

Review Article

Electrospinning: How to Produce Nanofibers Using Most Inexpensive Technique? An Insight into the Real Challenges of Electrospinning Such Nanofibers and Its Application AreasPravin Bhattarai^{*1,2}, Keshar Bdr. Thapa^{1,2}, Rajesh Bdr. Basnet³ and Saurav Sharma³¹National Public Health Laboratory (NPHL), Ministry of Health and Population, Teku, Kathmandu, Nepal²College of Biomedical Engineering and Applied Sciences, Hadigaun, Kathmandu, Nepal³Alliance Health Care Technologies & Services Private Limited, Anamnagar, Kathmandu, Nepal***Correspondence Info:**Pravin Bhattarai,
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E-mail: bhattaraip@nphl.gov.np**Abstract**

This paper highlights about the use of a very simple and inexpensive device for the production of nanofiber and its application in different biomedical areas. There are several literatures that explain the different methods for preparing as spun nanofibers but they are very time consuming and difficult in reproducing results unlike the robust electrospinning device discussed in this paper. Though easier to use, this methodology has still a lot of challenges that should be addressed in the upcoming researches. This paper briefly summarizes few challenges and application areas that require utmost attention to excel in the field of nanotechnology. Despite several application areas where the technique of electrospinning can be deployed in the fabrication of nanofibers, we have mainly emphasized energy related devices in the field of nanoelectronics.

Keywords: electrospinning, filtration, nanofiber, nanoelectronics, nanoscale materials, solar cell

1. Introduction

One aspect of present issues and great importance to the basic understanding of the performance of materials on their optical, chemical and mechanical properties is by the virtue of its size and dimension for real time application in different devices¹. In this regard, study of one-dimensional materials has increased significantly in various research institutes all over the world. Materials or structures having at least one of its dimension 100 nm or less are considered as one-dimensional materials. The novel chemical, physical and biological properties of one-dimensional materials can be attributed to its unique shape and nanofibrous morphology that can be easily deployed in optics, catalysis, data storage devices and biological scaffolds. Inadequacy of such properties in bulk materials where the particle is in the size of micron level further enhances study and use of one-dimensional materials. Currently, the present need marks the possibility of application of such attributes in nano-sized inorganic materials as quantum dots in a host device, e.g. MEMs, sensors, structural component in artificial organs and arteries, reinforced composites, electrodes, photocatalysts. Hence, there is still a lot of opportunities in the development of nanoarchitected ceramics that can be exploited for its use as high performance devices, tissue engineering scaffolds and a lot more.

Electrospinning is most widely chosen tool for synthesizing one-dimensional (1D) nanostructures which include ribbons, fibers, filled and hollow tubes from the fact that it's quite simple, easy to use and relatively inexpensive technique. The unique features of such as-spun nanofibers are one-dimensional morphology, extraordinary length, large surface area and highly porous structures^{2,3}.

2. Timeline of Electrospinning**A. History of Electrospinning**

Electrospinning is an old but yet fascinating technique. In the timeline of electrospinning, Rayleigh in 1897 first observed it, which was later studied by Zeleny in 1914 as electrospraying and was patented by Formhals in 1934. Although the term "electrospinning", derived from "electrostatic spinning", has been mostly used only during 1994 but its existence can be proven back since 1934. Since Formhals' first patent in 1934, he had published a series of patents until 1944 which included description of an experimental set-up for the production of polymer filaments using an electrostatic force⁴.

Table I: Timeline of electrospinning

Period	Inventors
1897	First observed by Rayleigh
1914	Studied in detail by Zeleny
1934	First patented by Antonin Formhals (1934-1944)
1952	Vonnegut and Newbauer invented a simple apparatus for electrical atomization and produced streams of highly electrified uniform roplets of about 0.1 mm in diameter.
1955	Drozin investigated dispersion of series of liquids into aerosols under high electric potential.
1966	Simon patented an apparatus for the production of non-woven fabrics that were ultrathin and very light in weight with different patterns using electrical spinning.
1971	Baumgarten made an apparatus to electrospin acrylic fibres.

The first patent (US Patent Number: 2116942) on electrospinning was filed by Antonin Formhals (1934) for the fabrication of textile yarns and a voltage of 57 kilovolt (kV) was used for electrospinning cellulose acetate using acetone and monomethyl ether of ethylene glycol as

solvent. Formhals spinning process consisted of a movable collector to collect as-spun threads in a stretched condition similar to that of a rotating drum in conventional spinning¹. More than 50 patents have been registered for electrospinning polymer melts and solutions in the past 60 years⁵.

