



Electrospinning Process Parameters and Application: a review



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Abstract

The provided text selection describes the fundamentals of the electrospinning process and its applications in various fields. It explains the process parameters, such as solution properties, processing conditions, and mechanical/physical parameters, that can be manipulated to stabilize the nanofiber production. The electrospinning process involves a high voltage power supply, a syringe pump, a syringe, a spinneret, and a metal collector. Increasing the intensity of the electrical field causes the formation of a conical shape known as the Taylor cone, from which a charged jet of fluid is ejected. The polymer jet undergoes an elongation process, reducing its diameter as the solvent evaporates. Stable environmental conditions are crucial for obtaining high-quality nanofibers. The electrospinning process parameters can be manipulated in three main categories: solution properties (viscosity, surface tension, and vapor pressure), processing conditions (voltage level, distance from needle tip to collector, needle diameter, feed rate, and collector type), and mechanical/physical parameters (such as the type of polymer and suitable solvents). Additionally, the text discusses the use of multiple syringes (needles) in electrospinning, spinners (flat and coaxial), and their impact on fiber morphology. It also highlights various applications of electrospun nanofibers in electronic devices, sensors, filtration, and drug delivery.

Keywords: Electrospinning, Parameters, application

Introduction

Electrospinning requires a high voltage power supply, a syringe pump capable of dispensing micro liters of solution, a syringe, a spinneret (a small diameter needle connected to the syringe with a Teflon tube) and a metal collector. The power supply is essential for overcoming the surface tension of the polymer solution in syringe needle tip and initiating the charged jet of the polymer solution. The positive terminal of the power supply is connected to the needle and negative (ground) is connected to the metal collector. [1-5]

As the intensity of the electrical field between the needle and collector is increased the hemispherical surface of the fluid at the needle tip elongates to form a conical shape known as the Taylor cone. When the

intensity of the electric field is further increased towards a threshold value, the repulsive electrostatic force overcomes the surface tension and the charged jet of the fluid is ejected from the tip of the Taylor cone. The discharged polymer jet undergoes an instability and elongation process, which allows the polymer in the jet to become very long and reduces the diameter of the extruded polymer fiber.

Meanwhile, the solvent that dissolved the polymer evaporates and the polymer in the jet is dried. The solvent evaporation depends on the distance between the tip and collector, the solution vapor pressure and the inside chamber temperature. Stable environmental conditions are therefore important in getting good quality nanofibers. Figure 1 illustrates the schematic diagram of the complete electrospinning setup.

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The electrospinning process parameters are easy to setup and when manipulated help the stabilization of the nanofiber. There are thus, essentially, three types of parameters that can be manipulated in the electrospinning process [6]

- (1) solution properties
- (2) processing conditions
- (3) mechanical and physical parameter

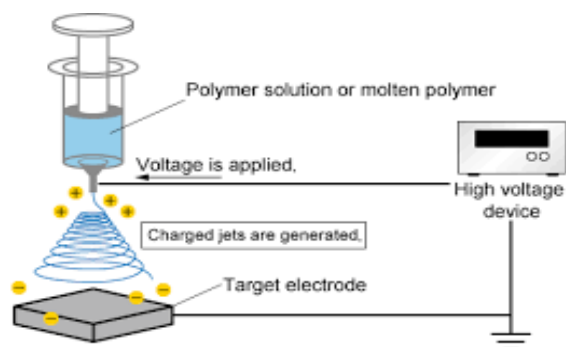


Fig. 1. Schematic diagram of the complete electrospinning process.

The chemical characteristics of the solution, such as its solubility and viscosity, are referred to as its properties. The ambient elements influencing the processing conditions are the morphology of the fiber. The feed rate, needle inner diameter, distance from needle tip to collector, kind of metal collector, and voltage intensity are among the mechanical parameters. The same methods have been applied in a variety of contexts and fields, including biotechnology, energy storage, biomedicine, and the environment. Electrospinning is a technique that can be used to

create protein nanofibrous structures, metal oxides, and pure polymer nanofibers. [7]

(1) Solution Properties.

