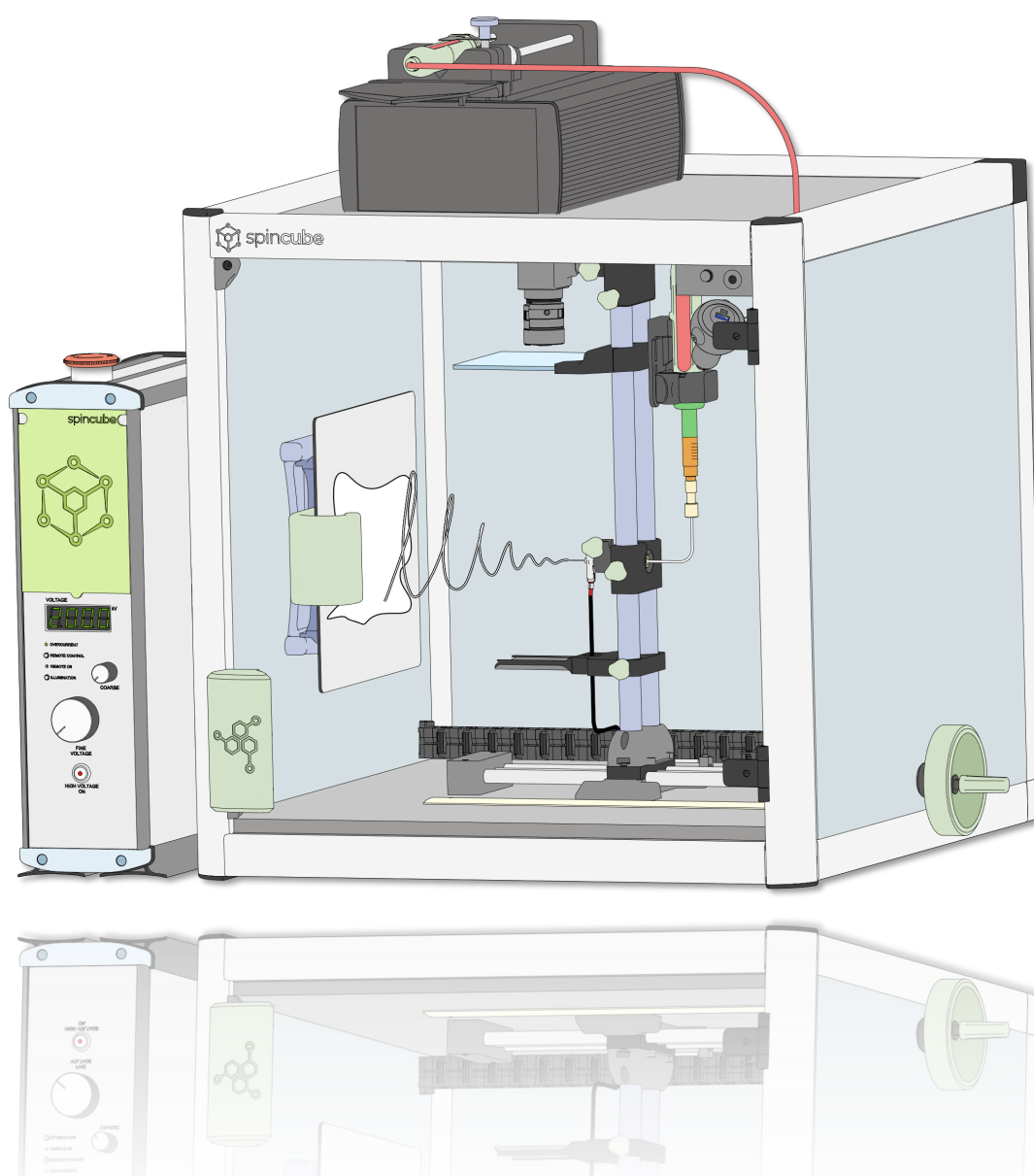


A comprehensive reference to:
Electrostatic fiber formation



1 What exactly are nanofibers?

Nanofibrous materials have gained significant attention in recent years due to their unique properties and potential applications in several industrial fields. These materials are composed of fibers with diameters in the nanometer range, which gives them a high surface area-to-volume ratio, high porosity, and excellent mechanical properties.

