

# Review of Polymer Solutions for Near-Field Electrospinning with Spatial Control

Antonio Osamu Katagiri Tanaka, Héctor Alán Aguirre Soto

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## Abstract

Near-field electrospinning (NFES) is identified to be a technique able to fabricate polymer nano and micro fibers with accurate placement. In the past years (2006-2019), several polymer solutions have been successfully electrospun into fibers through several variants of the conventional NFES process. Each NFES variant intends to tailor the process parameters in order to improve the fibers' properties. This paper presents a review on the research and related development of electrospun fibers, emphasizing the used polymers, solvents, and fiber characteristics. Relevant summary of polymer solutions and near-field electrospinning processing conditions is provided in this paper.

*Keywords:* polymer, solvent, near-field electrospinning, NFES, fibers, spatial control

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## 1. Introduction

Even though electrospinning is an old invention [1], it is currently a trending topic among researchers [2–4]. One of the reasons electrospinning is to be studied is its potential to fabricate polymer nano-fibers from a variety of polymers. The technique allows the production of

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thin continuous fibers with ease, with diameters down to 3 nm in some cases, which is something difficult to achieve by other techniques. Furthermore, the basic setup can be modified with ease to fabricate different fibers with diversified functionalities with different materials. The produced fibers can be aligned or unaligned. Besides, the electrospinning equipment is inexpensive and of small size, compared to the equipment of standard spinning techniques. On the other hand, the understanding of the electrospinning process has improved in the last years [5].

The main components of the electrospinning technique are the fluid control unit (e.g. syringe pump) and a voltage power supply. The process also requires a target electrode or combination of electrodes on which the fibers can be collected. Figure 1 describes a typical near-field electrospinning set-up [5]. Two sub-techniques can be derived from electrospinning depending on the distance between the dispensing electrode and the collector. The process in which the electrospun jet can be controlled near the tip is called NFES or near-field electrospinning [6]. Moreover, if the distance between the collector and the dispensing needle is greater, the configuration is known as FFES or far-field electrospinning [7]. Near-field electrospinning is considered to be an outstanding technique to fabricate polymer fibers with spatial control and it has suffered several modifications to improve the precision and accuracy of the fiber deposition. This paper intends to collect the NFES variants of electrospunable polymer solutions with spatial control in recent research.

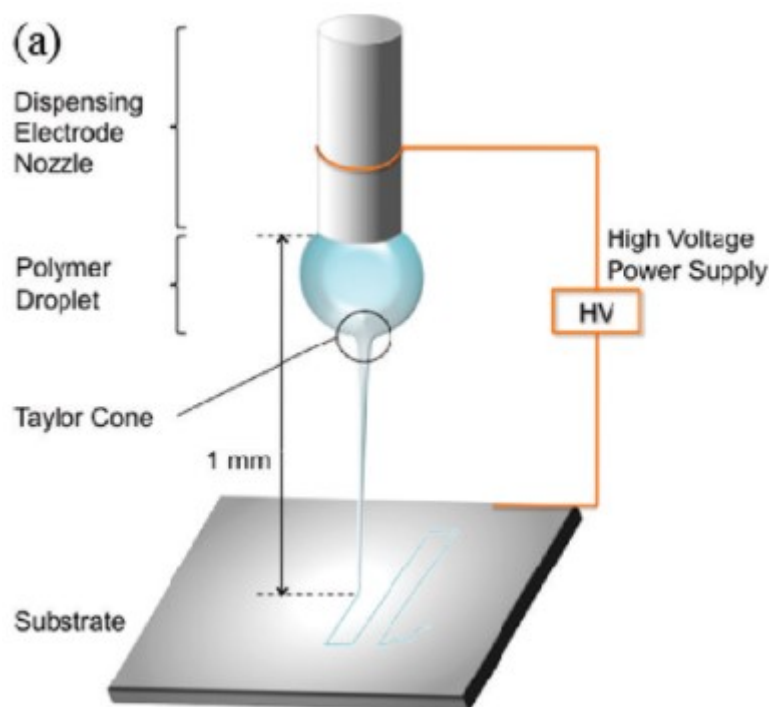


Figure 1: Typical near-field electrospinning set-up [8] .

