ISSN 1996-5044

Research Journal of

Nanoscience and Nanotechnology

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Research Journal of Nanoscience and Nanotechnology 3 (1): 19-33, 2013
ISSN 1996-5044 / DOI: 10.3923/rjnn.2013.19.33

Progress in Nanofiber's Fabrication by Electrospinning and Applications in Engineering and Technology

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ABSTRACT

The research and development of nanofibers has gained tremendous importance because of their unique properties and intriguing applications in the field of biomedicine (tissue engineering scaffold and drug delivery system), composites, sensor technology, optoelectronics, catalysis, filtration, protective clothing, energy storage, aerospace etc. Electrospinning is currently the only technique which can produce continuous fibers with diameters down to a few nanometers. This method (electrospinning) is simple and can be applied to polymers (solution and melt) and polymers loaded with active agents (e.g., enzyme), chromophores, or nanoparticles, as well as to metals and ceramics. Fibers can be oriented in an ordered way using special techniques or collectors, for specific applications e.g., filters, protective clothing. The electrospinning method can be modified to produce fibers with complex architectures, e.g., core-shell fibers or hollow fibers. It is evident by exponentially increasing research papers in commercial applications, due to wide scope of applicability and simple method of production of nanofibers lots of efforts are being made by the engineers and scientists to study the commercial applications.

Key words: Nanofibers, electrospinning, tissue engineering, polymer, scaffold

INTRODUCTION

Nanofibers are defined as fiber with diameter in the order of 100 nanometers. When the fiber-diameter of any material is reduced from "micrometers" (i.e., 10-100 μm) to "submicrons" and nanometers. Several amazing characteristics arise, such as very large surface area to volume ratio, flexibility in surface functionalities and superior mechanical performance (e.g., stiffness and tensile strength) compared with any other known form of material. Nanofibers can be produced by using various techniques such as template synthesis (Martin, 1996), phase separation drawing (Ondarcuhu and Joachim, 1998), self assembly (Whitesides and Grzybowski, 2002) and electrospinning (Huang *et al*., 2003). Among these techniques, the process of electrospinning seems to be the most promising because of the ease of fabrication, comparatively easy control over the process and easy scale up (Huang *et al*., 2003). Electrospinning, previously known as electrostatic spinning, was first studied by Zeleny (1914); however, the first patent describing the process of developing polymeric nanofibers via., electrospinning was obtained in by Formhals (1934). This technique was revived later by the work of Reneker and co-workers (Doshi and Reneker, 1995) as well as by Vancso and co-workers (Jaeger *et al*., 1998) during the last decade. Currently,

electrospinning is the only available technique for continuous production of nanofibers with diameter down to nanometers scale. Several factors control the electrospinning process which in turn influences the diameter and morphology of the resulting fibers. These factors include conductivity of solvent, solution viscosity, concentration, the nature and molecular weight of the polymer, net charge density, surface tension and ambient parameters (temperature, humidity and air velocity).

The nanofibrous matrix with high porosity, small pore size and very high surface area, is a potential candidate for developing novel filtration devices, materials for protective clothing, novel catalyst systems, light weight materials, controlled drug delivery devices and scaffolds for tissue engineering. It will be discussed here about the production of nanofibers by electrospinning, mechanism of fiber formation, controlling parameters in electrospinning, morphology control and important applications of nanofibers.

MECHANISM OF ELECTROSPINNING AND NANOFIBER PRODUCTION

Figure 1 explains the electrospinning setup depicting major components which includes a polymer (melt or solution) source, a high voltage power supply and a collection target/collector (a grounded conductor).

The polymer solution is contained in a syringe and flows through a capillary-like nozzle called spinneret. One electrode is connected to the spinneret tip and the counter electrode is attached to the collection target. The applied high voltage creates an electrically charged jet, where a polymer solution dries/solidifies and undergoes stretching during the course of travel to the target to produce fibers with a nanoscale diameter. A distance of 3-20 cm is generally maintained between the spinneret tip and collector (Li and Xia, 2004a), except for a near-field electrospinning where a distance of nearly 1 cm or below should be used. Direct Current (DC) power supplies are usually used but the use of Alternating Current (AC) potentials is feasible with an added advantage of improved fiber uniformity (Li and Xia, 2004a). The voltage applied is 1-30 kV in general.

