tension, and viscosity during fiber formation.

1.2 Electrospinning basis

1.2.1 Mechanism of electrospinning process

The fiber formation process during electrospinning can be divided into three stages: jet initiation, jet whipping instability, and fiber deposition [29]. As the first stage of electrospinning, jet initiation has been widely studied [30–32]. In the first milliseconds, the solution droplet begins to transform into a conical shape by the high electric potential. The round solution droplet tip becomes more and more sharp. Finally, a jet is emitted from the tip of the cone. Then the cone gradually changes back to the rounded shape, indicating the system transits from the jet initiation stage to the electrospinning stage. As long as the solution taken away by the solution jet is replenished promptly, the electrospinning process can last.

After ejection, the solution jet is straight for a short section (figure 1.3), which may extend from a few millimeters to several centimeters away from the nozzle tip along its axis direction. This ejected solution jet carries away electrical charges in the



Figure 1.3. Digital photo of an electrospinning solution jet, including a stable jet and jet whipping stage. Reproduced with permission from Elsevier [39].

form of uncompensated ions from the nozzle. Attributed to the effect of charge repulsion in the solution jet, free charges migrate radially onto the jet surface to satisfy the equilibrium condition. As a result, electrostatic force induced by the electric field is applied on the jet surface. The charged fluid jet accelerates under the action of the electric field, accompanied by the thinning of the fluid jet [31].

After the initial stable stage, the jet enters an instable stage (whipping) under the influence of the charges carried by the jet, which may involve bending, winding, spiraling, and looping movements. Many theoretical models have been proposed to describe this jet instability, and there is a prevalent belief that the jet is continuously elongated and becomes longer and thinner with continual stretching [33–37]. The existence of jet instability is due to the action of axisymmetric and nonaxisymmetric instabilities caused by the perturbations of surface charges. The axisymmetric instability derives from perturbation of the surface charges along the jet axis direction, which makes different segments of the jet be under different strengths of electrostatic force, resulting in an uneven jet. The nonaxisymmetric instability derives from perturbation of surface charges around the circumference of the jet, which induces a localized torque around the jet that accounts for the whipping motion. Under the condition where nonaxisymmetric instability plays the major role of instability during the electrospinning process, the jet is likely to be stretched uniformly [38]. The solution jets fly to the collector under electrostatic force, being accompanied by solvent evaporation, and deposit on the collector as dry fibers at last.

1.2.2 Effects of electrospinning parameters

Electrospinning process, fiber morphology, fibrous structure, and fiber production rate are governed by a number of parameters, e.g. applied voltage, flow rate, nozzle diameter, collecting distance, solution properties (e.g. polymer molecular weight, concentration, electrical conductivity, surface tension, solvent properties), and ambient conditions (temperature, humidity) (figure 1.4). Although, many of these electrospinning parameters are interdependent and there are interactions between them, we can derive a general trend of influences of some parameters on the electrospinning process.

1.2.2.1 *Applied voltage*

Applied high voltage is an essential factor in electrospinning, without which jet initiation does not happen. When the applied voltage is low, the electrostatic force is insufficient to overcome surface tension of the solution droplet, and as a result, no jet is stretched out and dripping happens. With increasing applied voltage, the electrostatic force increases, and eventually leads to jet initiation, and the electrospinning process starts. Generally speaking, the fiber diameter decreases with increasing voltage, and attributable to the growing stretching force [40, 41]. Applied voltage plays a far more important role in needleless electrospinning than in nozzle electrospinning. It can decrease the fiber diameter and increase the fiber production rate because high voltage can increase the jet number on the free solution surface of a spinneret and escalate the jet flying speed [42, 43].

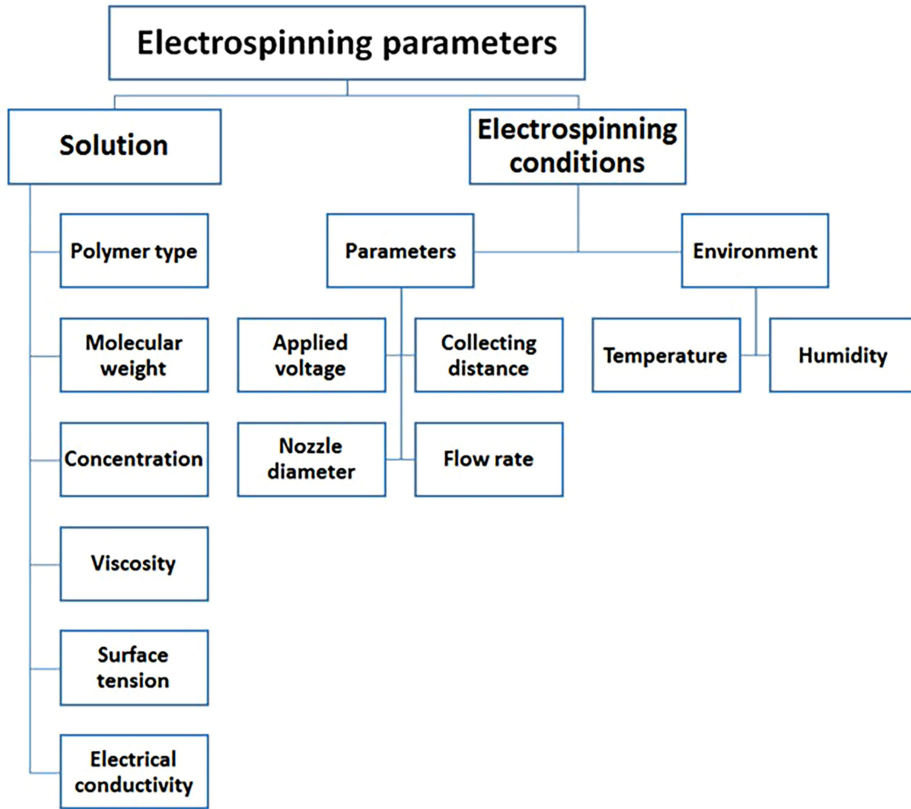


Figure 1.4. Important electrospinning parameters and conditions during the electrospinning process.

1.2.2.2 Collecting distance

In general, an optimum distance for electrospinning should be long enough for fiber stretching and solvent evaporation. Under the circumstance that applied voltage is constant, the change of distance between nozzle and collector will affect the electric field and fiber diameter accordingly. Another important influence of changing distance is on fiber morphology and structure. The solvent in the solution jet needs to have sufficient time to evaporate and turn the jets into dry nanofibers. When the collecting distance is very short, interconnected nanofibers are often collected [42–44]. This phenomenon happens more frequently in needleless electrospinning due to instantaneous generation of a large number of solution jets. It has also been found that interconnected nanofibers collected at a short distance could benefit the mechanical strength of nanofiber mats with improved and more durable performance in energy harvesting applications [44].

1.2.2.3 Flow rate

The flow rate of solution in nozzle electrospinning has a direct effect on fiber production rate: a large flow rate results in a high production rate and a small flow

rate results in a low production rate. A large flow rate will generally produce coarser nanofibers [45] because more solution is drawn out at the same time. It has been proposed that four electrospinning conditions may be observed with the increasing flow rate: discontinuous, continuous (stable), intermittent, and dripping. When the solution flow rate exceeds a critical value, the congestion of excess solution at the nozzle tip can affect the jet formation process, leading to the formation of an unstable jet, beaded fibers, and other defects, such as branching, splitting, and flattened fibers [46].

1.2.2.4 Solution properties

Polymer molecular weight has a substantial effect on electrospinning performances. In general, increasing molecular weight enables the polymer solution to be electrospun into uniform fibers at relatively low concentrations because a high molecular weight induces a large degree of chain entanglement [47]. It was found that polyvinyl alcohol (PVA) solutions produced fibers with morphologies ranging from beaded fiber, uniform fiber, to coarse non-uniform fibers with increasing concentration [48]. In another study, PA6 solution produced droplets at 5 wt% concentration, merged fibers at 15 wt% concentration, and smooth fibers at 25 wt% concentration [49].

When the solution concentration is very low, electrospray happens instead of electrospinning. At low solution concentrations, electrospinning usually produces defective fibers (discontinuous, merged, or beaded) because the surface tension of the solution overcomes the viscoelastic forces and electrostatic drawing force. When the solution concentration is sufficiently high, the chain entanglement of macromolecules is enough to overcome surface tension, and the fiber diameter increases with the rising concentration as there is more solid content in the solution [47, 50]. However, a too-high concentration makes electrospinning difficult due to high viscoelasticity [51], especially for needleless electrospinning that stops jet production at high solution concentrations. In addition, the critical voltage for electrospinning may go up when increasing the solution concentration [52]. The minimum solution concentration to produce smooth nanofibers is dependent on the polymer type, polymer molecular weight, and solvent used.