After Formhals's patent, a lot of researchers, Vonnegut and Newbauer, Drozin, Simons, Baumgarten have constantly enriched the potential of electrospinning by their novel works. A brief timeline on invention of electrospinning is shown in Table I. Since 1980s till present, with the advent of nanotechnology and simplicity to fabricate ultrathin fibers or nanomats of different polymers and varying diameters in the submicron or nanometer scale, electrospinning process has gained wide popularity. The increase in number of patents for different applications related to electrospinning and a large number (over 200) of universities and research institutes worldwide that includes the study of various aspects of the electrospinning process gives an insight in the popularity of the electrospinning process in recent years. Some companies such as espin Technologies, Nano Technics, and KATO Tech are constantly benefited by the utmost features derived from electrospinning, while companies such as Donaldson Company and Freudenberg have already applied the outcome of electrospinning process in their air filtration products since past two decades^{4,6,7}.

B. Electrospinning: The Set-up and Processing

i. Preparing Electrospinnable Solution

Ceramic nanofibers can be obtained directly by electrospinning a solution containing inorganic precursor and a solvent but this is rarely followed. Inorganic precursors such as metal alkoxides or metal salts have higher hydrolysis rates and inappropriate rheological properties unlike other precursors⁸. Due to this particular reason such practices leads to an uncontrolled electrospinning process. An easy way to overcome such hindrances is to add a polymer into the solution so that it ultimately helps to remove the rheological property imbalances and a catalyst, as an additive to control rapid hydrolysis of inorganic precursors.

Polymers such as poly (vinyl pyrrolidone) (PVP), poly (vinyl alcohol) (PVA), poly (vinyl acetate) (PVAc), polyacrylonitrile (PAN), poly (methacrylate) (PMMA) are usually dissolved in a relatively volatile solvent such as ethanol, water, isopropanol, chloroform and dimethyl formamide (DMF). Although literature includes wide use of all the above mentioned polymers but PVP, highly soluble in ethanol and water as well as showing good compatibility with many metal alkoxides and metal salts e.g. titanium isopropoxide and zirconium acetylacetonate, is one of the most widely used polymers⁹.

Few occasionally used additives, such as catalysts and salts are generally added in a small amount. Although added in a small amount, they facilitate the electrospinning process by stabilizing the precursors. These catalysts namely acetic acid¹⁰, hydrochloric acid¹¹, and propionic acid¹² facilitate a continuous spinning, avoiding blockage of the spinneret and by balancing both the hydrolysis and gelation rates. A salt, e.g. sodium chloride or tetramethyl ammonium chloride is significant for the bead free electrospun nanofibers as it increases the charge density on the liquid jet¹.

ii. Electrospinning of as Prepared Solution

A simple electrospinning equipment consists of a high-voltage power supply, a syringe pump (can be either 2.5 ml or 5 ml) with a metallic needle and a collector as shown in Figure 1. This high-voltage supply provides direct current (DC) voltage in a range of 0-50 kV. The prepared viscous solution is loaded into a syringe with a metallic needle attached to it. This is usually a programmed device guided by the help of a graphic user interface (GUI). This interface allows users to provide necessary input such as flow-rate, volume of syringe pump that can be varied as per user's requirement. The high voltage applied during the process can be varied manually with the help of a knob attached to the device itself. Now the electrospun nanofibers are deposited at a constant rate on the collector beneath the needle.

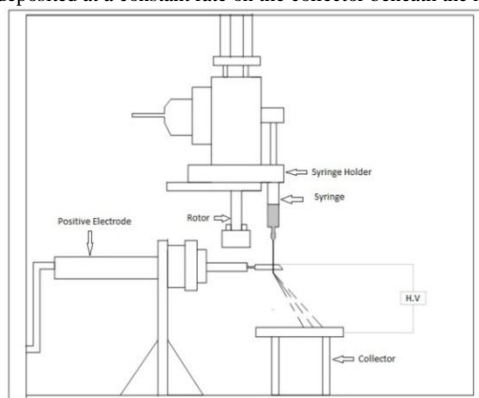


Figure 1: Electrospinning Set-up device

C. Principle of Electrospinning

i. Simple description:

Electrospinning apparatus consists of a high voltage power supply connected to a metallic spinneret so as to increase the electrostatic potential of the fluid. Increase in this electrostatic potential ultimately increases the liquid's surface charge. Surface tension of the liquid at the tip of the spinneret usually determines the normal shape of a volume of that solution but in the presence of external electrostatic potential, the liquid jet gets charged and this charge now repels the pre-existing surface tension bringing change in liquids original shape, now called as Taylor cone¹³.