The electric field is subjected to the capillary tube that contains polymer solution/melt (Fig. 1). The drop of polymer held at the tip of the spinneret under the action of surface tension (Fig. 2). On application of high voltage to the capillary, the electric field induces charges on the surface of the liquid and induced charges will get distributed over the surface of the liquid drop. At this stage, two

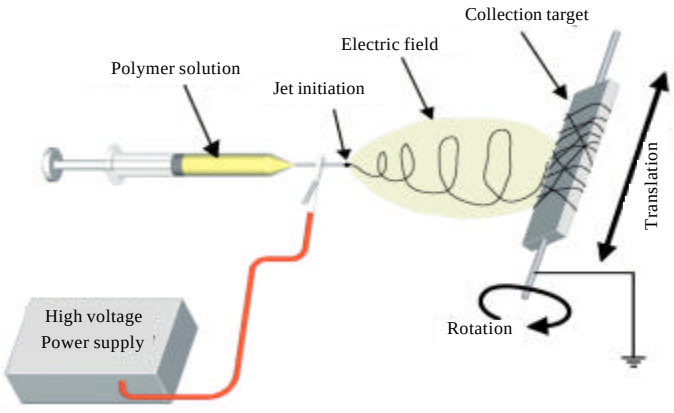

Fig. 1: Electrospinning setup where major components including a syringe filled with polymer solution/melt, a high voltage power supply and a collection target (i.e., grounded electrode) were illustrated (Sell *et al.*, 2007)

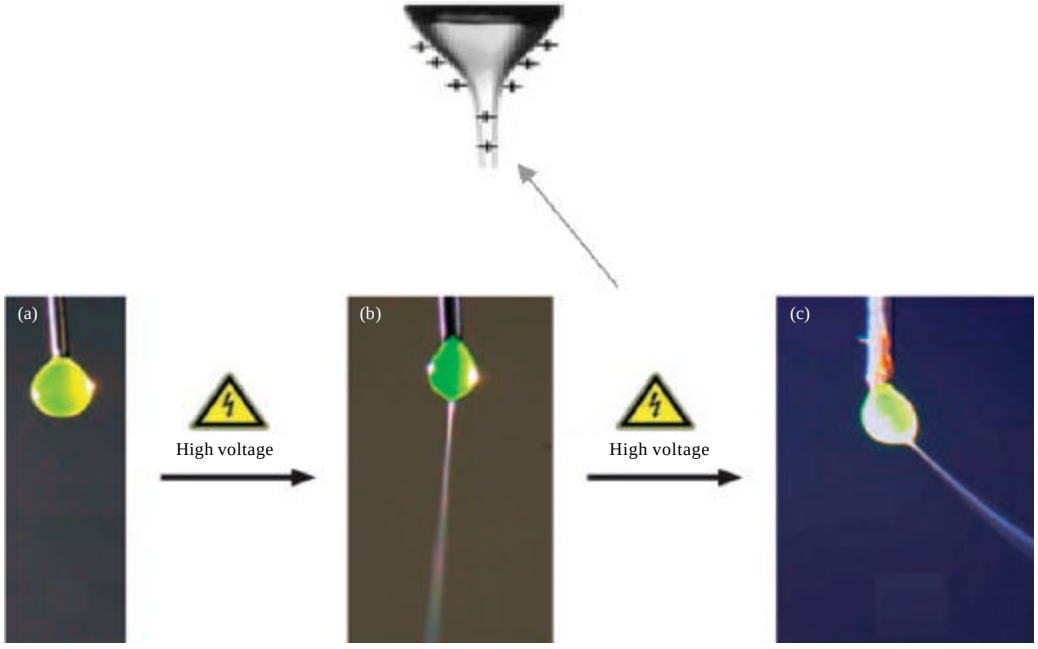

Fig. 2(a-c): A droplet of a 5% solution of polyethylene oxide (PEO) in water, dyed with fluorescein: (a) In the absence of an applied voltage, (b) At an applied voltage of 20 kV, with a jet perpendicular to the counter electrode/collector and (c) At an applied voltage of 20 kV, with a jet diagonal to the counter electrode/collector (Greiner and Wendorff, 2007)

major types of electrostatic forces are developed (1) Electrostatic repulsion between the surface charges and (2) Coulomb force exerted by the external field. Accumulation of like charges in the liquid droplet above a threshold level leads to mutual charge repulsion which in turn induces a stretching force in the droplet towards the collector (counter electrode). This stretching force works in the direction opposite to the surface tension. On increasing the intensity of the electric field gradually, the hemispherical surface of the fluid at the tip of the capillary tube elongates to form a conical shape known as the Taylor cone as shown in Fig. 2 with increasing field, a critical value is attained when the electrostatic interactions overcome the surface tension force and a charged jet of fluid is ejected from the tip of the Taylor cone (Fig. 2).

In the absence of an electric field (Fig. 2a) a droplet of a 5% solution of poly (ethylene oxide) (PEO) in water, is nearly spherical but at an applied voltage of 20 kV, the drop (Fig. 2b-c) is elongated and a jet of fluid is ejected to the direction of grounded electrode. In Fig. 2b, the jet is straight where the counter electrode is placed directly opposite to the spinneret. In Fig. 2c, with same applied voltage the jet is diagonal because the counter electrode is placed diagonal with the capillary tip.