The addition of surfactant and salt alters the surface tension and electrical conductivity of polymer solutions, and their influences on electrospinning may vary in different conditions. It has been reported that the addition of lithium chloride (LiCl), sodium nitrate (NaNO₃), sodium chloride (NaCl), and calcium chloride (CaCl₂) salts can increase solution conductivity, thus reducing electrospun PAN fiber diameter (the reduction is proportional to solution conductivity) [53]. On the contrary, the addition of salt in PA6 solution was found to increase fiber diameter; this is attributed to the increase in viscoelastic force within the solution jet [47]. The addition of surfactant can reduce solution surface tension, and as a result produce thinner nanofibers with better uniformity [54, 55]. Introducing surfactant (e.g. dodecylbenzene sulfonic acid, tetrabutylammonium chloride) in the solution could also produce nanofibers with special morphologies, e.g. tree-like [56] and nano-net morphologies [57].

1.2.2.5 Temperature and humidity of environment

Electrospun nanofibers normally have smaller fiber diameter with rising environment temperature during electrospinning because of the declined viscosity and surface tension of solution [58]. When using a low temperature (200–220 K) to prepare poly (lactic acid-*co*-glycolic acid) nanofibers, the porosity of electrospun nanofibers can be improved by four times because the ice crystals formed at low temperature serve as a removable void template to create additional pores [59]. Humidity can also affect fiber diameter and morphology [58, 60, 61]. It has been reported that polystyrene nanofibers prepared in a high-humidity environment exhibit porous morphology, because solvent evaporation from solution jets has a cooling effect and causes moisture condensation on the fiber surface, leading to breath-figure self-assembly [61].

In the following section of this chapter, state-of-the-art progresses of electrospinning techniques and apparatuses will be summarized (figure 1.5), covering nozzle electrospinning techniques (e.g. multicomponent electrospinning, multinozzle electrospinning, near-field electrospinning, melt electrospinning), needleless electrospinning techniques, and methods of controlled nanofiber deposition (e.g. aligned nanofibers, nanofiber yarns).

1.3 Nozzle electrospinning

In a nozzle spinneret-based electrospinning process, a spinning solution is transported to fiber electrospinning sites through a capillary channel or multiple channels.

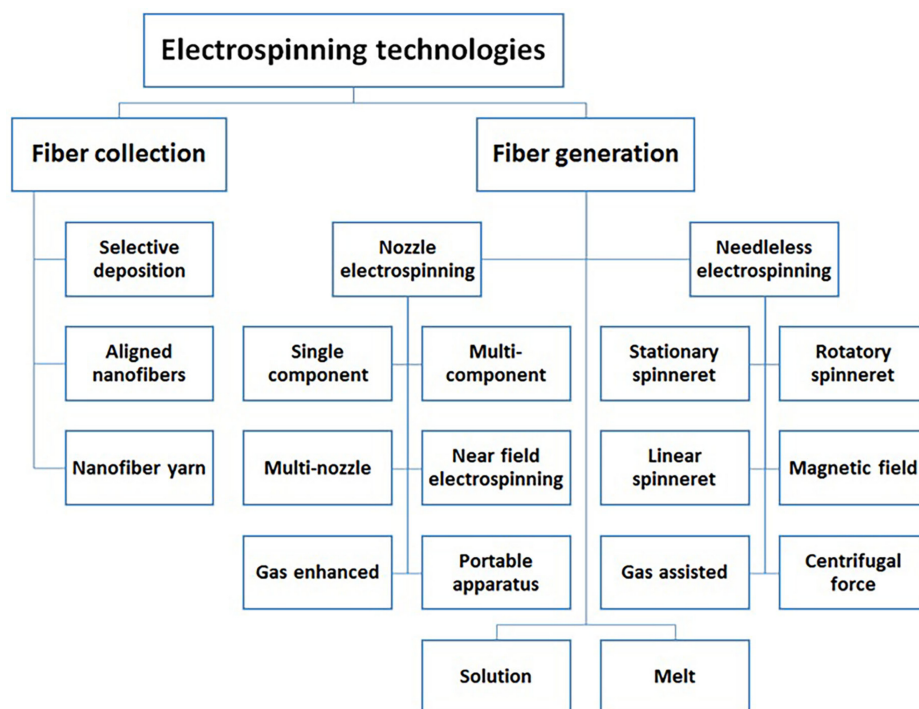


Figure 1.5. A summary of different electrospinning techniques.

This kind of electrospinning technique has many unique features, e.g. enabling incessant solution feeding and continuous electrospinning, the enclosed system avoiding unnecessary evaporation and maintaining the solution stability, tunable capillary diameter with easily controlled fiber diameter, and producing uniform nanofibers.

1.3.1 Single-component electrospinning

Single-component electrospinning refers to the circumstance that spinning solution is supplied through a single capillary nozzle, and jet initiation occurs at the nozzle tip when high voltage is applied. Although positive high voltage is generally used in electrospinning, negative high voltage has also been applied for making electrospun nanofibers. Tong *et al* investigated the effect of high voltage polarity on fiber quality of poly(hydroxybutyrate-co-hydroxyvalerate) (PHBV) nanofibers. They noticed that PHBV fiber diameter increased with increasing applied voltage for positive voltage electrospinning, but decreased for negative electrospinning; however, water contact angle, tensile strength, and stiffness of the nanofibers were barely affected by the polarity of applied voltage [54]. In another work of electrospinning zein/soy protein isolate (95/5), the diameter of positive electrospun nanofibers was smaller than that of negative electrospun nanofibers. With the increasing applied voltage, positive electrospinning produced thinner nanofibers while negative electrospinning did not show any clear effect on the fiber diameter [62].

In addition to the conventional setup where high voltage is connected to the nozzle spinneret, high voltage can also be connected to the collector to perform electrospinning. This works based on the inductive effect produced from the high potential applied by the collector. It has been found that when high voltage is connected to the collector and the nozzle is grounded, electrospinning proceeds successfully but requires higher critical voltage to start the spinning process, and results in enlarged fiber diameter and lower nanofiber production rate [63].

Direct current (DC) voltage is usually used for electrospinning when the high voltage power supply has either a positive high voltage output or a negative output. Alternating current (AC) voltage can not only implement electrospraying/electrospinning, but also has better controllability of fiber deposition. It has been found that AC electrospinning of poly(ethylene oxide) (PEO) significantly subsided the whipping phenomenon with a high degree of fiber alignment [64]. Unlike the conventional DC electrospinning in which a funnel shaped nanofiber mesh is formed, a visible thread can be observed emerging downstream from the needle in the AC electrospinning process. Furthermore, the fibrous thread is not attracted by the grounded electrode, and it can be easily deflected and collected [65].

1.3.2 Multicomponent electrospinning

In the textile industry, a single fiber with two components distributed in the radial direction, for example core-sheath and islands-in-the-sea structures, can find many unique applications. Although it is possible to obtain core-sheath-structured nanofibers induced by phase separation [66] or emulsion electrospinning [67, 68], normal

single-component electrospinning usually produces an electrospun nanofiber without an obvious core–sheath structure. This is attributable to the fact that two solutions inside the nozzle (diameter < 1 mm) have small Reynolds numbers. They can be identified as laminar flows and there is no mixing between them; as a result, they maintain their initial injection states [69]. Because of this, it is possible to conduct bicomponent electrospinning and fabricate nanofibers with tunable composition in the radial direction [70].

In a core–sheath electrospinning spinneret, an inner nozzle of small diameter and an outer nozzle of large diameter are positioned axially with the inner nozzle slightly protruding out of the outer nozzle. During the electrospinning process, the sheath solution is fed into the outer nozzle and the core solution is fed into the inner nozzle. At the nozzle tip, the outer solution forms a thin sheath that enclose the inner solution. Under the action of electrostatic force, both solutions are pulled into a compound Taylor cone of core–sheath structure. Then the core–sheath solution jet is stretched into thin core–sheath fibers by the electric field and deposited on the collector [70–72]. When the inner solution is replaced with a liquid (usually octane), hollow nanofibers (figure 1.6(a) and (b)) can be obtained after removing the liquid from the resulting core–sheath electrospun nanofibers [71]. Thanks to core–sheath electrospinning, many unspinnable materials, e.g. polydimethylsiloxane (PDMS) [73] and medicine [74], can be successfully processed into ultrathin fibers.

When two components distribute inside the bicomponent electrospinning nozzle in a side-by-side way, the fabricated nanofibers have a side-by-side structure [75]. Interestingly, self-crimped polyacrylonitrile nanofibers (figure 1.7) were produced when the polyurethane component in polyacrylonitrile/polyurethane side-by-side nanofibers was removed; this could provide an efficient way to tailor the structure of electrospun nanofibers [76].

