

Needleless Electrospinning Technology – An Entrepreneurial Perspective

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Abstract

Background/Objectives: Needleless electrospinning technology surmounts the shortcomings and bottlenecks of the conventional needle based electrospinning systems. Spinneret clogging and lower throughput in terms of productivity has been the setbacks of the conventional technology leading to fewer nanofibers based commercial products. Nanofiber based products finds a market in healthcare, energy and defence. Proof of concept leading to lab level prototype scaling up into industrial level of nanofiber production units is the focus of this paper. **Methodology/Analysis:** A simple search in the scopus web portal with a keyword 'electrospinning' resulted in 16,272 publications as of date. Same search for patent in US patent office portal resulted in 1939 granted patent documents. A comparison of years of technology time cycle (110 years), publications (16,272) and granted patents (1939) with the companies (31) working on electrospun products was made. **Findings:** Bubble, hollow tube, roller, wire based and slit-surface needleless electrospinning units are discussed on the basis of entrepreneurial product cycle of prototype development translating into commercial units. Nanofiber production at a mean diameter of 140 nm at commercial level using Poly (ethylene) oxide for Nano Drug Delivery Systems (NDDS) by wire based free surface electrospinning has been analysed. **Novelty/Improvements:** The analysis proves an entrepreneurial approach in the area of electrospinning as evidenced by number of patents match ups or exceeds publications in some region and also depicts its futuristic demand in several areas including healthcare and energy sectors.

Keywords: Electrospinning, Nanofibre, Nano Drug Delivery, Patent

1. Introduction

A patent¹ in early 21st century and a concept paper² during the same time period initiated the conventional electrospinning technology. Conventional electrospin-

ning involved needle spinneret for nanofiber production. In early 80s electrospinning process got revived³ over decades of time span of the granted patent. Several companies showed interest to manufacture nanofiber based products⁴. Number of publications and patents showed

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an exponential increase but not corresponding increase in nanofiber based products in the commercial market. A simple search in the scopus web portal with a key word search of electrospinning resulted in 16,272 publications as of date. Same search for patent in US patent office portal resulted in 1939 granted patent documents. Comparison of years of technology time cycle (110 years), publications (16,272) and granted patents (1939) with the companies (31) working on electrospun products a mismatch of technology maturity and product development is very much exhibited. Major setback for the conventional needle based electrospinning is its low throughput. A single needle based electrospinning process yielded productivity of only 0.005 g–0.01 g of fiber per hour per needle⁵. Inadequate astuteness of technology innovation and development translated into more company incorporations resulting in numerous electrospun nanofiber product commercialisations is inadequate. Needleless electrospinning was invented⁶ basically to overcome the shortcomings of conventional needle based electrospinning technology. From thereupon number of publications and patents is very much on the raise. Several start-up companies have shown interest for commercialisation of nanofibers based products from needleless electrospinning nanofiber products. Companies manufacturing conventional needle based electrospinning have diversified to manufacture needleless electrospinning commercial units. Analytics on the number of publications from SCOPUSweb portal and

the patents from United States patent office search with the term needleless electrospinning is given in Figure 1 and 2. Different types of spinnerets were tried as the proof of concept laboratory design models. All these spinnerets to produce electrospun nanofibers were of motorized moving category where clogging is minimized with very high production rates. In many instances for example using rotating disk type of spinneret, productivity showed 60 fold increase with respect to the conventional needle based electrospinning process⁷. Figure 3 shows different types of spinnerets with least resistance for fluid flow having a free flowing mechanism for the electrospin solution. Clogging of the needles a very important drawback in the conventional electrospinning mechanism which is minimized in this free surface needleless electrospinning technique.

2. Bubble Electrospinning

Liquid jets from soap bubble Technique to launch liquid jets from the apex of a soap bubble paved way resulting in concept and the development of bubble electrospinning. Bubble radius and surface tension of the liquid to be used depended on the applied voltage for the liquid jet formation. It was found radius of the soap bubble increased by increase of the applied voltage needed to launch the liquid jet.

Bubble electrospinning basically works on the launch of electrospun fiber jets from gas bubbles which are