Nanofibrous materials have shown promise in many different areas, including biomedical engineering, environmental remediation, energy storage, and filtration. For example, they can be used as scaffolds for tissue engineering, drug delivery systems, and wound dressings in biomedical applications. In environmental remediation, they can be used for water purification and air filtration. In energy storage, they have potential applications in batteries and supercapacitors.

Ultimately, the distinctive features of nanofibrous materials have made them an appealing research topic, and their prospective applications in a variety of disciplines make them highly relevant to modern-day research and development.

2 Preparation of nanofibers by electrospinning technique

Nowadays, numerous methods are accessible for producing nanofibrous materials, including mechanical fiber drawing from polymer melt, phase separation technologies, freeze-drying, and the utilization of self-assembling systems. Furthermore, electrostatic fiber formation (also known as electrospinning) is a promising and increasingly prevalent process. The benefit of electrospinning over its alternatives is that it can create nanofibrous systems from a diverse range of polymers in a short period of time and fiber diameters can be reliably achieved in the submicron range.

Although electrospinning is regarded as a modern process, the first device based on electrostatic fiber formation was described in J. F. Cooley's patent application in the early 1900s. However, the actual significance of this process was not appreciated at the time since the nature of the created material was unknown. After almost a century, electrospinning has drawn more attention as a result of the rapidly developing nanotechnology research and became one of the most significant preparative methods of nanofibrous systems.

Nanofibers can be created via electrospinning from polymer melts or solutions. While melt electrospinning may be carried out entirely from the polymer (solvent-free), and offers significant environmental advantages, solution electrospinning has a lower working temperature and can handle materials that are sensitive to heat. Throughout the fiber formation process the liquid raw material (precursor solution) is subjected to a high voltage ($5 < U < 50kV$) electric field. As a result, the polymer will have a $\oplus$ or $\ominus$ charge, and a grounded (i.e., zero potential; $\perp$) collector will serve as a counterpole. If the produced electric field is strong enough, the polymer solution (or melt) undergoes directed deformation, which is characterized by a so-called Taylor-cone. When the electric field strength [$E = U \times d^{-1}$, ($V \times m^{-1}$); where $d(m)$ is the distance between the poles and $U(V)$ is the voltage] reaches a critical level, one (or more) thin liquid jets ($\varnothing < 100\mu m$) emerge from the cone's tip, and fiber formation begins. The magnitude of the generated electric field strength has a significant impact on the resulting Taylor cone's shape (**Figure 1**).

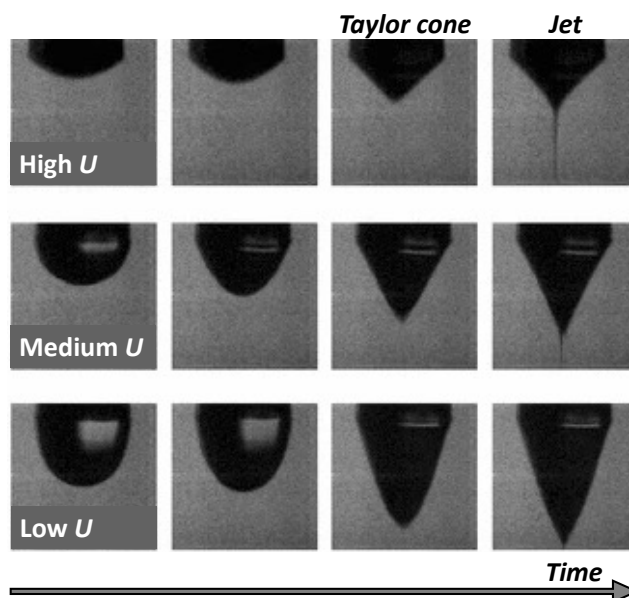


Figure 1: Taylor cone and jet formation as a function of time at different electric field strengths. Using variable voltage (U) at constant distance (d).