Viscosity, surface tension, and vapor pressure are examples of solution properties that are useful parameters in electrospinning. Both the diameter and the morphology of by changing the properties of the solution, the fibers. A selection of appropriate solvents for polymers and composite polymers are displayed in Table I.

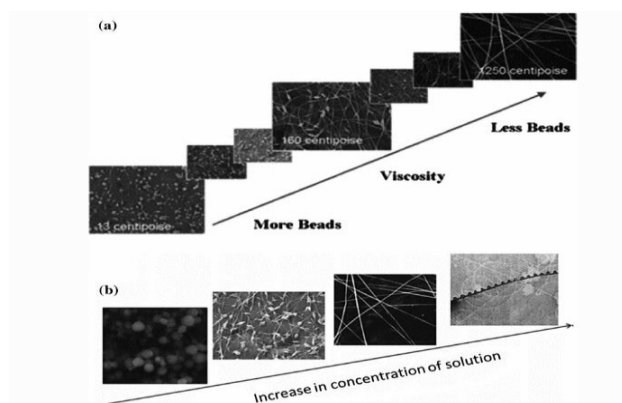


Fig. 2. a. SEM micrographs of electrospun nanofibers from different viscosities of solution, b. Evolution of electrospun nanofibers morphology with the increasing concentration of solution [8]

(2) processing conditions:

The processing takes into account variables like the voltage level, the distance between the needle tip and collector, the needle's inner diameter, the feed rate, and the kind of gatherer.

Table I. Polymers and its suitable solvents with concentration. (1)

S.no.	Polymer	Solvent	Concentration
1	Nylon6,6, PA-6,6	Formic acid	10 wt.%
2	Polyacrylonitrile, PAN	Dimethyl formamide	15 wt.%
3	Polyethylene-co-vinyl acetate, PEVA	Dimethyl formamide	14 wt.%
4	Polylactic acid, PLA	Dimethyl formamide	15 wt.%
5	Polystyrene, PS	Dimethyl formamide	18–35 wt.%
6	Polyvinyl alcohol, PVA	Distilled water	8–16 wt.%
7	Polybenzimidazole, PBI	Dimethyl acetamide	10 wt.%
8	Polyaniline (PANi)/Polystyrene (PS)	Chloroform	2 wt.%
9	Polyethylene Terephthalate, PET	Dichlormethane and trifluoroacetic	4 wt.%
10	Polymethacrylate, PMMA	Tetrahydrofuran, acetone, chloroform	10 wt.%
11	Polyvinylchloride, PVC	Tetrahydrofuran/dimethylformamide.	10–15 wt.%
12	Cellulose acetate, CA	Acetone, acetic acid, dimethylacetamide	12.5–20%
13	poly vinyl phenol, PVP	Tetrahydrofuran	20, 60% (w./v.)
14	Polycarbonate, PC	Dimethyl formamide:tetrahydrofuran (1:1)	10 wt.%
15	Polyaniline (PANi)/Polystyrene (PS)	Chloroform	2 wt.%
16	Polyamide, PA	Dimethylacetamide	4 wt.%
17	poly(ethylene-co-vinyl alcohol)	Isopropanol/water: 70/30 (%v/v)	2.5–20%w/v
18	Polyether imide, PEI	Hexafluoro-2-propanol	10 wt.%
19	Poly(vinylidene fluoride), PVDF	Dimethylformamide:dimethylacetamide (1/1)	20 wt.%,
20	Polyvinylcarbazole	Dichlormethane	7.5 wt.%

Electrospinning with Multiple Syringes (Needles)

The formation of a jet in a single needle electrospinning process is dependent on the dynamic balance of surface tension, electrostatic and columbic forces, air drag, and gravitational forces. The shape of the initiating droplet on the syringe needle tip can be changed by the applied voltage, flow rate, and solution viscosity in a single needle electrospinning process.