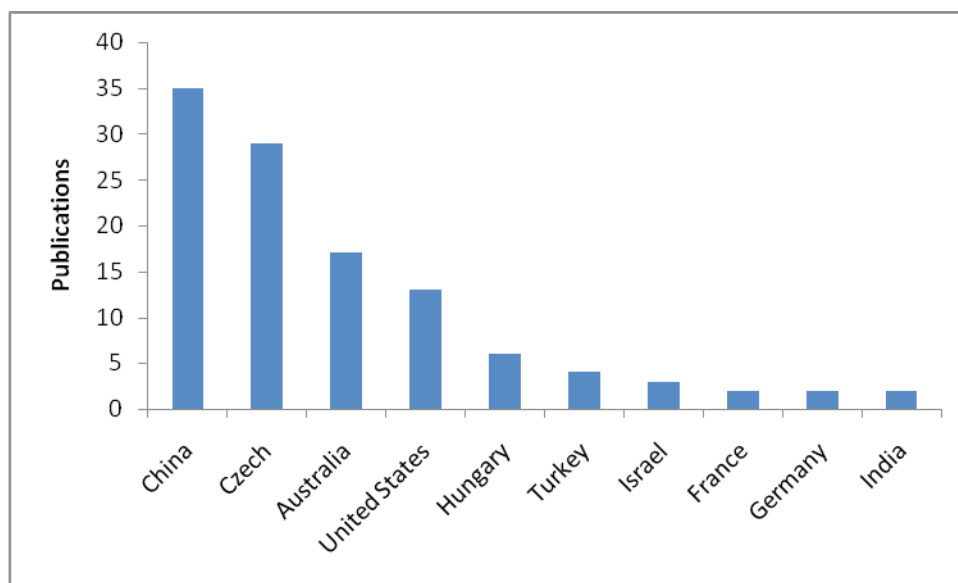


Figure 1. Country wise publications on needleless electrospinning.

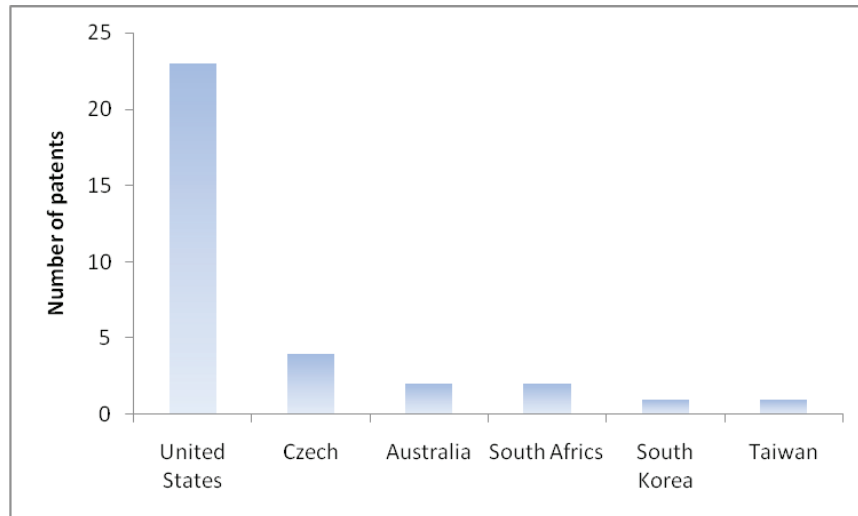


Figure 2. Country wise patents on needleless electrospinning.

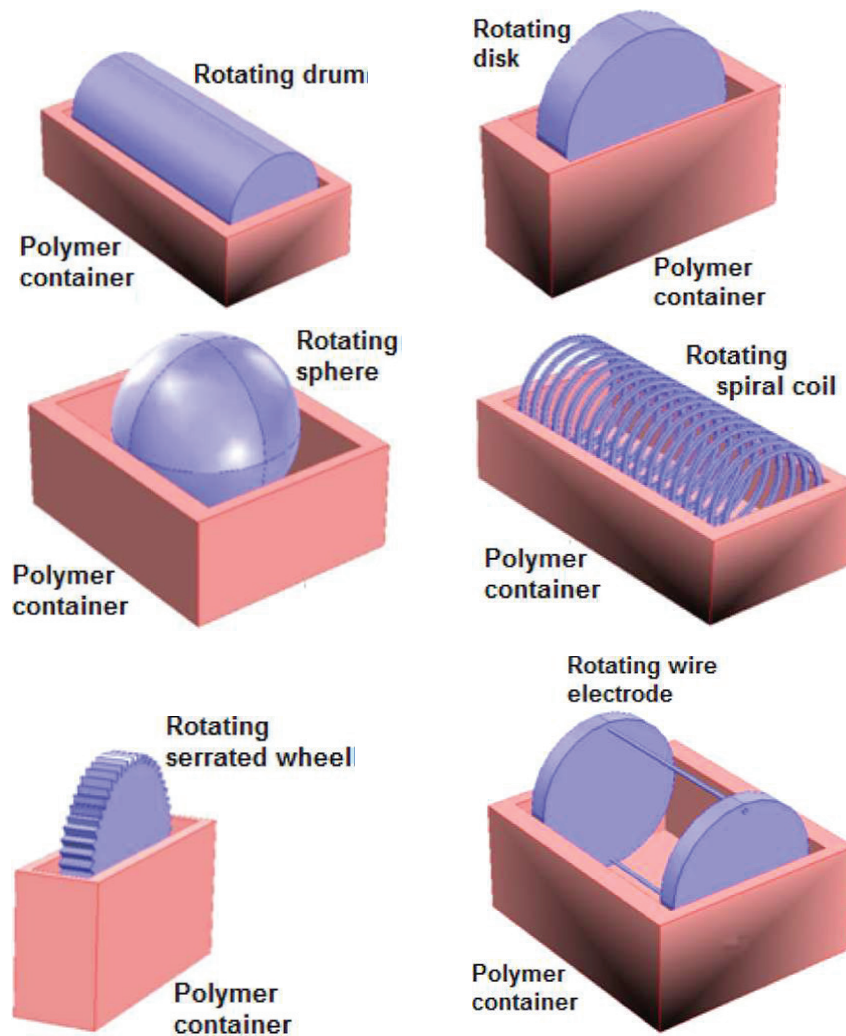


Figure 3. Different types of rotating spinnerets⁽⁷⁾.