The electric field between the charged liquid droplet and the grounded collector causes an electrically charged liquid jet (or melt jet) to shoot out of the cone's tip and fly toward the grounded collector (typically, the distance (l) between the cone and the collector is $5 < l < 25\text{cm}$). The liquid jet significantly lengthens and thins as it travels to the collector due to the electrical forces of attraction and repulsion. Due to the consequent increase in specific surface area, the solvent in a polymer solution evaporates rapidly, whereas a melt cools down as a result. In both scenarios, the polymer builds up on the grounded collector's surface in a solid form. The constant feeding of the precursor solution (or melt), which is handled by precision pumps (syringe pumps are commonly utilized on a laboratory scale), can assure the continuity of fiber formation. **Figure 2** displays the schematic layout of the spincube electrospinning device for laboratory application.

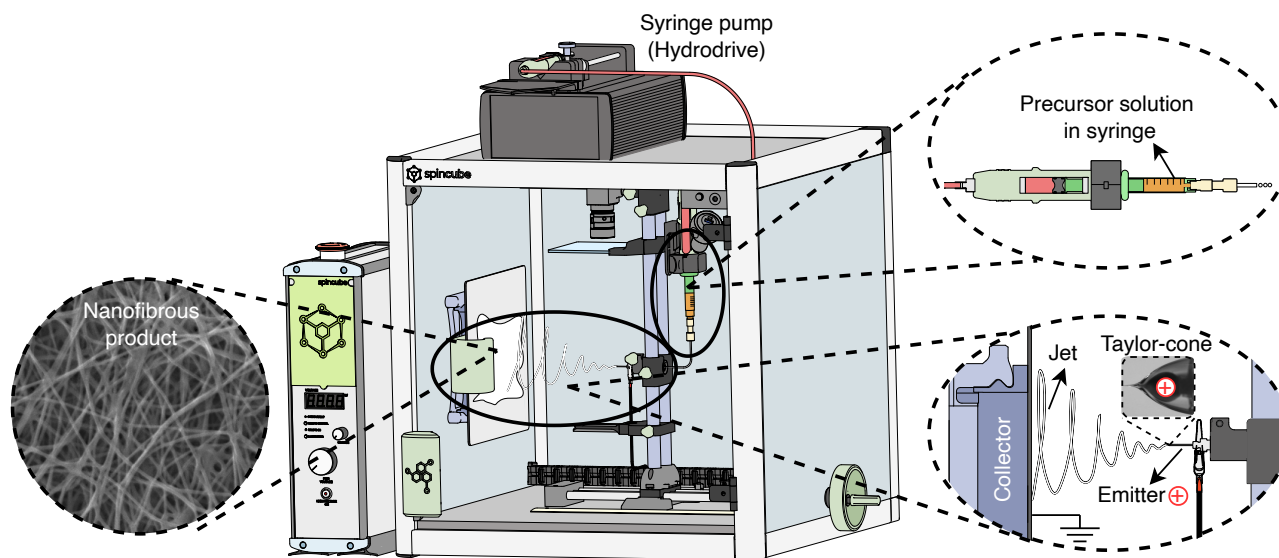


Figure 2: Schematic illustration of the spincube electrospinning apparatus and a typical electron microscopic image of the produced nanofibers.

In many cases, even though continuous delivery of the precursor is ensured, the liquid stream that exits the emitter is not continuous but instead periodically releases droplets. This process, known as electrostatic spraying (electrospraying), leaves behind primarily (polydisperse) submicron-sized spherical solid particles on the collector with no evidence of fiber production. While electrospraying has numerous beneficial applications (pharmaceutical formulations, the paint business, the food industry, etc.), it frequently causes serious issues when manufacturing nanofibrous materials, necessitating technical intervention.