Multicomponent nanofibers (figure 1.8) can be obtained via simply increasing the number of components inside the spinneret [77]. However, further increasing the component number to produce an islands-in-the-sea structure may be difficult

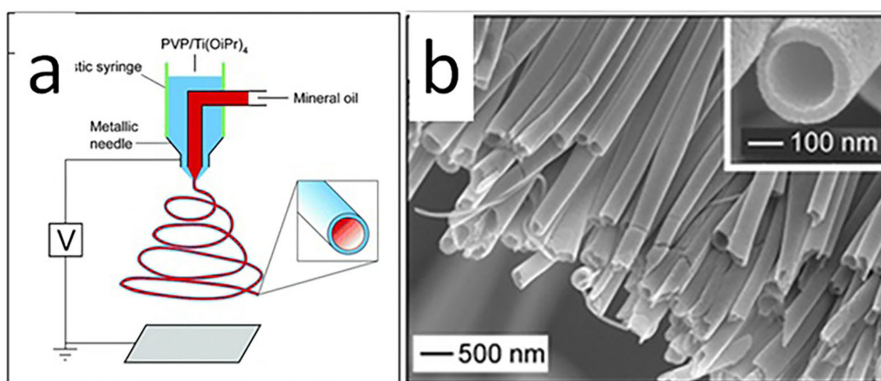


Figure 1.6. (a) Core–sheath electrospinning setup, and (b) SEM images of electrospun hollow nanofibers. Reproduced with permission from Royal Society of Chemistry [71].

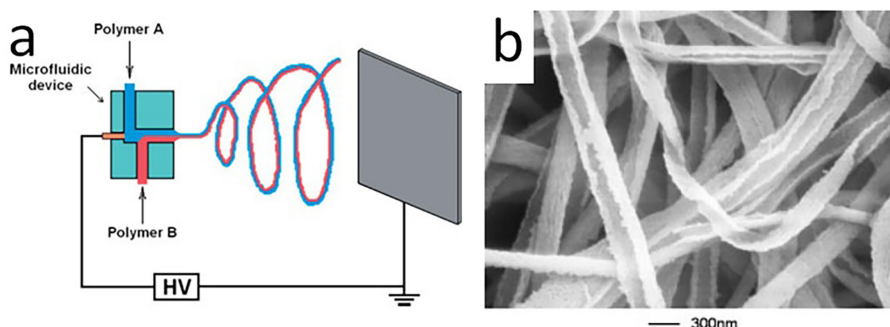


Figure 1.7. (a) Side-by-side electrospinning setup, and (b) SEM image of side-by-side bicomponent nanofibers with one component removed. Reproduced with permission from Wiley [76].

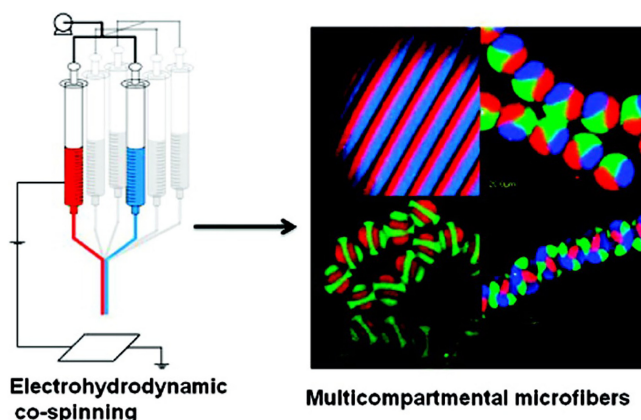


Figure 1.8. Schematic drawing of a multicompartmental electrospinning setup and prepared fibers. Reproduced with permission from American Chemical Society [77].

because the diameter of the nozzle is normally small (<1 mm); this makes it impractical to further prepare more complex spinneret, and demands large pressure to feed all the components evenly.

1.3.3 Multinozzle and porous spinneret

The single nozzle electrospinning generally has a low nanofiber productivity of $<0.3 \text{ g}^{-1} \text{ h}^{-1}$ per nozzle, which is far below the requirement for industrial nanofiber production. A direct and practical way of improving the nanofiber production rate is to increase the number of nozzles for electrospinning, namely by employing multinozzle electrospinning [78–81]. However, there is strong electrostatic repulsion between the jets in multinozzle electrospinning, which can easily affect the electrospinning process, causing poor fiber quality and an uneven fibrous membrane (figure 1.9(a)). Different nozzle position arrangements have been developed to reduce electrostatic repulsion and improve multinozzle electrospinning performances [82]. An auxiliary electrode can interfere with the electric field in multinozzle

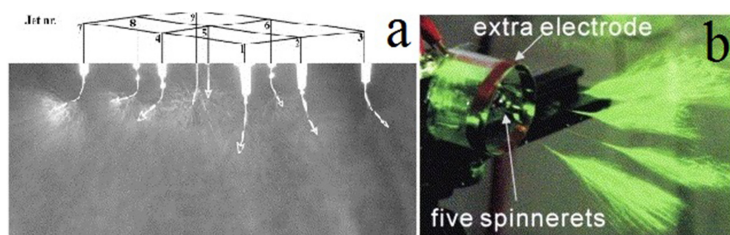


Figure 1.9. (a) A multinozzle electrospinning setup. Reproduced with permission from Elsevier [82]. (b) Cylindrical auxiliary electrode-assisted multinozzle electrospinning. Reproduced with permission from Elsevier [85].

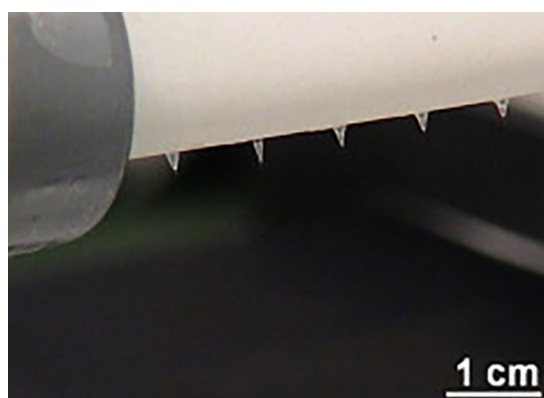


Figure 1.10. Horizontal tube electrospinning. Reproduced with permission from Elsevier [86].

electrospinning and improve its distribution (figure 1.9(b)); this improves electrospinning performances [83–85]. The presence of the external electrode could shrink the fiber deposition area and improve the fiber production rate.

Using a cylindrical tube with channels throughout the tube wall is an effective way to increase the solution channel number, thus increasing fiber production rate [86, 87]. It is still based on the capillary effect, which transports the spinning solution from inside of the tube to the outside, and the electrospinning happens on the tube surface. Tube electrospinning exhibits improved electrospinning productivity in comparison with single needle electrospinning, attributable to numerous nozzle number (figure 1.10). Although it is possible to improve the nanofiber production rate easily by increasing the tube length and number of channels, the gap between adjacent channels cannot be too small because strong jet interference can result in a nanofiber belt instead of a fiber web [86].

In electrospinning, ejected solution jet carries a large amount of charges, which drive the jet stretching and fiber deposition on the collector. The interference among solution jets in multijet electrospinning cannot be completely eliminated. In addition, solution and electrospinning conditions (e.g. solution type, solution

concentration, applied voltage) influence the electrostatic repulsion between ejected jets, and it is difficult to find an optimized distance between solution channels that can be adaptable to all electrospinning circumstances. If not resolved successfully, the electrostatic repulsion problem will be a serious barrier to the industrialization of multijet electrospinning.

1.3.4 Near-field electrospinning

Near-field electrospinning can be regarded as a technique of integrating nanolithography [88, 89] and electrospinning, and is a good example of interdisciplinary technological convergence. In a normal electrospinning system, the collecting distance is usually over 5 cm and the whipping stability is evident. Near-field electrospinning working on a much lower applied voltage can precisely control electrospun fiber deposition [90, 91]. In near-field electrospinning, the jet whipping instability is eliminated or greatly restricted due to a very short collecting distance (<5 cm). When a collector is controlled by a computer program with precisely positioned movement, the collected fibers can form predesigned patterns. Near-field electrospinning also has a few demerits; for example, it produces fibers with a much larger diameter than those in conventional electrospinning due to the insufficient jet stretching, and it has a small nanofiber production rate. The differences between near-field electrospinning and conventional electrospinning are presented in table 1.1.

Figure 1.11(a) illustrates a near-field electrospinning process, and the SEM image in figure 1.11(b) shows a collected nanofiber. The nanofibers fabricated by near-field electrospinning has a narrower diameter distribution than those fabricated by the conventional electrospinning process. This technique greatly expands the application fields of the electrospun nanofiber with ordered structures, e.g. into the fields of nanogenerators, tissue engineering, wearable sensors, and microelectromechanical systems [93–97].

Because of the precise fiber deposition in near-field electrospinning, it is feasible to fabricate a patterned 3D fibrous structure [95]. At microscale (figure 1.12), these fibers prepared by near-field electrospinning show precise deposition and they formed a well-organized 3D fibrous structure.

Table 1.1. Comparison of conventional electrospinning and near-field electrospinning [92].

