Electrospinning to Forcespinning™

A new process called Forcespinning™ has been developed to make nanofibers from a wide range of materials. This new method uses centrifugal force, rather than electrostatic force as in the electrospinning process. The Forcespinning™ method uses either solutions or solid materials that are solution or melt spun into nanofibers. Key parameters to control the geometry and morphology of the nanofibers include rotational speed of the spinneret, collection system and temperature. Orifices of the spinneret can have arbitrary geometric shapes to provide corresponding cross-section of nanofibers. The Forcespinning™ method has been successfully used to make nanofibers of poly-ethylene oxide, poly-lactic acid, bismuth, polypropylene, acrylonitrile-butadiene-styrene, polyvinyl pyrrolidone, and polystyrene among others.

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In 1902, Morton[1] and Cooley[2] discovered the electrospinning process, the way it is known now and filed US patents. Formhals[3] then investigated the problem for about a decade and obtained nine US patents on different aspects of electrospinning. These patents identified a number of key issues and their potential solutions to optimize their goal of manufacturing "fine fibers". Unfortunately, due to technology limitations, they did not realize the making of nanofibers nor their significant application potentials. In 1995, Doshi and Reneker re-introduced the electrospinning process[4], by then scanning electron microscope (SEM) was commonly available and they rightly place the significance of nanofibers in perspective. They clearly identified a myriad of applications for the electrospun nanofibers in as diverse fields as structures, textile, membrane, and biomedical engineering. When Doshi and Reneker re-introduced the century old process "electrical spinning", they also coined the more convenient term "electrospinning".

The electrospinning method as popularized by Doshi and Reneker[4] consists of driving a polymer solution jet through a high electric field rendering a meso-scale fluid jet into nano-scale fibers. The solvent selected to prepare the polymer solution has to have suitable vapor pressure, viscosity, and surface tension to promote fiber integrity. Besides these key factors electrospinning methods require an understanding of other parameters that affect formation of Taylor Cone and jet instability, namely, solution or melt conductivity, electrostatics, electric field strength, surface charge, and ionization field. In 1964,

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Sir Geoffrey Taylor[5] published the first detailed mathematical study on the subject of electro-hydrodynamic stability. Since then, several researchers[6] have developed various mathematical, rheological, dimensional, and neural network models to develop understanding of the complex electro-hydrodynamic process. These diversified approaches to understand the electrospinning process by numerous researchers over so many decades is an indication of the complexity of the method and resulting limitation in its commercial success. Electrospinning is so far the only method with potential industrial production, more than 50% of the research articles related to nanofiber production are based on electrospinning methods[7]. Some researchers have modified the traditional electrospinning method to enhance nanofiber production either through syringes connected in series or through a needleless electrospinning process[8-9]. Other techniques for nanofiber development have been reported such as: template synthesis, phase separation, self assembly, and other spinning methods (wet, dry and melt spinning), such as, recently reported articles where polymeric nanofibers are made by placing drops of a polymeric solution on a standard spin coater and nanofibers are created through the centrifugal forces that drive the polymer outwards[10-11]. Potentially, many materials can be spun into fibers with this method, though it may not be suitable for mass production, has large standard deviations on nanofiber diameter, and restricts fiber mat purity by the presence of polymer beads."

Methodology

The search for a method that would eliminate and/or minimize many of the limitations encountered on the above mentioned methods focused on: increase material choice, improve production rate, and lower fiber costs through an environmentally friendly process. In this new method[12-15] called Forcespinning™ the electric field, used in the electrospinning process, is replaced by centrifugal forces. The combination of centrifugal forces with multiple configurations of easily interchangeable spinnerets makes the Forcespinning™ a versatile method that overcomes many of the limitations of existing processes, namely, high electric fields and a solution that is typically dielectric. These changes significantly increase the selection of materials by allowing both non-conductive and conductive solutions to be spun into nanofibers. If necessary, high temperature solvent can also be used by heating the spinneret holding the material of interest. Additionally, a number of solid materials can be melted and spun without chemical preparation there is no need for solvent recovery since no solvent is involved.