Under the applied voltage, the charged jet is elongated and whips through air toward grounded target (Fig. 1-3) while the solvent evaporates and leaves behind a fiber with diameter ranging from micrometers to nanometers.

With a high-speed photograph (Fig. 3) of the jet which forms nanofiber, one can see that the jet travels a direct path towards the counter electrode for a certain distance at the beginning and

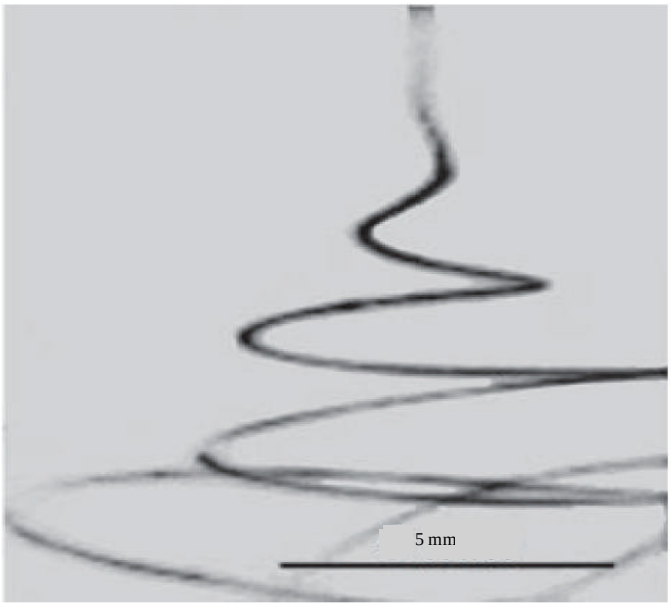

Fig. 3: High-speed photograph of a jet of polyethylene oxide PEO solution during electrospinning (Greiner and Wendorff, 2007)

thereafter the path changes significantly. The jet is moved laterally and forms a series of coils (whipping process) with increasing radius. The envelope of coils takes the form of a cone which opens towards the counter electrode. The fibers are randomly deposited on the grounded target/collector. Usually a circular and/or translational motion of the target is maintained to align the fibers in a specific orientation as well as to collect the fiber in a systematic way. The whipping process is the result of various types of instabilities of which most important is bending instability.

Nanofibers from polymer melt can also be obtained by electrospinning but in this case polymers are to be kept at an elevated temperature so that they remain in a molten state rather than dissolved in a solvent. Melt electrospinning eliminates the need for organic solvents and thus has the potential to increase throughput due to no loss in mass by solvent evaporation. A syringe pump is generally used for getting continuous flow of polymers through the syringe-needle or capillary. The spinning rate can be controlled by adjusting the flow rate of the polymer solution/melt and the magnitude of the electric field. A suitable distance between the needle tip and collector, gives time required for drying the fiber while whipping through air. Besides polymers, metals and ceramics is also applied for producing nanofibers by electrospinning.

CONTROLLING PARAMETERS IN ELECTROSPINNING

The spinning process depends on various parameters as described in the following.

System parameters:

- Molecular weight, molecular-weight distribution, architecture (branched, linear etc.), glass-transition temperature and solubility of the polymer
- Solution properties (viscosity, surface tension and electrical conductivity)

Process parameters:

- Electric potential, flow rate and concentration
- Distance between the capillary and collector
- Ambient conditions (temperature, humidity and air velocity in the chamber)
- Properties of the substrate
- Motion of collector/target
- Geometry of the electrodes

Morphology and diameter of fiber: The morphology and diameter of the fiber can be controlled by controlling the above parameters (Table 1). Formation of beads is a common phenomenon/problem in electrospinning. Beads, rather than fibers, are formed during electrospinning for certain properties of solution and process parameters. The beads are arranged on fibers like pearls on a string (Fig. 4-5). In some cases, the fibers are not round but flat ribbons and in other cases, the fiber diameter is not uniform: There is a broad distribution of fiber diameters.

By varying the above controlling parameters through vigorous experimentation, one can find the right conditions (parameters) for making nanofibers with uniform diameters as well as can avoid the formation of beads. Reckener and co-workers (Fong *et al*., 1999) have studied the effects of solution properties on the bead formation.