Since the spinneret and the collector are oppositely charged there occurs a potential difference. As we know that the electric stresses concentrate on the surface of a conductor forming a sharp point, thus in this case there is also a sharp point called as taylor cone. When the concentrate at this point, ejection of a fluid jet due to the greater electrical attraction at the tip occurs. Now, this charged fluid is driven towards the direction of the local electrostatic field. After travelling certain distance, produced jet is susceptible to various instabilities e.g. Rayleigh instability and off-axis bending instability¹⁴. To obtain good quality nanofibers one should always take great care of such instabilities. The effect of such instabilities during fiber forming processes is discussed later in this chapter.

Once the jet is present in air it starts thinning due to various forces acting on it. This thinning of the jet leads to higher surface area due to which the solvents associated with this fluid start evaporating and finally the fluid jet gets converted into a solid fiber. The orientation of this fiber can vary according to the alignment of polymer molecules in presence of electrostatic field and also by the degree of draw ratio of the fiber during its time in the air.

ii. Jet Instabilities:

It plays vital role in the fiber forming processes as it governs breaking of jet causing difficulties in the process of electrospinning. But such effects can be controlled by regulating solution properties such as polymer concentration. Increasing polymer concentration checks breaking of jet due to the stronger inter-molecular interaction¹⁵.

According to Hohman and co-workers there are three types of instabilities¹⁵: 1) Axisymmetric Plateau-Rayleigh instability; 2) Axisymmetric instability caused by having a conductive jet; 3) Bending or 'whipping' instability

D. Parameters of Electrospinning

Here, the different variables involved during the electrospinning process are discussed. Certain variables which when not controlled directly affect the fiber forming ability and failure to deliver good quality nanofibers during electrospinning process. Thus there is a great need to look into these variables so as to produce our desired type nanofibers. Several literatures categorize these parameters into different subheadings e.g. Mit-upatham and co-workers have differentiated as solution parameters, process parameters and environmental conditions¹⁶. Solution parameters include the solution properties which are used as the feedstock while process parameters are directly associated with those related to the design, geometry and operation of the electrospinning apparatus and finally the environmental conditions such as atmospheric conditions and some local variables in which the spinning is performed.

Table II: Parameters of Electrospinning process¹⁷

Solution Properties	Controlled Variables	Ambient Parameters
Viscosity/Concentration	Flow rate	Temperature
Conductivity	Electric field strength	Humidity
Surface tension	Distance between tip and collector	Air velocity
Polymers molecular weight	Needle tip design	
Dipole moment	Collector composition and geometry	
Dielectric constant	NA	NA

Apart from Mit-Uppatham classification of the processing variables, Doshi and Reneker have also categorized it similarly in three but different subheadings namely solution properties, controlled variables, and ambient parameters. The entire variable name that falls under each heading is listed in the Table II.

i. Solution Properties

The solution properties such as viscosity, conductivity, surface tension etc. affect the fiber forming ability as well as alter the morphology of as-spun fibers. Solution viscosity alone is one of the biggest properties found to alter the fiber size and morphology during electrospinning of nanofibers. Reasonable number of studies has shown the presence of defects such as beads and droplets instead of fibers at low polymer concentrations^{18,19}.

ii. Controlled Variables

Few studies have been done to correlate some fundamental relationship between flow rate and fiber size. These studies have revealed that smaller diameter fibers are easier to obtain if the flow rates can be lowered²⁰ or else nanofibers revealed some beaded morphology. This bead formation takes place when fibers don't get enough chance to dry before reaching the collector²¹⁻²⁴.