## 2. NFES articles' summary

The key advance that allows for lowering of the voltage and attendant increase in patterning control of NFES is the use of a superelastic polymer ink. Solutions of such a polymer ink contain long entangled polymer chains that promote stretchability and are expected to augment continuity of the electrospun jets. This facilitates the continuous electrospinning of the polymer jet into nanofiber.

The polymer jet does not self-initiate under the influence of the voltage because the electrostatic force cannot overcome the surface tension at the droplet - air interface. Therefore, the electrospinning process was initiated by introducing an artificial instability at the droplet - air interface with a glass microprobe tip (1 to 3  $\mu\text{m}$  tip diameter) that resulted in a very high local electric field, sufficient to overcome the interfacial surface tension, giving rise to the formation of the Taylor cone and initiation of the polymer jet.

Therefore, we believe that 2 wt % PEO solution has the proper balance of viscosity, conductivity, and resistance to hardening that allows for low-voltage continuous electrospinning.

At 300 V and lower, the perturbation phenomenon was eliminated and straight deposition patterns were obtained (as seen in Figure 4c,d). This could be achieved with slow stage speeds (20-40 mm/s) clearly demonstrating that the low-voltage NFES technique substantially reduces bending instabilities

Another advantage of lower voltage operation lies in reduction of the diameter of the jet, leading to thinner nanofibers. This is most likely due to the lower electrostatic forces at play that reduce the feed rate of the polymer, thus reducing jet thickness. Therefore, the voltage can be manipulated to directly control the thickness of the nanofibers

Further investigation into the effect of voltage on the thickness of nanofibers reveals an increasing linear trend of average diameter versus voltage as shown in Figure 6. This trend is opposite to that observed in far-field electrospinning

We further demonstrate the ability to integrate low-voltage NFES "writing" capability with 3D substrates by suspending nanofibers on carbon micropost arrays on a Si substrate. In a typical example, we use posts of a height of 40  $\mu\text{m}$ , a diameter of 30  $\mu\text{m}$ , an interpostal distance of 100  $\mu\text{m}$ . [8]

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Table 1: Electrospun Polymer Solutions - Solution and Process Parameters

Polymer(s)	Solvent(s)	NFES Variant	Process Parameters and Fiber Characterization	Ref.
Poly(ethylene oxide) (PEO MW = 4 000 000)	Deionized water	Low-Voltage NFES (LV NFES)	<b>Solution Concentration:</b> 1, 2, and 3 <i>wt%</i> PEO <b>Nozzle:</b> 27 gauge type 304; stainless steel needle <b>Solution deposition rate:</b> lower than $1\mu L/h$ <b>Nozzle-to-substrate distance:</b> 1mm <b>Substrate composition:</b> Pyrolyzed SU-8 carbon and Si <b>Applied voltage:</b> polymer jet initiated at 400-600 V and dispensed at 200-400 V <b>x-y stage velocity:</b> 10-40mm/s <b>Fiber Diameter:</b> 50-425nm <b>Distance between adjacent fibers:</b> <i>Not determined</i>	[8]
Poly[2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylenevinylene] (MEH-PPV) with Poly(ethylene oxide) (PEO)	acetonitrile, toluene mixture (65/35); acetic acid, toluene (17/83); pure toluene	Typical process	NFES <b>Solution Concentration:</b> 10mg of MEH-PPV in 2mL of toluene; 500mL of MEH-PPV solution with 250mg of PEO in 3.5mL of acetonitrile; 500mL of MEH-PPV solution with 250mg of PEO in 3mL of acetic acid / toluene (17 / 83). The resulting MEH-PPV/PEO concentration is 1:100 <b>Nozzle:</b> mm-diameter tip Tungsten spinneret in a 26 gauge needle <b>Solution deposition rate:</b> 50 $\mu L/h$ <b>Nozzle-to-substrate distance:</b> 500 $\mu m$ <b>Substrate composition:</b> SiO <sub>2</sub> /Si (oxide thickness = 800 nm) <b>Applied voltage:</b> around 1.3kV <b>x-y stage velocity:</b> 50cm/s <b>Fiber Diameter:</b> 100nm <b>Distance between adjacent fibers:</b> around 100 $\mu m$	[11]

