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Research note

Electrospinning as a versatile method for fabricating coreshell, hollow and porous nanofibers

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Abstract Electrospinnng is one of the most conventional methods for producing nano fibers in different forms, such as core-shell hollow and porous nanofibers. These forms open new windows on innovative applications for nanofibers like ultra filtration, fuel cells, membranes, tissue engineering, catalysis and drug delivery or release and nanofluidics and hydrogen storage. In the presented paper, developments in the electrospinning method toward fabrication of core-shell, and hollow and porous nanofibers are presented. Different spinnerets like coaxial and side by side are considered. Furthermore, experienced methods for producing these novel fibers, such as Nonsolvent-Induced Phase Separation (NIPS) and phase separation, are described. It is concluded that there is rapid development and achievement in the improvement of nanofibers for new applications, and electrospinning has become a forerunner in this field.

1. Introduction

Several desirable characteristics, such as high surface area to volume ratio, flexibility in surface functionalities and superior mechanical properties, can be achieved, when the diameter of the polymer fiber is reduced to nanoscale [1]. So far, many different techniques of fabricating polymer nanofibers, such as electrospinning, melt blowing, phase separation, self assembly and template synthesis, have been introduced for this purpose [2–7].

Electrospinning provides a straightforward electrohydrodynamical mechanism [7–10] to produce fibres with diameters less than 100 nm [11], even up to 5 nm [12]. Under the influence of an electric field, a pendant droplet of the polymer solution at the spinneret is deformed into a conical shape [13,14]. In other words, the electrospinning process can considered the ability to fabricate nanofibers by an electrically charged jet of polymer solution or polymer melts [15–22]. The earliest set up of electrospinning used auxiliary electrodes to direct the electrospinning jet onto rotating collectors. Later, some researchers used different collection devices [23–29] and manipulated electric fields [7]. For example, Dalton et al. deposited electrospun polymer melts in an innovative way to form a layered tissue structure [30].

The electrospinnability of a polymer solution is limited by the viscosity, conductivity, and applied solvents, as well as the conformation and molecular weight of the polymer. Some polymers are not spinnable because of limited solubility in a proper solvent for electrospinning, having proper polar characteristics. To overcome this problem, an unspinnable polymer can be fabricated as nanofibers by co-spinning with a spinnable polymer solution [9,31].

Different structures of nanofiber, such as coreshell, bicomponent, hollow and porous structures, could be produced by using special designs of spinnerets. Functionalizing nanofibers with super active surface properties can be produced by controlling nanofiber body size, mass and content. Special nanofiber morphologies and textures can be utilized in advanced applications, such as nanofluidics, catalysis, drug delivery and release, nano supports, energy storage and gas sensors [32,33]. In this paper these types of nanofiber structure, with an emphasis on their formation mechanisms and functionality, are reviewed.

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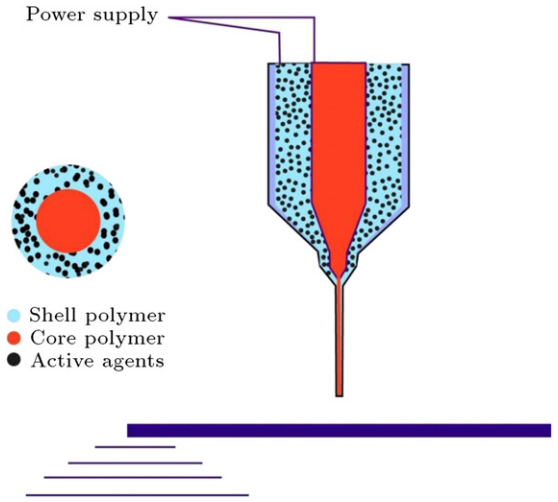

Figure 1: Schematic illustration of the electrospinning setup with two coaxial capillaries for spinning core-shell nanofibres.

2. Coreshell nanofibers

To impart functional properties onto the surface of the nanofibers (Shell), while keeping the intrinsics properties of the nanofibers (Core), coreshell nanofibers are introduced. In these kinds of nanofiber, the exterior layer may include active agents for imparting functional properties, such as shells holding immobilized/migratable specific enzymes [34].

Different methods, including multistep template synthesis [34,35], surface initiated atom transfer radical polymerization 'ATRP' [36] and coaxial electrospinning [37–45], are introduced for fabricating core-shell nanofibers. Among them, coaxial electrospinning is generally counted as one of the most versatile methods for fabricating these kinds of nanofiber [46].