produced on the surface of the electrospin polymeric solution. The inventive technology was a simple petri plate set-up shown in Figure 4. The petri plate holds the polymeric solution as the reservoir. The electrostatic force is applied by using a power supply connected copper wire electrode with the other end attached to the rim of the polymeric solution reservoir. The produced nanofibers were collected on a stainless steel wire mesh acted as the grounded collector surface. Like the conventional needle based electrospinning, a specific distance between the petri plate and the stainless steel wire mesh, was maintained for nanofibers collection. Bubble formation on the surface of the polymeric solution which was a flat open surface was initiated by blowing an inert gas into the polymer solution, ending up in bubble formation shown in Figure 5. Metallic needle tip connected to an empty syringe served as an air generator, by slowly pushing the empty syringe, air present inside the syringe formed bubbles which rose to the surface and developed into nanofiber jets upon in contact with electrostatic force developed into jets for electrospun fiber formation⁸.

In terms of the applied voltage, a voltage potential was applied between wire electrode fixed in the petri dish and the wire mesh acting as the collector, polymer solution becomes positively charged with respect to the collector. Adjustments and fine tuning were done to the applied potential difference till a liquid jet formation was launched from the apex tip of the bubble in the surface of the polymeric solution. Characterisation and measurement of the

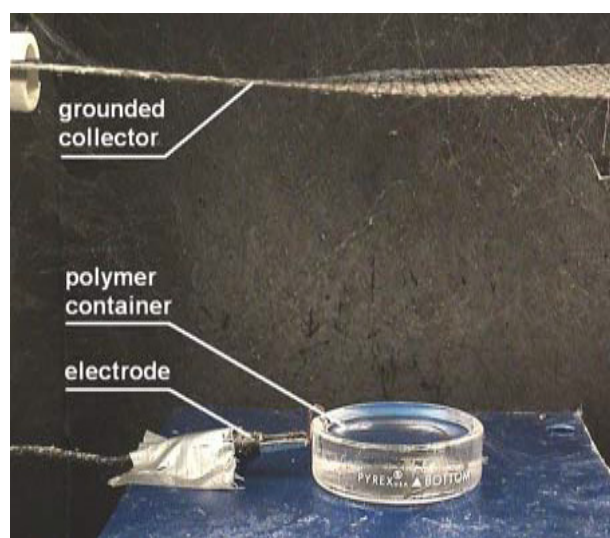


Figure 4. Petri dish as a fiber generator in bubble electrospinning⁽⁸⁾.

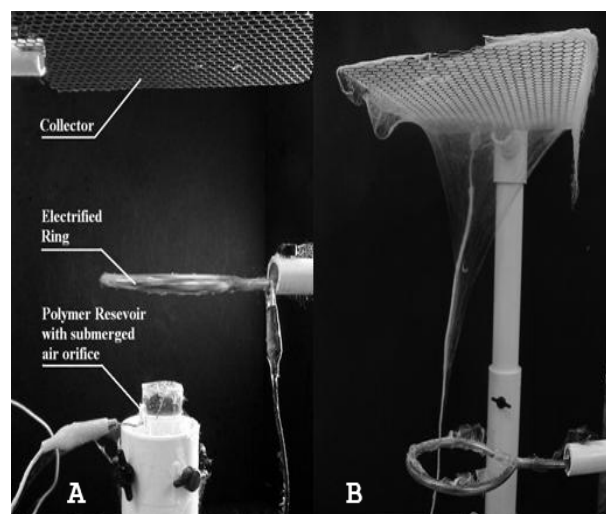


Figure 5. Proof of concept apparatus for bubble electrospinning⁽⁹⁾.

intrinsic properties of the polymer solution were measured by using Brookfield viscometer and a technique called drop weight for viscosity and surface tension of the polymer solution. Morphological characterisation of the bubbles was imaged with a high speed frame rate camera.

Based on the in-house technology development for bubble electrospinning using petri dish, a prototype was fabricated⁹. In this equipment, an electrified ring served dual purpose to direct the electrospun fibers towards the collector as well to increase the distance between the bubble and the collector wire mesh. Measurement of the diameter of the electrospun fibers had diameters in the range of 1.0–5.0 microns. The mass production rate was in the range of 8.0–10.0 g/hr per jet. Lower diameter of the produced fibers was achieved by optimization of the formulation of the polymer solution which resulted in the success of a granting of a patent¹⁰ in bubble electrospinning.

A patent¹¹ claimed the invention of bubble electrospinning in 2007. Upon comparison with the conventional needle based electrospinning, this type predominantly depends on the intrinsic property of the polymer solution to be used for the manufacture of nanofibers. Size geometry of the produced bubbles determines the nanofiber morphology. The induced electrostatic force overcomes the intrinsic property of the polymer solution namely, surface tension to form nanofibers. The main drawback of this setup was the difficulty in controlling the trail of bubbles on the surface of the polymer solution¹².