	Conventional electrospinning	Near-field electrospinning
Material	Solution, polymer melt	Solution, polymer melt
Applied voltage (kV)	10–30	0.2–12
Collecting distance (mm)	50–500	0.5–50
Fiber diameter (μm)	0.01–1	0.05–30

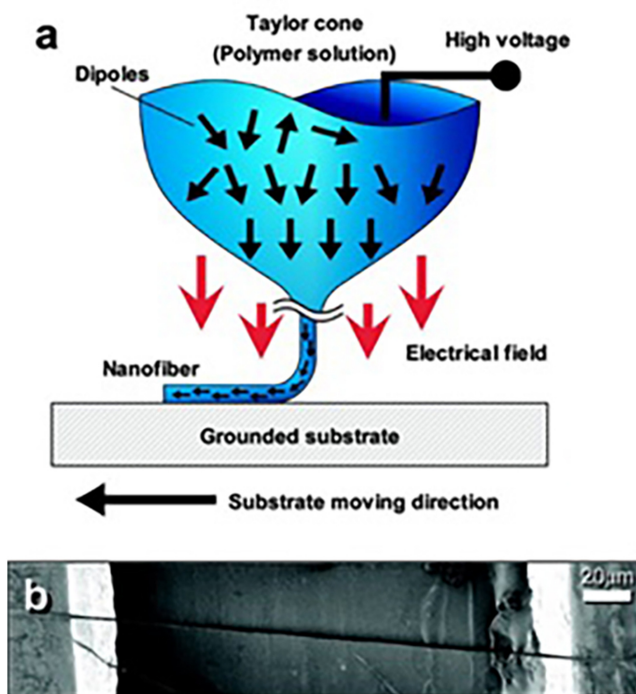


Figure 1.11. (a) Schematic diagram of near-field electrospinning process, and (b) SEM image of a single polyvinylidene fluoride nanofiber formed across two electrodes. Reproduced with permission from American Chemical Society [93].

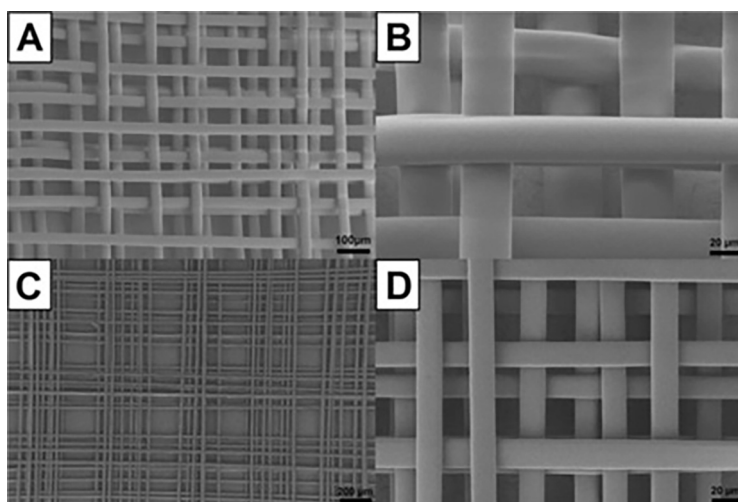


Figure 1.12. (a) SEM images of deposited poly(2-ethyl-2-oxazoline) fibrous structures. Reproduced with permission from Elsevier [98].

1.3.5 Gas-enhanced electrospinning

Applying auxiliary gas to an electrospinning process can diminish the influence of Coulombic repulsion force in multinozzle electrospinning [99]. As a result, gas-enhanced electrospinning can produce thinner nanofibers [100–102], improve nanofiber collection [103–105], and increase nanofiber production rate [106]. It has been found that the application of nitrogen gas containing solvent vapor can help to eliminate the whipping motion of the solution jet and lead to the collection of highly aligned nanofibers on the fast-rotating cylindrical collector [103]. The Zetta electrospinning system was developed in another work; it can process both polymer solution and polymer melt into nanofibers. Figure 1.13 shows that Zetta electrospinning uses airflow to enhance electrospinning with a much-improved nanofiber production rate [106].

When preparing thick nanofiber membranes by extending electrospinning time, it is very easy to build up electrostatic charges on the collector because polymer nanofibers are electrically nonconductive. These accumulated electrostatic charges can weaken the electric field, prevent fiber deposition, and produce coarser fibers. Solvent accumulation inside the nanofibrous membrane is another problem, which, if not removed rapidly, can cause jointed nanofibers or even film after a long period of electrospinning. Gas-enhanced electrospinning can efficiently solve these problems [107]. Airflow has also been applied to assist melt electrospinning (figure 1.14), resulting in 10% thinner polylactic acid fibers compared to fibers produced by unassisted melt electrospinning [100].

1.3.6 Melt electrospinning

Melt electrospinning technology was reported in as early as 1936 in a patent filed by Charles Norton from the Massachusetts Institute of Technology [108]. In spite of its early appearance, melt electrospinning had not drawn as much attention as solution electrospinning because it needs to maintain an elevated temperature during the electrospinning process and polymer melts have low conductivity and high viscosity. In recent developments, various heating methods have been applied in melt electrospinning, e.g. electrical heating [109, 110], air heating [111, 112], circulating fluid heating [113], and laser heating [114, 115]. Melt electrospinning does not involve any solvent and is an environmentally friendly process, which makes it

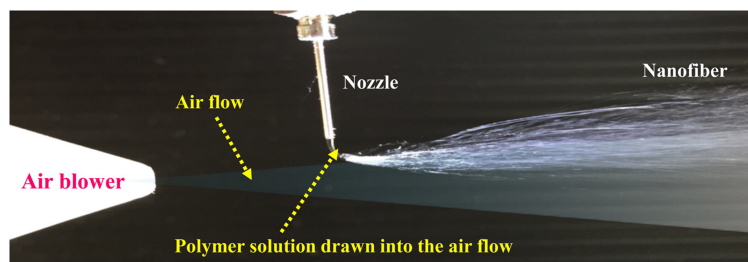


Figure 1.13. Zetta electrospinning process. Reproduced with permission from American Chemical Society [106].

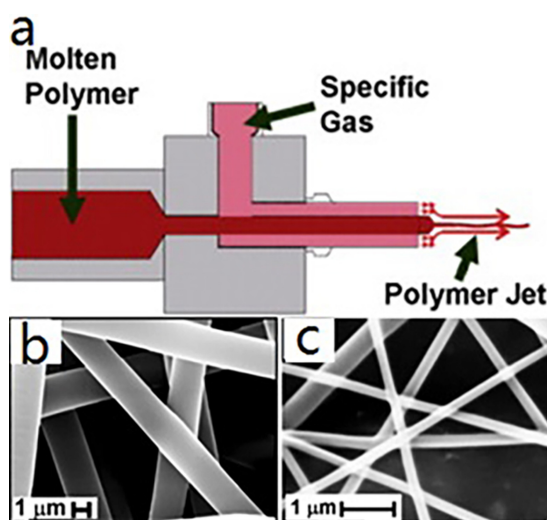


Figure 1.14. (a) Schematic diagram of gas-enhanced melt electrospinning; SEM images of fabricated polylactic acid fibers (b) without a gas-assisted system and (c) with a gas-assisted system. Reproduced with permission from Elsevier [100].

favorable for various applications, e.g. filtration [116], sensors [117], textiles [118], and especially biomedical applications [113, 119–121]. Conversely, solution electrospinning uses flammable, toxic solvents; its operation can cause environmental issues and hazard risks, which indicate that melt electrospinning could play a more important role in nanofiber production and applications.

1.4 Needleless electrospinning

In spite of great efforts to improve electrospinning productivity, conventional capillary electrospinning can only produce a very limited quantity of nanofibers, and is unable to meet the ever-increasing demand for industrial applications. Needleless electrospinning offers a solution to this problem. Needleless electrospinning (also referred to as free surface electrospinning) is a special type of electrospinning. Instead of forming a jet from a capillary tip, needleless electrospinning forms a large number of solution jets directly from an open liquid surface. Needleless concept appeared in as early as 2004 [122], when Yarin reported upward needleless electrospinning of nanofibers from a two-layer system. Since then, this technology has attracted tremendous interests attributable to its ability for large-scale nanofiber production.

During a needleless electrospinning process, numerous jets are generated instantaneously from the spinneret surface, exempt from influences of the capillary effect [123]. The electrospinning liquid (electrically conductive) on the spinneret surface self-organizes on a mesoscopic scale to form waves, and jet initiation happens from wave crests when the applied voltage exceeds a critical value. The electric field intensity profile around the spinneret and in the electrospinning zone play a far more

important role in needleless electrospinning than in needle electrospinning, in terms of jet initiation, jet stretching, nanofiber productivity, and morphology.

Most of the works on needleless electrospinning have been focusing on understanding how spinneret design affects electrospinning performances and nanofiber production. It has been established that a large curvature can generate a high-intensity electric field, which increases fiber production rate. Based on their motility, needleless electrospinning spinnerets can be classified into the following categories: stationary needleless spinneret, linearly moving needleless spinneret, and rotatory needleless spinneret. In addition to these three types of needleless spinnerets, there are some other needleless electrospinning designs working on different jet initiation mechanisms.

