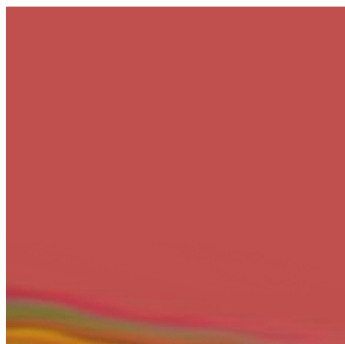
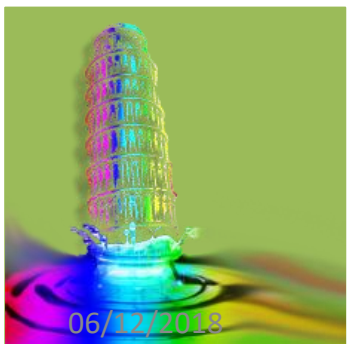




CENTRO E. PIAGGIO
Bioengineering and Robotics Research Center



Electrospinning

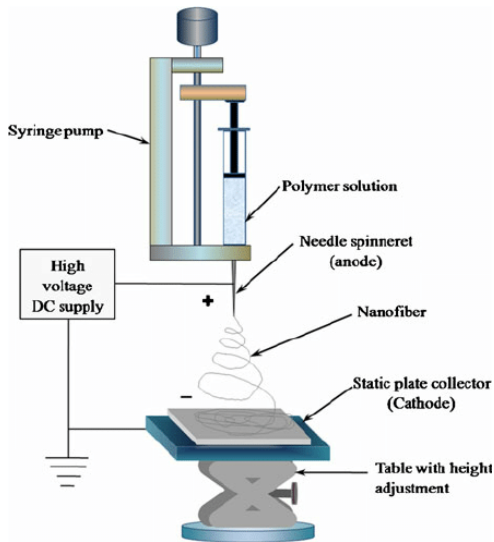
Anna Lapomarda
anna.lapomarda@ing.unipi.it

+ Overview

- Solution electrospinning (Electrospinning)
- Melt electrospinning
- Electrospaying

+ Standard electrospinning set ups

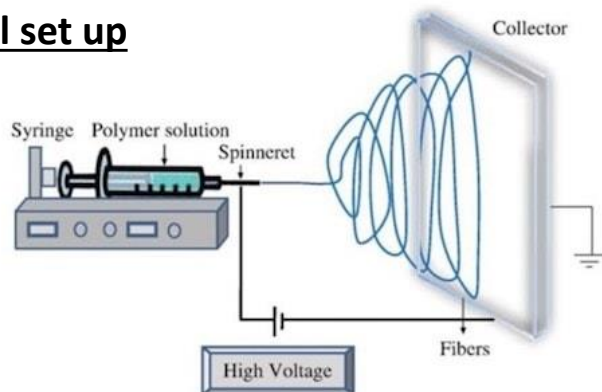
Vertical set up



Main components:

- High voltage power supply (generally 5-50 kV)
- Spinneret (e.g. a pipette tip)
- Syringe containing the polymer solution and connected to a pump
- Grounded collecting plate (usually a metal screen plate or rotating mandrel)

Horizontal set up



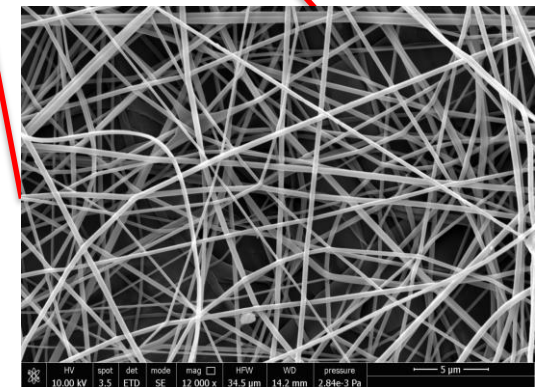
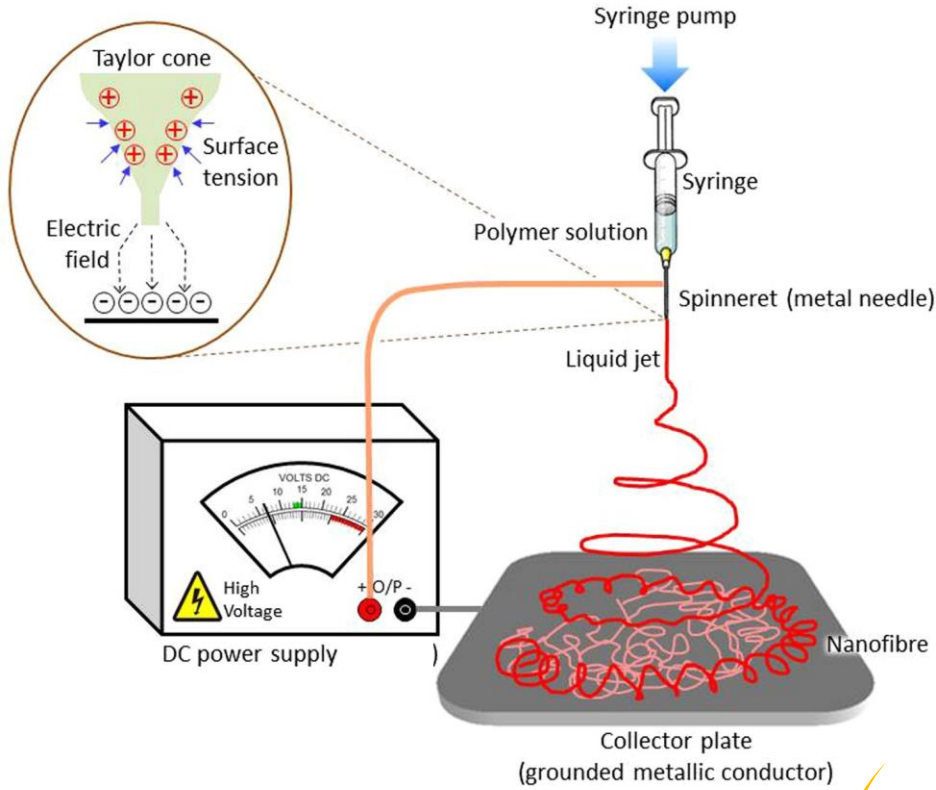
Some producers:

<http://www.mpstrumenti.eu/portfolio/electrospinning-machine-sdnfnanon-mecc-co-ltd/>

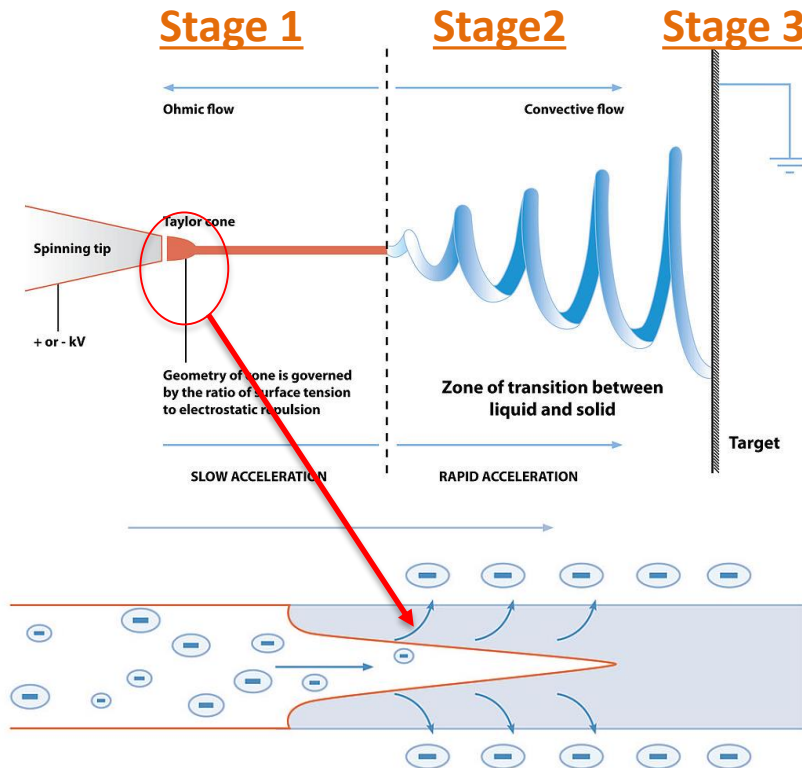
<http://espinnanotech.com/#>

<http://www.linaribiomedical.com/index.php>

+ Working principles



+ Fiber Formation



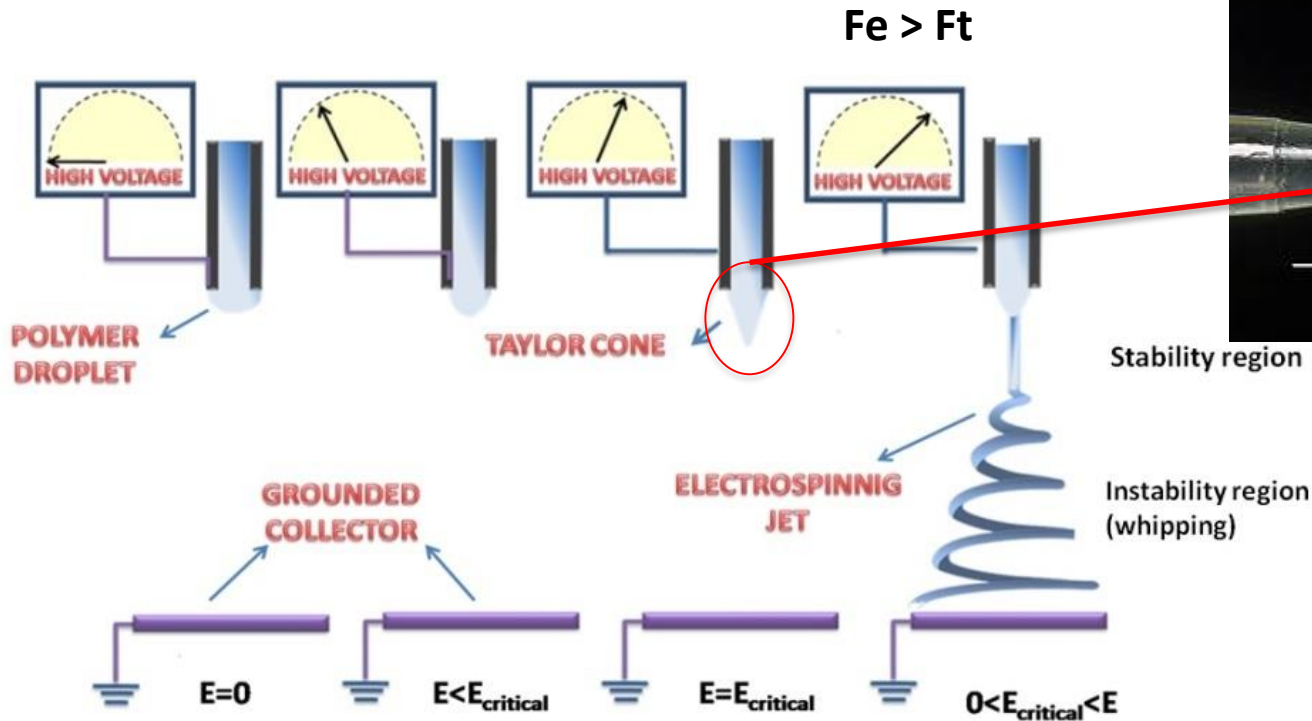
Recent theoretical and experimental studies have demonstrated that the electrospinning process generally consists of three stages:

1- jet initiation and elongation of the charged jet along a straight line;

2- growth of electrical bending instability (also known as whipping instability) and further elongation of the jet, which may or may not be accompanied with the jet branching and/or splitting;

3- solidification of the jet into micro/nanofibers and deposition on collector.