A secondary electrode shortens the distance between the jet deposition areas. When the number of jets in the electrospinning system is increased, one more columbic repulsion force between the jets is involved, resulting in more complex processes. [9]

Because of the mutual columbic repulsion forces, each jet deposits nanofibers in distinct zones on the collector. In addition to that, the fiber collection is determined by the force balance between the jet-jet interactions and the secondary electric field. The production rate is higher than with the single needle process, as expected. Figure 3 depicts a diagram of multi needle electrospinning and its samples.

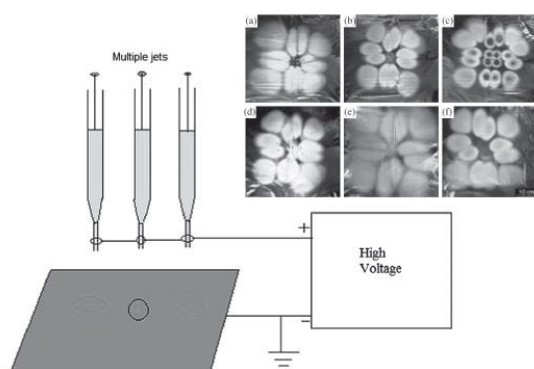


Fig. 3. Multi-jet electrospinning setup and collection pattern on the collector

Spinnerets, both flat and coaxial

Because of its simple design, low manufacturing cost, and lower cost of operation, the conducting single flat spinneret is commonly used in electrospinning instead of the multi spinnerets system. clogging probability and morphology and the proper size of resultant nanofibers. Figure 4 (A) depicts a schematic diagram of a flat base spinneret. If the electrospinning experiment is performed with lower boiling point solutions, it will solidify before reaching the collector or forming the Taylor cone at the tip's end. This issue can be solved in two ways: changing the solvent and changing the tip or needle. The co-solvent technique has been used by many researchers. the tip modification by using coaxial spinnerets and gas jackets to insulate the solutions, and so on.

Different types of spinnerets are used to create different fiber morphologies, such as double layer and hollow nanofibers. Spinneret coaxial (Fig. 4 (B)) in electrospinning is most commonly used to create

hollow nanofibers. [10] Wang and Li et al. created hollow silica and titanium nanofibers. The outer spinneret material forms a cover around the inner spinneret material in their electrospinning system. Manipulation of the operating conditions and parameters stabilizes the Taylor cone.

Because of the high voltage, only fibers that stretch and have smaller dimensions are collected. The needle tip uses gas jackets to stabilize the Taylor cone at high voltage situations. This setup has been used to create hollow fibers as well as metal and metal oxide coatings on fibers. The needle has been used with the gas jacket, resulting in Fig.4. Various spinnerets, as well as their components and schematic diagrams. fibers with an appealing surface morphology.

The schematic diagram of the gas jacket tip is shown in Figure 4 (C). Gibson, Gupta, and Ramakrishna et al. created an electrospinning device in which two polymers are extruded side by side, as shown in Figure 4 (D). This process produces fibers with properties from each of the polymeric components. These fibers, which combine properties from two different components, can be useful in protective clothing applications and other applications. The viscosity and conductivity of each polymer solution are critical processing parameters for this bi-component electrospinning technique. [11]

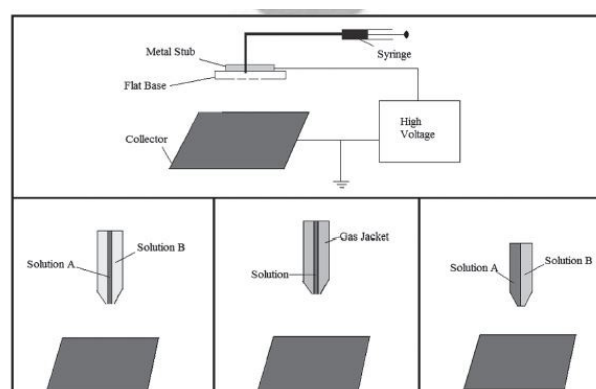


Fig. 4. Various spinnerets and their components and their schematic diagrams.