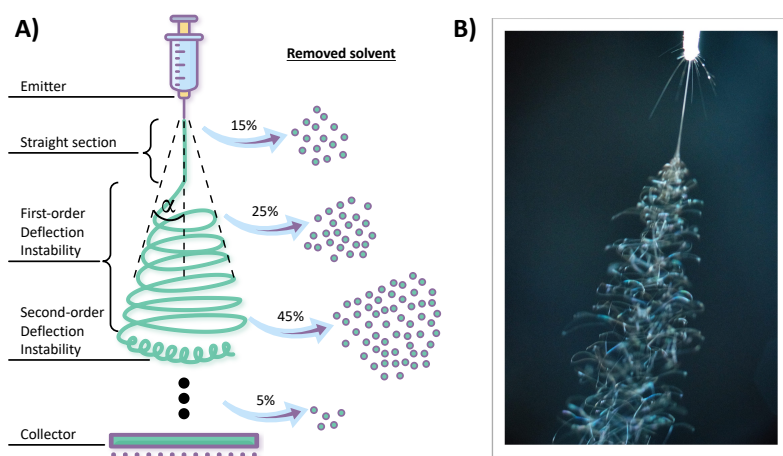


Figure 3: a) Schematic figure of jet instabilities and b) photo image of jet motion by a high-speed microcamera.

The exiting jet also exhibits a few naturally occurring instabilities, the most recognizable of which is the so-called *deflection*. First, slight deflection is created throughout the spinning process as a result of random events or potentially disruptive impacts from outside environmental factors (fluid resistance, changing temperatures, humidity, etc.). Because of Coulomb repulsion, the deflection steadily rises, causing the jet to spread. The characteristic whipping motion causes the jet to take a thinning spiral-like structure. The

widening of the helix from the emitter to the collector is rather symmetrical, as seen in **Figure 3**, and may be well defined by the increasing diameter and the angle (α) of the helix's longitudinal axis. The precursor solution's solvent loss in the emitter-collector segment is not uniform; it is most prominent during the first- and second-order deflection instabilities, where around 70% of the total solvent content is lost. The majority of the remaining solvent from the jet is also eliminated as it reaches the collector, causing solid material to be formed on the collector.

In addition to the liquid jet's inherent deformation, instability may also occur because of the precursor solution's composition and the equipment's settings and parameters. The so-called *beads*, which are the best described as bean-like thickenings of nanofibers, are one of the distinctive outcomes of these deformations. These beads can be easily identified by examining scanning electron micrographs of the nanofibrous material (**Figure 4**). Unwanted bead formation is caused by constant changes in surface tension and electric field strength, which can generally be prevented by adjusting the precursor solution composition and other fiber formation parameters.

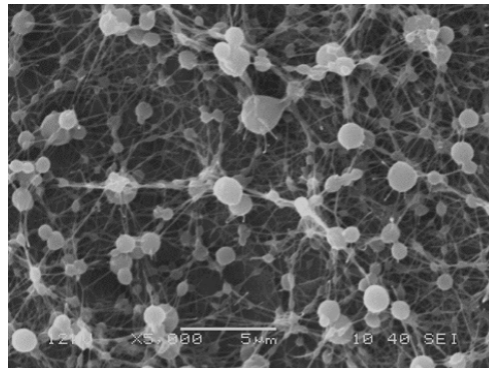


Figure 4: A scanning electron microscopic image of bead formation on nanofibers.

3 The main parameters of electrospinning

A nanofibrous material of sufficient quality has the following characteristics: sub-micron diameter fibers with a constrained size range, free of heterogeneities (such as beads), and the capacity to be produced with a satisfactory yield. A number of distinct factors that interact during fiber production may also affect the characteristics and quality of the fibrous product. Although these characteristics are the consequence of complicated processes, they may be categorized in various ways to aid in the design of the optimization process (**Table 1**).