+ Electrospinning: Stage 1 (1)



Critical tension (in kV)
$$V_c = \frac{4H^2}{h^2} \left(\ln \left(\frac{2h}{R} \right) - 1.5 \right) (1.3\pi R\gamma)(0.09)$$

H = distance from the needle tip to the collecting screen

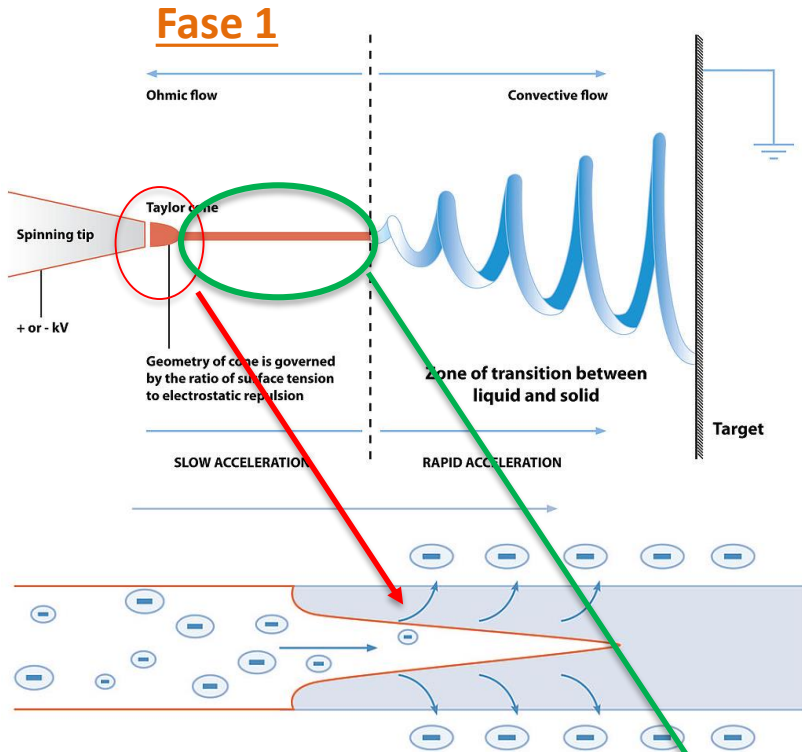
h = length of the liquid column

R = needle outer radius

γ = surface tension of the solution

0.09 = conversion factor to express the tension in kV

+ Electrospinning: Stage 1 (2)



Before the growth of electrical bending instability, the straight, tapered segment of the jet leaving the tip is accelerated as the Coulomb forces, acting on the charges carried with the leading segments, pull the jet toward the collector.

Lunghezza critica del getto

$$L = \frac{4kQ^3}{\pi\rho^2I^2} (R_0^{-2} - r_0^{-2}) \quad \text{and} \quad R_0 = \left(\frac{2\sigma Q}{\pi k\rho E} \right)^{\frac{1}{3}}$$

Q = flow rate

σ = surface charge

k = conductivity (dimensionless)

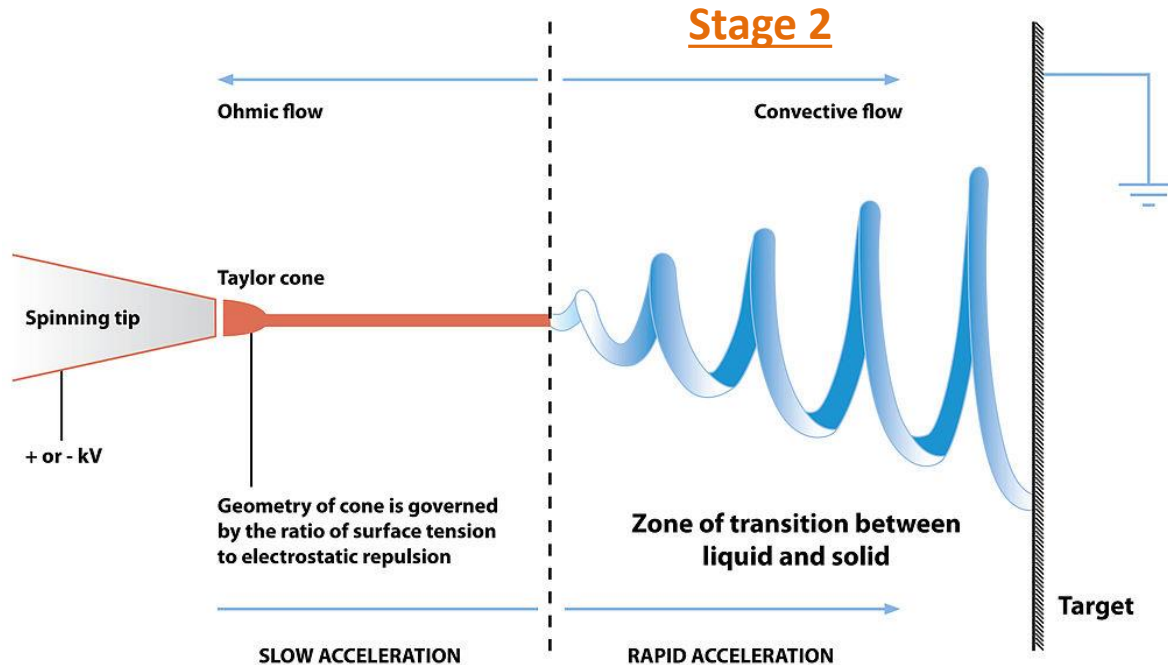
E = applied electric field

I = current passing through the jet

ρ = liquid density

r_0 = initial radius of the jet

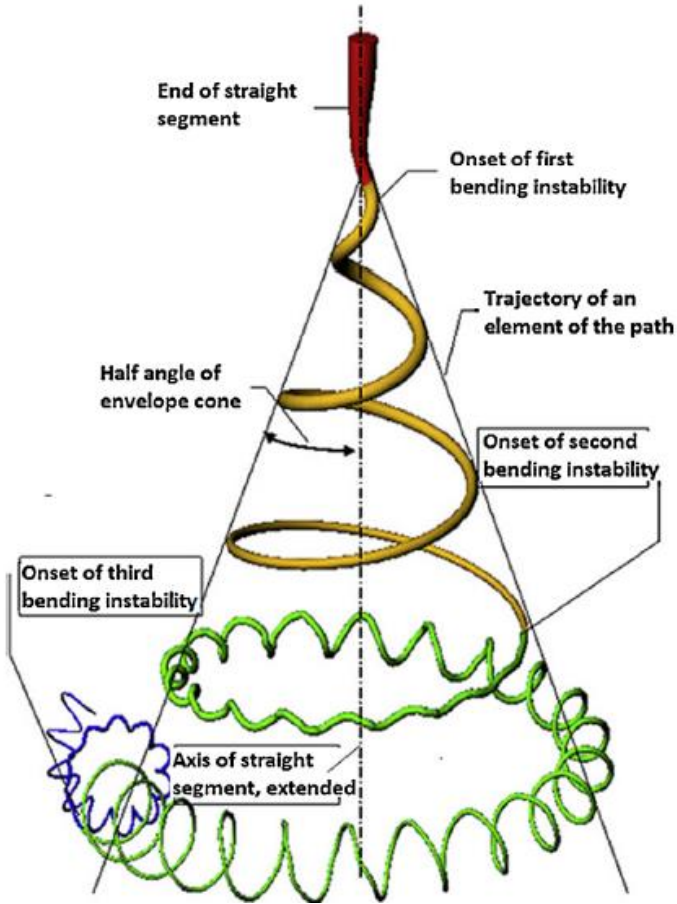
+ Electrospinning: Stage 2 (1)



As the diameter of the jet, in the straight segment, decreases monotonically with distance from the tip, the jet becomes very long and thin, and the characteristic time required for excess charge to redistribute itself along the full length of the jet becomes longer. The location of excess charge, within or on the fluid, then tends to change with the elongation, bending and otherwise deforming the jet. That means the long jet becomes unstable due to the growth of various instabilities.



Electrospinning: Stage 2 (2)



Three kind of instabilities can be observed:

1- Rayleigh instability: it is an axisymmetric instability dominated by surface tension and suppressed at high electric fields when the applied electric field E_0 and the surface charge density exceed a threshold given by:

$$(\varepsilon - \varepsilon')E_0^2 + \frac{4\pi^2\sigma^2}{\varepsilon'} = \frac{2\pi\gamma}{h}$$

where γ is the surface tension, h is the radius of the jet, ε and ε' are the dielectric constant inside and out-side the jet separately, and $\varepsilon/\varepsilon' \gg 1$.

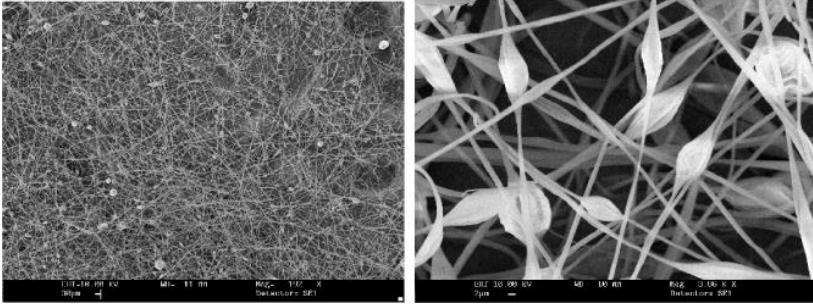
2- axisymmetric instability: it occurs at higher electric field than the Rayleigh instability

3- non axisymmetric instability (whipping instability): it describes long wave perturbations of a liquid column driven by the lateral electric force and the aerodynamic interaction at high electric field

The 2nd and 3rd instabilities are due to fluctuations in the dipolar component of the charge distribution. They are electrically driven and essentially independent of the surface tension of the liquid. It is noted that the electric field strength will be proportional to the instability level. Namely, the Rayleigh instability occurs when the electric field is the lowest, whereas the bending instability corresponds to the highest electric field .

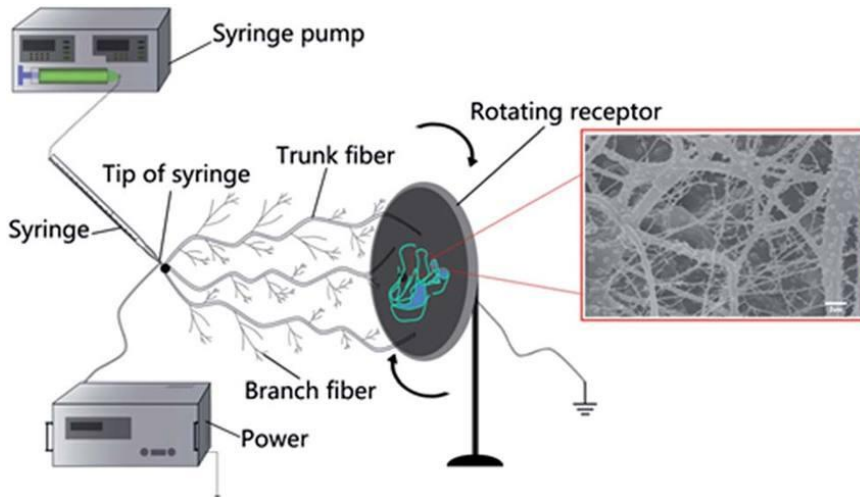
+ Electrospinning: Stage 2 (3)

BEADS



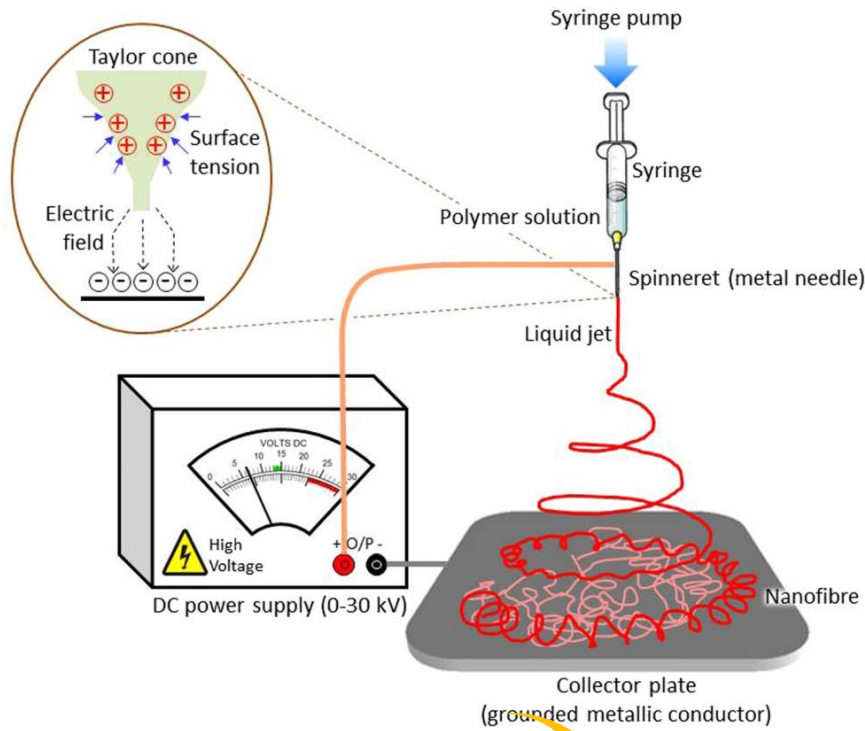
When the excess electrical charge carried by the jet is reduced, the capillary instability, which causes a cylindrical fluid jet to break up into droplets, may occur and lead to formation of beaded nanofibers. **Beads formation occurs when the charge per unit area is small.**

BRANCHING

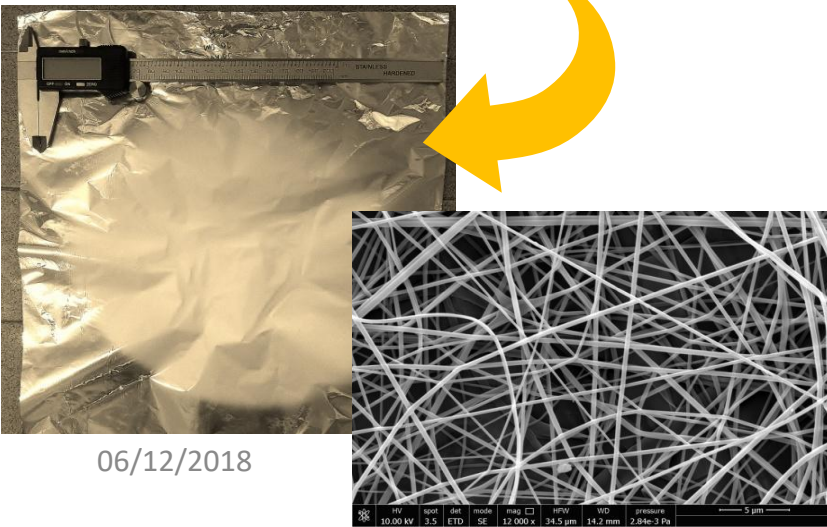


The formation of branches is observed more frequently in **more concentrated and more viscous solutions**, and at electric fields higher than the minimum field required for producing a single jet.