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<i>Table 1 continued</i>				
	Poly(ethylene oxide) (PEO)	Water	Scanning Electrospinning and NFES	Tip <b>Solution Concentration:</b> 7wt% PEO [12] <b>Nozzle:</b> Needle outer diameter of 200 $\mu m$ and inner diameter of 100 $\mu m$ <b>Solution deposition rate:</b> 0.1 $\mu L/h$ <b>Nozzle-to-substrate distance:</b> 500 $\mu m$ <b>Substrate composition:</b> <i>Not determined</i> <b>Applied voltage:</b> polymer jet initiated at 1.5 kV and dispensed at 600V <b>x-y stage velocity:</b> 120mm/s <b>Fiber Diameter:</b> 709 $\pm$ 131nm; 49-74nm when applied voltage is 800V <b>Distance between adjacent fibers:</b> <i>Not determined</i> <b>Notes:</b> 108m yield in 15min with a fiber diameter of 709 $\pm$ 131nm
6	Poly(vinylidene fluorid) (PVDF)	N,N Dimethyl- formamide (DMF)	Helix Electrohydro- dynamic Printing (HE-printing)	<b>Solution Concentration:</b> 1.8g PVDF in 4.1g of DMF and 4.1g of acetone. The resulting concentration is 18% PVDF. [13] <b>Nozzle:</b> Needle outer diameter of 510 $\mu m$ and inner diameter of 260 $\mu m$ <b>Solution deposition rate:</b> 400nL/min <b>Nozzle-to-substrate distance:</b> 10-50mm <b>Substrate composition:</b> Poly(dimethylsiloxane) (PDMS) on Ecoflex <b>Applied voltage:</b> 1.5–3kV <b>x-y stage velocity:</b> 0-400mm/min <b>Fiber Diameter:</b> about 1.5-3 $\mu m$ <b>Distance between adjacent fibers:</b> <i>Not determined</i>
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<i>Table 1 continued</i>				
~	Polyhedral Oligomeric Silsesquioxane-Poly(Carbonate-Urea)Urethane (POSS-PCU) and Polyhedral Oligomeric Silsesquioxane-Poly(Caprolactone-Poly(Carbonate-Urea)Urethane) (POSS-PCL-PCU)	Dimethyl acetamide (DMAC) and 1-Butanol	Electrohydro-dynamic 3D Print-patterning or Electrohydro-dynamic Jetting	<b>Solution Concentration:</b> POSS-PCU and POSS-PCL-PCU used in 20%w/w concentration in DMAC [14] <b>Nozzle:</b> needle of 750 $\mu m$ in diameter <b>Solution deposition rate:</b> less than 1 $\mu L/min$ <b>Nozzle-to-substrate distance:</b> about between 500 $\mu m$ to 2mm <b>Substrate composition:</b> <i>Not determined</i> <b>Applied voltage:</b> 8.0-10.0kV <b>x-y stage velocity:</b> 10mm/s <b>Fiber Diameter:</b> 5-50 $\mu m$ <b>Distance between adjacent fibers:</b> 250 $\mu m$
	Poly(ethylene oxide) (PEO)	Distilled water	Electrohydro-dynamic Writing or Mechano-electrospinning (MES)	<b>Solution Concentration:</b> 6wt% PEO [15] <b>Nozzle:</b> <i>Not determined</i> <b>Solution deposition rate:</b> 1200nL/min <b>Nozzle-to-substrate distance:</b> 7.5mm <b>Substrate composition:</b> <i>Not determined</i> <b>Applied voltage:</b> polymer jet initiated at 2 kV and dispensed at 0.8-1kV <b>x-y stage velocity:</b> around 400mm/s <b>Fiber Diameter:</b> 200-350nm <b>Distance between adjacent fibers:</b> 5 $\mu m$