1.4.1 Stationary needleless spinnerets

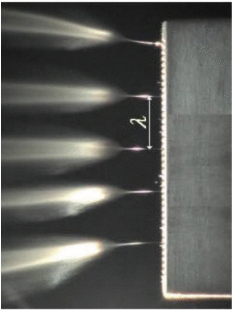
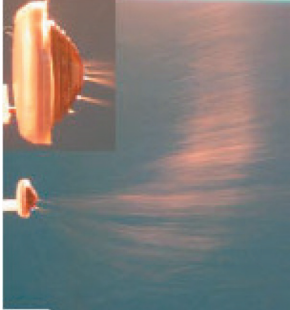
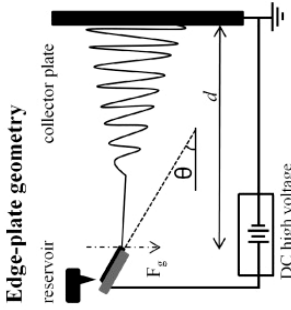
Stationary needleless spinnerets refer to those spinnerets that do not move during the electrospinning process while the spinning solution is fed into them or the spinnerets work in batch mode. This kind of electrospinning setup is relatively simple and the electrospinning processes can be implemented with minimum efforts. Spinnerets in this category include, wire, twisted wire, conical wire, bowl, sharp edge, cylinder, slit, curved slot, stepped pyramid, and cleft (shown in table 1.2).

1.4.2 Rotatory needleless spinnerets

During electrospinning using stationary spinnerets, there is no effective control over the solution distribution on the spinneret surface. As a result, electrospinning conditions and fibrous membrane uniformity may vary as electrospinning continues. When rotatory needleless spinnerets are used for electrospinning, the spinnerets can spread the solution evenly onto their surface through rotation and ensure continuous electrospinning. The solution layer thickness can be easily regulated by the rotating speed with improved electrospinning stability and nanofiber uniformity. Jet initiation in needleless electrospinning can be summarized in four stages. (1) A thin solution layer is formed on the spinneret surface because of spinneret rotation. (2) Rotation causes perturbations on the solution layer, inducing the formation of conical spikes. (3) When high voltage is applied, these spikes centralize the electric force, intensifying perturbation to form Taylor cones. (4) Solution jets are stretched out from Taylor cones, resulting in nanofiber formation. Table 1.3 lists the common rotatory spinnerets and fiber-generating sites, including cylinder (roller), disc, ball, coil, cone, and wire frame.

With the help of finite element analysis (FEA), the electric field intensity profile in the electrospinning area can be simulated and used for optimizing spinneret design [42, 136, 137]. It has been found that an auxiliary structure on the primary spinneret structure can centralize the electric field around with increased intensity. Therefore, auxiliary structures were introduced onto common needleless spinnerets to enhance electric field strength and improve electrospinning performances; these structures include the Von Koch curve fractal structure [142], needles on a disc or helix slice [143, 144], barbed roller [145], probed cylinder [146], and threaded rod [147].

Table 1.2. A summary of stationary needleless electrospinning spinnerets and their spinning process.

Spinneret	Fiber generation	Fiber generation area	Ref
Cleft	 (Reproduced with permission from AIP Publishing)	Cleft surface	[123]
Conical coil	 (Reproduced with permission from Wiley)	Coil wire surface and gap	[43]
Plate	 (Reproduced with permission from Elsevier)	Plate edge	[124]

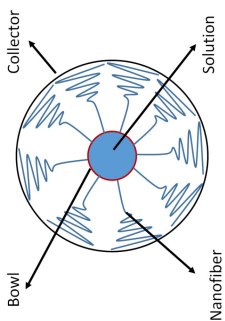
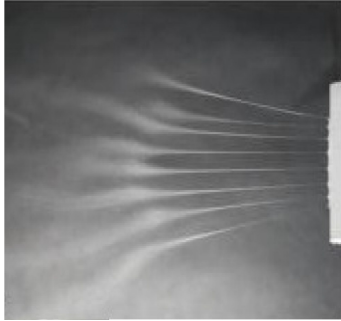
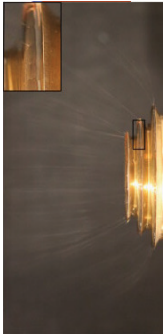
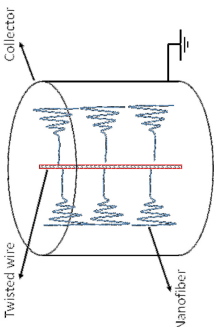
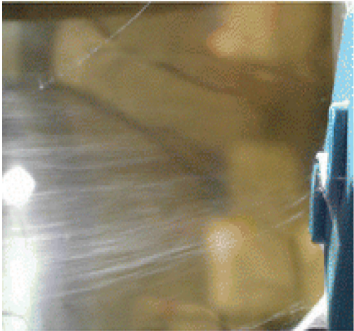


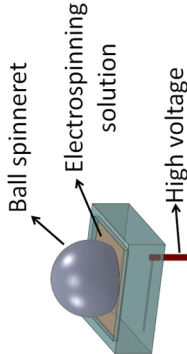
Bowl		Bow edge	[125]
Slit		Slit	[126–129]
Stepped pyramid		Pyramid edge	[130, 131]
(Reproduced with permission from Elsevier)			
(Continued)			

Table 1.2. (Continued)

Spinneret	Fiber generation	Fiber generation area	Ref
Twisted wire		Wire surface	[132]
Curved slot		Slot	[133]

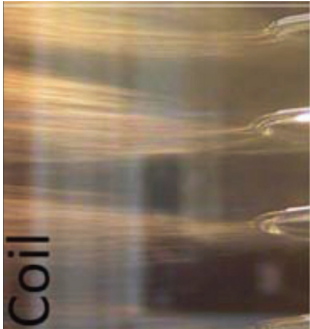
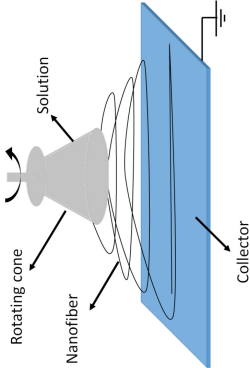
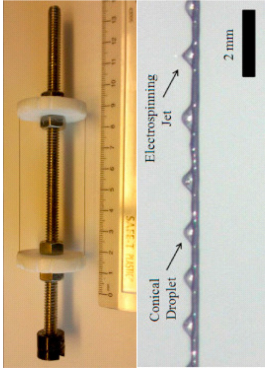
(Reproduced with permission from Springer Nature)

Table 1.3. A summary of rotatory needleless electrospinning spinnerets and their spinning process.

Spinneret	Fiber generation	Fiber generation area	Ref
Cylinder roller	 (Reproduced with permission from Wiley)	Cylinder surface	[42, 134]
Disc	 (Reproduced with permission from Wiley)	Disc rim	[42]
Ball	 (Reproduced with permission from Wiley)	Ball surface	https://snrfibers.com [135]

(Continued)

Table 1.3. (Continued)

Spinneret	Fiber generation	Fiber generation area	Ref
Coil		Coil wire surface	[136, 137]
Cone		Cone edge	[138]
Wire frame		Wire surface	[139–141]

(Reproduced with permission from Elsevier)

1.4.3 Linear needleless spinneret

The jet initiation sites on linear needleless spinnerets are distributed in parallel to the linear direction of the spinnerets, and a typical design is wire electrospinning. Although the wire spinneret remains stationary, there is a solution-feeding device moving linearly along the wire to feed spinning solution evenly along the wire, thus ensuring continuous electrospinning. In another design, a horizontal bead chain was used to produce nanofibers, and the chain moved in parallel to the flat collector. The beads, as an auxiliary structure, can centralize the electric field and improve electrospinning performances [148].

The applied voltage needed to start jet initiation and electrospinning in needleless electrospinning is usually much higher than that in conventional needle electrospinning because it requires a much higher electrostatic force to stretch out a solution jet from the small curvature surface of the needleless spinneret [42]. In a recent work of electro-aerodynamic field aided needleless electrospinning, two auxiliary forces of additional electric field and airflow were used to assist the electrospinning process (figure 1.15). The high-intensity electric field was generated between the slot and the inductive electrode (5 cm distance). In this way, electrospinning ran at a voltage equivalent to that in needle electrospinning (e.g. 10–30 kV) [149]. The auxiliary airflow diverted the nanofibers away from the inductive electrodes and directly to the collector.

With the fast advances of electrospinning technology, commercialized needleless electrospinning technologies are already on the market (e.g. technologies produced by Elmarco, Fanavaran Nano-meghyas, Revolution Fibers, SPUR Company, Shanghai Yuntong Nanomaterials Technology Co., Ltd, Stellenbosch Nanofiber Company, INOVENSO). Such technologies produce nanofibers based on different mechanisms but all have a large-scale nanofiber production capacity.

1.4.4 Magnetic field-assisted needleless electrospinning

Apart from the above-mentioned needleless spinnerets that relying on specific geometric shapes to conduct electrospinning, there are also a number of needleless electrospinning technologies that utilize additional forces such as gas blowing, a magnetic field, or centrifugal force to implement electrospinning.