+ Stage 3: Fiber solidification and deposition



During the process of jet elongation, the solvent evaporates, leaving behind a charged polymer fiber. The solid polymer fibers are then deposited on a grounded collecting screen. During the fiber deposition process, a characteristic instability termed as **buckling instability** may occur due to the longitudinal compressive force from jet impingement on a solid flat surface.

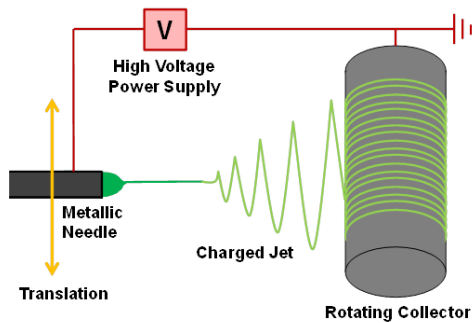


+ Types of collectors (1)



ASSEMBLY-ALIGNED FIBROUS MESH

Rotating drum



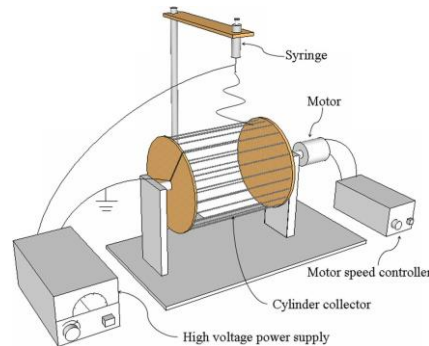
Advantage

- Simple set-up
- Large area of aligned fibres can be fabricated

Disadvantage

- Highly aligned fibrous assemblies are difficult to fabricate
- Fibre breakage may occur if rotating speed is too high

Rotating wire drum collector



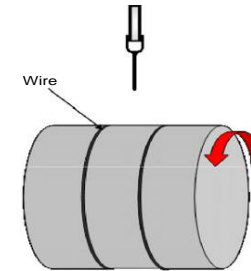
Advantage

- Simple set-up
- Highly aligned fibres are possible

Disadvantage

- Thicker layer of aligned fibres are not possible
- Fibres may not be aligned throughout the whole assembly

Drum collector with wire wound on it



Advantage

- Simple set-up
- Highly aligned fibres are possible
- Area of aligned fibres on the wire is adjustable by varying wire thickness

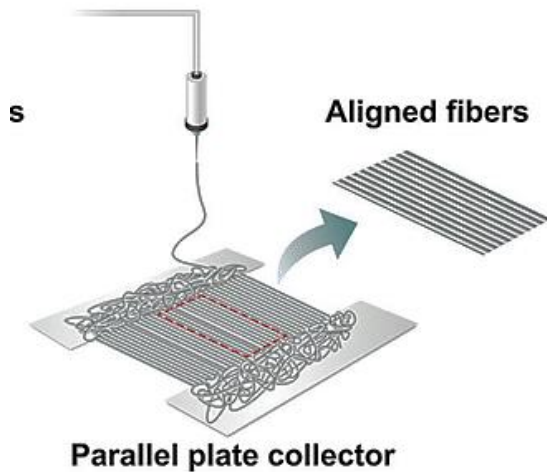
Disadvantage

- Aligned fibres are concentrated on the wire instead of the whole drum

+ Types of collectors (2)

ASSEMBLY-ALIGNED FIBROUS MESH

Parallel electrodes



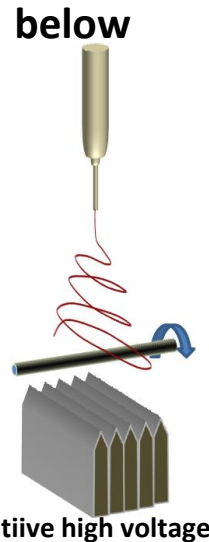
Advantage

- Simple set-up
- Highly aligned fibres are easy to obtain
- Aligned fibres are easily transferable to another substrate

Disadvantage

- Thicker layer of aligned fibres are not possible
- There is a limit in the length of the aligned fibres

Rotating tube collector with knife-edge electrodes below



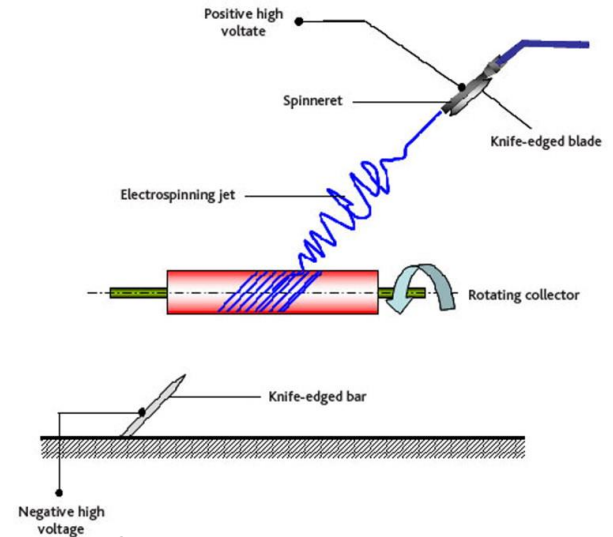
Advantage

- Highly aligned fibres possible
- Aligned fibres covered the whole tube
- Thicker layer of aligned fibre deposition is possible

Disadvantage

- Set-up requires a negative electrode to be effective
- Only possible for small diameter tube

Controlling electrospinning jet using knife-edged electrodes



Advantage

- Highly aligned fibres possible
- Able to control the direction of fibre alignment on the tube
- Spinneret with Knife edged blade
- Thicker layer of aligned fibre deposition is possible

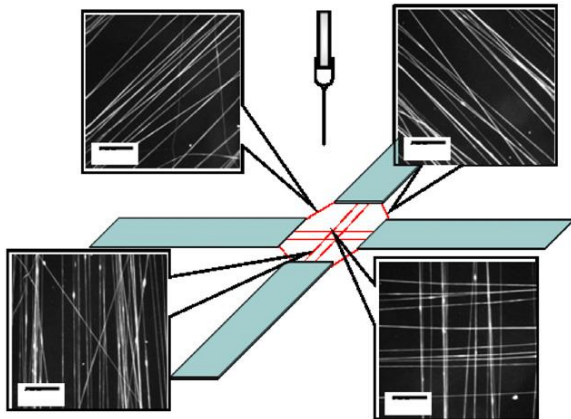
Disadvantage

- Set-up requires a negative electrode to be effective
- Only possible for small diameter tube

+ Types of collectors (3)

ASSEMBLY FIBER ARRAY

Array of counter electrodes



Advantage

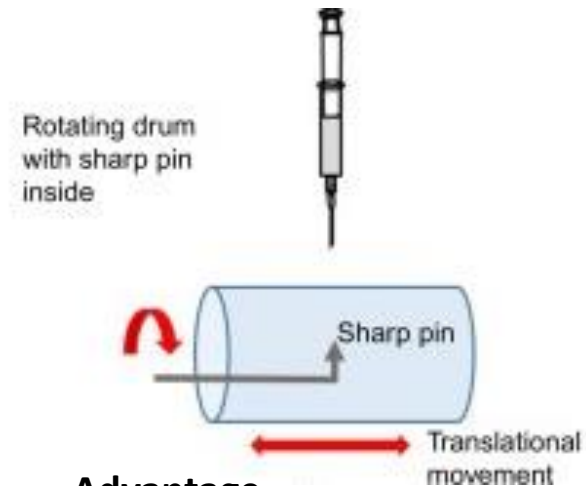
- Simple set-up

Disadvantage

- Fibre patterning is not consistent throughout the assembly
- Area of the assembly is limited
- Thicker fibrous assembly is not possible

06/12/2018

Rotating drum with sharp pin inside



Advantage

- Large area of arrayed fibres can be fabricated

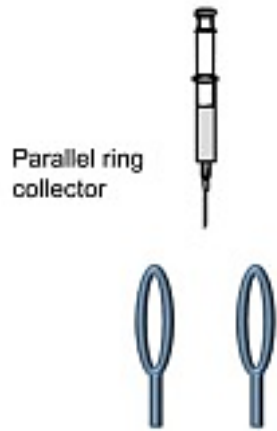
Disadvantage

- Set-up is complicated
- Thicker area of arrayed fibre assembly may not be possible

+ Types of collectors (4)

ASSEMBLY YARN

Ring collector placed
in parallel



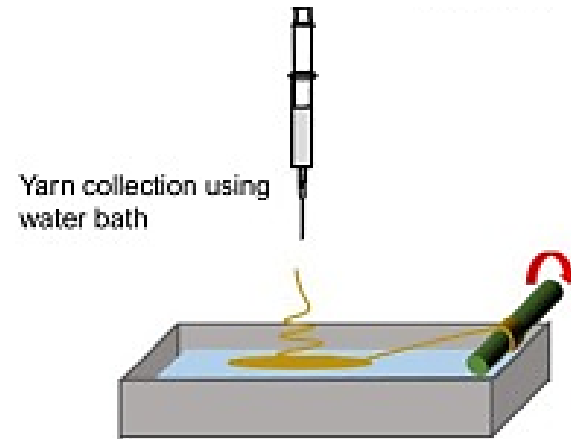
Advantage

- Simple set-up
- Twisted yarn can be fabricated

Disadvantage

- Fabricated yarn is of limited length
- One of the rings has to be rotated to twist the fibres that are deposited into yarn

Yarn collection using water bath



Advantage

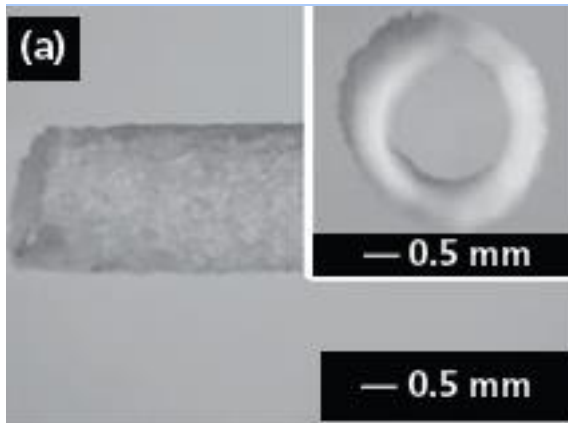
- Simple set-up
- Long continuous yarn can be fabricated
- Fibres in the yarn are generally well aligned

Disadvantage

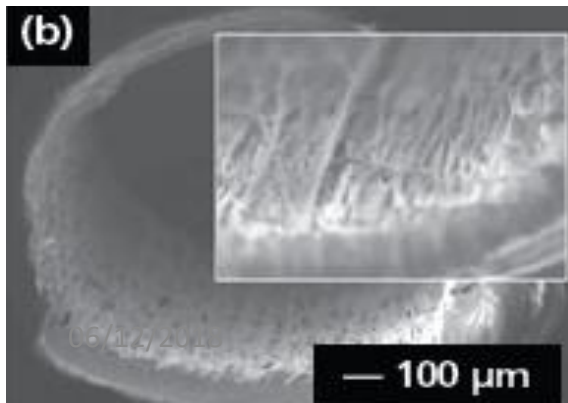
- Yarn collection speed is relatively slow

+ Types of collectors (5)

It is also possible to electrospin the material directly on object with a predefined shape and dimensions, in order to obtain three-dimensional nanofibrous structures.



