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# Experimental Study on Tip-Induced Electrospinning

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**Abstract:** Tip-induced electrospinning (TIE) is demonstrated to produce nanofibers with high throughput. Probe array was plunged into the plane polymer solution and then withdrawn quickly, and due to the effects of viscous force and the electric force, Taylor cones were generated from the position where the probes left, and then mass nanofibers were obtained on the collector. The experimental results show that the threshold voltage under different electrode distances increases with the increase of the concentration, and the mean throughput of polyethylene oxide (PEO) nanofibers is up to 1.961 2 g/h when the solution tank size is 142 mm × 50 mm and the applied voltage is 63 kV. And the maximum deposition error of nanofibers is within the range of 31.08%—43.23% on the condition that the applied voltage changes from 45 kV to 63 kV.

**Keywords:** electrospinning; tip-induced; nanofibers; high-throughput

# 基于针尖诱导的静电纺丝实验分析

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**摘 要:** 针尖诱导电纺技术(TIE)利用针尖阵列插入聚合物溶液表面后快速抽离，由于高压强电场和黏性力作用在液面抽离处诱导形成泰勒锥，进而在收集板上得到大量纳米纤维，实现纳米纤维的批量制造. 实验结果表明在不同电极间距下，电纺阈值电压随着溶液浓度的增加而增大. 当工作电压为63 kV，溶液槽尺寸为142 mm ×50 mm时，电纺聚氧化乙烯(PEO)溶液的纤维产量达1.961 2 g/h. 当工作电压从45 kV增加到63 kV时，纤维沉积均匀性变化范围为31.08% ~43.23%.

**关键词:** 静电纺丝; 针尖诱导; 纳米纤维; 高产量

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Nanofibers with high surface area to volume ratio, superb chemical and electrical characteristics, etc, have stimulated scientists to explore their properties and potential applications in battery separator [1-2], flexible electrode [3-4], filtration [5-6], fiber-based sensors [7-8], and tissue engineering scaffolds [9-10]. As a simple and well-established technique for fabricating nanofibers cheaply and continuously, electrospinning becomes a hotspot in the

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field of nanotechnology research. However, its ultra-low production rate prohibits its potential applications. To overcome this disadvantage, Yang *et al*[11] reported bubble-electrospinning, in which gas is injected into polymer solution to form multiple bubbles on solution surface and multiple jets travel toward the collector from the bubbles in the strong electric field, but the gas flow is required to be controlled accurately and the diameter of nanofibers varies greatly. Other methods like multi-jet array electrospinning and tipless electrospinning[12] were researched to increase production rate, but it is shown that with multi-jet array electrospinning, lots of tiny liquid droplets are easily formed on the collector because of the electric field interference between spinnerets[13], and tipless electrospinning needs periodical cleaning of spinneret electrode. All of those drawbacks make them hard to meet the demand of industrial production. Then, in this paper, tip-induced electrospinning (TIE)[14], which has low driven voltage, high output and is free of cleaning, is proposed as a potential technology for mass-production of nanofibers. In TIE, probe array is dipped into the surface of polymer solution and withdrawn periodically, Taylor cones are thus induced and then jets are issued from the top of Taylor cones due to the high electric field.

## 1 Experimental details

As illustrated in Fig. 1, the setup for tip-induced electrospinning consists of high-voltage source, grounded collector, exhaust fans, solution tank, probe array and control module. A 142 mm × 50 mm solution tank was placed in the center of the setup. The high-voltage source (ES80P-20W/DDPM, Gamma, America), whose anode and cathode were connected to the solution tank and the collector, respectively, was applied to supply high voltage ranging from 0 to 80 kV. The probe array was connected to the rotating arm controlled by control module. As illustrated in Fig. 2, probes were plunged into and withdrawn quickly from the plane polymer solution, and Taylor cones came into being from the position where the probes left, as described in Fig. 2(a). When the probe tips left about a few millimeters from the solution surface, the induced liquid cones was disconnected from the tips and then stretched to become jets in strong electric field, as shown in Fig. 2(b). Two fans were installed above the collector to exhaust and assist the deposition of the as-spun nanofibers. The ambient temperature, humidity and the diameter of probes is about (23 ± 2) °C, 45 ± 5% RH and 250 μm, respectively. Polyethylene oxide powder (PEO, average molecular mass = 300 000 g/mol, Dadi Fine Chemical Co., Ltd., China) dissolved in the mixture of deioned water and ethanol (the mass ratio of deioned water and ethanol was 3∶1) was used as working solution. The mass of nanofibers was measured by electronic balance (BS124, Sartorius Co., Germany) and the morphology of nanofibers was characterized by scanning electron microscopy (LEO-1530, Leo Electron Microscopy Ltd., Germany).