Applications

Electronic Devices

When electrospun nanofibers are derived from metals and metal composites, they have electrical and electrooptical applications. Nanofibers were created by Johnson et al. Polyaniline doped in polyethylene oxide (PAn/PEO) produces fibers with varying diameters. Scanning conductance microscopy (SCM) was used to measure the conductivity of the nanofibers. They were able to create insulating fibers with diameters as small as 15 nm. They reported that the bulk conductivity of PAn/PEO single fibers is 102 S/cm for 70 nm diameter fibers and 103 S/cm for 20 nm diameter fibers. [12]

As a result, the conductivity of nanofibers is proportional to their diameter. Pinto *et al.* were successful in demonstrating Low source-drain voltages were observed in the field-effect transistor (FET) behavior of doped PANn/PEO composite nanofibers. It was recently discovered that an electrically bi-stable device can be built using a donor-acceptor system between nanoparticles (NPs) and active media According to this principle, Wang *et al.* [13]

Electrospinning was used to create silver nanoparticles/tetracyanoquinodimethane (TCNQ) composite fibers. These fibers exhibit an intriguing electrical behavior. The current is very low if the potential is less than +29 V, indicating that the nanofibers have a high impedance. Similarly, when the potential is high or greater than +29 V, the current is very high, causing the device to switch from a low-conductivity OFF to a high-conductivity ON state. In Ag NP/TCNQ composite fibers, these bi-stable properties were stable and reproducible. Electrospinning can also produce metal nanoparticles such as silver, gold, and palladium, as well as polymer composite materials, which are useful in microelectronics. Electro-optic applications make use of surface modified TiO₂ nano fiber polymers. [14]

Sensors

Sensors are widely used in environmental protection, such as industrial process control, safety, and the detection of toxic chemicals and reducing gases that may be harmful to the environment. the natural world. The sensor must be capable of detecting objects and responding quickly. Because of their large surface area to volume ratio, electrospun fiber sensors have higher sensitivity to any detecting particles. Doping and surface modifications can be used to introduce sensing materials into electrospun nanofibers.

The composite polymer nanofibers have applications in both chemical and gas sensors.

When compared to pure PPy film, Bai *et al.*'s composite PPy/PMMA non-woven mat performed better in gas sensing to ammonia and chloroform vapors. This property is primarily due to the high specific surface area of the electrospun composite nano fibers. Zheng and co. [15]

The gas sensitivity property of α -Fe₂O₃ nano fibers was investigated. These fibers act as sensors for C₂H₅OH, CH₃OH, and NH₃, H₂, CH₄, C₂H₂, HCHO, CO, and NO₂ are the most common. Their sensitivity is affected by the operating temperature. When compared to other reducing gases, the α -Fe₂O₃ nanofiber sensor is more sensitive to ethanol. Wang *et al.* created a PAA sensing material grafted with pyrene methanol (PM) nanofibers to detect the metal ions Hg²⁺, Fe³⁺, and the explosive 2,4-dinitrotuloene (DNT) in water. The PM grafted PMMA polymer nano fibers were extremely sensitive in detecting DNT.

Filtration

Because of their novel properties such as high porosity and interconnectivity, electrospun nanofibers are promising candidates for membranes in biotechnology and environmental engineering systems. as well as their large surface area Because the electrospun fiber diameters are comparable to the mean free path of air molecules, these nanofibers can be used in water and air filtration systems. The electrospun fibers' high filtration efficiency is due to their submicron size. [16]