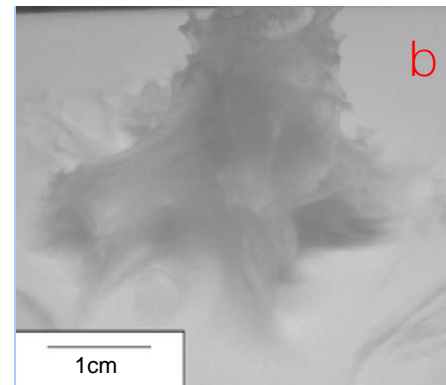
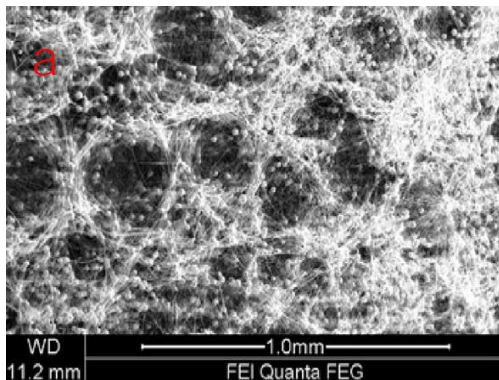
Vessel of poly(propylene carbonate) with 2mm inner diameter



Vessel obtained by rolling an electrospun structure and sealing the extremities with dichloromethane.

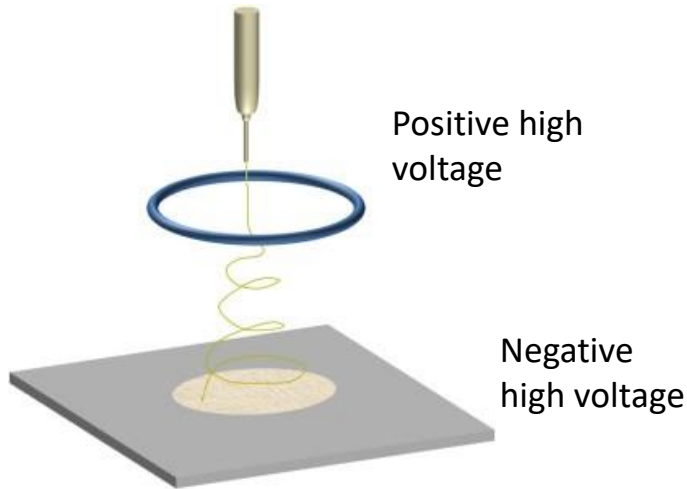
+ Static collector without the use of auxiliary electrodes

It is possible to obtain two-dimensional patterned structures or even three-dimensional structures using a static collector. Formation of **honeycomb (a)** and **dimpled (b)** have been obtained. This is attributed to the build-up of **electrostatic charges on the deposited collector, which prevents incoming electrospun fibres from depositing directly on the collector**. These fibres would form honeycomb or dimpled structures as they accumulate and dry just above the collector before they are laid down on it. The conditions required to form such three-dimensional structures are still unclear and more studies have to be carried out to determine their formation. However, as the fibres that formed this structure are very loosely packed, it can be easily compressed. Thus, it is not suitable in applications where mechanical strength is required to maintain the integrity of the three-dimensional shape.

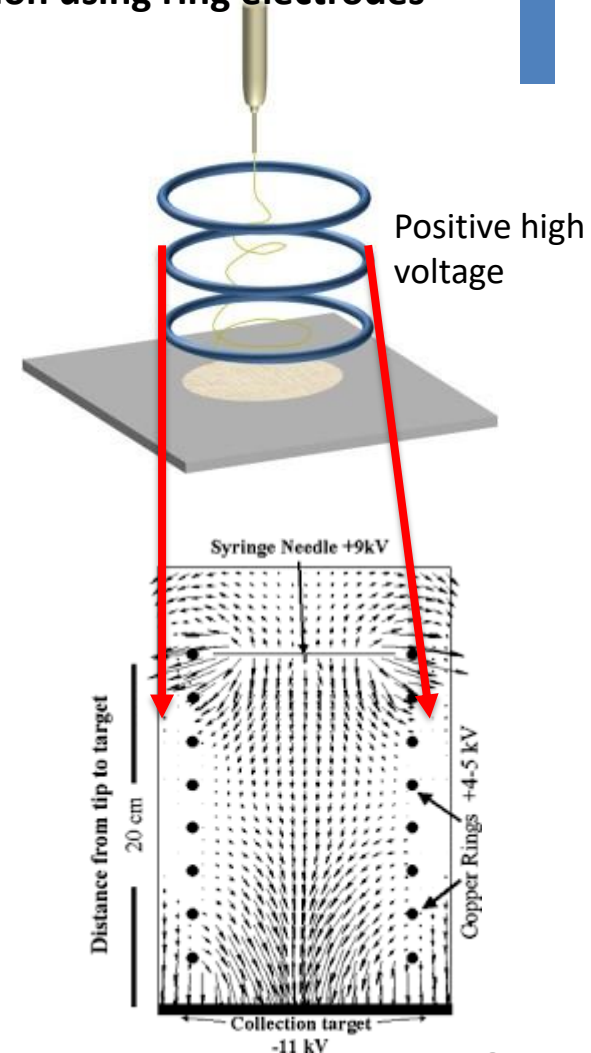


+ Utilizzo di elettrodi ausiliari per controllare la direzione del getto

Controlled deposition using single ring



Controlled deposition using ring electrodes



To manipulate the external electric field so as to exert some control on the electrospinning jet, the shape, position and polarity of the charges applied to the auxiliary electrode(s) have to be considered. Rings, placed below the tip of the spinneret, as auxiliary electrodes, with the same positive charge as that of the solution have been used. The positively charged rings that were evenly spaced out between the tip of the spinneret and the collector created a cylindrical 'electrical wall' that discouraged the charged electrospinning jet from travelling out of it. To create a pulling force on the electrospinning jet through the charged rings, a negative charge was applied to the collector

+ Choice of materials

Materials used in electrospinning:

- Natural polymers
- Synthetic polymers
- Composites
- Ceramics
- Semiconductors

Polymer solutions are directly electrospinnable instead the other categories of materials require a post processing of the fibers. For example ceramic nanofibers require a sintering process.

+ Natural polymers

Natural polymers normally exhibit better biocompatibility and low immunogenicity, compared to synthetic polymers, when used in biomedical applications. A strong reason for using natural polymers for electrospinning is their inherent capacity for binding cells since they carry specific protein sequences, such as RGD (arginine/glycine/aspartic acid). Scaffolds fabricated from natural polymers promise better clinical functionality. However, partial denaturation of natural polymers has been reported in recent years that demands concern.

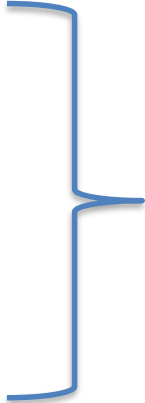
Typical natural polymers include:

- Collagen
- Chitosan
- Gelatin
- Silk protein
- Fibrinogen
- Hyaluronic acid

+ Synthetic polymers

Synthetic polymers often offer many advantages over natural polymers as they can be tailored to give a wider range of properties such as, necessary mechanical properties (viscoelasticity and strength), and desired degradation rate

- PGA
- PLA
- PCL
- PU
- PP
- PET
- PE
- PMMA
- PS
- Nylon



These materials are not used in melt electrospinning

+ Solvents used for electrospinning

Solvent performs two crucial roles in electrospinning:

-to dissolve the polymer molecules for forming the electrified jet. The selection of an appropriate solvent system is indispensable. The intermolecular interaction in a polymer–solvent system (binary system) is either attractive or repulsive which depends solely on the type of solvent. Moreover, it should maintain the integrity of the polymer during the entire process.

-to carry the dissolved polymer molecules towards the collector. This aspect depends on the solvent properties such as, solvent volatility, vapour pressure.

Particularly:

- **Solvent vapour pressure** plays a critical role in determining the evaporation rate and the drying time.
- **Solvent volatility** plays a significant role in the formation of nanostructures as it influences the phase separation process.
- **Solvent viscosity** and **surface tension** influence the fiber morphology and size

The solution viscosity is determined by the concentration of the polymer, but the value of surface tension depends on both the polymer and solvent. It has been recognized that surface tension seems more likely to be a function of solvent compositions, but is negligibly dependent on the polymer concentration. A lower surface tension of the solvent is not always necessarily suitable for electrospinning properties of solvents have a profound effect on fiber diameter.

+ Common solvents used in solvent electrospinning

Polymers	Solvents	Fiber diameter (nm)	Applications
Silk fibroin/PEO	Water	590 ± 60	Bone tissue engineering
Gelatin	Acetic/Formic acid	109–761	Biomaterial scaffold
Collagen type I	HFP ^a	100–600	Materials science and engineering
Collagen type II	HFP	496	Cartilage engineering
Gelatin/PVA	Formic acid	133–447	Controlled release of drugs
Chitosan	Acetic acid	130	Biomedical applications
PVA	Water	250–300	Drug delivery
Chitosan/PVA	Formic acid , TFA ^b , HCl	330	Tissue engineering
Cellulose acetate	Acetone, DMF ^c , Trifluoroethylene (3:1:1)	200–1000	Filtration
HA/Gelatin	DMF/Water	190–500	Tissue engineering
Fibrinogen	HFP	80 ± 30	Wound repair
Polyamide-6	m-Cresol + Formic acid	98.3 ± 8.2	Biomedical applications
Polyurathane	Water	100–500	Tissue engineering
Polycaprolactone	DMF + Methylene chloride	200	Wound healing
Collagen/chitosan	HFP/TFA	300–500	Biomaterial scaffolds
Chitin	HFP	163	Wound healing
PCL/Gelatin	TFE ^d	470 ± 120	Wound healing
Polyaniline/Gelatin	HFP	61 ± 13	Tissue engineering

^a Hexafluoro isopropanol.

^b Trifluoro acetic acid.

^c Dimethyl formamide.

^d Trifluoro ethylene.

+ Properties of solvents used in solvent electrospinning

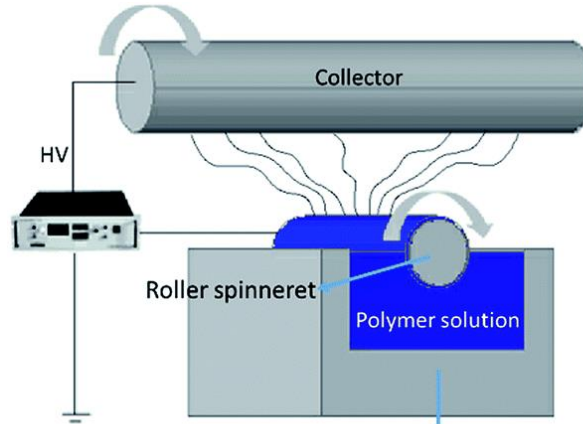
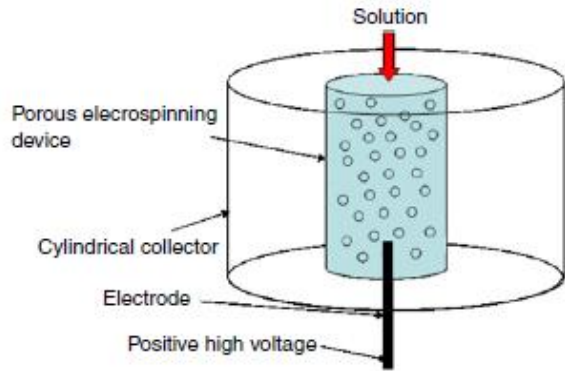
Solvents	Surface tension (mN/m)	Dielectric constant	Boiling point (°C)	Density (g/ml)
Chloroform	26.5	4.8	61.6	1.498
Dimethyl formamide	37.1	38.3	153	0.994
Hexafluoro isopropanol	16.1	16.70	58.2	1.596
Tetrahydrofuran				
Trifluoro ethanol	26.4	7.5	66	0.886
Acetone	21.1	27	78	1.393
Water	25.20	21	56.1	0.786
Methanol	72.8	80	100	1.000
Acetic acid	22.3	33	64.5	0.791
Formic acid	26.9	6.2	118.1	1.049
Dichloro methane	37	58	100	1.21
Ethanol	27.2	9.1	40	1.326
Tri fluoro acetic acid	21.9	24	78.3	0.789
	13.5	8.4	72.4	1.525