It was observed that the morphology of the fibers depends strongly on surface tension, viscosity and net charges which the jet carries with itself. However, the bead formation is believed to be the result of balancing three forces (1) Surface tension force which tries to minimize the surface area by converting the liquid jet into one or many spherical droplets (beads), (2) The electrostatic repulsion between the charges which tends to increase the surface area thus preventing the bead formation and (3) The viscoelastic force which resists any rapid change in the shape and favours the formation of fibers with smooth surfaces. Thus it is obvious that the formation beads can be avoided by suppressing the effect of surface tension by the last two forces. Viscoelasticity can be

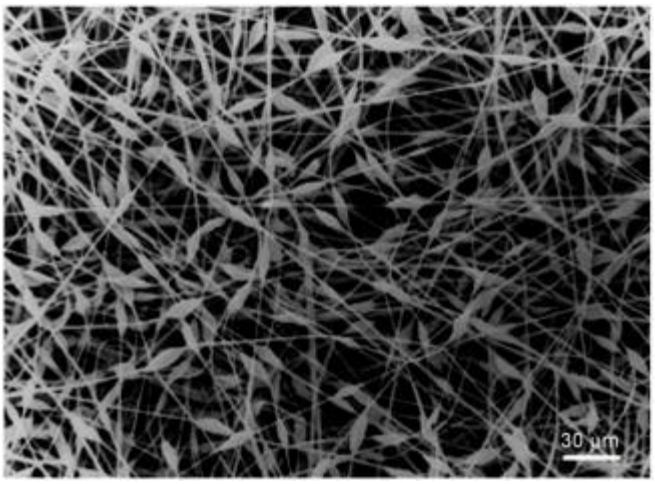

Fig. 4: SEM image of the irregularly shaped fibers of polystyrene (PS) produced by electrospinning from THF solution (Greiner and Wendorff, 2007)

Table1: Effect of each parameter on fiber morphology

Parameters	Fiber morphology	Theoretical interpretation of the phenomenon observed	References
System			
Viscosity	At lower viscosity beads are formed along with the fiber Increase in viscosity leads to an increment in fiber diameter	At lower viscosity surface tension predominates over the columbic repulsive force (stretching force). Hence, the solution tends to remain in the spherical state leading to beads formation	Lyons *et al*. (2004) and Zong *et al*. (2002)
Polymer concentration	Fiber diameter increases with increase of concentration	Higher polymer concentration leads to higher polymer chain entanglements along the polymer chain. This increased entanglement reduces the stretching that the jet can undergo before reaching the collector	Jiang *et al*. (2004)
Molecular weight	Reduction in the number of beads and droplets with increase of molecular weight	Higher molecular weight i.e., longer chains of polymer render it the tendency to maintain the jet at even lower concentrations	Geng *et al*. (2005)
Conductivity	Decrease in fiber diameter with increase in conductivity	Higher conductivity implies higher charge accumulation under the potential applied which leads to greater columbic repulsion between the like charges developed, leading to increased stretching that fiber undergoes, hence a decrease in fiber diameter	Jiang *et al*. (2004)
Process			
Applied voltage	Decrease in fiber diameter with increase in voltage	Greater voltage increases the charge density in the polymer jet aiding stretching of the fiber on its course to the collector	Kim *et al*. (2005)
Distance between tip and collector	Generation of beads at too small or too large distance An intermediate distance between the two will give uniform nanofibers	With increase in tip to collector distance the field per unit length decreases leading to dominance of surface tension over the columbic repulsive force thus giving raise to beads	Jiang *et al*. (2004)
Feed/Flow rate	Fiber diameter decreases with decrease in flow rate, high flow rate induces beads in the fiber	The volume of polymer dispensed from the tip of is higher at higher flow rate, leading to tracing of the surplus polymer solution over the jet initiated to give a thicker fiber	Zuo *et al*. (2005)