The fiber fineness, matrix structure, thickness, and pore size all influence filtration quality. Larger particle sizes than the pore size of Because of the sieve effect, the fiber will be blocked on the membrane surface. If the particles are smaller than the pore size, they will penetrate the membrane and can still be collected by the membranes through adsorption, electrostatic attraction, and fiber surface properties. Furthermore, due to the Brownian motion effect, very fine particles may be captured. Polystyrene (PS) nanofibers were electrospun from recycled expanded polystyrene (EPS) and combined with micro glass fibers to create a new material Shin *et al.* formed a membrane for the removal of water droplets from a water-in-oil emulsion. Depending on the total PS concentration in the membrane, the fibers were able to capture particles. [17]

They also discovered that adding nylon to electrospun fibers improves the water capture ability of glass fibers. Nanofibers are a type of fiber. They can be used as scaffolds in ultrafiltration (UF) for oil/water emulsion separation. The ultrafine membranes in oil/water separation systems are composed of three layers: the top layer of nonporous hydrophilic fibers, the middle layer of PVA nanofiberous mats, and the third layer of nonwoven substrate particles. These highly hydrophilic nonwoven nanofiber membranes have excellent filtration properties. [6]

These membranes are used to separate microparticles from water. The processing determines filtration capacity, pore size, and chemical stability.

Electrospun nanofibers with functional surface modifications are a more efficient way of filtering heavy metal ions and small molecules. The surface chemistry of the membranes is altered by these functional materials.

The surface of cellulose nanofibers is dyed Cibacron Blue F3GA, and the functionalized nanofiber membrane has a high affinity for bovine serum. Higher capture abilities for serum albumin (BSA) and bilirubin. An alkali treatment can increase the hydrophilicity of cellulose fibers. This regenerated cellulose is more chemically and thermally stable than natural cellulose fibers. The mechanical properties of a 112 Al₂O₃ doped PVDF composite electrospun fiber membrane increase by 50% when the hydrophilicity increases. [18]

drug delivery

Various techniques are used to incorporate drugs into electrospun fibers (Figure 5). The drug release profile is heavily influenced by drug loading, so selecting the best loading method is critical. essential for the desired application. [19]

The simplest approach is the direct blending between the polymer and the drug by the dissolution of the two components in a suitable solvent. Blending has the highest loading rate compared to other techniques. The strength of the polymer drug interaction will govern the release profile together with the drug solubility properties. Balancing hydrophobicity of drug and polymer is a crucial task for constant release over a defined time window.

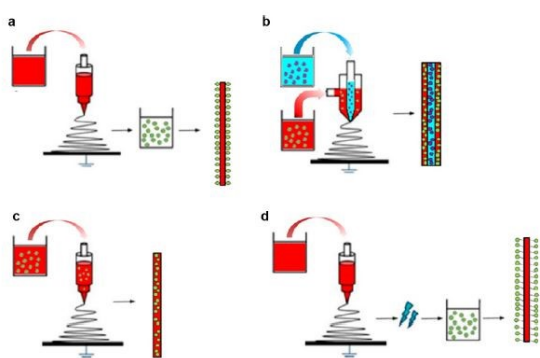


Fig 5: Schematic representation of different approaches for the drug loading. (a) physical absorption after the electrospinning. (b) Blend solution between drug and polymer. (c) Coaxial electrospinning and (d) chemical surface modification after the electrospinning process [20]

The disadvantages of this technique are primarily associated with the presence of the organic solvent, which is frequently capable of denaturing bioactive molecules. Furthermore, a burst release of the drug is commonly observed. [21]

Emulsion electrospinning offers a potential alternative, allowing the drug to be encapsulated inside micelles and formed into core-shell nanofibers. Typically, drug-containing micelles are formed by the addition of a supernatant to a drug-containing water solution. A vigorous mixing of the formed micelles with a polymer oil solution result in a stable emulsion suitable for electrospinning. The benefits are primarily two: the first is reduced contact between the bioactive molecule and the organic solvent, allowing the use of various combinations of hydrophilic drugs and hydrophobic polymers; and the second is the ease with which uniform core-shell structures can be formed without the use of specialized coaxial apparatus. [22]