A coaxial jet is formed by a coaxial spinneret when two different liquids flow through outer and inner capillaries simultaneously. Both capillaries are connected to a high voltage power supply and nanofibers are consolidated during solvent evaporation and stretching. The feeding ratio of two components affects the uniformity and stability of the jet flow Core. The shell thickness of the nanofibre can also be controlled by flow rates of liquids in the inner and outer capillaries during coaxial electrospinning (Figure 1). Other parameters, such as size of core-shell capillaries, applied electric field, volume feed rate, immiscibility of core-shell liquids, and their viscosity and conductivity, also play a crucial role in determining the uniform formation of core-shell jets and the morphology of the produced nanofibres in this electrospinning method [47].

Recently, both miscible and immiscible polymer blends have been electrospun into nanofibers by the phase separation phenomena [48]. The complex internal structure of nanofibers is formed via spontaneous phase separation during the electrospinning process.

Achieving continuous and uniform core-shell nanofibres can be ascertained by proper stretching of the droplet (Cone Taylor). Core deformation or its breakage into droplets may occur due to viscous force caused by the shell transforming itself into droplets (due to weakness of electric fields) [46], or its quick stretching, thus, exerting strong viscous stress tangential to the core [37,41].

Many studies have utilized a coaxial jet and produced special nanofibers through it. Generally, the polymer with lower surface tension is pumped to the outer capillary. For example, in the case of polystyrene (PS) and polyaniline (PANI), PS lay on PANI due to lower surface tension [49]. In another experiment, poly (ethylene oxide) PEO and chitosan (CS) were studied and fabricated nanofibers were proposed for biomedical fields like wound care and tissue engineering. It was proved that blending ratio, molecular weight of chitosan, and processing temperature were important factors in fabricating the final structure [50]. Pyridine (Py)-co-urethane as shell, and caprolactone (CL)-co-urethane as core, were coaxially electrospun for fabricating shape memory nanofibers. In this study, the ratio of core polymer and shell were modified for best shape recovery. The fabricated fibers exhibited high dimension stability and good shape recovery under thermal-induced tests [51]. Collagen as the shell, and poly-ε-caprolactone (PCL) as the core, were used for forming a nonwoven mat. According to cell culture results, collagen-coated PCL nanofibrous mat were compatible with fibroblast cell migration and proliferation in comparison with other controls (pure PCL fibers and single collagen and PCL nanofibrous or their mixtures) [52]. There also other studies into the use of electrospinning in the fabrication of polymeric, ceramic, and composite nanofibers, with core-sheath, hollow, or porous structures, as well as efforts made to improve their morphological homogeneity, functionality, and device performance [53,54].

Other efforts are also presented for fabricating core-shell nanofibers besides utilizing a coaxial spinneret. Zander et al. prepared poly methyl methacrylate (PMMA) — poly acrylonitril (PAN) fibers by using a conventional single-nozzle electrospinning technique. In this study, PMMA and PAN were used as core and shell, respectively [55]. The same technique was also used for poly (ethylene oxide) (PEO) and chitosan (CS). In this study, the fraction effect of each component in the solution was investigated. They found that the core-shell structure transformation is caused by different phase separation mechanisms with a continuous decrease of PEO fraction [56].

Side by side nanofibers are another kind of bicomponent fiber showing fascinating properties. Despite the coaxial spinneret, the capillaries for this purpose are placed side by side and two polymer solutions just come into physical contact at the end of the spinneret tip. The fabricated fibers benefit from both intrinsic properties of the two polymers, simultaneously. For instance, one of the sides is able to absorb chemicals, while the other side is capable of electrical conducting. Here, nanofibers, due to their excellent fineness, can act as nanosensors for highly sensitivities applications. They would be swollen when in contact with or in the absorption of chemical agents. As the other part's physical properties are not affected by the other side's absorption, the nanofibers would sag. This bending may then be used to switch an electrical circuit (like a siren circuit in the case of chemicals leakages) [32,33,57].

3. Hollow nanofibers

Hollow nanofibres are applicable for innovative and very specific usages, such as nanofluidics and hydrogen storage [58]. Usually, two different methods, including the Chemical Vapor Deposition (CVD) method [34,59] and direct co-axial spinning method [30], are employed for producing these kinds of nanofiber.