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<i>Table 1 continued</i>				
Poly(ethylene oxide) (PEO)	Deionized water and the ethanol with a volume ratio of 3:1	Airflow-assisted Electrohydrodynamic Direct-writing (EDW)	<b>Solution Concentration:</b> 8wt% PEO <b>Nozzle:</b> Outer airflow passage diameter: 1mm Airflow gas pump pressure: 25kPa Inner liquid passage diameter: 0.21mm <b>Solution deposition rate:</b> 30 $\mu$ L/h <b>Nozzle-to-substrate distance:</b> 2mm <b>Substrate composition:</b> Silicon <b>Applied voltage:</b> about 2kV <b>x-y stage velocity:</b> 1-20mm/s <b>Fiber Diameter:</b> 3.73 $\pm$ 1.37 $\mu$ m <b>Distance between adjacent fibers:</b> 5.13 $\pm$ 6.67 $\mu$ m	[16]
Poly(Vinylidene Fluoride) (PVDF)	Acetone and Dimethyl Sulfoxide (DMSO)	3D Electrospinning	<b>Solution Concentration:</b> 17wt% PVDF; 1.7g of PVDF, 5g of acetone, 0.5g of Capstone FS-66, 5g of DMSO <b>Nozzle:</b> Needle inner diameter of 100 $\mu$ m <b>Solution deposition rate:</b> 14 nL/min <b>Nozzle-to-substrate distance:</b> 750 $\mu$ m <b>Substrate composition:</b> A4 size commercial printing paper (Double A) <b>Applied voltage:</b> 1.9kV <b>x-y stage velocity:</b> 10mm/s <b>Fiber Diameter:</b> Not determined <b>Distance between adjacent fibers:</b> Not determined	[17]

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<i>Table 1 continued</i>				
Poly(9-Vinyl Carbazole) (PVK)	Styrene	Typical process	NFES	<b>Solution Concentration:</b> 3.96wt% PVK in styrene [18] <b>Nozzle:</b> Needle inner diameter of 100 $\mu m$ <b>Solution deposition rate:</b> 500nL/min <b>Nozzle-to-substrate distance:</b> around 2.5mm <b>Substrate composition:</b> Si/SiO <sub>2</sub> <b>Applied voltage:</b> 3-4kV x-y stage velocity: 13.3cm/s <b>Fiber Diameter:</b> 289.26 $\pm$ 35.37nm <b>Distance between adjacent fibers:</b> 50 $\mu m$ <b>Notes:</b> 15m yield in 2min
Polystyrene (PS)	1,2,4-Trichloro benzene	Electrohydrodynamic (EHD) jet printing		<b>Solution Concentration:</b> 1 to 5wt% PS [19] <b>Nozzle:</b> Glass nozzle inner diameter of 2 $\mu m$ and outer diameter of 2.66 $\mu m$ <b>Solution deposition rate:</b> Si <b>Nozzle-to-substrate distance:</b> 20, 30, 40 $\mu m$ <b>Substrate composition:</b> <b>Applied voltage:</b> 500 to 400V in 25V increments <b>x-y stage velocity:</b> 0.01-10mm/s <b>Fiber Diameter:</b> about 60-170 $\mu m$ <b>Distance between adjacent fibers:</b> <i>Not determined</i>
Poly(ethylene oxide) (PEO)	<i>Not determined</i>	Typical process	NFES	<b>Solution Concentration:</b> 3wt% PEO [20] <b>Nozzle:</b> <i>Not determined</i> <b>Solution deposition rate:</b> <i>Not determined</i> <b>Nozzle-to-substrate distance:</b> 500 $\mu m$ <b>Substrate composition:</b> Si <b>Applied voltage:</b> 1000V <b>x-y stage velocity:</b> 20cm/s <b>Fiber Diameter:</b> 300nm <b>Distance between adjacent fibers:</b> 25 $\mu m$

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<i>Table 1 continued</i>				
Poly(ethylene oxide) (PEO)	Distilled water		Multinozzle NFES	<b>Solution Concentration:</b> 5wt% [21] <b>Nozzle:</b> four-nozzle and six-nozzle array with needle spacing changes from 1.5mm to 3.5mm <b>Solution deposition rate:</b> 1-3 $\mu$ L/min <b>Nozzle-to-substrate distance:</b> 2mm <b>Substrate composition:</b> Not determined <b>Applied voltage:</b> 1.7-2.7kV <b>x-y stage velocity:</b> Not determined <b>Fiber Diameter:</b> 5.47 $\mu$ m <b>Distance between adjacent fibers:</b> 3-5 mm
Poly(ethylene oxide) (PEO)	Distilled water		Multinozzle NFES	<b>Solution Concentration:</b> 5wt% [22] <b>Nozzle:</b> Dual-28G-needle array with needle inner diameter of 0.18mm and outer diameter of 0.36mm; with needle spacing changes from 2.0mm to 3.0mm <b>Solution deposition rate:</b> 0.2 $\mu$ L/min <b>Nozzle-to-substrate distance:</b> 3.0-4.0mm <b>Substrate composition:</b> Not determined <b>Applied voltage:</b> 2.0-3.0kV <b>x-y stage velocity:</b> 20mm/s <b>Fiber Diameter:</b> Not determined <b>Distance between adjacent fibers:</b> 218-326 $\mu$ m