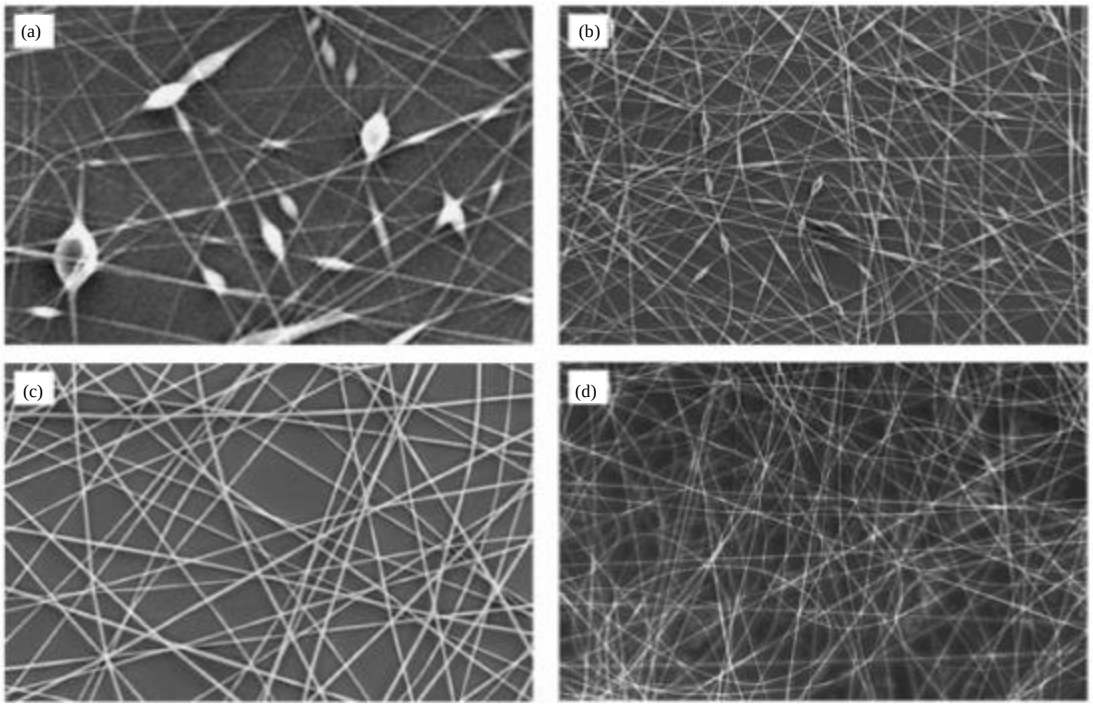

Fig. 5(a-d): SEM images of polyvinyl pyrrolidone (PVP) nanofibers that were electrospun from PVP solutions in a mixture of ethanol and water (16:3 v). The weight percentage of PVP in the solution was (a) 3, (b) 5, (c) 7 and (d) 5. In preparation of sample d, 0.35 mg mL^{-1} of tetraethyl ammonium chloride was added to the solution before electrospinning (Li and Xia, 2004b)

increased by increasing the solution concentration; the repulsive forces between charges can be increased by adding salts in solution and the surface tension force can be decreased by using solvent with low surface tension and/or by adding surfactant to the solution. Reckener and coworkers (Fong *et al*., 1999) formed PEO (poly-ethylene oxide) nanofibers where they eliminated the bead formation by adding salt to the solution, by increasing the solution concentration which increases viscosity and by using solvents of low surface tension. Li and Xia (2004b) investigated the morphology of (PVP) polyvinyl pyrrolidone fibers depending on solution concentration.

Figure 5 shows SEM images of nanofibers at various concentrations. It is clear that with increasing solution concentration there is a decrease in density of beads (Fig. 5a-b) and at a higher concentration the formation of beads is completely avoided (Fig. 5c). Bead-free nanofibers were also formed at lower solution concentration but with an addition salt tetraethyl ammonium chloride (Fig. 5d).

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Normally, the diameter of the fiber decreases with decreasing solution concentration (i.e., increasing viscosity), increasing capillary-collector distance and increasing flow rate and field

strength. Very high viscosity solution can create flow problems; at very high flow rate of solution there will be a lot of solvent which might not evaporate before deposition on the collector and at a very strong electric field there will be atomization of the solution which prevents continuous fiber-formation. Thus one needs to optimize various parameters need to manufacture nanofibers of desired diameter and morphology.

POLYMER SYSTEMS FOR THE PRODUCTION OF NANOFIBRES

A large number of different polymers have been electrospun and extensive research is going on to optimize the solution, process and ambient parameters that affect the electrospinning process, to develop polymeric nanofibers of appropriate physical, chemical and mechanical properties. Literature shows that most of the initial polymeric nanofibres were based on non-degradable polymers such as polyamides (Schreuder-Gibson *et al*., 2002), polyurethanes (Tsaia *et al*., 2002), polyesters (Megelski *et al*., 2002), polycarbonates (Huang *et al*., 2003) and a wide range of vinyl polymers (Huang *et al*., 2003).

Non-woven nanofibre matrices based on some of these polymers are currently used for developing novel filtration devices. Different type of polymers have been used to develop protective military clothing, ultra light weight materials for space application, high efficiency catalysts, nanoelectronic devices, nanosensors and various biomedical applications including medical prosthesis, scaffolds for tissue engineering and controlled drug delivery devices (Huang *et al*., 2003). The development of nanofibre matrices from natural and synthetic biodegradable polymers marked a significant step towards extending their application in the biomedical field. Some of the applications of nanofibers are summarized in Table 2.

APPLICATIONS OF NANOFIBERS

Nanofibers have the potential to find numerous applications in various fields of science and engineering. Some of the important applications are summarized in Fig. 6 and discussed in the following.