Among various parameters listed under controlled variables, the effect of field strength or applied voltage is considered as one of the most studied parameter. In order to initiate an electrospinning process, electric field strength in a range of 0.5 and 1.5kVcm⁻¹ is generally prescribed¹⁵. At low voltages, bead free spinning is possible as the jet originates from the Taylor cone itself rather than from the surface of the liquid within the needle tip when voltage exceeds the typical spinning range. These large beaded structures at higher voltages are generated when the Taylor cone recedes at the tip of the needle. Beads formation exceeds according to the increase of the electric field strength^{20,25}. Increased voltages not only produce beaded structures but also lead to the formation of several large diameter jets²⁶.

Distance between tip and collector is also very much responsible in controlling the fiber diameters and morphology. Varying the distance not only affects the electric field strength but also determines the actual flight time to obtain a fiber. Few cases have revealed possibility of obtaining thinner fibers by increasing the distance between tip and collector as it provides sufficient time for the jet to be stretched under the influence of electric field²⁷. But this very concept has also been reported in few other cases to obtain thicker fibers due to reduction of the electric field²⁰. Beading has been observed at distances that are either too close or too far^{18,28}.

Needle tips of various configurations and designs to produce distinguished fiber have been investigated. Li and Xia work reveals the use of a coaxial, two-capillary spinneret to produce hollow nanofibers²⁹. The literature also reveals the use of multiple tips that were specially designed to increase the production rate of electrospinning of poly (ethylene oxide) (PEO)³⁰.

Collectors with various geometries such as parallel plates^{31,32}, frame collectors⁴, conductive rings³³ and rotating cylindrical drum collector^{34,35} have been designed and used. This varying geometry helps in controlling the deposition patterns or the extent of the bending instability¹³ that enables the collection of fibers in different forms such as thin strips or yarns³⁶.

iii. Ambient Parameters

Few studies have revealed the effects of ambient parameters in the spinning process. They found an inverse relationship between the temperature and fiber diameter. This decreased fiber diameter with the increase in temperature has been attributed to the decrease in the viscosity of the polymer solution³⁷. Casper *et al* work revealed some small circular pores on the surface of the fibers due to increase in the humidity. Later, these small pores coalescence due to further increase in the humidity³⁸. Spinning performed under vacuum condition yielded fibers and yarns with larger diameters due to higher electric fields³⁷.

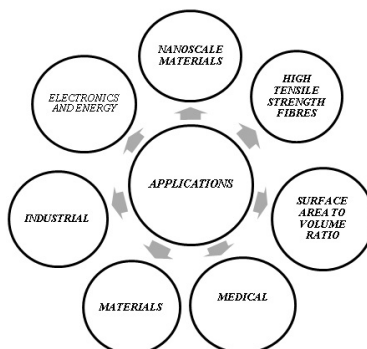


Figure 2: Application areas of Electrospun nanofiber

3. Applications of Electrospun nanofibers

Increasing number of patents and journals in the field of electrospinning everyday reveals its significance for real world applications.³⁹ The growing interest in electrospinning field can be realized in three different aspects:

a) Fabrication of various types of fibers in the nanoscale range or having a nanoscale surface texture provides substrate for various interaction modes with other materials as compared to macroscopic materials.

b) As-spun nanofibers undergo higher stretching before reaching the collector. This higher drawing ratio is expected to produce fibers having a highly oriented molecular structure with fewer rooms for defects, thus achieving its expected maximum strength. A simple relationship, surface area to volume ratio is inversely proportional to average fiber radius (surface area to volume ratio = $2/r$), illustrates an inherently high surface area to volume ratio for electrospun fibers having diameter in nanoscale. These very fascinating and unique properties of electrospun fibers give rise to applications in filtration, cellular matrices, catalyst substrates, ultra-strong composites, bioreactors, functional textiles, drug encapsulation, wound dressings and stent manufacture¹³. Kotha *et al* focuses in the improvement of tensile and compressive properties as well as in fracture toughness by the use of uncoated or acrylic coated 30 μm fibers for its use as reinforcements in bone cements⁴⁰. Zirconia fibers, aesthetically viable as well as having satisfactory regulatory concerns further enhances its application in this field. Jing *et al* as produced ultrathin nanofiber is believed to have great applicability as a fiber template or coaxial electrospinning to create ultrathin tubular structures⁴¹. Azad⁴² has also mentioned about the importance of 8YSZ in nanofibrillar structure as a key ceramic component of relevance to fuel cells. Li *et al* has prepared hollow 8 YSZ fibers that has important application in catalytic combustion⁴³. It is shown in Figure 2, the possible areas where electrospun nanofibers can have potential application in the present state.