3. Hollow Tube

Multiple hanging drops were used to produce multiple jets by inducing an applied electrical charge on the polymer solution hanging drops. Hollow perforated plastic tube shown in Figure 6 was used as the polymer solution reservoir resulting in hollow tube needleless electrospinning. The array of orifices on the hollow tube became the source of jets resulting in electrospun fiber formation¹³.

Intrinsic and extrinsic parameters like the conventional needle based electrospinning, polymer solution concentration, applied voltage, distance from the tube to the grounded collector, one another parameter for the hollow tube electrospinning, hole spacing being the added parameter determines the fiber diameter and production rates of the produced fiber. The mass production rate of nanofibres using porous tube was several fold higher in comparison with the needle based electrospinning technique.

A porous hollow tube made of Polytetrafluoroethylene (PTFE) was used as a prototype set-up shown in Figure 7 for creating the electrospinning jets to be used for the production of nanofibers. This porous hollow tube works as the polymer solution reservoir and the polymer solution is ejected outside the tube by the external air pressure which is applied from the top of the polymer solution

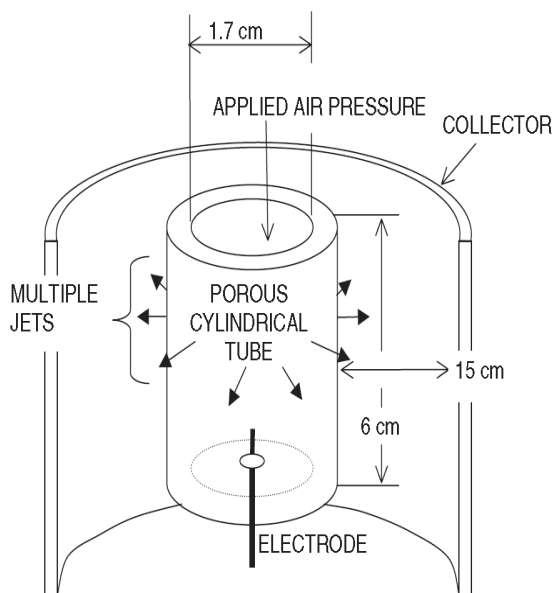


Figure 6. Flow diagram of hollow cylinder based needleless electrospinning technique⁽¹³⁾.

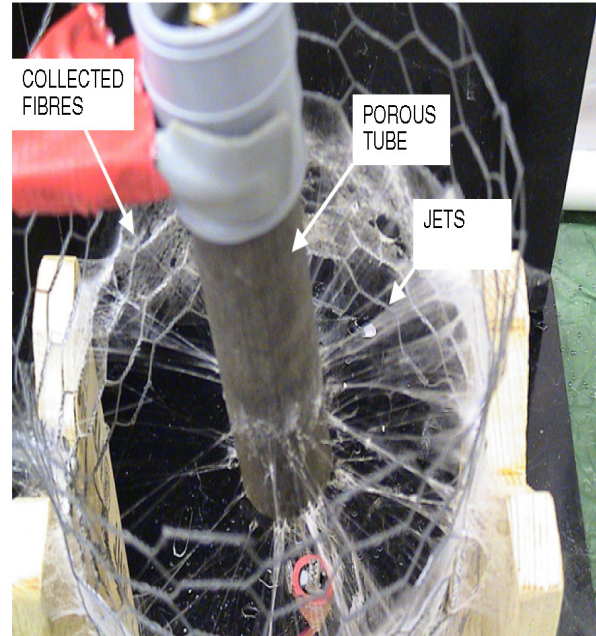


Figure 7. Proof of concept apparatus for hollow tube electrospinning⁽¹³⁾.

reservoir. Applied air pressure was less than 10 kPa. The average pore size was in the range of 20-40 μm . This range of pressurized air had sufficient force to push the polymer solution without forming films on the surface of the hollow tube porous surface. As the geometry decides, more number of holes, more number of jets. Few hundreds of jets get ejected at a single time from the hollow tube porous surface¹⁴. Each hole represents and thereby produces a single jet which finally forms one long continuous fiber. Taylor cone formation of the jets are produced as the jets reach a distance of a few centimetres from the cones, the electrical bending instability becomes dominant and the jet forms an expanding coil to form nanofibers.

Pores in the hollow tube porous surface wall essentially to provide as a conduit for the flow of the polymer solution whereby a sufficient flow resistance is provided so that that the polymer solution does not gets flooded out or flows at a very high flow rate and wet the outer tube surface thereby forming a liquid film. By application of this sufficient force, the polymeric solution flowed at an adequate rate through the porous wall and formed small drops on the surface. Charging of the sample polymeric solution was carried out using a wire electrode. At the sealed end this wire electrode providing a variable high voltage power supply was fixed. This wire electrode had

the capacity to charge the polymer solution to an applied voltage was in the range of 20-60 kV.

Adjustments and optimization of the process parameters resulted in much smaller fiber diameter, one of the main parameters being gap distance between the tube and collector surface. Morphological characterization of the produced nanofibers was determined using Image J software and was found to be in the range of 300-600 nm. By conventional needle based electrospinning, rate of nanofibers produced of about 0.02 gh^{-1} whereas using a 2 cm section of polymer filled porous tube produced nanofibers at a rate greater than 5 gh^{-1} which is about 250 times more than the rate of the conventional needle based electrospinning technique¹⁴. The used porous tube has an easier construction, operational and control facility facilitating scaling up and potential commercialization proportions.