Table 1: The main parameters affecting electrostatic fiber formation.

		set value	
parameter	<i>low</i>	<i>optimal</i>	<i>high</i>
precursor	molecular mass	no fiber formation	no fiber formation
	viscosity	no fiber formation	clogging
	conductivity	polydispersity	decreasing fiber diameter
	surface tension	fewer beads	unstable jet, electrospraying
process parameters	electric field strength	no fiber formation	beads, electrospraying
	emitter-collector distance	thickening, widening, beads	beads
	feed rate	drying, clogging	dripping, beads, increasing fiber diameter

3.1 The effect of precursor solution

The fiber production process and the characteristics of the resulting fibrous material are greatly influenced by the characteristics of the precursor solution (mixture). The possibility of fiber production is mostly determined by the molecular weight of the polymer and its solubility in a particular solvent. The requirements of the intended use, such as solubility, stability (particularly in technical or synthetic applications), or biocompatibility, biodegradability, and toxicity (especially in biomedical applications), can make the choice of polymer challenging. The development of fibers is also largely governed by the characteristics of the solvent, such as boiling point, dielectric constant, conductivity, and surface tension. Without a need for completeness, the most frequently employed polymers and solvents for the fabrication of nanofibers are listed in **Table 2**. It is vital to remember that polymer mixtures as well as single polymers are frequently employed. Similar to this, the desired nanofibrous structure may be created using binary or even ternary solvent mixes.

Table 2: The most commonly used polymers and solvents for electrospinning.

Polymer	Solvent	Application
polyurethane	dimethyl-formamide	safety clothes
polycarbonate	dimethyl-formamide / tetrahydrofuran	sensors
polyaniline / polystyrene	chloroform / camphorsulfonic acid	electric conductor
polyvinyl phenol	tetrahydrofuran	antibacterial coating
polystyrene	tetrahydrofuran	membrane filters
polylactic acid	dichloromethane	anti-scarring membrane
polyvinyl alcohol	water	drug delivery
polyvinyl pyrrolidone	ethanol	drug delivery
cellulose acetate	acetone, acetic acid	membrane filters
collagen	hexafluoro-2-propanol	artificial tissue

It is crucial to determine the molecular weight and ideal concentration of the polymer(s) after choosing the solvent(s) and polymer(s). In each case, there is a minimum polymer concentration at which fiber formation can start, although frequently, polymer solutions that are thinner than the ideal concentration can result in the formation of beads. Excessively high polymer concentrations can raise surface tension, also resulting in undesired thickenings and beads.

As a result of extremely high polymer concentrations – and the parallel increase in the viscosity of the precursor solution – the internal friction forces (the number of second-order interactions between the polymer chains) increase to such a degree that the electrostatic forces are no longer able to overcome them. It is possible to increase the diameter of the nanofibers by increasing concentration within a certain range. This effect can be clearly observed in the electron microscopic images of electrospun matrices of polyvinyl alcohol, where beads and larger fiber diameters can be seen in (**Figure 5: a-c**) with an ever-widening size distribution (**Figure 5: d**).

In addition to the concentration of the polymer, its molecular weight also has a significant impact on the rheological and electrical characteristics of the precursor solution. In general, high molecular weight promotes fiber production by providing the necessary viscosity of the precursor solution. However, there is a molecular weight limit; a molecule that is too big will result in thick fibers with a wide size distribution, and a too viscous precursor solution can make continuous and precise dosing difficult, lowering fiber production yield. Naturally, viscosity, concentration, and molecular weight are all closely related.