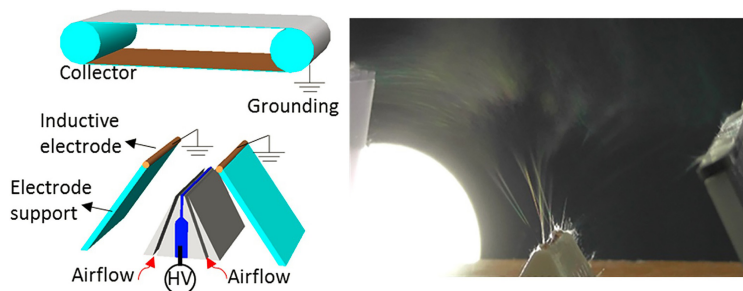


Figure 1.15. Schematic drawing of electro-aerodynamic needleless spinning setup and its electrospinning process (HV means high voltage). Reproduced with permission from IOP Publishing [149].

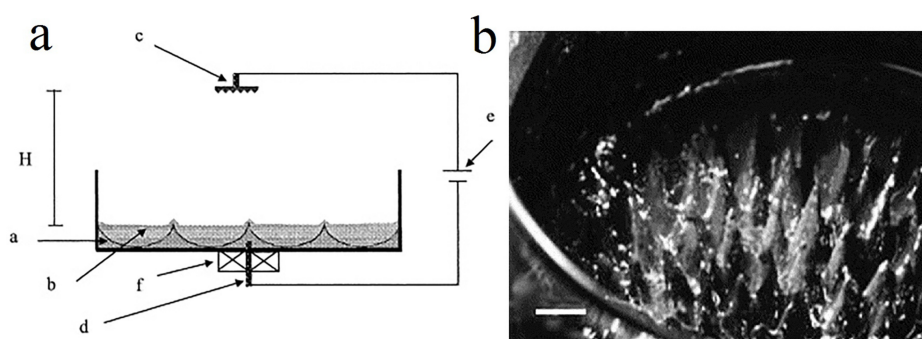


Figure 1.16. (a) Schematic drawing of magnetic field-assisted needleless electrospinning. (b) Spikes formed on the silicone oil-based magnetic fluid under the action of a permanent magnet. Reproduced with permission from Elsevier [122].

Yarin and Zussman [122] reported an electrospinning technique that used a magnetic field to initiate the jet formation (figure 1.16). The spinning liquid comprised two layers: a bottom ferromagnetic fluid layer, and a top polymer solution layer. When an external magnetic field was applied to the ferromagnetic fluid and an electric field applied to the polymer solution, the ferromagnetic fluid triggered the formation of steady vertical spikes, which perturbed at the interlayer interface and solution layer. When the applied voltage was high enough, solution jets were stretched out from the spikes.

1.4.5 Gas-assisted needleless electrospinning

Because of low cost and high safety, airflow has been used to assist jet initiation in electrospinning. A gas-jet electrospinning technique (also referred to as bubble electrospinning) was developed in 2007 [150]; it used gas to create bubbles on the liquid surface, which increased surface curvature and facilitated jet initiation. Since the introduction of this, gas-initiated electrospinning has attracted great interest [151, 152]. In nozzle electrospinning [153] and needleless electrospinning [154], high-speed gas can improve nanofiber production rate because airflow can enhance solution jet stretching, thus facilitating jet initiation. In addition to the gas-assisted solution electrospinning, melt electrospinning can also benefit from additional airflow [100, 155].

1.4.6 Centrifugal force-assisted needleless electrospinning

Centrifugal spinning has been developed over many years, and was originally extensively used for producing glass fibers [156]. Recently, centrifugal spinning has been applied to prepare nanofibers [157–159]. Many parameters in centrifugal electrospinning, e.g. voltage, spinneret rotation speed, solution feed rate, distance between spinning head and collector, and solution concentration, can affect the electrospinning process and nanofiber quality. The combination of mechanical rotation and electric field makes it very effective in fabricating aligned nanofibers

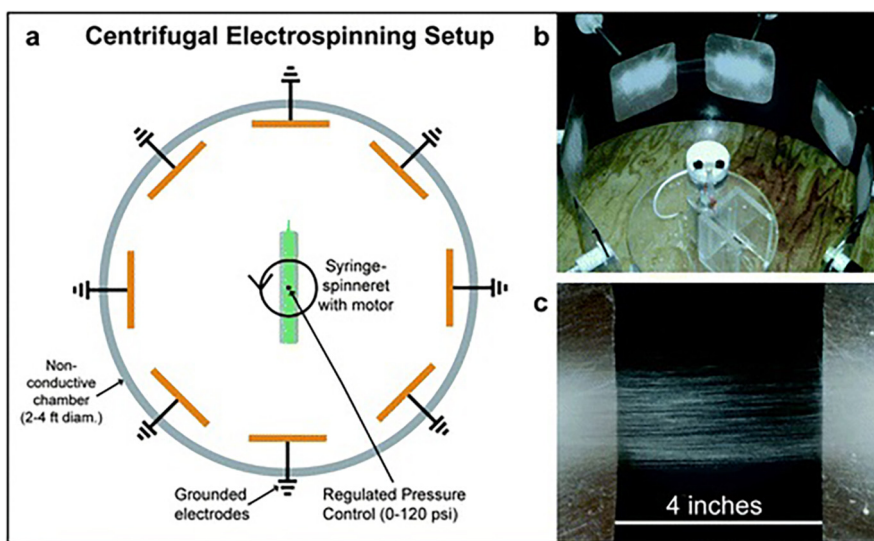


Figure 1.17. (a) Schematic drawing and (b) digital photo of centrifugal electrospinning process; (c) SEM image of centrifugal poly(vinylidene fluoride) (PVDF) fibers deposited between two grounded electrodes. Reproduced with permission from Royal Society of Chemistry [162].

(figure 1.17) [160–162]. The combination of centrifugal spinning and electrospinning has many advantages [162, 163]:

- For high-concentration solution or polymer melt, it is hard to achieve jet initiation in normal electrospinning because of the high viscosity; however, centrifugal force can easily transport these fluids.
- Higher nanofiber production rate.
- Finer nanofibers.
- Lower jet initiation voltage.

1.5 Nanofiber collection

Electrospun nanofibers are generally collected as randomly distributed nanofiber mats, the morphology of which is similar to a nonwoven fibrous mat. In lots of studies, such a fibrous structure was described as a fiber web, fiber sheet, nonwoven fiber, and membrane. To meet specific applications, nanofibers are collected into different structures from nonwoven fibers. Numerous setups have been developed to manipulate nanofiber deposition. As a result, aligned nanofibers, nanofiber yarn, and 3D nanofiber structures have been achieved.

1.5.1 Selective nanofiber deposition

Due to the large amount of electrostatic charges carried by nanofibers, there is significant Coulombic repulsion force between these fibers. As a result, the nanofiber deposition area on the collector is usually large and with low fiber collection efficiency. An auxiliary electric field could improve the control over nanofiber

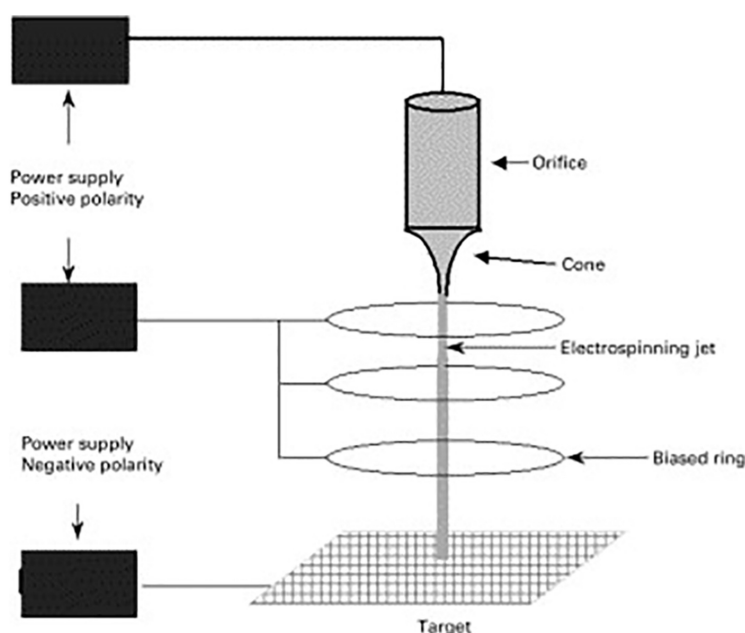


Figure 1.18. Auxiliary electric field-assisted fiber deposition. Reproduced with permission from Elsevier [164].

deposition [83, 84]. Charged rings have been used to restrain nanofiber disposition [164] to a small area; the schematic diagram of this electrospinning setup is shown in figure 1.18. When an auxiliary electrode was used in electrospinning to control fiber deposition, nanofibers in a 2D pattern or 3D structure could be fabricated [165, 166]. During the electrospinning process, the Taylor cone was stabilized and the jet whipping was converged sufficiently [165]. This method could provide a practical strategy for the fabrication of nanofibers with elaborate structures.