Coaxial electrospinning is a loading technique as well as a technique for the formation of core-shell nanofibers. As previously stated, the coaxial technique's applicability necessitates a specific

apparatus and optimization time. It does, however, provide an infinite combination of polymers for the core and shell, as well as a modular platform for the loading of different drugs in different compartments of the fiber. Furthermore, coaxial loading of a single drug has the significant advantage of inserting the drug into the core polymer while the shell acts as a physical barrier preventing burst release. The main drawbacks are related to parameter optimization and the technique's difficult scalability. [23]

Another method for drug loading is to immobilize the bioactive molecule on the surface after the electrospinning process. As a result, every contact between the active molecule and the environment can be avoided. organic solvent, preventing any unwanted degradation. Another advantage is that the original degradation and mechanical properties of the polymeric matrix are preserved. To achieve a longer release over time, however, strong non-covalent bonding between the polymer and the drug is required, as well as a cross-linking process. [24]

Celebioglu et al. (2020) use supramolecular complexation of drugs prior to spinning as another strategy for the fabrication of fast dissolving oral drugs. In this instance, Before direct spinning of the solution, the use of cyclodextrin (CD) allowed the formation of a complex with hydrocortisone. Because of their increased solubility and rapid dissolution, the former CD-hydrocortisone fibers proved to be a very promising material for oral delivery applications. [25]

Ocular application for example

Various formulations for ocular drug delivery have been investigated in previous decades. Most ocular formulations can be classified according to the area of the eye where the drug is intended to be delivered. Topical eyedrops, ointments, and emulsions, nanoparticulate drug delivery systems, drug-loaded contact lenses, and ocular implants and patches are common formulations for the anterior segment of the eye. Drug delivery to the posterior segment of the eye is frequently attempted using intravitreal or periocular administration of nanoparticle systems, in situ forming implants, and solid implant hydrogels. [26]

Electrospun materials have been widely used in the anterior segment of the eye for therapeutic purposes. Electrospun drug delivery systems have been used to treat a variety of diseases, including infection, inflammation, aging, and neurodegeneration. There has been a significant amount of research done in the field of novel electrospun materials for anterior and posterior segment drug delivery (Table II).

Because most ocular disorders involve multifactorial disease progression and complex pathogenesis, an appropriate drug delivery system should be able to encapsulate multiple drugs at the same time while allowing controlled and sustained release.

Table II: Recent developments in electrospun materials in ocular drug delivery. (23)

Anterior/posterior segment delivery	Polymer(s) used	Therapeutic tested	Proposed clinical application
Anterior segment	Poly lactic acid	Cyclosporine	Alkali-injured cornea
Anterior segment	Chitosan, PVA and Eudragit® RL100	Ofloxacin	Microbial keratitis
Anterior segment	PVA and gelatin	Propolis	Microbial keratitis
Anterior segment	PLGA and PVP	Pirfenidone and moxifloxacin	Corneal abrasion
Anterior segment	PLC/PEG	Besifloxacin hydrochloride	Bacterial keratitis
Posterior segment	PCL	Fluocinolone acetonide	Retinal inflammation
Anterior segment	PCL	Dexamethasone	Ocular inflammation
Anterior segment	PAMAM dendrimers and PEO	Brimonidine tartrate	Glaucoma
Anterior segment	Pullulan/Gellan	Fluorescein	
Anterior segment	Sodium hyaluronate and PVP	Ferulic acid and ϵ -polylysine	Corneal infections
Anterior segment	Chitosan PVA and PVP	Ofloxacin	Corneal infections
Anterior segment	Chitosan PVA and PVP	Azithromycin	Corneal infections
Anterior segment	PVP	Azithromycin-loaded poly(lactic-co-glycolic acid) copolymer/Pluronic NPs	Corneal infections
Anterior segment	Silk fibroin	Epigallocatechin gallate	Corneal regeneration
Anterior segment	PEG-PPG-PEG	Azithromycin	Corneal infections
Anterior segment	Chitosan, Eudragit S100 and Zein	Triamcinolone acetonide	Glaucoma
Posterior segment	Poly(caprolactone)	Bevacizumab	AMD
Posterior segment	PEG/PCL	Pigmented-epithelium-derived factor	Retinal regeneration
Anterior segment	Polyvinyl Alcohol and hydroxypropyl- β -cyclodextrin	Voriconazole	Voriconazole
Anterior segment	PLA (PLA)/(PVA)	Dexamethasone	Ocular inflammation
Anterior segment	PLGA and PEG	Dorzolamide	Glaucoma
Anterior segment	PVA, acrylic resin, PVP	Voriconazole	Keratocysticosis
Anterior segment	PCL and poly(butylene succinate)	Ofloxacin	Ocular infections