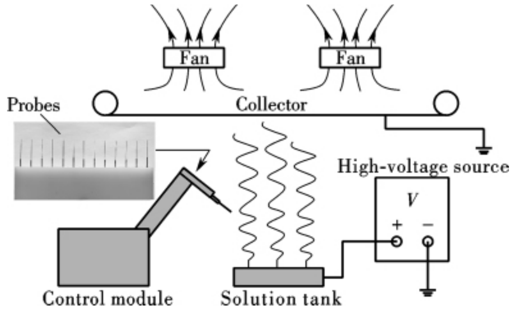
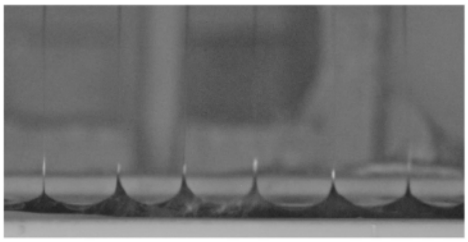
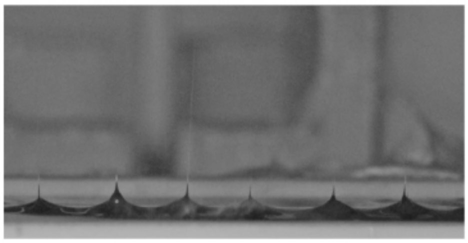



**Fig. 1 Schematic diagram of TIE setup**



(a) Probe array withdrawing from the solution surface



(b) Multiple jets induced from liquid cones

**Fig. 2 Optical view of TIE process**

## 2 Results and discussion

The effect of electrode distance on the threshold voltage is discussed first, in which threshold voltage is defined as the minimum applied voltage to sustain jet for more than 10 s in one induction cycle, and only one probe is used here to avoid the disturbance from other jets. The probe was withdrawn from plane solution at a speed of about 0.31 m/s. As illustrated in Fig. 3, when the solution concentration was 10% and the electrode distance increased from 20 cm to 40 cm, the threshold voltage of TIE changed from 25.5 kV to 30 kV. For the concentration of 14% and 16%, the threshold voltage varied from 37 kV to 46 kV and from 50 kV to 64 kV, respectively and the same trend can be achieved. It can be concluded that the threshold voltage increased with the increase of the concentration due to larger viscosity. Obviously, for longer electrode distance and higher concentration, higher applied voltage is needed.

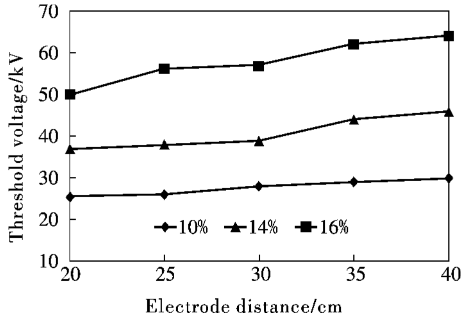



**Fig. 3　Curve of threshold voltage *vs* electrode distance at different concentrations of PEO solutions**

The throughput of the probe array (13 probes) was also explored under the condition that the induction interval of probe array was about 1.2 s and the probe array was withdrawn from plane polymer solution at a speed of about 0.37 m/s. The concentration of PEO solution and the electrode distance was 14% and 35 cm, respectively, to achieve good morpology of nanofibers. As depicted in Fig. 4, the mean throughput capacity after electrospinning for 30 min was increased from 0.386 8 g to 0.980 6 g when the applied voltage was changed from 45 kV to 63 kV. It reveals that the duration of jets sustained longer with the increasing voltage and the output increased sharply when the applied voltage was higher than 60 kV.