+ **Electrospinning solution delivery system**

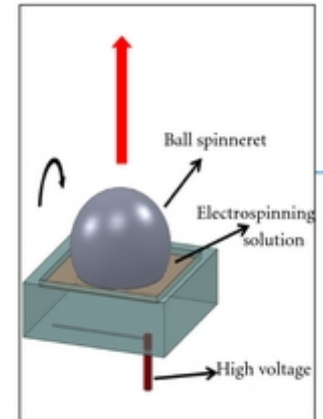
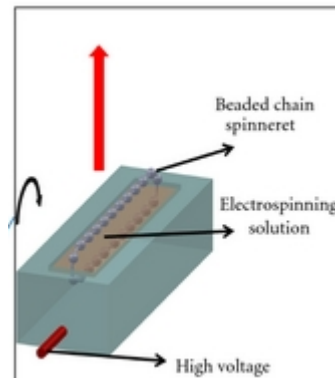
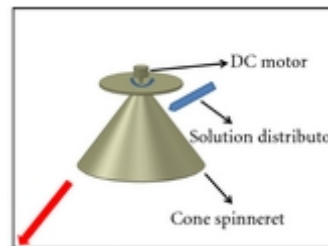
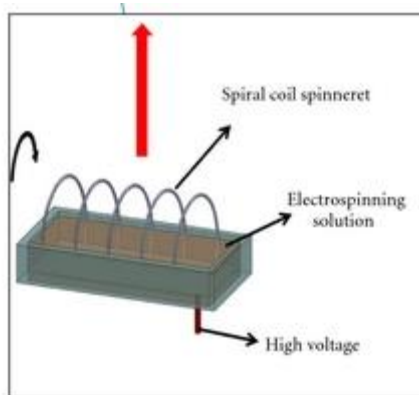
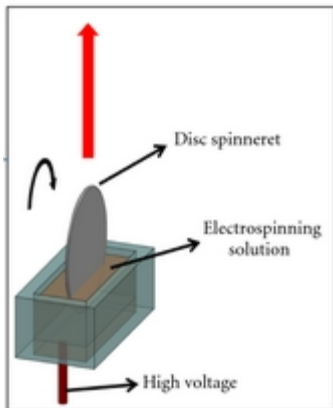


- **Needleless electrospinning**
- **Spinneret modification**

+ Needleless electrospinning (1)

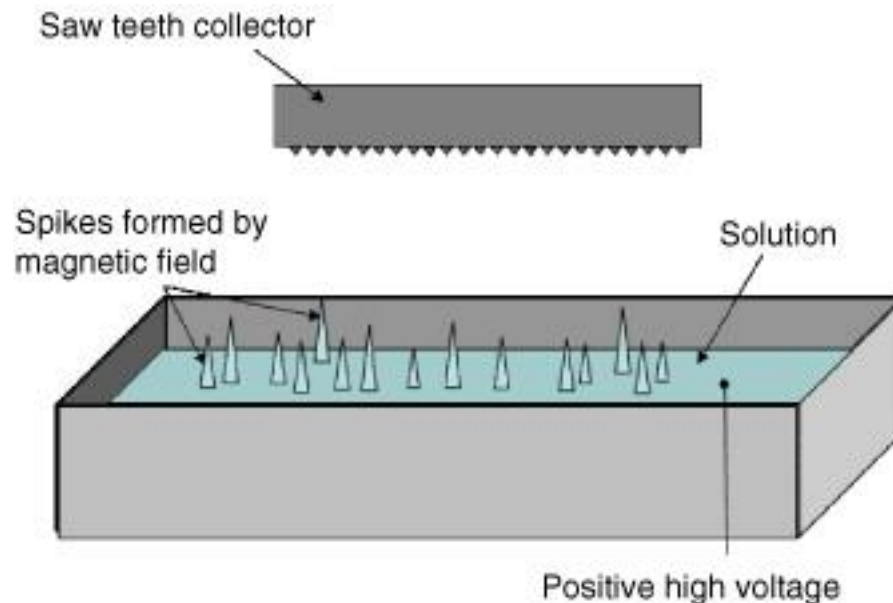


Cross section of solution tank



+ Needleless electrospinning (2)

Multiple spikes electrospinning source



Advantage

- No clogging of solution at the source
- High production rate of fibres

Disadvantage

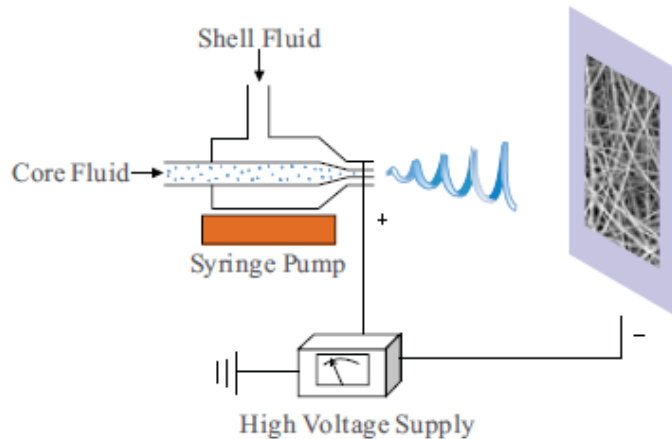
- Complicated set-up
- Variation of fibre diameter may be large



Spinneret modification



Coaxial spinneret

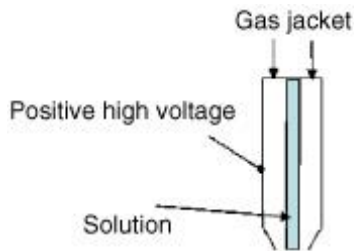


Advantage

- Single fibres made out of two different materials can be electrospun
- Hollow fibres can be fabricated by removing the core material
- Materials normally not electrospinnable can be made into nanofibre by using an electrospinnable outer material

Disadvantage

- Materials to be electrospun must be chosen carefully to reduce mixing of the materials



Gas jacket electrospinning

Advantage

- Single fibres made out of two different materials can be electrospun

Disadvantage

- Materials to be electrospun must be chosen carefully to reduce mixing of the materials



Effects of various parameters on electrospinning



The electrospinning process is solely governed by many parameters which significantly affect the fibers morphology and diameters. These parameters are classified into the following categories:

Solution parameters

- Molecular weight
- Concentration*
- Viscosity
- Surface tension
- Conductivity/surface charge density

Process parameters

- Voltage
- Flow rate
- Type of collector
- Tip to collector distance
- Diameter of the tip

Ambient parameters

- Temperature
- Umidity

* This parameter does not affect the Melting electrospinning process

+ Solution Parameters (1)

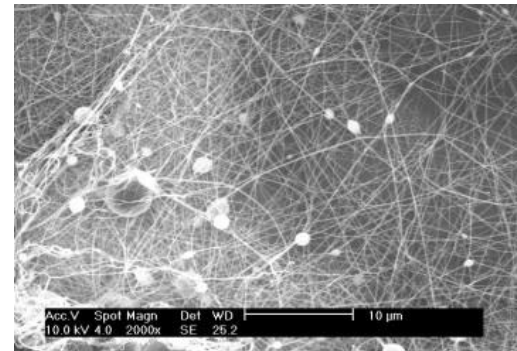
Concentration

In the electrospinning process, for fiber formation to occur, an optimum solution concentration is required : $C_{\min} < C < C_{\max}$

It has been found that at low solution concentration, a mixture of beads and fibers is obtained and as the solution concentration increases, the shape of the beads changes from spherical to spindle-like and finally uniform fibers with increased diameters are formed because of the higher viscosity resistance.

If $C < C_{\min}$

Beads are obtained instead of fibers



If $C > C_{\max}$

The formation of continuous fibers are prohibited because of the inability to maintain the flow of the solution at the tip of the needle resulting in the formation of larger fibers

+ Solution Parameters (2)

Molecular weight of the polymer

It has a significant effect on rheological and electrical properties such as viscosity, surface tension, conductivity and dielectric strength. This parameter affects the morphology of electrospun fiber: high molecular weight polymer solutions have been used in electrospinning as they provide the desired viscosity for the fiber generation. It has been observed that too low molecular weight solution tends to form beads rather than fibers and a high molecular weight solution gives fibers with larger average diameters. **Molecular weights of the polymer reflects the number of entanglements of polymer chains in a solution**, thus solution viscosity. It has been observed that high molecular weights are not always essential for the electrospinning process if sufficient intermolecular interactions can provide a substitute for the interchain connectivity obtained through chain entanglements, and using this principle, researchers have prepared oligomer-sized phospholipids from lecithin solutions into nonwoven membranes through electrospinning.

Viscosity

Solution viscosity plays an important role in determining the fiber size and morphology during spinning of polymeric fibers. It exists polymer-specific, optimal viscosity values for electrospinning and this property has a remarkable influence on the morphology of fibers. It has been found that with very low viscosity there is no continuous fiber formation and with very high viscosity there is difficulty in the ejection of jets from polymer solution, thus there is a requirement of optimal viscosity for electrospinning. The solution viscosity has been strongly related to the concentration of the solution and the relationship between the polymer viscosity and/or concentration and fibers obtained from electrospinning has been studied in a number of systems, including poly(lactic-co-glycolic acid) (PLGA).

+ Solution Parameters (3)



Surface tension

solvent compositions of the solution plays a critical role in the electrospinning process and by reducing the surface tension of a nanofiber solution; fibers can be obtained without beads. Different solvents may contribute different surface tensions. Generally, the high surface tension of a solution inhibits the electrospinning process because of instability of the jets and the generation of sprayed droplets. The formation of droplets, bead and fibers depends on the surface tension of solution and a lower surface tension of the spinning solution helps electrospinning to occur at a lower electric field. However, not necessarily a lower surface tension of a solvent will always be more suitable for electrospinning. Basically, surface tension determines the upper and lower boundaries of the electrospinning window if all other variables are held constant.

+ Solution Parameters (4)

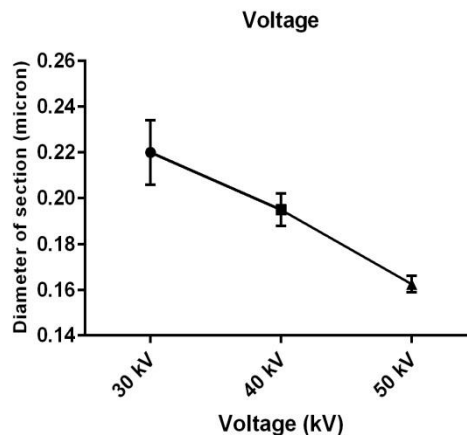
Conductivity/surface charge density

Polymers are mostly conductive, with a few exceptions of dielectric materials, and the charged ions in the polymer solution are highly influential in jet formation. **Solution conductivity is mainly determined by the polymer type, solvent used, and the availability of ionisable salts.** It has been found that with the increase of electrical conductivity of the solution, there is a significant decrease in the diameter of the electrospun nanofibers whereas with low conductivity of the solution, there results insufficient elongation of a jet by electrical force to produce uniform fiber, and beads may also be observed. Highly conductive solutions are extremely unstable in the presence of strong electric fields, which results in a dramatic bending instability as well as a broad diameter distribution. Generally, electrospun nanofibers with the smallest fiber diameter can be obtained with the highest electrical conductivity and it has been found that the there is drop in the size of the fibers is due to the increased electrical conductivity. It was observed that the jet radius varied inversely with the cube root of the electrical conductivity of the solution. It is possible to vary the conductivity of the polymer by adding ionic salt like KH_2PO_4 , NaH_2PO_4 , and NaCl .



Process parameters (1)

Voltage



In the electrospinning process a crucial element is the applied voltage to the solution. Only after attainment of threshold voltage (V_c), fiber formation occurs, this induces the necessary charges on the solution along with electric field and initiates the electrospinning process. There is a little dispute about the behaviour of applied voltage in the electrospinning process. Some researchers have suggested that when higher voltages are applied, there is more polymer ejection and this facilitates the formation of a larger diameter fiber. Other authors have reported that an increase in the applied voltage (i.e., by increasing the electric field strength), increases the electrostatic repulsive force on the fluid jet which ultimately favours the narrowing of fiber diameter. In most cases, a higher voltage causes greater stretching of the solution due to the greater columbic forces in the jet as well as a stronger electric field and these effects lead to reduction in the fiber diameter and also rapid evaporation of solvent from the fibers results. At a higher voltage there is also greater probability of beads formation.

+ Process Parameters (2)

Flow rate

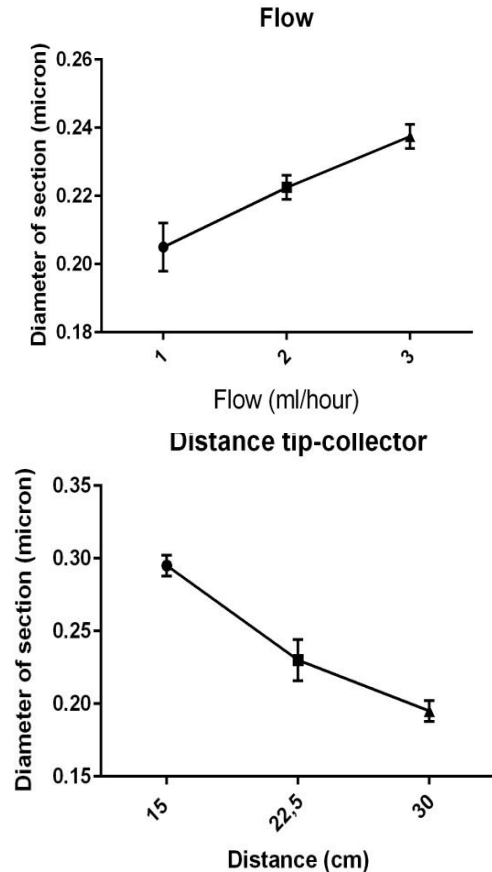
The flow rate influences the jet velocity and the material transfer rate. A lower feed rate is more desirable as the solvent will get enough time for evaporation. There should be always a minimum flow rate of the spinning solution. It has been observed that the fiber diameter and the pore diameter increases with an increase in polymer flow rate. High flow rates result in beaded fibers due to unavailability of proper drying time prior to reaching the collector.