To produce hollow nanofibers by the CVD method, the first precursor polymer is transformed to nanofiber or a "template" by a conventional electrospinning method. Then, they would be coated with proper polymers or metals. Finally, hollow fibers

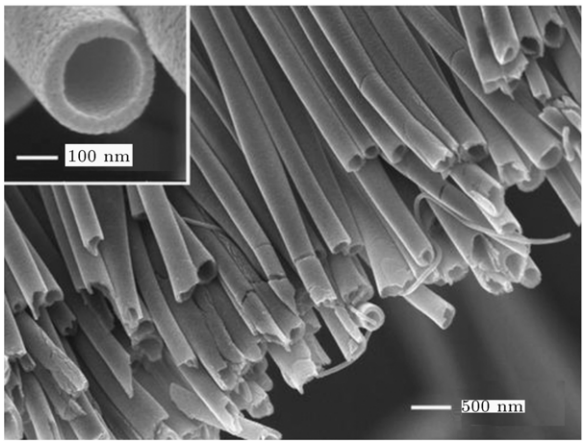

Figure 2: SEM image of TiO_2/PVP poly hollow nanofiber.
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are fabricated by dissolving the template material and drying them with centrifugal rotation dryers or by calcining in furnaces [60–65]. It is reported that, like conventional electrospun nanofibers, the quality of such resultant hollow nanofibres can also be controlled by electrospinning parameters [65].

The second mentioned method, i.e. co-axial spinning, is more regarded by researchers for fabricating hollow nanofibers. In the co-axial spinning method, nanofibers are produced by the same as procedure as that for fabricating core-shell nanofibers. But, the core material is dissolved with a selective solvent at the end of the process [65–67]. TiO_2/PVP poly (vinyl pyrrolidone) hollow nanofibers are formed (Figure 2) in this way by removing the oil phase as core [37]. In another study, poly(vinyl pyrrolidone) (PVP) and tetra butyl titanate (Ti $(OC_4H_9)_4$) were prepared as outer or shell solutions and paraffin oil was used as the inner or core material. Hallow microsized TiO_2 fibers with a hollow structure were obtained, when the organics and inner materials were removed. The photocatalytic activity of such microsized TiO_2 fibers is enhanced for degrading acetaldehyde gaseous by adding interior hollow channel numbers, proved by the Brunauer–Emmett–Teller (BET) analysis [68]. It was found that electrospinning parameters such as feed ratio can have an effect on the size and wall thickness of these nanofibres. Successful hollow nanofibre synthesis is possible by choosing the right solvent and controlling the heating rate [69].

Hollow nanofibers can also be filled with different substances for expanding their applications [53,70]. Poly-ε-caprolactone PCL nanofibrous was fabricated for controlled release of bovine serum albumin (BSA) or lysozyme. They used poly (ethylene glycol) (PEG) as core materials and proved that the releasing rate could be controlled by adding PEG, water soluble macromolecules in the sheath material [71]. Hydrophilic hollow nanofibers with periodic bumps were fabricated by Loscertales et al. They manipulated inorganic and hybrid (inorganic/organic) fibers and sol–gel chemistry for fabricating such fibers [72].

Yu et al. fabricated hollow carbon nanofibers (HCNFs) by coaxial electrospinning of poly (styrene-co-acrylonitrile) (SAN) and poly (acrylonitrile) (PAN) solutions. PAN was used as shell and SAN was found to be a very suitable material for the core. Considering the intrinsic properties of SAN, it showed a good thermal sustainability that prevented the PAN shell from shrinking during the stabilization and carbonization process. They found that solution concentration and flow rate were effective in controlling the outer diameters and wall thickness of HCNFs [73].

Highly porous polymeric hollow nanofibers were prepared by the coaxial electrospinning method [56]. They used silicon oil as the core material and a mixture of two polymers (PMMA, PC) as the shell. It is found that solvents and concentrations have major effects on the diameter and wall thickness of the produced hollow fibers. High dielectric constants of the solvent reduced the diameter of the hollow fibers. The wall thickness and average pore size were increased with increasing the polymer molecular weight of the polymer, but the specific surface area slightly decreased. Highly porous polymeric hollow fibers are used for catalysis, optoelectronics, nanofluidics, drug delivery and biosensor systems [56].

4. Porous nanofibers

The ranges of application for porous nanofibers are more extended and general in comparison with hallow nanofibers. Due to their high surface area, they are used for filtration, fuel cell [74,75], membrane [76], tissue engineering [77,78], catalysis and drug delivery and release [46,79]. Porous nanofibers can be generated with special topology by selecting particular solvents or solvent mixtures, or polymer mixtures under controlled environmental mediums. In one approach, immiscible components in a common solvent are electrospun and then one of the polymers is dissolved from the spun material to obtain porous nanofibers. This system is based on phase separation caused by different evaporation rates. Some other methods are also introduced for producing porous nanofibers, including Vapour-Induced Phase Separation (VIPS), Nonsolvent-Induced Phase Separation (NIPS) [80] and Thermally Induced Phase Separation (TIPS) [80–82], selective dissolution [79], rapid phase separation [13,83] and selective pyrolyzate composite formation [79,84].