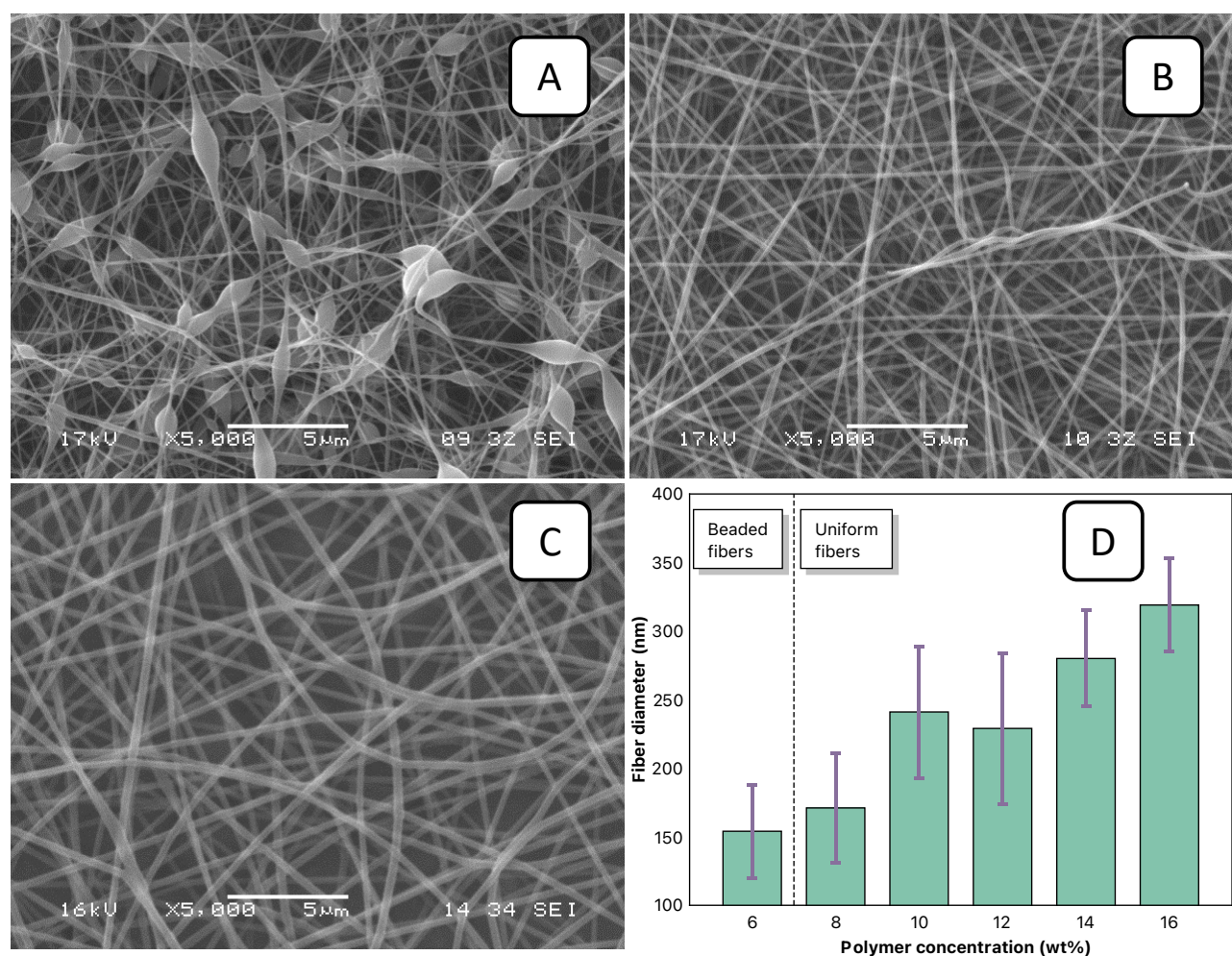


Figure 5: The effect of polymer concentration on the fiber morphology and fiber diameter in the electrospinning of polyvinyl alcohol (PVA; $M_W = 130kDa$; Degree of hydrolysis = 98%), where a) 6, b) 10, c) 14 wt% PVA concentrations were studied, d) fiber diameter distribution at the range of 6 – 16 wt% PVA concentration.