By electrospinning nanofibers onto a columnar-shaped collector, instead of flat collector as usual, tubular nanofibers can be obtained [167, 168]; these will have promising potentials in biomedical and industrial applications. Figure 1.19 shows complex nanofiber tubes collected on columnar collectors.

In addition, when a patterned substrate is used to collect nanofibers, the electric field does not distribute uniformly on the substrate, the collected nanofibers show predesigned arrangement on the substrate [169–171]. Figure 1.20(a) shows that the tips on the substrate can centralize the electric field, and as a result, more nanofibers are drawn to these tips and collected PEO nanofibers show a patterned structure (figure 1.20(b)).

1.5.2 Aligned nanofibers

When nanofibers are collected unidirectionally, the tensile strength of nanofiber mats in the fiber-aligned direction can be significantly improved. Collecting nanofibers using a high-speed rotating drum collector or disc is an efficient way to obtain aligned nanofibers [103, 172, 173]. Aligned nanofibers can also be collected using a

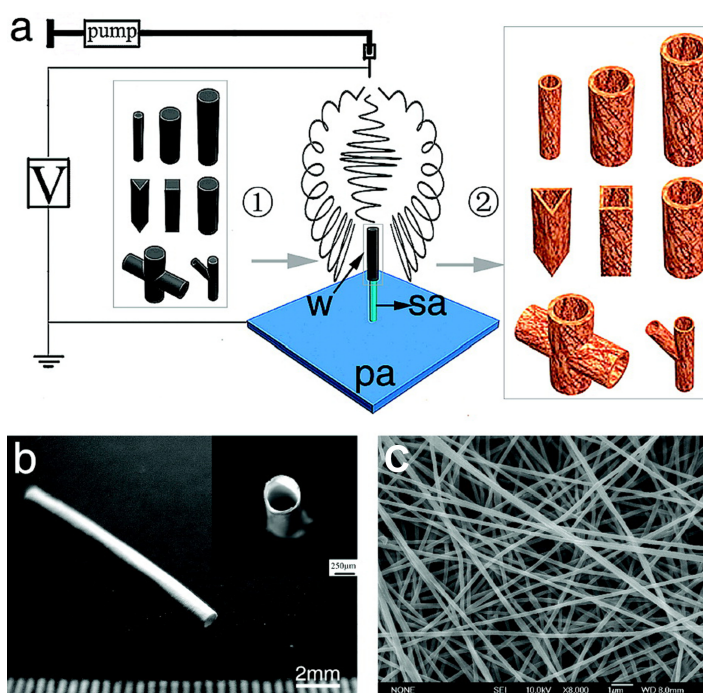


Figure 1.19. (a) Schematic illustration of columnar collectors for fabricating fibrous tubes. (①: columnar collectors and ②: fibrous tubes.) (b) Fabricated fibrous tube (diameter = 500 μm, inset is the cross-section image). (c) SEM image of nanofibers. Reproduced with permission from American Chemical Society [168].

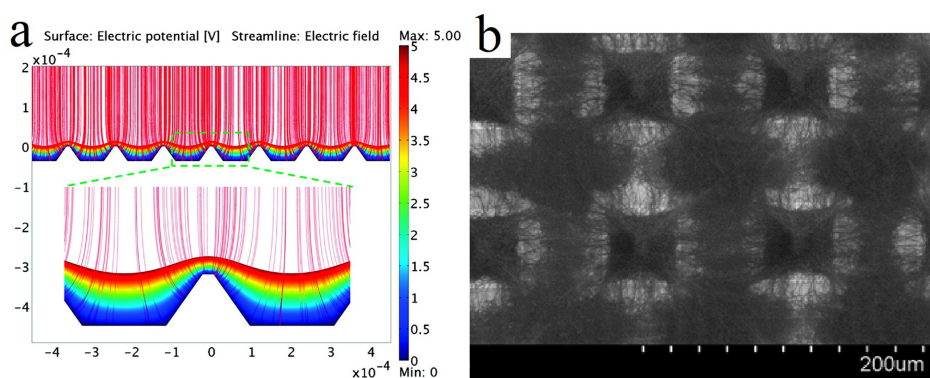


Figure 1.20. (a) Simulation of electric potential and electrical field distribution on the substrate, (b) SEM image of PEO fiber deposited on a patterned substrate for 5 min. Reproduced with permission from American Chemical Society [169].

stationary parallel electrodes collector [174–176] or tip collector [177]. Figure 1.21 shows aligned short nanofibers collected by two parallel Si substrates. When using two magnet bars as the collector, it is also possible to collect aligned wavy polymeric nanofibers [178].

In another work, an electrode with counter polarity voltage applied was used to govern fiber deposition [179]. Fiber placement and alignment on both microscale and nanoscale can be achieved through controlling the shape and magnitude of the electric field of the counter electrode. This technology demonstrates the ability to make nanofiber membranes with tailored porosity (figure 1.22).

AC voltage has the potential of minimizing jet whipping instability and enhancing fiber alignment. A method named biased AC electrospinning used amplified AC voltage to perform electrospinning with improved fiber alignment. When the electrospinning process is driven by DC voltage, it has a high level of whipping instability due to the action of repulsive Coulombic force. Meanwhile, AC power carries both positive and negative charges, and both attractive and repulsive Coulombic forces take effect during electrospinning and lead to a weakening of overall whipping instability [180].

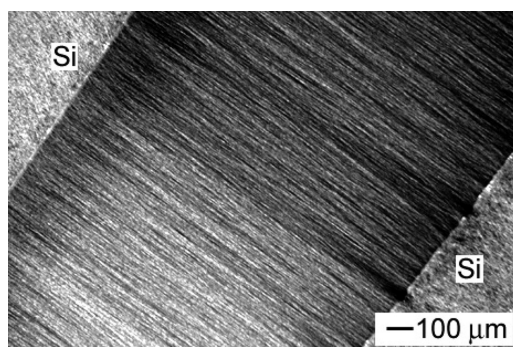


Figure 1.21. SEM image of aligned nanofibers collected across two parallel Si substrates. Reproduced with permission from American Chemical Society [174].

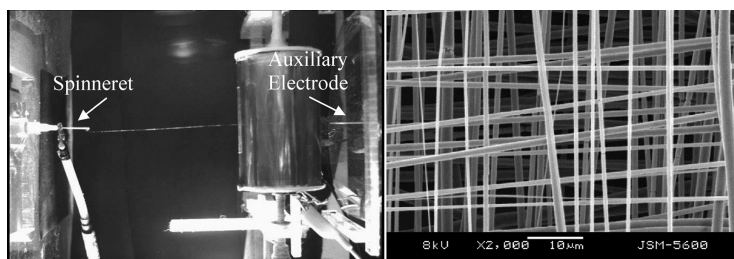


Figure 1.22. A straight fiber being collected with auxiliary counter electrode, and SEM image of collected fibers. Reproduced with permission from American Chemical Society [179].

1.5.3 Nanofiber yarns

The first attempt in electrospinning nanofiber yarns goes back to the 1930s; the involved apparatus contained a fiber spinning wheel and a fiber yarn collecting device [181]. Recently, the production of uniaxial nanofiber bundles or twisted nanofiber yarns by electrospinning has drawn increasing interest because yarns can be woven or knitted into 2D fabrics or 3D structures with tailored structure, mechanical strength, and porosity. Nanofibers collected in a yarn form can find applications in the traditional textile industry, and creates possibilities for a greater number of new applications. Especially, the large-scale production of nanofiber yarns is becoming more and more imperative. The most popular setups for nanofiber yarn/bundle production are listed in table 1.4, and can be divided into the following categories:

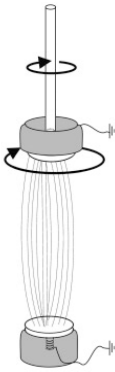
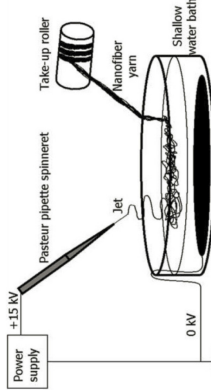
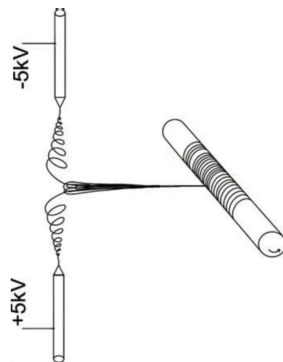
- Collect aligned nanofibers in a short length first (e.g. two rotating discs, high-speed collector) and then twist them into twisted short nanofiber yarns [182–185].
- Collect short twisted yarns directly using two rotating tube collectors [186].
- Collect continuous nanofiber bundles with the help of airflow [187, 188].
- Use a water bath as the collector to obtain continuous nanofiber bundles [189–191].
- Use an auxiliary electrode to govern nanofiber collection for obtaining nanofiber bundles [192–194].
- Apply both positive and negative potentials in electrospinning to improve nanofiber alignment and collect continuous nanofiber bundles [195–197].
- Use a rotating funnel as the collector to obtain continuously twisted nanofiber yarns directly [198–203].
- Wrap electrospun nanofibers around conventional filaments or yarns to obtain composite nanofiber yarns [204, 205].
- Use AC potential to electrospin nanofibers instead of DC potential, and then collect continuous nanofiber bundles [206].