Filtration application: Filtration has profound application ranging from the regular household application to industrial scale unit operations (Suthar and Chase, 2001). To reach high filter

Table 2: Polymeric nanofiber in different applications

Polymer	Polymer nanofiber	Applications	References
Synthetic	Poly(ε caprolactone)	Bone tissue engineering	Yoshimoto *et al*. (2003)
	Poly(ε-caprolactone)	Multilineage differentiation of human mesenchymal stem cells	Li *et al*. (2005)
	Cellulose acetate	Adsorptive membranes/felts	Zhang *et al*. (2008)
	Polyvinyl alcohol	Drug delivery	Zeng *et al*. (2005)
Natural	Cellulose	Affinity membrane	Ma *et al*. (2005)
	Chitin	Wound healing	Noh *et al*. (2006)
	Silk fibroin	Skin tissue engineering	Min *et al*. (2004)
	Fibrinogen	Wound healing	Wnek *et al*. (2003)
Composite	Gelatin/polyaniline	Tissue engineering scaffold	Li *et al*. (2006)
	chitin/polyvinyl alcohol	Tissue engineering scaffold	Shalumon *et al*. (2009)
	poly(e-caprolactone)/chitosan	Tissue engineering scaffold	Malheiro *et al*. (2010)
	PLGA/collagen	Bone tissue engineering	Ngiam *et al*. (2009)

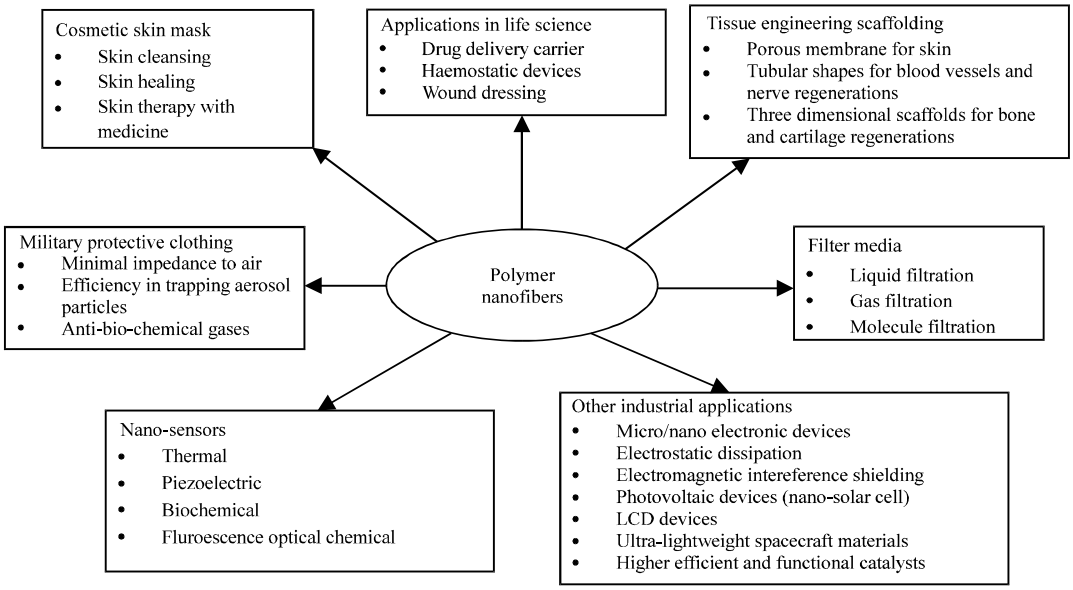

Fig. 6: Potential applications of electrospun polymer nanofibers (Huang *et al.*, 2003)

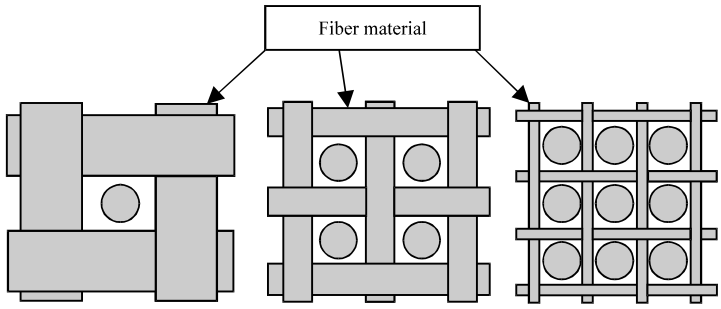

Fig. 7: Efficiency of a filter increases with decrease in fiber diameter (Huang *et al.*, 2003)

efficiencies, it is generally necessary that the sizes of the channels and pores in the filter material be adjusted to the fineness of the particles to be filtered. To filter increasingly finer particles, a transition from fibers with diameter in the micrometer range to fibers with diameters in the nanometer range is required.