A schematic of the new system is shown in Fig. 1a (sketch) and b (prototype). Main components of the system are the spinneret, thermal system, collection devices, environmental chamber, control system, motor, and brake. Multiple spinnerets have been designed such as the one presented in Fig. 1c, where a polymer is continuously fed into a cavity and the solution or melt is centrifugally forced through the designed orifices (cross sections and L/D are controlled) to make nanofibers. The control system drives the system to the speed and temperature set- points specified by the user. The system is controlled through a programmable logic controller (PLC). The PLC has a keypad and a liquid crystal display to allow the user to enter the set-points, run, stop, and monitor the operation of the system. The controller is also interfaced with a computer that allows the user to remotely control the process[12-15].

The chassis assembly of the system contains the motor and the oven; the spinneret is connected at the end of the shaft using a threaded coupling; the brake stops the motor in case of power failure and it is also useful to hold the shaft still when mounting or dismounting the spinneret. The thermal system includes both heating (heater) for melting and cooling (thermo-electric cooler) for low temperature operations. The environmental chamber allows maintenance of non-ambient conditions including vacuum, inert gas, and aseptic environment. It may be noted that the environmental chamber also acts as a safety wall for the operator of this dynamic system.

In summary, the process consists of depositing the material either as a solution or as granular solids that are melted in the spinneret and then spun to make nanofibers. The control parameters that affect fiber diameter include spinneret selection, nozzle configurations, rheological properties, rotational speed, temperature, collection system, and environment.

Initial results

A well studied system, namely, solution spinning of aqueous PEO was used first[6] to make nanofibers. While typical concentration of PEO in DI water is in the range of 5% to 12% for electrospinning, the concentration for this new method varies significantly (from 7% to 16%) depending on the spinneret type, molecular weight, and rotational speed, among others. The concentration of all polymers in the solutions systems used in the Forcespinning™ method has been observed to be considerably higher than the reported ones for electrospinning methods. Corresponding rotational speeds for PEO were in the range of 3,000 to 5,000 RPM. Fig. 2a shows a SEM micrograph of the first PEO nanofibers (scale bar 20μ) obtained by Forcespinning™ (average diameter of 300 nm).

Melt spinning of commercially available injection moldable polystyrene (PS) was also performed to check the versatility and

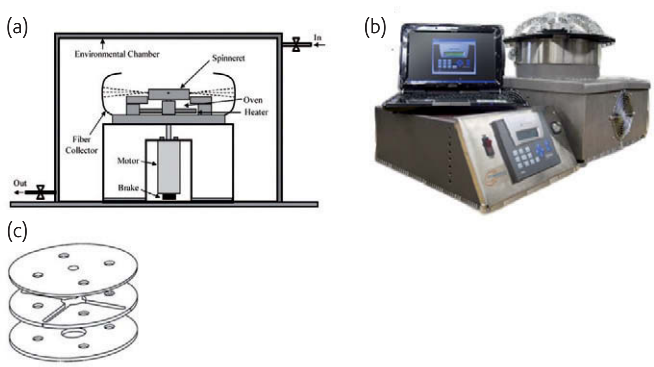

Fig. 1 Sketch (a), Actual Prototype (b) and Spinneret sample (c), of the Forcespinning™ Method.

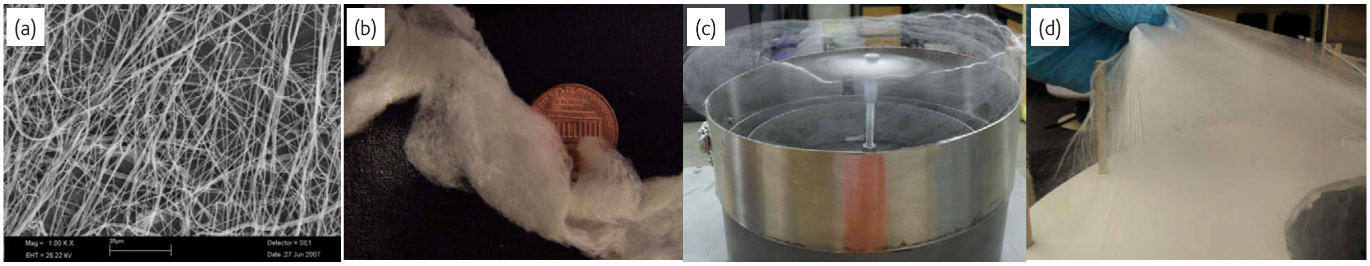

Fig. 2 (a) PEO Nanofibers (Solution) and (b) PS Mat (Melt) produced by the ForcespinningTM Method, (c) nanofiber webs and (d) free standing nonwoven mats also produced by the presented method.