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<i>Table 1 continued</i>				
Poly(ethylene oxide) (PEO)	Distilled water		Multinozzle NFES	<p><b>Solution Concentration:</b> 5 wt% [23]</p> <p><b>Nozzle:</b> Dual-28G-needle array with needle inner diameter of <math>180\mu m</math> and outer diameter of <math>360\mu m</math>; with needle spacing changes of <math>2.0mm</math></p> <p><b>Solution deposition rate:</b> <math>0.2\mu L/min</math></p> <p><b>Nozzle-to-substrate distance:</b> <math>4.0mm</math></p> <p><b>Substrate composition:</b> chromium-plated glass</p> <p><b>Applied voltage:</b> <math>2.5kV</math></p> <p><b>x-y stage velocity:</b> <math>20mm/s</math></p> <p><b>Fiber Diameter:</b> <i>Not determined</i></p> <p><b>Distance between adjacent fibers:</b> 2.3002- 2.7224mm</p>
Poly(ethylene oxide) (PEO)	<i>Not determined</i>	<i>deter-</i>	Typical process	NFES <p><b>Solution Concentration:</b> 2wt% [24]</p> <p><b>Nozzle:</b> G30 needle with inner diameter of <math>0.15mm</math></p> <p><b>Solution deposition rate:</b> <i>Not determined</i></p> <p><b>Nozzle-to-substrate distance:</b> 1-3mm</p> <p><b>Substrate composition:</b> Silicon</p> <p><b>Applied voltage:</b> 1250V</p> <p><b>x-y stage velocity:</b> <i>Not determined</i></p> <p><b>Fiber Diameter:</b> <i>Not determined</i></p> <p><b>Distance between adjacent fibers:</b> <math>20\mu m</math></p>

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<i>Table 1 continued</i>					
Gelatin (porcine skin)	Acetic and Acetate	Acid Ethyl	Typical process	NFES	<b>Solution Concentration:</b> 11wt% gelatin, 30wt% wa- ter, 35.4wt% acetic acid, 23.6wt% ethyl acetate <b>Nozzle:</b> 19G needle tip with outer diameter of 1.08mm  <b>Solution deposition rate:</b> <i>Not determined</i> <b>Nozzle-to-substrate distance:</b> 1.25mm <b>Substrate composition:</b> Poly(Dimethylsiloxane) (PDMS) films <b>Applied voltage:</b> 1000V <b>x-y stage velocity:</b> <i>Not determined</i> <b>Fiber Diameter:</b> around 2-3 $\mu$ m <b>Distance between adjacent fibers:</b> 40 $\mu$ m
Poly(ethylene ox- ide) (PEO)	Water/Ethanol (v/v = 60/40)		Typical process	NFES	<b>Solution Concentration:</b> PEO concentrations of 16% adn 18% <b>Nozzle:</b> 40 $\mu$ m <b>Solution deposition rate:</b> <b>Nozzle-to-substrate distance:</b> 1mm <b>Substrate composition:</b> Planar silicon <b>Applied voltage:</b> 1.7kV <b>x-y stage velocity:</b> 0.36m/s <b>Fiber Diameter:</b> 5.15 $\mu$ m <b>Distance between adjacent fibers:</b> <i>Not determined</i>