4. Roller Electrospinning

A rotating roller attached to a high applied voltage supply produces nanofibers. Cylindrical geometry facilitates free flow and circumvents fluid flow resistance, hence roller was the choice. Rolling unit served dual purpose, as a polymer solution reservoir as well as a nanofiber generator. The rolling unit was partially dipped and covered by polymer solution filled rectangular tank¹⁵. The applied high voltage generator unit was connected to the roller. The collector was positioned on the top and perpendicular to the rotation of the roller. The collector was a stationary unit which was grounded to create a potential difference. A nonwoven substrate material moves along the grounded collector electrode, thereby facilitating the production of the electrospun nanofibers as a

continuous free streamlined process shown in Figure 8. As the applied voltage reaches and exceeds a critical field strength value, as the process is electrospinning and no difference from the conventional needle based electrospinning process in terms of concept, Taylor cones were formed which were ejected into polymer solution jets transforming into nanofibers travelled towards the collector electrode, getting collected in the collector present above the roller¹⁶. Unlike the conventional needle based electrospinning technique, simultaneous formation of numerous Taylor cones was observed from the roller surface which acts as a rotating spinning electrode. Polymer solution viscosity defines the Taylor cone formation and more Taylor cones were formed. Higher viscosity resulted in ejection of stronger jet and the jets travelled a longer

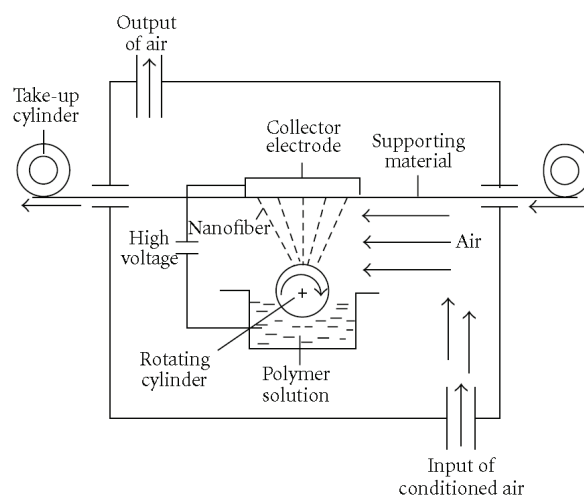


Figure 8a. Flow diagram of roller based needleless electrospinning technique⁽¹⁵⁾.

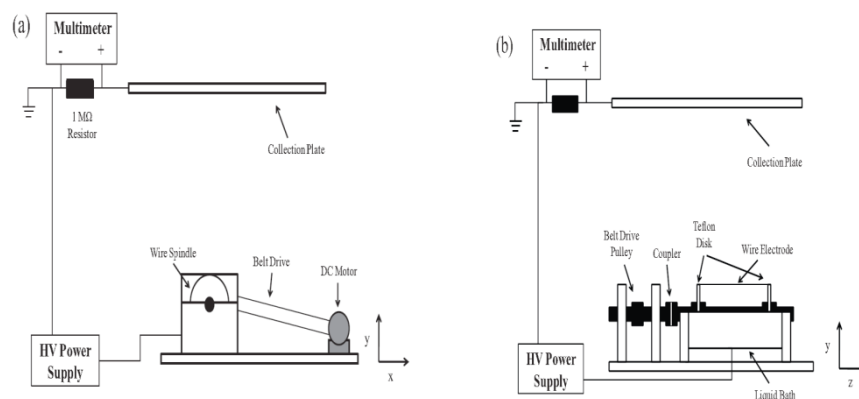


Figure 8b. Flow diagram (a) side view (b) front view of free surface wire based needleless electrospinning technique⁽¹⁹⁾.

pathway ending up in more Taylor cones per area unit of spinning surface. Spinning performance is elevated by the formation of numerous Taylor cones. Determination of Number of Taylor Cones per square metre of the spinning surface (NTC/m²), spinning performance for one Taylor Cone (SP/TC) and Total Spinning Performance (SP) was accurate with easily available measurement techniques.

Independent and dependent process parameters were discussed. These parameters were not of much different from the conventional needle based technique. In terms of the polymer properties, chemical moiety and concentration played a critical role. Solvent played a role in terms of the intrinsic property of the solution being, electrical conductivity, surface tension, dielectric constant and viscoelastic components. These parameters are very much qualitatively measurable with precision and accuracy thereby scaling up is very much easy. The equipment process parameters being applied high voltage and jet travel distance between the electrodes are adjustable and controllable with high veracity. Roller speed played a critical role in terms of nanofiber properties. In variation with conventional needle based electrospinning technique, in this roller based electrospinning technique, new quantitative measurement parameters were evaluated, Taylor cone density per surface area, spinning performance for one Taylor cone, total spinning performance, Fiber Diameter Uniformity Coefficient (FDUC) and Non Fibrous Area coefficient (NFA)¹⁷.