Tip to collector distance

It has been found that a minimum distance is required to give the fibers sufficient time to dry before reaching the collector, otherwise with distances that are either too close or too far, beads have been observed. It has been reported that flatter fibers can be produced at closer distances but with increase in distance rounder fibers have been observed

Type of collector

A collector serves as a conductive substrate where the nanofibers are collected. Generally, aluminium foil is used as a collector but due to difficulty in transferring of collected fibers and with the need for aligned fibers for various applications, other collectors such as, conductive paper, conductive cloth, wire mesh etc were used. The fiber alignment is determined by the type of the target/collector and its rotation speed



+ Ambient parameters

Humidity

It has been found that at very low humidity, a volatile solvent may dry rapidly as the evaporation of the solvent is faster. Sometimes the evaporation rate is so fast than compared to the removal of the solvent from the tip of the needle and this would create a problem with electrospinning. As a result, the electrospinning process may only be carried out for a few minutes before the needle tip is clogged. It has also been suggested that the high humidity can help the discharge of the electrospun fibers.

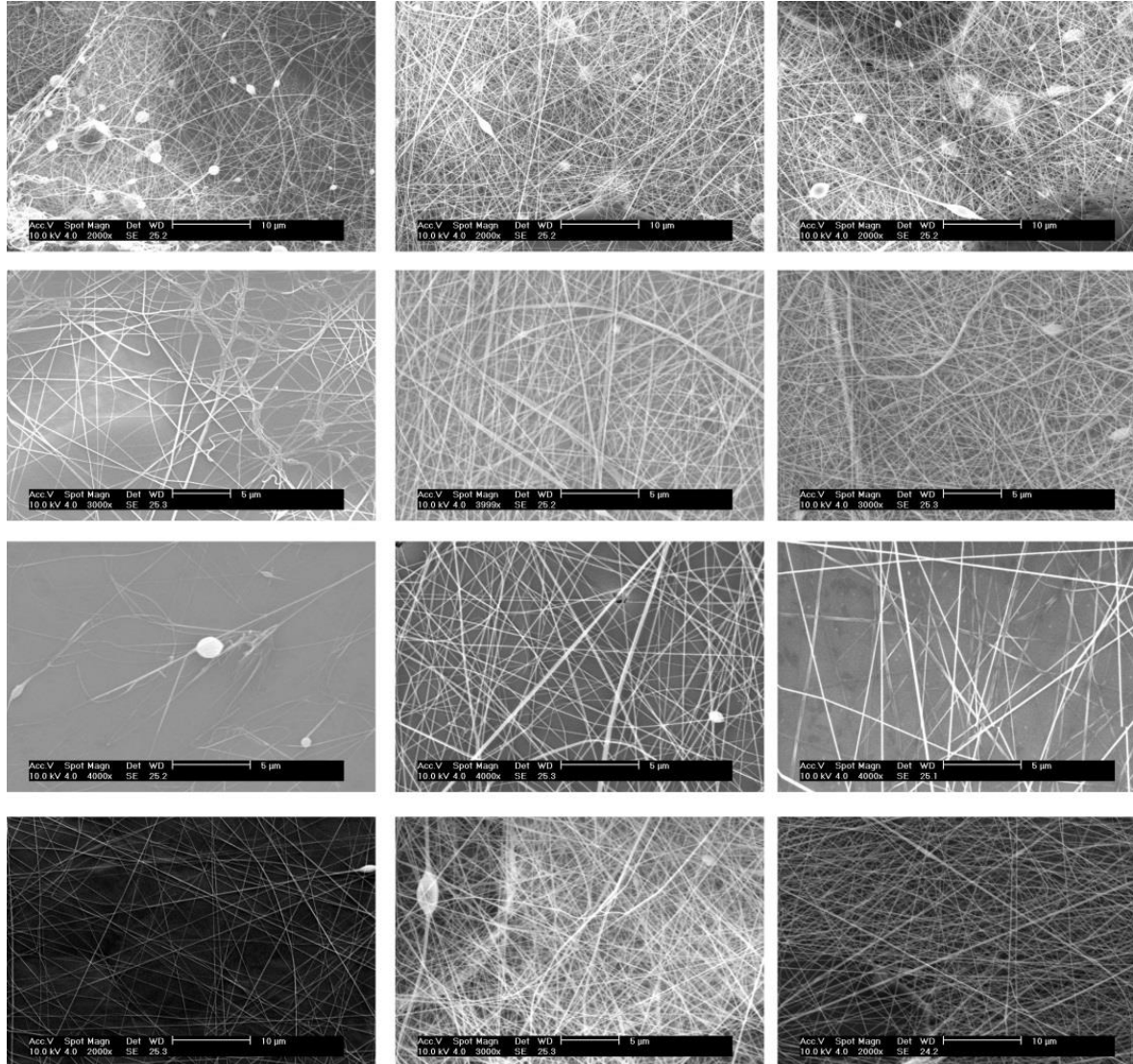
Temperature

It has been showed that an increase in temperature is associated to a decrease fiber diameter, and this is due to the decrease in the viscosity of the polymer solutions at increased temperatures (there is an inverse relationship between viscosity and temperature).

+ Electrospinning parameters and their effects on fiber morphology

Parameters	Effect on fiber morphology
<i>Solution parameters</i>	
Viscosity	Low-beads generation, high-increase in fiber diameter , disappearance of beads.
Polymer concentration	Increase in fiber diameter with increase of concentration.
Molecular weight of polymer	Reduction in the number of beads and droplets with increase of molecular weight.
Conductivity	Decrease in fiber diameter with increase in conductivity.
Surface tension	No conclusive link with fiber morphology, high surface tension results in instability of jets.
<i>Processing parameters</i>	
Applied voltage	Decrease in fiber diameter with increase in voltage.
Distance between tip and collector	Generation of beads with too small and too large distance, minimum distance required for uniform fibers.
Feed rate/Flow rate	Decrease in fiber diameter with decrease in flow rate, generation of beads with too high flow rate.
<i>Ambient parameters</i>	
Humidity	High humidity results in circular pores on the fibers.
Temperature	Increase in temperature results in decrease in fiber diameter.

+ Electrospinning results



+ Melt electrospinning

Melt electrospinning allows new approaches to various applications, overcoming technical restrictions governed by solvent accumulation and toxicity. In melt electrospinning, instead of a solution, the polymer melt is introduced into the capillary tube and has to be performed in a vacuum condition and for that the capillary tube, the traveling of the charged melt fluid jet, and the metal collecting screen must be encapsulated within a vacuum.

Advantages:

- No requirement of dissolution of polymers in organic solvents and their removal/recycling
- A higher throughput with no loss in mass by solvent evaporation
- Generation of sub-micron scale fibers of polymers which lack appropriate solvents at room temperature
- Cheaper procedure

Disadvantages:

- High viscosity (many orders of magnitude greater than that of a polymer solution)
- Required very high processing temperature to maintain the polymers melted
- Inability to attain fibers in nanometer range

These disadvantages may preclude their being used for tissue engineering or drug delivery applications.

+ Materials used in melt electrospinning

Polymer	Processing temperature (°C)
Polypropylene	220–240
Poly(ethylene terephthalate)	270
Poly-(ethylene glycol-block- ϵ -caprolactone)	58.2
Polyethylene	200–220
Poly(methyl methacrylate)	130–157
Polyamides (nylon)	220
Polystyrene	240



Characterizations of electrospun nanofibers



The characterization of fibers produced by the electrospinning process remains one of the most difficult tasks as the chances of getting single fibers are rare. Generally in electrospinning, the polymers used are

- **Geometrical characterizations (physical and structural)**
- **Mechanical characterization**
- **Chemical characterization**

+ Geometrical characterization

Determination of the morphological properties of the electrospun structures

- **Shape and diameter of the fiber**
- **Fiber orientation**
- **Fiber morphology (e.g. cross-section shape and surface roughness)**
- **Porosity and pore size**

For the characterization of geometric properties, techniques such as scanning electron microscopy (SEM), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), and atomic force microscopy (AFM) are used.

Porosity is another geometric parameter and the porosity and pore size of nanofiber membranes are important for its applications in filtration, template for tissue engineering, protective clothing, etc. The pore size measurement can be conducted by a capillary flow porometer, mercury porosimetry. Generally electrospun structures show very high porosity of about 90%.

+ Chemical characterization

The characterization of the **molecular structure of a nanofiber** can be done by **Fourier transform infra red (FTIR)** and **nuclear magnetic resonance (NMR) techniques**. If we blend two polymers together for the fabrication of nanofibers, not only can the structure of the two materials be detected but the intermolecular interaction can be determined by the use of these methods.

Surface chemical properties of nanofibers can be evaluated by its hydrophilicity, which can be measured by measuring the contact angle of polar or apolar solvents.

+ Mechanical characterization

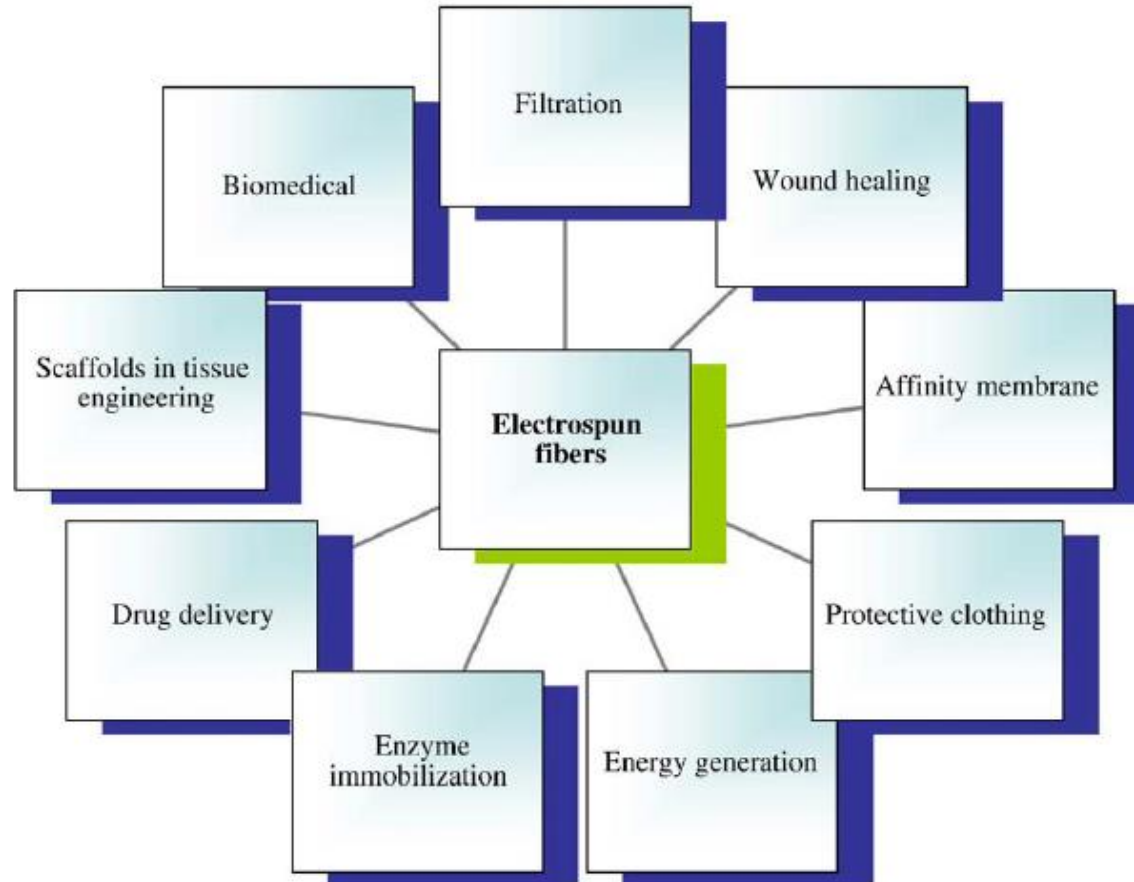
Precise measurement of mechanical properties of the nanofibrous matrix is crucial, especially for biomedical applications, for example as scaffolds, because the scaffold must be able to withstand the forces exerted by growing tissue or during physiological activities and related biomechanics. Mechanical characterization is achieved by applying tensile test loads to specimens prepared from the electrospun ultra fine non-woven fiber mats. During mechanical characterization of single nanofibers sufficient care must be taken in sample mounting in order to avoid severe damage or sample manipulation. A variety of approaches has been applied towards mechanical characterization of nanofibers and nanowires by employing **nanoindentation, bending tests, resonance frequency measurements, and microscale tension tests.**

The anisotropy of the structure depends on the type of collector used (e.g. static, dynamic).