Fibrous filter media provide advantages of high filtration efficiency and low air resistance. Filtration efficiency is determined to a great extent by fiber fineness (Fig. 7). In the industry, coalescing filter media are studied to produce clean compressed air. These media are required to capture oil droplets as small as 0.3 micron. It is realized that electrospinning is rising to the challenge of providing solutions for the removal of unfriendly particles in such submicron range. Since, the channels and structural elements of a filter must be matched to the scale of the particles or droplets that are to be captured in the filter, one direct way of developing high efficient and effective filter media is by using nanometer sized fibers in the filter structure (Graham, 2002). In general, due to the very high surface area to volume ratio and resulting high surface cohesion, tiny particles of the order of <0.5 mm can be easily trapped in the electrospun nanofibrous structured filters and hence the filtration efficiency can be improved.

In addition to fulfilling the more traditional purpose in filtration, the nanofiber membranes fabricated from some specific polymers or coated with some selective agents can also be used as, for example, molecular filters. Further improvement is possible by electrostatically charging the nanofibers (Gibson *et al.*, 2001). The fibers are often already charged during electrospinning but this charge generally dissipates through contact of the fibers with an electrical ground.

Semiconductor nanofibers: One-dimensional metal oxides have been extensively researched due to improved electro-optical, electro-chromic, ferroelectric, catalytic and gas sensing properties. Bognitzki *et al.* (2001) reported coating of electrospun poly (lactic acid) (PLA) fibers to manufacture polymer-metal hybrid nano and mesotubes. The above experiment was shortly followed by Caruso *et al.* (2001) who coated poly (L-lactide) electrospun fibers with amorphous titianium dioxide sol-gel (Caruso *et al.*, 2001). In this report hollow titania fibers were produced by removal of the thermally degradable polymer. The first report of electrospinning metal oxide composite fibers was made in 2002 (Dai *et al.*, 2002). Alumina borate solution was mixed with polyvinyl alcohol (PVA) to form a viscous gel that was electrospun. Composite nanofibers were calcinated above 1000°C to form pure $Al_4B_2O_9$, $Al_{18}B_4O_{33}$ and stable phase of a-Al_2O_3 was reported to form at 14000°C. Another group combined the methodology for forming mesoporous TiO_2 with the technique of electrospinning to produce mesoporous titanium dioxide fibers (Madhugiri *et al.*, 2004).

Protective clothing application: There has been a noticeable increment in funding from the government for the development of suitable protective clothing in military which is expected to help maximize the survivability, sustainability and combat effectiveness of the soldier system to overcome extreme weather conditions, ballistics, nuclear, biological and chemical warfare[48]. It has also been found application in designing breathing apparatus and protective clothing with the particular function of against chemical warfare agents whose inhalation and absorption through the skin can risk an individual's life. At present, the protective clothing used to overcome the warefare agents contains charcoal absorbents and has its limitations in terms of water permeability, extra weight-imposed to the article of clothing. Because of their great surface area, nanofiber fabrics are can neutralize chemical agents and also provide a free flow of air and water vapour without acting as a barrier (Tomer *et al.*, 2005). Electrospinning can be used to produce nanofibrous mats containing fibers in layers one after the other and also conferred with high porosity but very small pore size, providing good resistance to the penetration of chemicals and harmful agents in aerosol form.

Energy, electrical and optical applications: Polymeric conductive nanofibers also have potential in fields like electrostatic dissipation, corrosion protection, electromagnetic interference shielding, photovoltaic device, fabrication of tiny electronic devices or machines such as Schottky junctions, sensors and actuators etc. Since, rate of electrochemical reactions is proportional to the surface area of the electrode and nanofiber have an inherent advantage of high surface area to suffice the requirement (Schreuder-Gibson *et al.*, 2002; MacDiarmid *et al.*, 2001). Conductive nanofibrous membranes are also quite suitable for use as porous electrodes in developing high performance batteries and Polymer Electrolyte Membrane Fuel Cells (PEMFCs) due to its high porosity and inherent large total surface area. Polymer batteries have been developed for cellular phones to replace conventional, bulky lithium batteries. The components of polymer batteries are a carbon anode, a lithium cobalt oxide cathode and a polymer gel electrolyte. A conductive

nanofiber offers noteworthy properties of polymer batteries, for example, less electrolyte leakage, high dimension flexibility and high energy density per weight (Yun *et al*., 2001). However, there is still a need to improve energy density per weight of polymer batteries to increase their market share.

Catalysis and enzymatic reactions: Catalysts and enzymes can be embedded on nanofibers by co-electrospinning the polymer and enzyme or catalyst. Also the catalyst or enzymes can be separately reacted with nanofibers to have nanofiber supported catalyst or enzyme system. The enzyme/catalyst encapsulated nanofibers are more reactive compared to that in a membrane film cast from the same solution (Greiner and Wendorff, 2007). Unlike solubilized or nanoparticle supported enzymes/catalysts, enzyme/catalyst carrying fibers could be easily recovered from the reaction system for recycling purpose.