Bundled formation of nanofibers were observed using roller electrospinning technique, bundle formation increases the throughput of the electrospinning process, hence making this innovative technology very productive leading to commercialization¹⁸.

5. Wire Electrode

In this set-up innovated by Massachusetts Institute of Technology, free surface electrospinning is worked upon for high throughput production of electrospun nanofibers. Entrainment of drops on a high voltage charged metallic wire emits the polymeric jet leading to nanofiber production which is collected over a nonwoven substrate. An electrified liquid bath containing the solution to be electrospun to produce nanofibers, moves perpendicular to the highly charged wire electrode. Through an orifice, the polymer solution drips to the wire electrode forming droplets, these highly charged entrained droplets forms a Taylor-like cone emitting a series of numerous jets. More in-depth analysis revealed the electrospin solution

upon dripping through the orifice of the solution bath, resulted in a liquid coating on the wire electrode. These liquid coatings were unstable forming an entrainment of independent individual well separated droplets on the metal wire. Plateau-Rayleigh instability was associated with de-wetting of the film formation of the coated liquid. This prototype involves several wires attached to a rotating spindle. In this prototype, immersion principle was used. The rotating spindle was partially immersed in a fluid bath involving the process of immersion. The applied high voltage led to entrainment, de-wetting, jet formation ending up in nanofibers formation collected over the collecting substrate^{19,20}.

The actual experimental prototype was fabricated using a wire electrode spindle consisting of Teflon disks, stainless steel wires, high voltage power supply translated into a commercial unit. Upon several modifications, a commercial unit working on free surface wire electrode electrospinning unit was developed by Elmarco Ltd. shown in Figure 9. In the variant model, several wire electrodes are used with the movement of fluid bath resulting in a very high throughput of nanofiber productivity²¹. Polyethylene oxide was electrospun using free surface wire electrode electrospinning to be used as nanocarriers for Nano Drug Delivery Systems (NDDS). NS Lab™ was purchased from Elamrco limited, Czech for the manufacture of NDDS. Ultrapure water system was used for electrospinning. Different concentrations were tested for bead-string removal and to achieve high nanofiber density. Intrinsic and extrinsic parameters were optimized to achieve high throughput. As applied voltage was increased, nanofiber throughput increased within a specific time period. Increasing applied voltage will have a detrimental effect on the embedded therapeutic protein or even the pharmaceutical small molecule; hence a compromise was attained between throughput and pharmaceutical efficacy. Figure 10 shows the Field Emission Scanning Electron Microscopy imaging of Poly(ethylene) oxide having molecular weight of 600,000 Da using water system. Applied voltage was kept at 35 kV. Mean nanofiber diameter of 140 nm was achieved. Nanofiber diameter was measure using ImageJ software. Use of ionic salts and ionic solvent system is hypothesized to increase the conductivity of the electrospin solution resulting in high throughput and increased fiber density per square area. Currently technology development is currently on-going for an efficient nanocarrier system with increased drug load per square area.

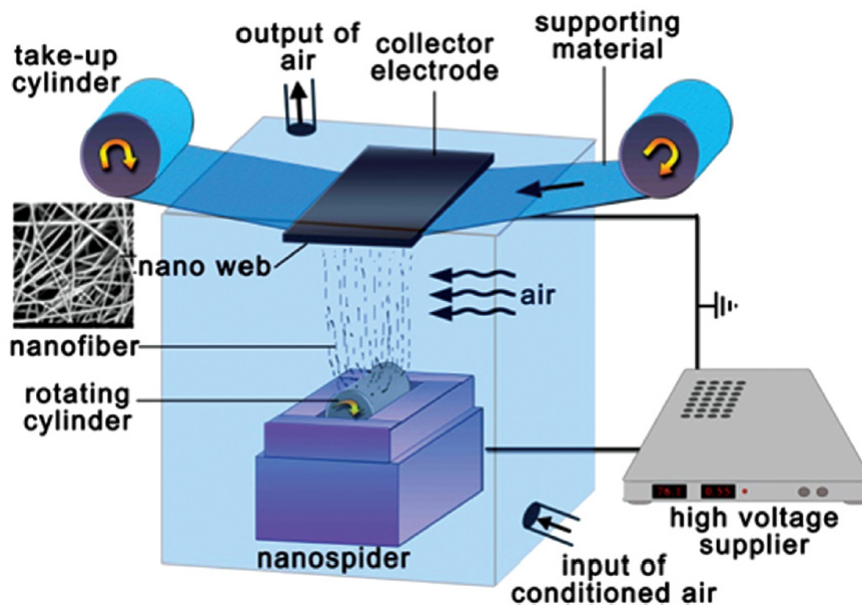


Figure 9a. Commercial unit for roller based needleless electrospinning⁽¹⁶⁾.