Moreover, the mechanical properties are affected by the fiber dimension: the elastic modulus decreases with the diameter of the fiber

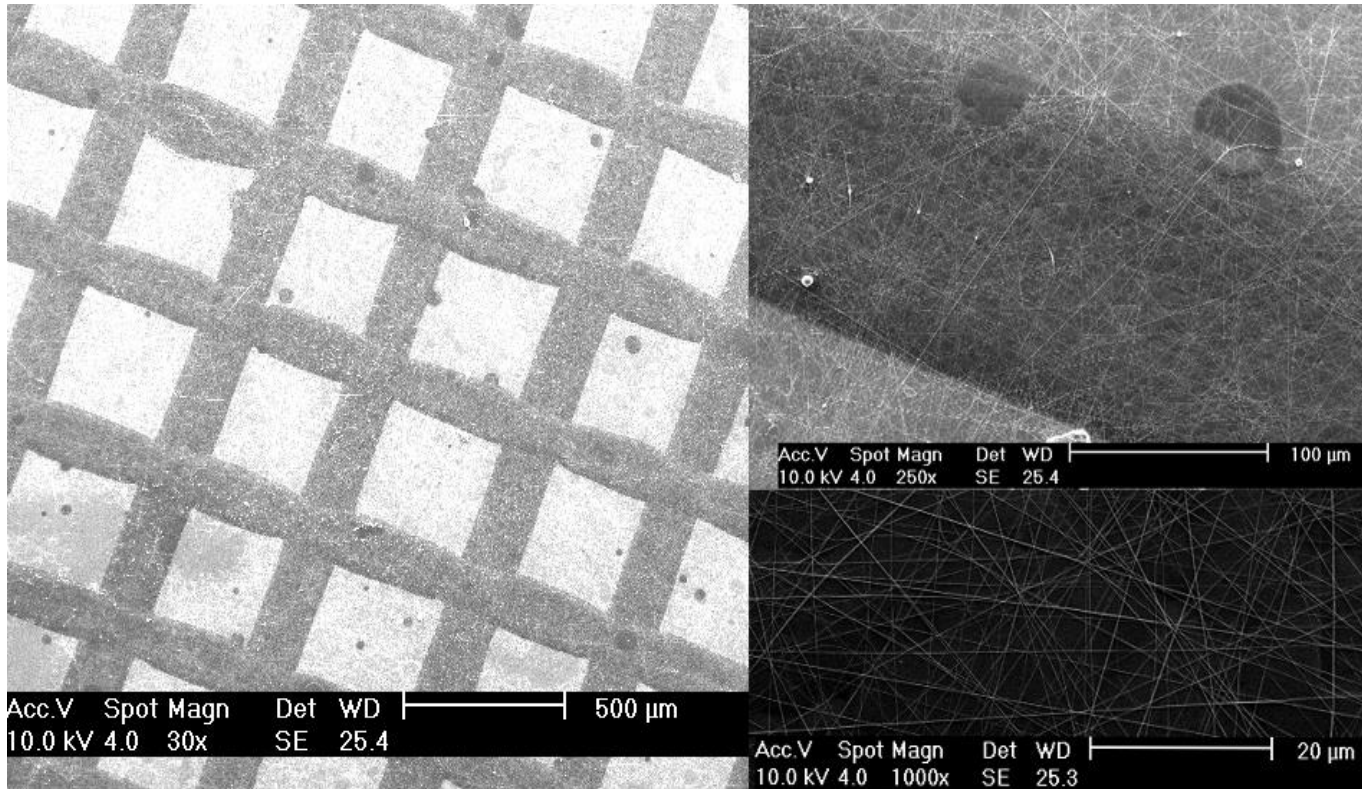
Polymers	Ultimate strength
Collagen II	3.3 ± 0.3 MPa
poly(ϵ -caprolactone)	40 ± 10 MPa
Gelatin	4.79 MPa
Cross-linked gelatin	12.62 ± 1.28 MPa
Silk fibroin	7.25 MPa
Poly(vinylchloride)/Polyurethane (25/75)	6.30 MPa
Polyethylene oxide	10 ± 0.2 MPa

+ Electrospinning applications



+ Applications

PAM²-Electrospinning

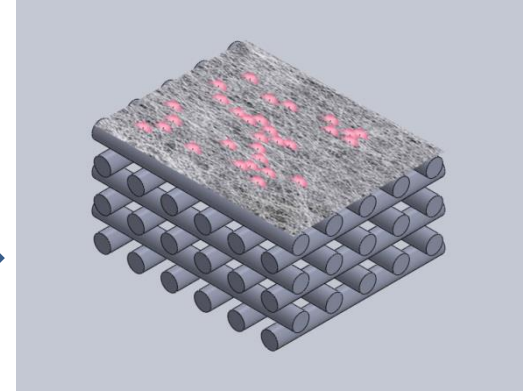
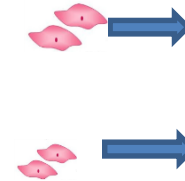




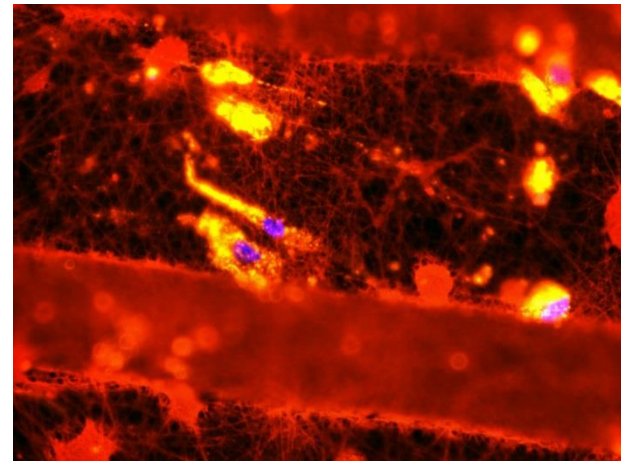
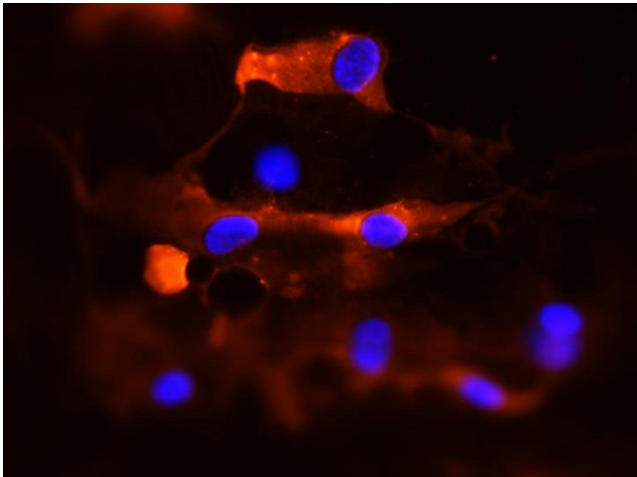
In-vitro assay



- Cell Density
- Viability Assay: MTS assay
- Proliferation Assay: EDU



Used cells: Saphenous Vein Pericytes





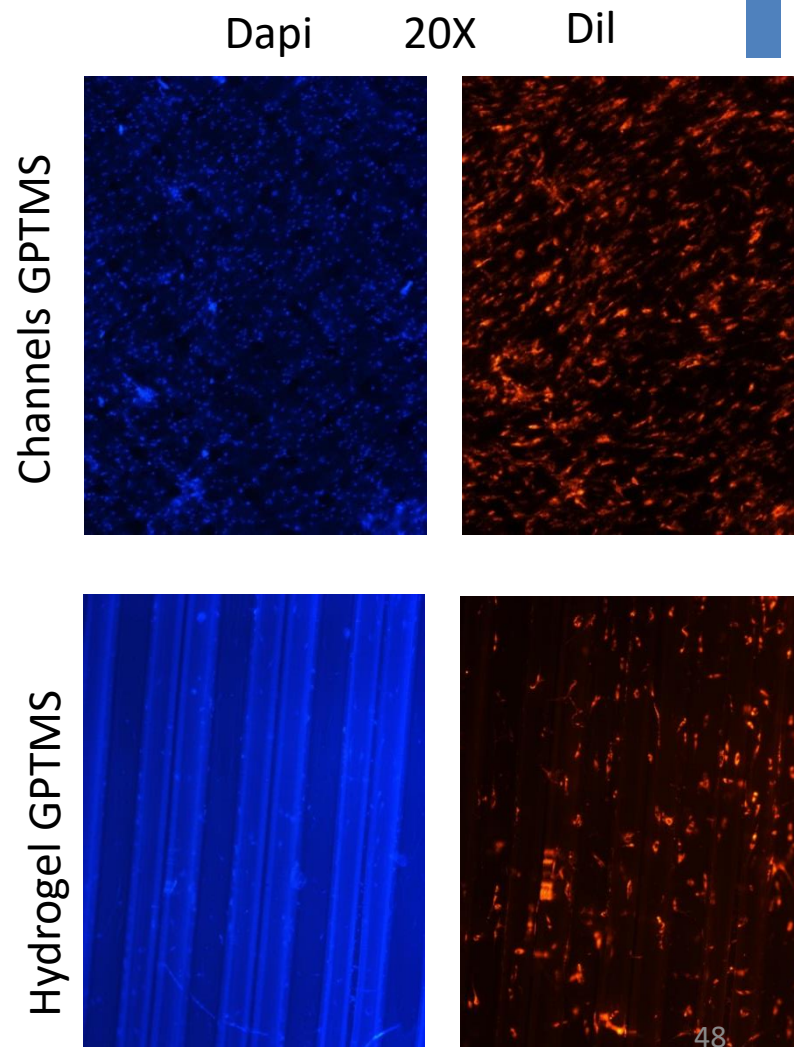
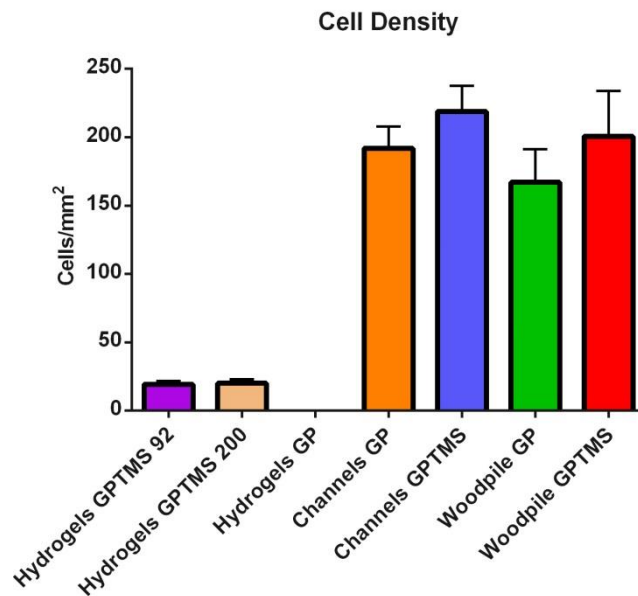
In-vitro assay



Cell Density

Cells marked with:

- Dapi: Nuclei
- Dil: Membranes



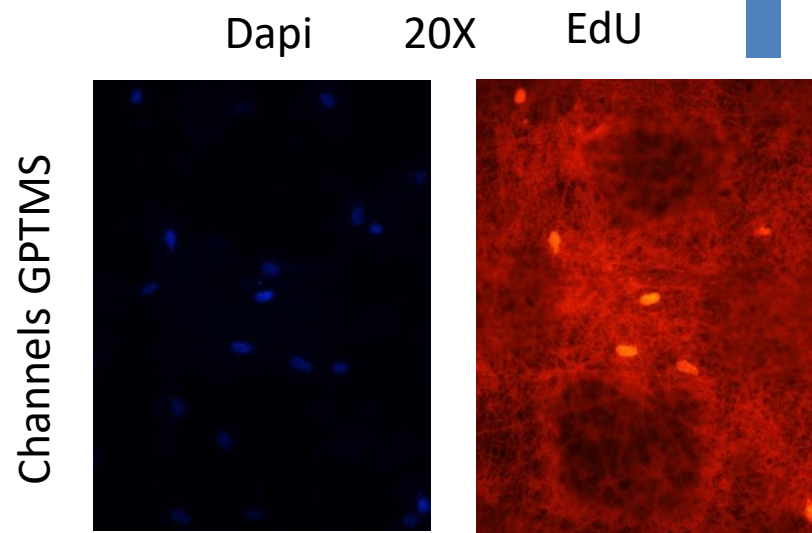
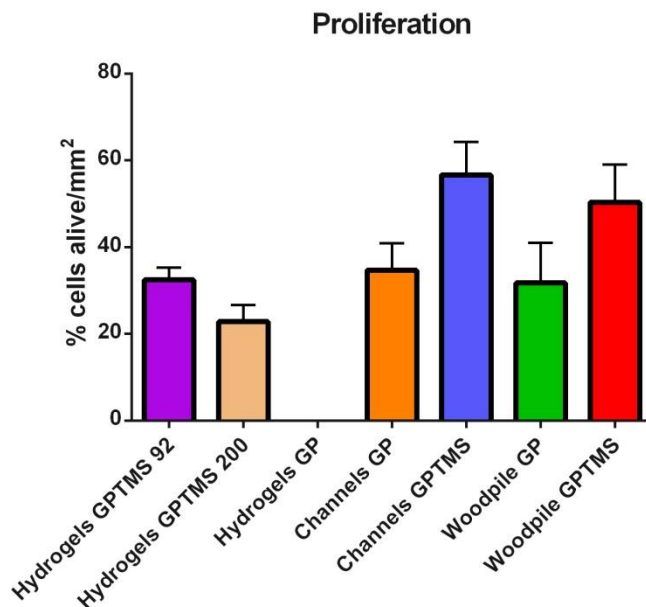