Biomedical applications: Nanofibers have a multitude of possible applications in medicine and pharmacy; most importantly in drug delivery system, tissue engineering and wound healing:

Drug delivery system: The drug molecules can be embedded in the nanofibers by electrospinning polymer solution containing drugs (Li and Xia, 2004a; Greiner and Wendorff, 2007). The drug loaded nanofibers have shown a good control of drug release over time and drug-supported nanofibers will be a good drug delivery system for tumor therapy, as well as for inhalation and pain therapy (Greiner and Wendorff, 2007).

Tissue engineering: The basic units forming tissues or organs are cells and the cells are surrounded with ECM (extracellular matrix). The ECM consists of nanoscale fibers which offer structural integrity to tissues (Sell *et al*., 2007). The nanofibers can be used to prepare 3D scaffolds in which cells can proliferate, migrate and differentiate into various tissues or organs. Figure 8 shows an articular cartilage developed in a 3D scaffold prepared from nanofibers made of collagen type 2.

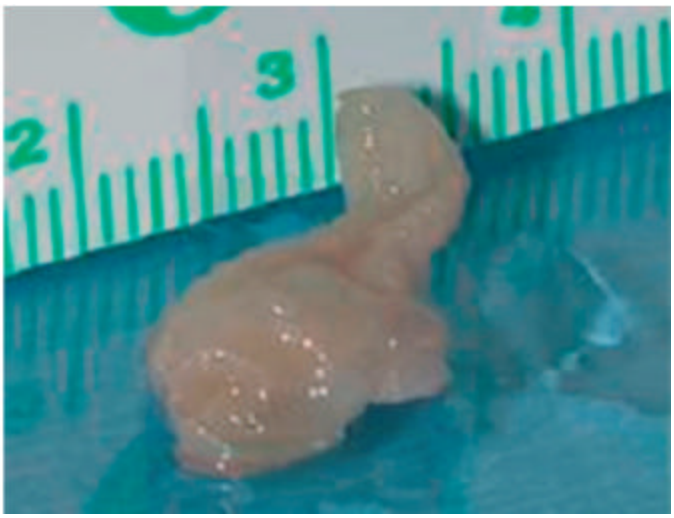

Fig. 8: Articular cartilage sample developed from normal human chondrocytes and electrospun collagen type 2 in a slow-turning lateral vessel bioreactor (Sell *et al*., 2007)

Fig. 9: Handheld device for the electrospinning of wound dressings. Inset: PEO fibers electrospun from aqueous solution onto a hand (Greiner and Wendorff, 2007)

This proves the efficacy of nanofibers as a potential candidate for tissue engineering. The other tissues like nerve, blood vessels, ligament, bone, urological tubes, etc., can be prepared by using 3D scaffold made of nanofibers (Greiner and Wendorff, 2007).

Wound healing: Wounds covered with polymer membranes that encourage formation of normal skin growth and eliminate formation of scar tissue that would occur in a traditional treatment, heal faster. It was found that large wounds such as burns and abrasions heal very fast when the wound is wrapped with a thin mat of nanofibers having pores below the dimensions of bacteria. The healing is faster when the mats are made of drug-loaded fibers. The mat having the pores with dimensions below bacteria prevents the entry of bacteria to the wound area while allowing air or moisture. Smith *et al.* (2001) described a process in which a fiber mat can be directly electrospun onto the affected skin areas. A handheld electrospinning device (Greiner and Wendorff, 2007) has been developed for the direct application of nanofibers onto wounds (Fig. 9). This can be used as a household item and will be available soon in the market (Greiner and Wendorff, 2007).

CONCLUSION

Electrospun nanofibres having unique properties (large surface to volume ratio, high density of pores and excellent surface adhesion) are being applied in many areas. Its application range extends from biotechnology, filtration and protective materials, medical prostheses, electronics, energy generation to defense security purposes and many others. Though, electrospinning has become an essential technique for spawning one-dimensional nanostructures but still there are many methodological issues which need to be resolved. For example, so far, still after having so much development in this technique, it is quite not easy to electrospin uniform nanofibers with diameters below 100 nm, especially at the scale of 10-30 nm. Moreover, the correlation between the

secondary structure of electrospun nanofibers and the processing parameters need to be investigated more systematically. The mechanical properties, photoelectric properties and other properties of single fibers should be analytically studied and optimized. Due to low production rate and low mechanical strength, the commercial application of electrospun fibers has been slow down. It is also necessary to develop new materials with various functions based on the knowledge of the electrospinning process and properties of electrospun nanofibers. It is hoped that, with further research and development, electrospinning will be the most significant nanotechnology of the century and will be extensively applied in various fields.

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