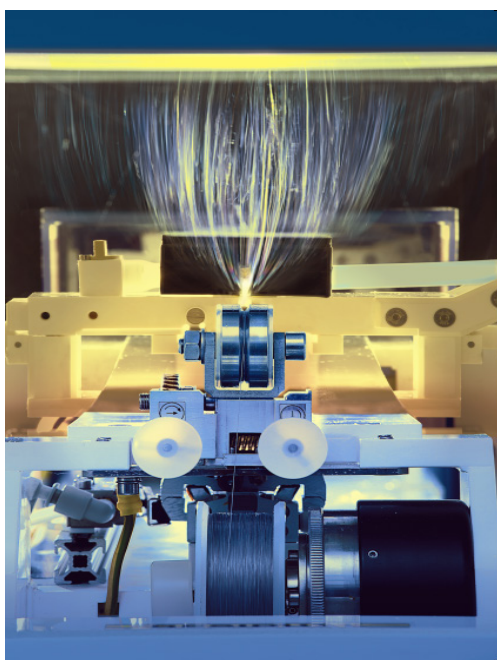


Figure 9b. Commercial unit for free surface wire based needleless electrospinning⁽²¹⁾.

6. Slit-Surface Electrospinning

Slit-surface electrospinning pioneered core-shell nanofiber production. In this cutting edge technology two different polymers were used for the production of core-

shell nanofiber morphology. This system is one of the best for NDDS. Slit-surface electrospinning co-localized two different electrospinning solutions through a slit surface ejecting multiple core-sheath cone-jets^{22,23}. The two different electrospin solutions namely core and sheath were delivered to the slits through their corresponding and respective nozzles ascertaining the co-localization of both the core and sheath materials resulting in core-sheath fiber formation as they eject into the upper free space. The emitted jets were at very rate throughput of a maximum of 1 L/h. Unlike the conventional needle based electrospinning system, slit-surface electrospinning system needs applied high voltage in the range of 70-90 kV, whereas the conventional system works in the range of 10-30 kV. As critical electric applied field strength has attained depending and based on the electrospin's intrinsic properties, through slit-surface pathway resulting in Taylor-like cone jets ejection.

Fluid dynamics plays a very critical role in the success of the slit-surface electrospinning process product delivery. It was hypothesized that viscous shear forces generated from flow of the electrospun sheath solution entrains the core solution, at the interface, to form a core-sheath cone-jet. Optimization of viscous shear force was controlled by adjusting the intrinsic and extrinsic variables of the electrospin solution and process parameters. Critical parameters were intrinsic property of the

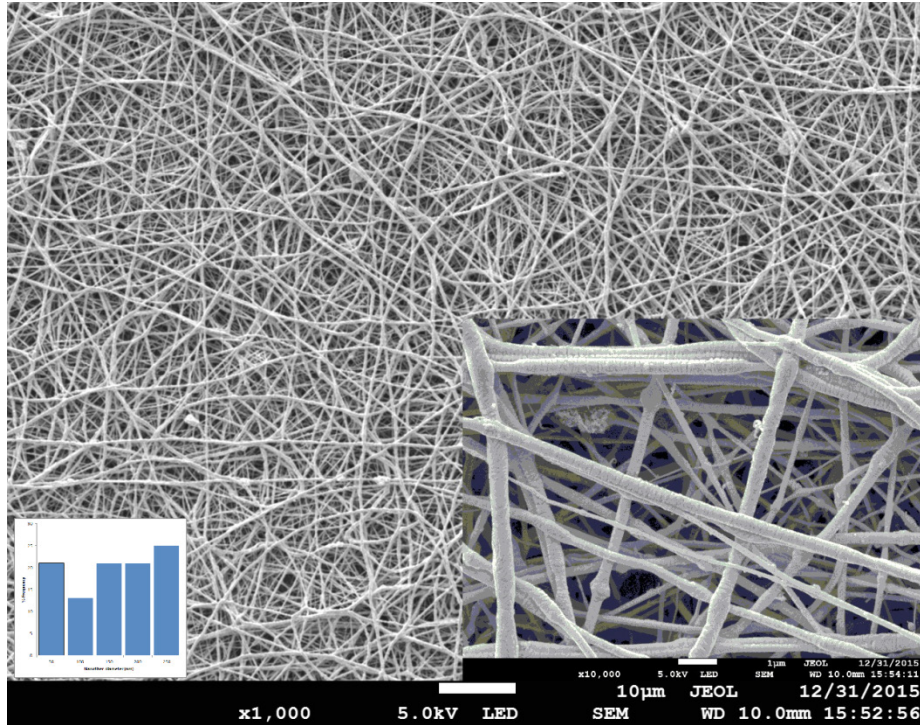


Figure 10. Field Emission Scanning Electron Microscopy imaging of Polyethylene oxide nanofibers produced by free surface wire electrode electrospinning.

electrospin solution, namely concentration related with flow rate and viscosity. Extrinsic parameter the nozzle geometry also played a critical role for the maximum proportion of the core-sheath structure formation. Greater success was achieved even at very high flow rates of the electrospin solution. The core-sheath nanofibers were collected onto a collector which was grounded to form an electric field between the charged slit fixture and collector leading to dissipate charges carried by the collected fibers.

1,000 fold increase and enhanced manufacturing throughput was achieved in the production of core-sheath nanofiber formation in comparison with the conventional needle based electrospinning system. Figure 11 shows the prototype apparatus of the slit-surface electrospinning system. As it shows even the embedded drug particle dexamethasone was imaged in the produced core-shell nanofiber. Two patents were granted for slit-surface electrospinning technology^{24,25}.