In context to energy harvest and storage materials, as-spun nanofibers can be classified as:

i. Cathode material for Li-ion batteries:

According to the report of Dimesso *et al* the cathode material of Li-ion batteries can be successfully prepared using the 3-D structures of LiFePO_4 coated nanofiber nonwovens⁴⁴. Such uniformly distributed LiFePO_4 crystallites over the surface area of carbon nonwoven fibers exhibited good performances delivering a discharge capacity of 156 & 152 mAh/g at discharge rates of c/25 & c/10 at room temperature, respectively. This finding correlates to the 92% of the theoretical value of the former and 89.5% of the later discharge rates. Thus, such electrospun cathode materials can undoubtedly correspond to the material of a high electronic conductivity and a high “free” surface area of the carbon nanofibers.

ii. Dye Sensitized Solar Cells (DSSCs):

TiO_2 has been a material of choice and most widely used in the field of energy and solar harvesting. This TiO_2 when electrospun into vertical nanofibers have been reported to behave as a photoelectrode⁴⁵⁻⁴⁷. The first step to prepare such vertical nanofibers of TiO_2 is to electrospin, followed by post-treatment of nanofibrous TiO_2 ribbons and cutting of such vertically aligned nanofibers so as to give a 3D structure. These as-prepared vertical nanofibers can have a height of around 27 μm but can be easily raised from 10 to 100 μm with an average area of 0.2 cm^2 and 90 ± 30 nm diameter. The conversion efficiency, short circuit current, and open circuit voltage of such TiO_2 nanofibers were measured as 2.87% and 5.71 mA/cm^2 , 0.782 V, respectively. Optimizing the varying parameters of the nanofibers e.g. porosity, diameter, or height can likely enhance the photo-conversion efficiency of such TiO_2 nanofibers.

iii. Filtration:

Electrospun nanofiber based filters have been proven to have very higher filtration efficiency at relatively small decrease in permeability long time back. E.g., Gibson *et al*⁴⁸ has already revealed the extreme efficiency of electrospun nanofibers mats in trapping airborne particles of size varying from 0.5 to 200 μm . Similarly, Barhate and Ramakrishna *et al* have mentioned that a nylon-6 electrospun nanofiber mat (100 μm thick, 0.24 μm average pore size) have been proven to have a slightly higher filtration efficiency (99.993 %) than a commercially existing type, high efficiency particle air (HEPA) filter (99.97%, thickness 500 μm , average pore size 1.7 μm)⁴⁹. In the context of evolution, it has been recently discovered that the filtration by a single thick layer nanofiber mat can be obscured by the filters made up of multiple thin nanofiber layers in terms of the pressure drop during filtration. Moreover, 3D polyurethane (PU) nanofiber via multilayered electrospinning has also been proposed in terms of better filtration efficiency⁵⁰.

The filtration mechanism of such filters are different in context to the size of the air borne particles, for e.g. particles of larger diameters (> 400 nm), are simply blocked by the filter surface, while the particles of diameters smaller than the surface pores are collected by either interception, impaction, static electrical attraction or Brownian motion effect (for < 20 nm).

4. Challenges

1) Production of nanofibers in a consistent fashion; 2) Easily reproducible results; 3) Control mechanism over the diameter of fibers and their size distribution; 4) Ability to reproduce the nanofibers in specific positions followed by the particular orientation at industrial level; 5) Production in large scale; 6) Should have a deep comprehension of electrostatic and fluid dynamics; 7) Very narrow range of acceptable solvents, often most proven as toxic

Apart from the physical challenges to be met, there are still a lot other things to control at the time of producing such nanofibers. E. g., variables that have been discussed earlier like solution conductivity, spinneret design, and electric field intensity have a very wide control over the morphology, diameter, and orientation of such as obtained nanofibers. So, to control such variables during the entire run of nanofiber production remains as a great challenge in the field of nanotechnology.

5. Conclusion

Finally, we can conclude that despite a lot of challenges existing in the field of electrospinning, it's necessary to optimize the processing variables so as to obtain highly oriented nanofibers reproducibly. Besides, the major application area of energy harvesting can be expanded in the terms of more efficient self-renewable energy based nanoelectronics system.

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