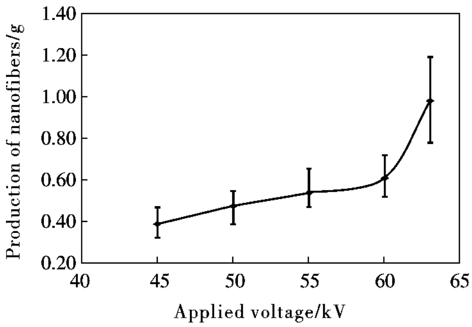



**Fig. 4　Curve of throughput of nanofibers *vs* applied voltage by electrospinning for 30 min** (The concentration of PEO solution is 14% and the electrode distance is 35 cm)

Six aluminum foils (100 mm × 100 mm) were attached onto the collector (734 mm × 724 mm) to estimate the deposition uniformity by the index of maximum deposition error, as shown in Fig. 5. The maximum deposition error can be calculated by Eq. (1).

$$\delta = \frac{\max\{ |m_A - \overline{m}_i| \}}{m_A} \times 100\% \qquad (1)$$

where $\delta$ is the maximum deposition error; $m_A$ is the average mass of the row; $\overline{m}_i$ is the average mass of nanofibers on aluminum foil, and $i$ is the number of aluminum foil (1—6). The maximum deposition error is listed in Tab. 1. Such deposition uniformity can be greatly improved by increasing the number of probes and the length of solution tank.

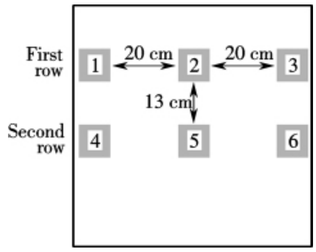
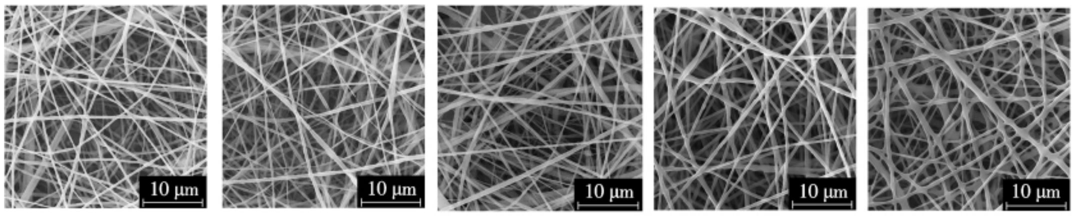



**Fig. 5　Schematic diagram of the arrangement of aluminum foils**

**Tab. 1　Maximum deposition error of nanofibers**

| Applied voltage/kV | The maximum deposition error/% | |
|---|---|---|
| | First row | Second row |
| 45 | 42.41 | 32.30 |
| 50 | 41.76 | 39.58 |
| 55 | 43.23 | 38.98 |
| 60 | 33.07 | 38.04 |
| 63 | 18.86 | 31.08 |

As shown in Fig. 6, when the applied voltage was 45 kV, 50 kV, 55 kV, 60 kV and 63 kV, the average diameter of nanofibers was 396 nm, 413 nm, 528 nm, 602 nm and 751 nm, respectively. And the diameter distribution of the nanofibers was 100—800 nm, 100—800 nm, 200—900 nm, 300—1 000 nm and 300—1 300 nm, respectively, as illustrated in Fig. 7. When the applied voltage was increased to 63 kV, many fibers contacted each other and formed intersections. The reason of such phenomenon is that when the applied voltage was increased, the stronger electric field induced largr diameter of Taylor cone and more instable movement of the as-spun nanofiber, which leads to insufficient evaporation of solvent.




(a) 45 kV (b) 50 kV (c) 55 kV (d) 60 kV (e) 63 kV

**Fig. 6 SEM images of nanofibers on No. 5 Al foil under different applied voltages** (The concentration of PEO solution is 14% and the electrode distance is 35 cm)

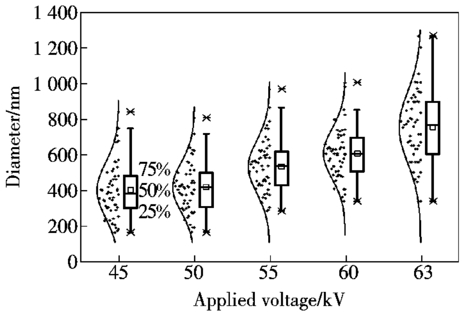



**Fig. 7 Diameter distribution of the electrospun nanofibers under different applied voltages** (The concentration of PEO solution is 14% and the electrode distance is 35 cm)

## 3 Conclusion

TIE experiments were conducted to investigate the throughput and the nanofiber deposition uniformity. The experimental results reveal that the threshold voltage under different electrode distances increases with the increase of the concentration. Its mean throughput of nanofibers is up to 1.961 2 g/h when the voltage is 63 kV and the maximum deposition error of nanofibers in the same horizontal direction is within the range of 31.08%—43.23%. The average diameter of nanofibers increases from 396 nm to 751 nm when the applied voltage varies from 45 kV to 63 kV. Higher voltage leads to larger average diameter and wider diameter distribution.

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