7. Perspective

Publications and patent analytics prove an entrepreneurial approach as number of patents match ups or exceeds publications in United States of America. Within a spe-

cific timeline, proof of concept design scaling up to prototype model translating into a commercial unit for sales depicts the technology maturity, corporate videos of the commercial units are available in the social media²⁶⁻³⁰. First publication on needleless electrospinning was published in 2004, within a decade a start-up company has come up with products in the pipeline using needleless electrospinning technology for healthcare applications. AxioCore™ fiber technology involves core-sheath nanofibers produced by slit-surface electrospinning promises to be a cutting edge technology for healthcare applications³¹. Nanofiber based products with more diversified applications would really prove this cutting edge technology as a commercial success. Scaling up of the apparatus to commercial level units is not that difficult and hence attaining very high production rates for nanofiber based products is highly attainable for even diversified applications.

8. Competing Interest

One of the authors, Balu Ranganathan discloses financial interest in Palms Connect Sdn Bhd, holds stock options in the company.

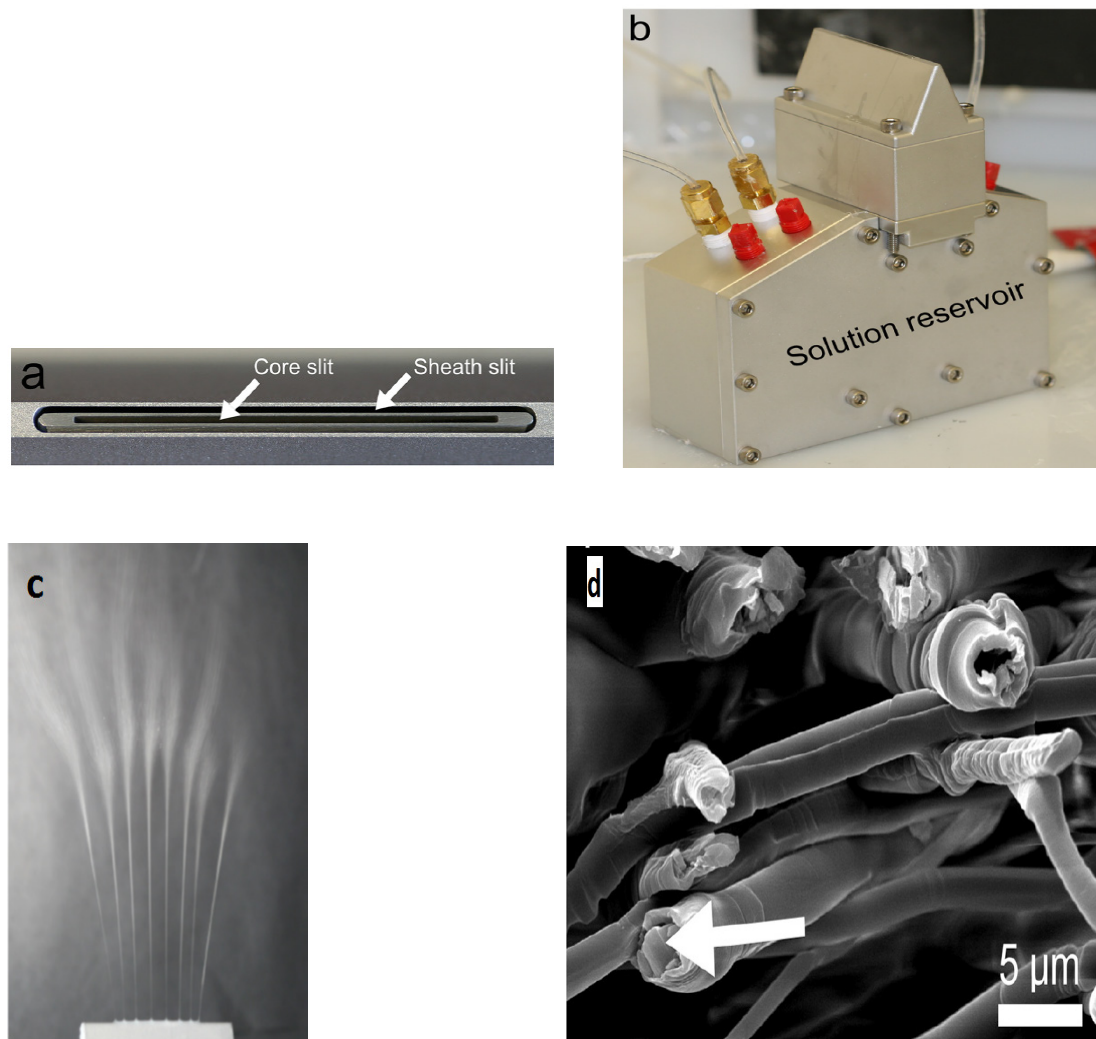


Figure 11. Slit-surface electrospinning prototype set-up. In figure 10-d shows Cross-sectional image showing of core-sheath fiber structure. The arrow points to a dexamethasone drug particle⁽²²⁾.

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