robustness of the method. Resulting fibers at 3,000 RPM are shown in Fig. 2b. To ensure the flexibility of the new method several materials have been used such as laboratory grade bismuth powder which was melt spun resulting in a sample of 500 nm nanofibers. Polypropylene (PP) nanofibers were also successfully made, PP is difficult to impossible to make using conventional electrospinning process since it does not have a room temperature solvent. The developed system allows for flexibility to easily collect material in a variety of ways such as webs (Fig. 2c) or free standing non-woven mats (Fig. 2d) depending on the selected spinneret and collection method. When collected as webs, the aligned fibers can also be spun in yarns that can then be woven into many desired applications. The initial Forcespinning™ prototype offers a nanofiber mass scale production method (1g/min per spinneret orifice, several orifices/spinneret).

This method opens the doors to boost development of the previously mentioned potential applications and numerous more. In addition to being a simple and flexible system, it offers low cost mass production of a broad range of polymeric, metallic, ceramic or composites fibers in the nano, sub micron and single digit micron scale.

Conclusions

To overcome present limitations encountered in typical electrospinning processes, a novel process was developed and tested. This new method called Forcespinning™ uses centrifugal forces, rather than electrical forces as in electrospinning. The technological challenge of bringing the fibers down to the nanometer scale was resolved by a set of designed spinnerets that when combined with the developed thermal systems, speed, and collection systems allow for a novel production system. Forcespinning™ is versatile to use both solutions and melted materials. The availability to melt the materials in the spinneret eliminates the restrictions imposed by cost (solvent recovery step) and environmental (material contamination) effects of hazardous polymer solvents. A prototype that fits in a standard chemical hood was developed to make fine fibers in the nano, submicron and single digit micron scale from a number of ordinary materials such as PEO, bismuth, PP, PS, PC, PLA, ABS, PPV, PVP, and nano-reinforced polymer composites. The new method is simple to change materials and allows automatic control of process parameters. Initial results on PEO nanofibers showed that the average fiber diameter depends on the inverse of the rotational speed squared. It is anticipated that the safety, simplicity, and versatility of this method will make it another practical method to make nanofibers allowing the development of applications with new nanofibers that cannot be made using electrospinning methods. mt

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REFERENCES

1. Morton, W. J., *Method of Dispersing Fluids*, US 705,691, 1902.
2. Cooley, W. J., *"Apparatus for Electrically Dispersing Fluids,"* US 692,631, 1902.
3. Formhals, A., *"Method and Apparatus for the Production of Artificial Fibers"* US 2,158,416, 1939.
4. Doshi, J. and Reneker, D. H., *J of Electrostat* (1995) **35**, 151.
5. Taylor, G. I., *P R Soc London* (1964) **280**, 383.
6. Reneker, D. H. and Yarin, A. L., *Polymer* (2008) **49**, 2387.
7. Polymer Nanofibers – *An Overview of Applications and Current Research into Processing Techniques*; www.azonano.com/details.asp?ArticleID=1280; 2005.
8. Petras, D., *et al.*, *"Method and device for production of nanofibres from the polymeric solution through electrostatic spinning"* Patent Application Publication US 2008/0307766 A1, Dec. 18, 2008.
9. Robertson, J. A. and Scott, A. S., *"Electrospraying/electrospinning array utilizing a replacement array of individual tip flow restriction"* US Patent Application Publication US 2008/0131615 A1, June 5, 2008.
10. Weitz, R. T., *et al.*, *Nano Lett* (2008) **8** (4), 1187.
11. Jancic, R. and Aleksic, R., *Mater Lett* (2000) **42** 350.
12. Lozano, K. and Sarkar, K., *Methods and apparatuses for making superfine fibers*, US20090280325 A1.
13. Lozano, K. and Sarkar, K., *Superfine fiber creating spinneret and uses thereof*, US20090280207 A1.
14. Lozano, K. and Sarkar, K; *superfine fiber creating spinneret and uses thereof*, US20090269429 A1.
15. Lozano, K. and Sarkar, K., *Superfine fiber creating spinneret and uses thereof*, US20090232920 A1.