In-vitro assay

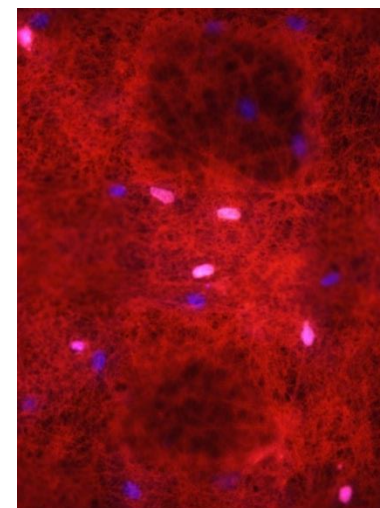


Cells marked with:

- Dapi: Nuclei
- EdU: proliferative Nuclei

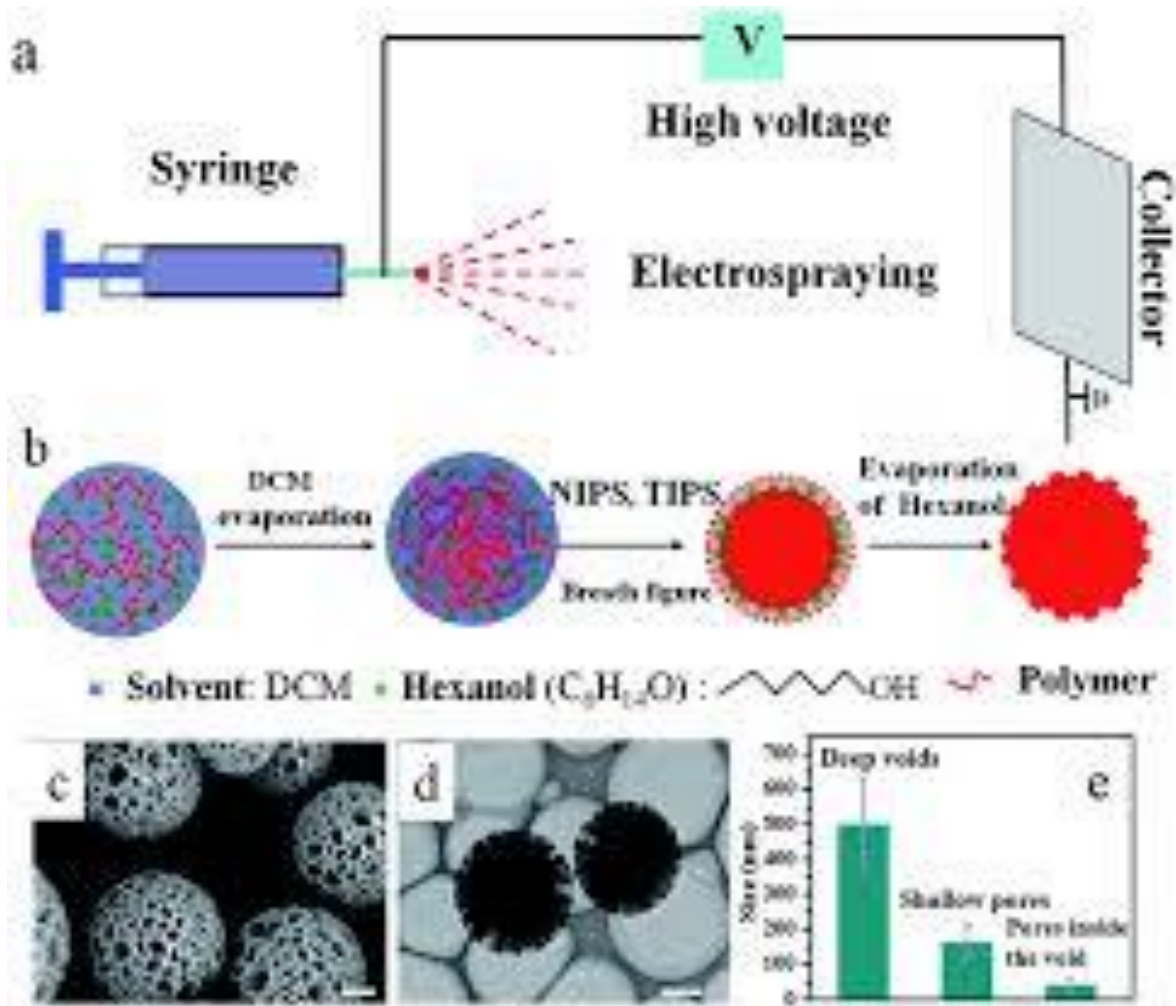


Merged



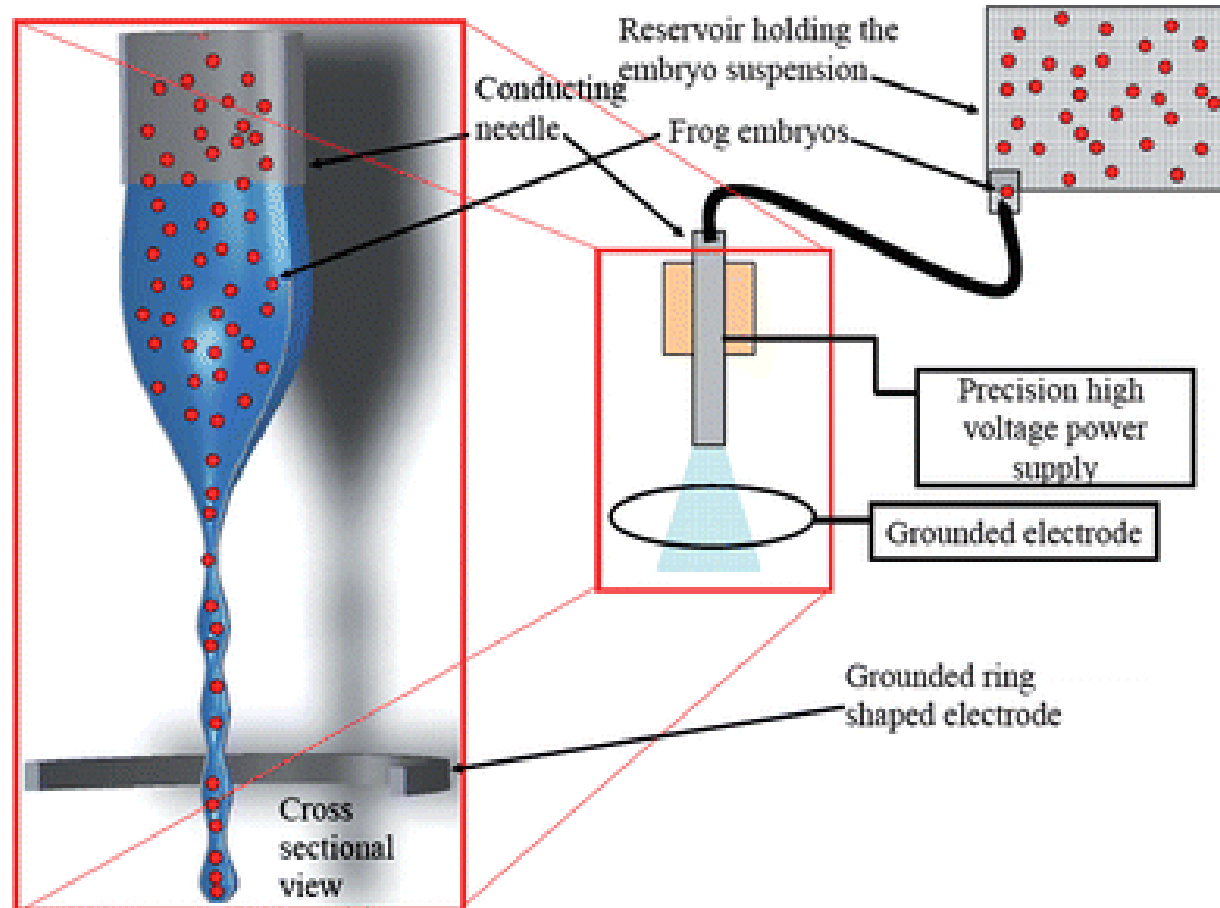
+

Electrospraying

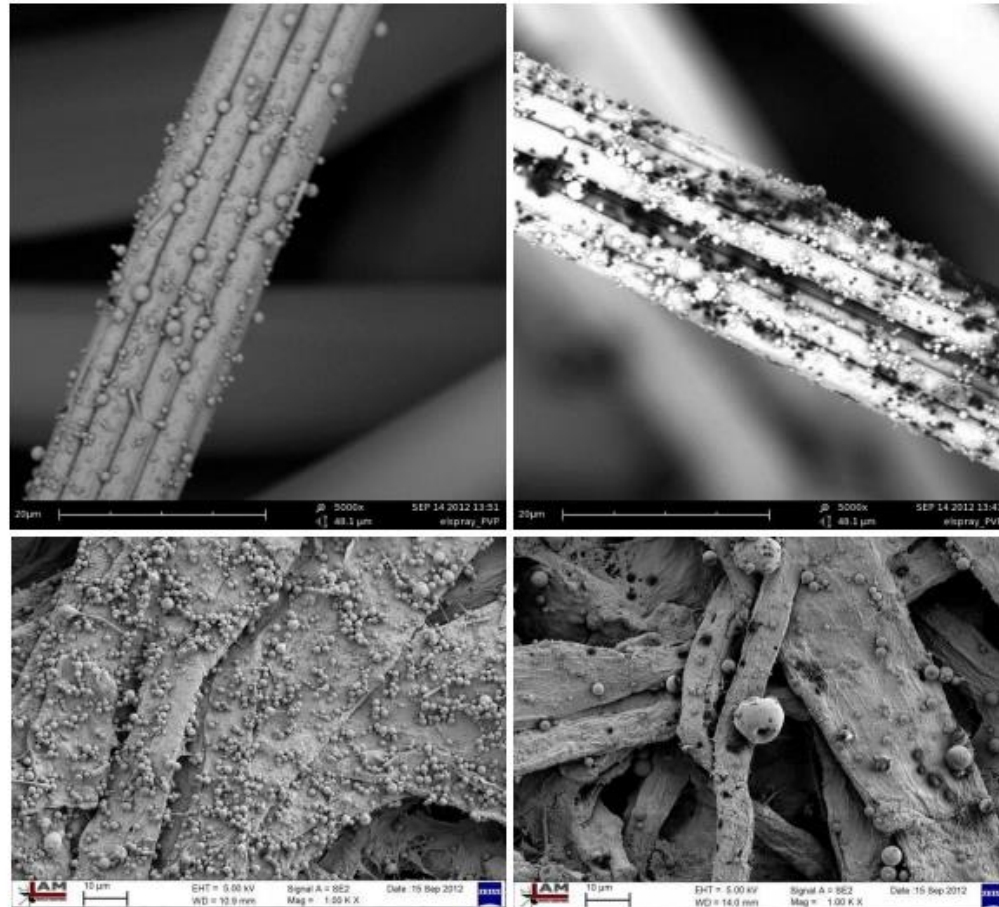




Electrospraying



+ Electrospraying - Results





References



Bhardwaj, Nandana, and Subhas C. Kundu. "Electrospinning: a fascinating fiber fabrication technique." *Biotechnology advances* 28.3 (2010): 325-347.

Teo, Wee E., and Seeram Ramakrishna. "A review on electrospinning design and nanofibre assemblies." *Nanotechnology* 17.14 (2006): R89.

Sun, B., et al. "Advances in three-dimensional nanofibrous macrostructures via electrospinning." *Progress in Polymer Science* 39.5 (2014): 862-890.