Baskakova et al. [27] reported the development of a PCL-based nanofibrous matrix containing acyclovir, ciprofloxacin, and cyanocobalamin for the treatment of cytomegalovirus infections.

Electrospun matrices also offer an attractive solution for the fabrication of medical devices for the long-term delivery of biologics. Unlike the solvent-cast material, the electrospun matrix is highly malleable, flexible and amendable by different techniques. Jiang et al (25) . reported the fabrication of a hollow intravitreal medical device loaded with the anti-VEGF agent bevacizumab. The fabrication of the capsule involved the bilayer ES of chitosan and PCL matrix loaded with HEPES salts followed by heat-based sintering of the fibrous matrix for crosslinking of the matrix, offering control of drug release. The addition of salt was performed to control the pore formation of the capsular matrix that could be used as a release modifier (Fig.6). The bilayer matrix of the capsule offered control over the release of bevacizumab for up to 9 months, which is highly desirable. However, the biodegradation of the matrix was very slow, wherein no significant difference in the morphology of the capsule was observed over the course of 9 months in terms of the fiber morphology and thickness of the capsule (80–90 μm).

Summary

Electrospinning is a process that uses a high voltage power supply, syringe pump, syringe, spinneret, and metal collector. The process involves creating a

charged jet of polymer solution by overcoming surface tension with the power supply. The jet undergoes elongation and drying, resulting in the formation of nanofibers. The process parameters that can be manipulated include solution properties, processing conditions, and mechanical/physical parameters. Various solvents and polymers can be used in electrospinning, and solution properties like viscosity and surface tension are important. Processing conditions such as voltage level, distance, feed rate, and collector type also affect the process. Electrospinning can be done with multiple syringes and different types of spinnerets to create different fiber morphologies.

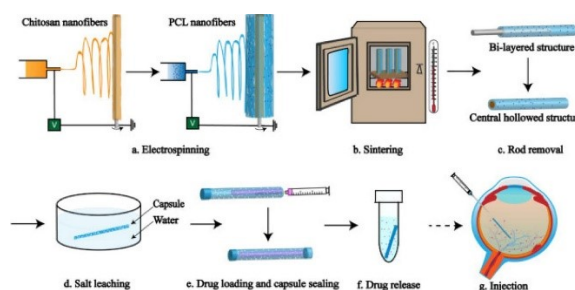


Fig 6: Fabrication of novel electrospun intravitreal implants loaded with anti-VEGF protein by Jiang and co-workers

Applications of electrospun nanofibers include electronic devices, sensors, filtration, and drug delivery. Nanofibers derived from metals and composites have electrical and electrooptical applications. Nanofiber sensors have higher sensitivity due to their large surface area, and they can be used for gas and chemical sensing. Electrospun nanofibers have potential in filtration systems for air and water, owing to their high porosity and submicron size. Functional surface modifications enhance their filtration efficiency and the ability to filter heavy metal ions and small molecules. Electrospun fibers can also be used for drug delivery, with various techniques for incorporating drugs into the fibers. Direct blending of the drug and polymer is a common method.

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Conflict of Interest

There is no conflict of interest in the publication of this article.

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