Surface tension is also an important factor in fiber production. It is typically true that bead formation may be avoided and decreased by lowering surface tension. Additionally, continuous fiber formation may be assured even at low electric field strengths when surface tension is reduced. As a result, knowing the lower and higher surface tension limits for the precursor solution is critical in electrospinning applications. If the polymer and solvent cannot be changed or adjusted, the production of nanofibers can be enhanced with various surface tension-reducing additives. Examples of such compounds include co-solvents and nonionic surfactants.

Another important parameter is the conductivity of the precursor solution, which is mostly governed by the particular type of polymer and the quality and quantity of ionizable salts present in the precursor solution. When the conductivity is weak (or inadequate), the electrostatic force field is unable to appropriately lengthen the fluid jet as it exits (the Taylor cone does not form), which prevents the formation of fibers. Sufficient conductivity can guarantee continuous fiber creation, while on the other hand, increasing the conductivity allows for thinner fibers. However, the application of an excessively strong electric field (an "overcharged" system) leads to instability and very polydisperse fiber diameters.

3.2 Effect of process parameters

After assembling the appropriate precursor system, the settings of the electrospinning equipment must also be fine-tuned. The magnitude of the applied electric field, the collector-emitter distance,

the emitter geometry, the collector geometry along with its motions (such as a rotary drum collector), and the precursor feed rate are all setup factors that must be considered. Modern software, such as **spinstudio** allow for simultaneously monitoring and adjustment of these parameters (see **Figure 6**).