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<i>Table 1 continued</i>			
Poly(ethylene oxide) (PEO)	Water/Ethanol (v/v = 3/1)	Electrohydrodynamic Direct-Write (EDW)	<p><b>Solution Concentration:</b> 14wt% PEO [27]</p> <p><b>Nozzle:</b> Stainless needle with inner diameter of 210<math>\mu m</math> and outer diameter of 400<math>\mu m</math></p> <p><b>Solution deposition rate:</b> 50<math>\mu L/h</math></p> <p><b>Nozzle-to-substrate distance:</b> 2mm</p> <p><b>Substrate composition:</b> Poly(ethylene terephthalate) (PET)</p> <p><b>Applied voltage:</b> 3kV</p> <p><b>x-y stage velocity:</b> 700mm/s</p> <p><b>Fiber Diameter:</b> 15-35<math>\mu m</math></p> <p><b>Distance between adjacent fibers:</b> 70<math>\mu m</math></p>
Poly(ethylene oxide) (PEO)	Deionized water	Mechano-Electrospinning	<p><b>Solution Concentration:</b> 3wt% PEO [28]</p> <p><b>Nozzle:</b> Stainless steel nozzle with inner diameter of 160<math>\mu m</math> and outer diameter of 310<math>\mu m</math></p> <p><b>Solution deposition rate:</b> 50nL/min</p> <p><b>Nozzle-to-substrate distance:</b> 2-5mm</p> <p><b>Substrate composition:</b> Silicone</p> <p><b>Applied voltage:</b> polymer jet initiated at 2kV and dispensed at 1kV</p> <p><b>x-y stage velocity:</b> 200-400mm/s</p> <p><b>Fiber Diameter:</b> from 344<math>\pm</math>32 to 214<math>\pm</math>27nm</p> <p><b>Distance between adjacent fibers:</b> Not determined</p>

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<i>Table 1 continued</i>				
Poly(co-Glycolic acid (PLGA)	Dimethyl Carbonate (DMC)	Tethered Electrohydrodynamic Spinning (TPES)	Pyro-	<b>Solution Concentration:</b> <i>Not determined</i> [29] <b>Nozzle:</b> nozzle-free <b>Solution deposition rate:</b> The drop reservoir is placed directly on a flat substrate <b>Nozzle-to-substrate distance:</b> Taylor's cone is focused and put in direct contact with the collector <b>Substrate composition:</b> Poly(tetrafluoroethylene) (PTFE) coated glass slide <b>Applied voltage:</b> pyro-electric field of between $2.7 \times 10^7$ V/m and $5.5 \times 10^7$ V/m <b>x-y stage velocity:</b> <i>Not determined</i> <b>Fiber Diameter:</b> 304.7nm <b>Distance between adjacent fibers:</b> <i>Not determined</i>
Poly(ethylene oxide) (PEO) with Tetrabutylammonium tetrafluoroborate (TBF) and SU-8 2002	N,N-Dimethylformamide (DMF)	Typical process	NFES	<b>Solution Concentration:</b> SU-8/PEO/TBF blend with 0.75wt% PEO, 1wt% TBF; the blend is diluted with 30vol% DMF $\mu m \mu m$ [6] <b>Solution deposition rate:</b> <i>Not determined</i> <b>Nozzle-to-substrate distance:</b> <i>Not determined</i> <b>Substrate composition:</b> Brass disk with a diameter of 38mm <b>Applied voltage:</b> 980V <b>x-y stage velocity:</b> <i>Not determined</i> <b>Fiber Diameter:</b> <i>Not determined</i> <b>Distance between adjacent fibers:</b> <i>Not determined</i>

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<i>Table 1 continued</i>				
Poly(ethylene oxide) (PEO)	Water:Ethanol (3:2)	Suspension	NFES	<b>Solution Concentration:</b> 14wt% PEO [30] <b>Nozzle:</b> stainless steel needle (25 G) with inner diameter of 0.25mm <b>Solution deposition rate:</b> 3nL/s <b>Nozzle-to-substrate distance:</b> between 0.5 and 10mm with 0.5mm increments <b>Substrate composition:</b> Planar silicon electrodes <b>Applied voltage:</b> 1.6kV <b>x-y stage velocity:</b> 50, 150, and 250mm/s <b>Fiber Diameter:</b> 300nm <b>Distance between adjacent fibers:</b> 0.1 and 0.5mm
Poly(ethylene oxide) (PEO)	Deionized water	Typical process	NFES	<b>Solution Concentration:</b> 10wt% PEO [31] <b>Nozzle:</b> 32G metal needle <b>Solution deposition rate:</b> (Jet impact speed of 5mm/s ) <b>Nozzle-to-substrate distance:</b> 0.5mm <b>Substrate composition:</b> p-type silicon wafer <b>Applied voltage:</b> 400V <b>x-y stage velocity:</b> 5mm/s <b>Fiber Diameter:</b> <b>Distance between adjacent fibers:</b> 50μm

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### 3. Polymer Solution and Process Parameters

### 4. Applications

### 5. Fiber Characterization

### 6. Conclusion

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