Wendorff et al. produced PLA/PVP porous nanofibres by a vapour-induced phase separation method. They controlled the density of porosity by changing the amount and ratio of two polymers [59]. Gupta et al. produced porous nylon-6 fibers from the Lewis acid base complication of gallium trichloride (GaCl3) and nylon-6 using electrospinning, followed by GaCl3 removal [85]. Xia et al. produced highly porous nanofibres by a poly (styrene) (PS) solution in a mixture of DMF/THF as the core liquid and PVP/TiO_2 solution in ethanol as the shell liquid [37]. Two polymers of PS and PVP/TiO_2 matrix were separated and the PS phase was removed by calcining fibers to form highly porous nanofibres. In another method, porous structures are obtained by phase separation of the polymer and the solvent in a bath of liquid (such as nitrogen), and, thus, porous nanofibres are formed by removing the solvent under vacuum [86]. Kim et al. reported electrospun polymer (poly (L-lactide) (PLLA), Polystyrene (PS) and poly (vinyl acetate) (PVAc)) nonwoven mats with porous surface morphology by varying the collector temperature. They found that the surface morphology, porous structure, and properties, such as pore size, depth, shape and distribution of nonwoven mats, were influenced by the collector temperature [87]. Nayani et al. manipulated a nonsolvent-induced phase separation (NIPS) method for producing highly porous and hollow poly(acrylonitrile) (PAN) fibers [80]. In NIPS, the phase separation is induced by solvent/nonsolvent exchange. A highly porous structure is obtained when the polymer is immersed in a nonsolvent bath (water). They found that the specific surface area of porous PAN fibers (Figure 3) increased in comparison with the conventional

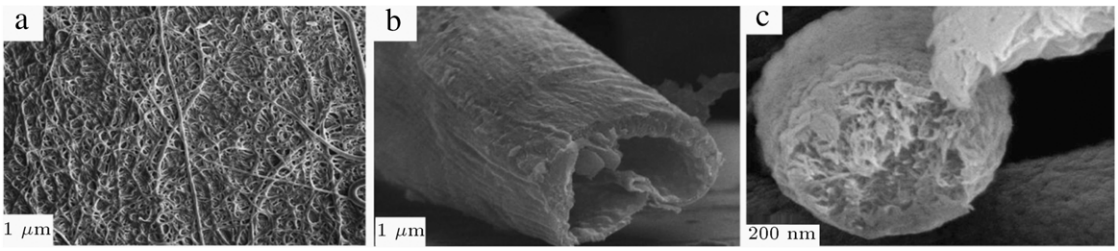

Figure 3: (a) SEM images of porous hollow fibers collected in ethanol/acetone. (b) Image of hollow PAN fiber collected with methyl ethyl ketone as a collecting bath. (c) Image of cross of a porous PAN fiber.
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electrospinning technique. In another study, PAN porous fibers were fabricated in one step by electrospinning a ternary system of PAN/DMF/water. The porous structure was formed when the spinodal decomposition phase separation occurred. In addition, the diameter of fibers increased by increasing the surface tension and viscosity of PAN solutions. It was observed that the BET surface area of porous PAN nanofibers with water was higher than the nonporous PAN nanofiber without water under the same conditions [88].

5. Conclusion

Nanofibers show highlighted properties due to their increased specific surface area, and electrospinning is one of the promising methods for producing such fibers. There are many developments for increasing their performance abilities, and so nanofibers in different forms, like coreshell, hollow, and porous, are presented. One important event in this field is introducing the coaxial spinneret (side by side and core sheath). Nanofibers produced by this method can be applied to tissue repair, wound healing and drug delivery systems.

Finally, these high potent fibers, i.e. nanofibers, constantly find their way into new fields, and coreshell, hollow and porous nanofibers are the result of such developments. Nevertheless, certain essential studies are still required and some challenges remain to be faced. Converting these advanced nanofibers to yarn is an important issue should which should be considered. Therefore, resultant nanofibers must have desirable properties. Mechanical behavior, such as tensile strength, plays a key role in determining the ability of yarn production. There are yet some unknown and uncontrollable parameters which need further attention, and studies should be undertaken into such influencing factors on the roughness of nanofibers.

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