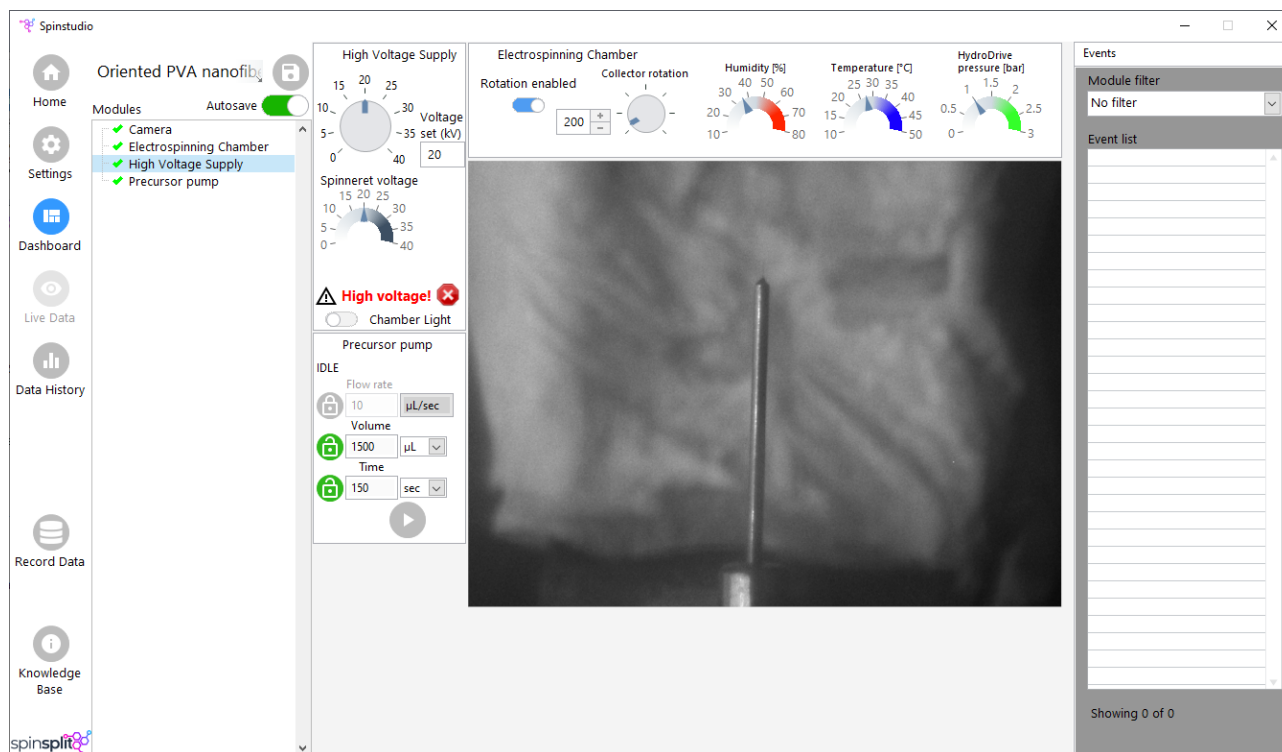


Figure 6: View of the main dashboard of **spinstudio**, where the main device parameters can be monitored and adjusted.

The magnitude of electrical voltage, which normally ranges from 5 to 40 *kV*, is a vital point during fiber development. Increasing the voltage often helps fiber formation since it boosts output, increases the tensile force applied to the fluid jet, and thus decreases the average fiber diameter. An excessively strong electric field, on the other hand, might result in an electrical discharge, which can be damaging to both the sample and the instrument. Because a DC generator can create lower fiber diameters than an AC power supply, it is more commonly employed. Positive polarity generators are more frequently utilized because they can reach lower fiber widths than negative polarity generators.

The distance between the electrodes (also known as, the collector-emitter distance) has a nearly linear impact on the magnitude of the electric field created in the device. Reducing the distance between the electrodes or raising the voltage (usually 0.5 – 5 *kV/cm* is employed) can both strengthen the electric field. Of course a certain minimum separation is needed between the collection and the emitter to ensure solvent evaporation. Additionally, undesirable bead formation might result from a path length that is either too short or too lengthy. It can also be seen that the nanofibers flatten (develop into ribbon-like fibers) at shorter distances. This may be the result of the fibers' inability to completely dry during their brief flight, which leaves them considerably more flexible when they land on the collector. Additionally, because of the collector's increased field strength, the fibers impact it at a faster rate, making it easier for them to deform. By selecting a solvent with a lower boiling point (more volatile solvent), this phenomenon can be prevented. However, solvents that evaporate too quickly make continuous dosing more challenging because the liquid on the emitter may quickly dry up and become clogged, which will prevent fiber formation. On the other hand, the cross-section of the fibers gets closer to the standard circular shape as the distance grows.

The feed rate of the precursor solution is another deciding factor since it directly impacts production. Although faster feed rates enhance the amount of fibers generated per unit of time, the solution may start to drip if the intake is too quick. Furthermore, the solvent may not be able to evaporate quickly enough, lowering the quality of the product caught on the collector.

The collector's design and arrangement are also significant since they influence productivity and

fiber orientation (for example, a rotating drum collector can assure unidirectionally oriented fibers). Collectors are often made of a grounded, well-conducting material, the most common of which is aluminum, although there are examples of collection surfaces made of plastic, cloth, or paper. Other collector elements, such as lattice, needle, and porous plates, have been developed in addition to flat surfaces.

4 Conclusion

In conclusion, electrospinning is a versatile and promising technique for producing nanofibers with unique properties. The process is influenced by various key parameters, including solution properties, process conditions, and equipment parameters. By optimizing these parameters, it is possible to control the morphology, size, and properties of the resulting nanofibers. Understanding the effect of each parameter is crucial for achieving consistent and reproducible results. Additionally, further research and development in this field could lead to the production of nanofibers with new and improved properties, opening up new opportunities for applications in fields such as biomedicine, energy, and environmental science among many others.

If you have any further questions concerning electrospinning, please contact us or read our customer research on [our website!](#)

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