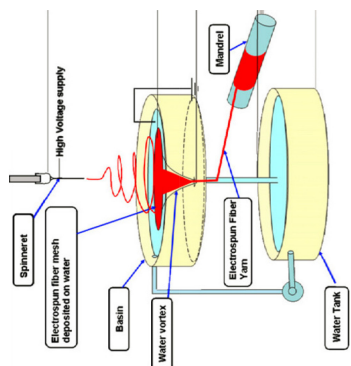
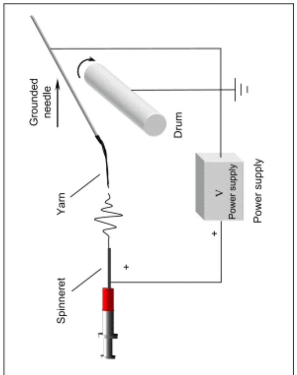
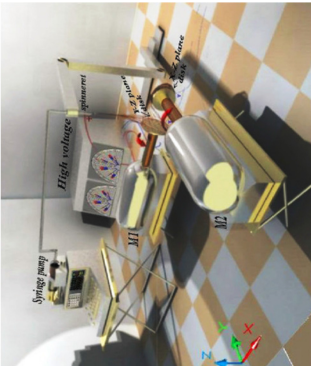
In addition to solution electrospinning, direct yarn production has also been realized in melt electrospinning. Polypropylene nanofiber yarn was continuously manufactured by a melt electrospinning method, wherein suction airflow was used to facilitate the formation of aligned nanofiber strand. The twisting of yarn strands was realized through a tailored rotating collector [188]. In another interesting work, AC electrospinning was used to generate nanofiber bundles directly in the absence of a collector [206]. Smoke-like nanofibers were generated from a rod electrode. Because of the existence of both positive changes and negative changes, they are easy to manipulate and can be effectively wound into nanofiber yarns (figure 1.23).

1.6 Summary and outlook

The science and engineering of nanofiber electrospinning technologies have advanced rapidly in recent years. Many successful attempts to improve electrospinning yield, nanofiber and nanofiber membrane quality, controlled nanofiber

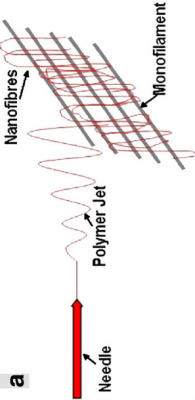
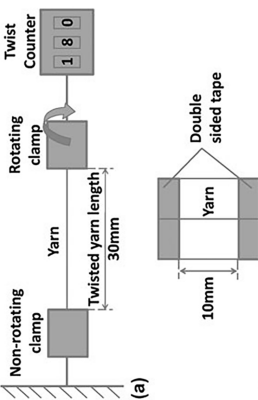
Table 1.4. A summary of nanofiber yarn electrospinning setups, mechanisms, and features.

Electrospinning setup example	Mechanism	Continuous production (Y/N)	Collected form	Representative work
 (Reproduced with permission from Elsevier)	Two separated rotating disc	N	Twisted yarn	[182]
 (Reproduced with permission from Elsevier)	Water bath collection	Y	Nanofiber bundle	[189, 190]
 (Reproduced with permission from Elsevier)	Opposite polarity electrospinning	Y	Nanofiber bundle	[195–197]

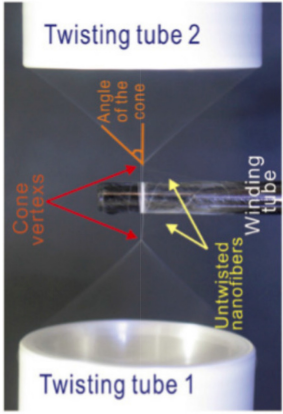
 <p>(Reproduced with permission from Elsevier)</p>	Water bath collection	Y	Nanofiber bundle	[191]
 <p>(Reproduced with permission from Elsevier)</p>	Grounded bar-induced collection	Y	Nanofiber bundle	[192, 193]
 <p>(Reproduced with permission from Elsevier)</p>	Two rotating discs perpendicular to each other	Y	Nanofiber bundle	[194]

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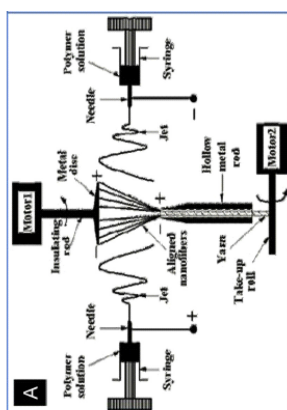
Table 1.4. (Continued)

Electrospinning setup example		Mechanism	Continuous production (Y/N)	Collected form	Representative work
	<p>(Reproduced with permission from Elsevier)</p> 	Parallel filament collection	Y	Nanofiber-covered filaments	[204]
		Short aligned nanofiber strip	N	Twisted yarn	[183–185]
		Two rotating tubes and a winding tube	Y	Twisted yarn	[186]

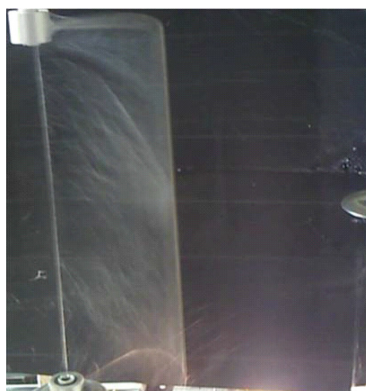
(Reproduced with permission from Cambridge University Press)



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Opposite polarity electrospinning, rotating funnel collection	Y	Twisted yarn	[198–201]
Needleless electrospinning, rotating collection	Y	Nanofiber-covered yarns	[205]

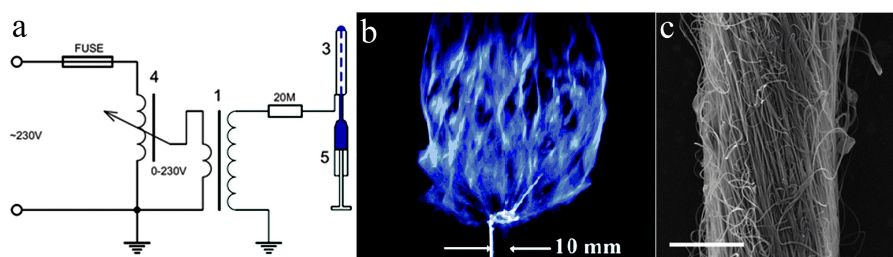


Figure 1.23. (a) Schematic diagram of the AC electrospinning setup, (b) generated polyvinyl butyral nanofiber plume, and (c) SEM image of nanofiber yarn (scale bar = 5 μm). Reproduced with permission from Royal Society of Chemistry [206].

deposition, and secondary nanofibrous structure have contributed to the exciting development of various advanced electrospinning technologies. Commercial production of electrospun nanofibers has also been realized.

In spite of the enormous amount of efforts made to further electrospinning technology, there are still some major challenges in this field, e.g. small production rate of nanofiber yarn, high electrical hazard risk associated with needleless electrospinning, and large fiber diameter in melt electrospinning. Future research on electrospinning should primarily focus on the large-scale production of high-quality nanofibers with improved safety and efficiency.

1. Near-field electrospinning has the ability of producing delicate fiber structures in 2D or 3D arrangements; however, the fiber diameter is much larger than that of conventional needle electrospinning and the fiber production rate is much lower. An important trend in near-field electrospinning is to become more productive, e.g. though multijet synchronous printing, and to have the ability of fabricating nanofiber-based real structures.
2. The current yarn electrospinning technology is either based on needle electrospinning that has a low nanofiber production rate or has a low yarn production efficiency in needleless electrospinning. The large-scale production of nanofiber yarn can greatly expand the application of electrospun nanofibers.
3. The needleless electrospinning technologies generally require high voltage to initiate jet ejection and ensure continuous electrospinning. The high voltage can lead to electrical discharge, and even fire/explosion hazards. The utilization of auxiliary fields, e.g. a magnetic field, airflow field, or centrifugal force, will effectively facilitate the electrospinning process. Results of this would include reducing the critical voltage, thinning the fiber diameter, and improving nanofiber production rate; therefore, this is a promising future direction for energy-efficient, safe production of nanofibers on a large scale.
4. Melt electrospinning is an environmentally friendly process, and melt electrospun fibers are of great importance in biomedical, filtration, and textile areas. Due to the deficiency of free charge in polymer melt, the fiber diameter of melt electrospinning is normally at the micrometer scale. The

utilization of airflow or centrifugal force can effectively improve melt jet stretching and produce thin melt electrospun fibers. The production of thinner fibers in melt electrospinning will be an important research direction in the future.

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