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Electrospinning Theory

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1.1 Nanotechnology and Nanofibers

Nanotechnology is a science and technology that studies the properties and applications of materials with structure sizes ranging from 1 to 100 nm. After the invention of scanning tunneling microscope in 1981, a molecular world with a length of 1 to 100 nm was born. The ultimate goal is to directly construct products with specific functions with atoms or molecules. Therefore, nanotechnology is a kind of technology that uses a single atom or molecule to manufacture material.

Nanotechnology is an interdisciplinary and comprehensive subject. The research content involves the broad field of modern science and technology. Nanoscience and technology mainly include seven relatively independent and mutually penetrating disciplines (nano-system physics, nanochemical, nanomaterials, nanobiology, nanoelectronics, nanoprocessing, and nanomechanics) and three research fields (nanomaterials, nanodevices, and nanoscale detection and characterization). The preparation and research of nanomaterials are the basis of the whole nanotechnology. Among them, nanophysics and nanochemistry are the theoretical basis of nanotechnology, and nanoelectronics is the most important content of nanotechnology.

1.1.1 Development History of Nanotechnology

In 1993, the first International Nanotechnology Conference (INTC) was held in the United States, which divided nanotechnology into six branches: nanophysics, nanobiology, nanochemistry, nanoelectronics, nano-processing technology, and nanometrology, which promoted the development of nanotechnology. Because of the particularity, magic, and universality of this technology, many outstanding scientists from all over the world have been working hard for it.

Nanotechnology generally refers to the technology of materials, design, manufacturing, measurement, control, and products at the nanometer level (0.1–100 nm).

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Nanotechnology mainly includes nano-level measurement technology, nano-level surface physical and mechanical properties detection technology, nano-level processing technology, preparation technology of nanoparticles, nanometer material, nanobiological technology, and nanoassembly technology.

In the 1970s, scientists began to put forward ideas about nanotechnology from different perspectives. In 1974, scientist Tony Gucci first used the term nanotechnology to describe precision machining.

In 1981, scientists invented the scanning tunneling microscope, an important tool for nanoresearch, which revealed a visible atomic and molecular world for us and played a positive role in promoting the development of nanotechnology.

In 1990, scientists at IBM’s Almaden Research Center successfully rearranged individual atoms, making a key breakthrough in nanotechnology.

In July 1990, the first International Conference on nanoscience and technology was held in Baltimore, the United States, marking the official birth of nanoscience and technology.

In 1997, American scientists successfully used single electrons to move single electrons for the first time. Using this technology, it is expected that in 20 years, a quantum computer with a speed and a storage capacity that is thousands of times higher than that of today will be successfully developed.

In 1999, scientists from Brazil and the United States invented the world’s smallest “scale” when conducting carbon nanotube experiments. It can weigh an object one billionth of a gram, which is equivalent to the weight of a virus. Soon after that, German scientists developed a scale capable of weighing individual atoms, breaking the record set jointly by American and Brazilian scientists.

By 1999, nanotechnology gradually entered the market, and the annual turnover based on nanotechnology products reached US$50 billion. Some countries have formulated relevant strategies or plans, and invested huge sums to seize the strategic heights of nanotechnology.

At present, the research and application of nanotechnology are mainly in materials and preparation, microelectronics and computer technology, medicine and health, aerospace and aviation, environment and energy, biotechnology, and agricultural products. The equipment made of nanomaterials has lighter weight, stronger hardness, longer service life, lower maintenance cost, and more convenient design. Nanomaterials can also be used to make materials with specific properties or materials that do not exist in nature, and to make biomaterials and biomimetic materials.

1.1.2 Introduction to Nanofibers

Nanofiber: refers to a linear material with a certain length-to-diameter ratio with a diameter of nanoscale and a large length. In addition, the fiber modified by filling nanoparticles into ordinary fibers is also called nanofiber. In a narrow sense, the diameter of nanofibers is between 1 and 100 nm, but in a broad sense, fibers with a fiber diameter of less than 1000 nm are called nanofibers.

At present, the thinnest nanofibers are single-carbon atom chains. Chinese scientists have been able to produce carbon nanotubes with a diameter of less than 0.4 nm, which is the leading level of the world. This kind of carbon nanotube is known as the king of nanomaterials. The reason is that this material, which is so thin that it is difficult to be observed by ordinary instruments, has magical abilities: super high strength, super flexibility, and strange magnetism. Due to the short spacing of carbon atoms and small diameter in carbon nanotubes, the fiber structure is not prone to defects. Its strength is 100 times that of steel, and its density is only 1/6 of that of steel, 200 times that of ordinary fibers. The rope made of carbon nanotubes can be pulled from the earth to the moon without being broken by its own weight; it has strange conductivity. Carbon nanotubes have both metal and semiconductor conductivity. Even different parts of a nanotube can show different conductivity due to structural changes. Using it to make rectifying tubes can replace silicon chips, which will cause great changes in electronics and make computers very small; nanodevices made of carbon nanotubes can be used to assemble nanorobots, mosquito planes, and tanks, etc. Carbon nanotubes can be used as hydrogen storage materials to develop hydrogen into clean energy for human services. In addition, carbon nanotubes can also be used as stealth materials, catalyst carriers, and electrode materials. Nanofibers can support the arrangement of "nanomachines" and connect the integrated and arranged "nanomachines" into a large-scale system.

1.1.3 Main Characteristics of Nanofibers

1. The smaller the size of surface effect particles, the larger the surface area. Because the surface particles lack the coordination of adjacent atoms, the surface energy becomes extremely unstable, and it is easy to combine with other atoms, showing strong activity.
2. Small size effect: when the size of particles is small enough to be close to or smaller than the wavelength of light wave, the de Broglie wavelength of conducting electrons, and the coherence length transmission depth of superconducting state, the periodic boundary conditions will be destroyed, and the acoustic, optical, electromagnetic, and thermodynamic properties of particles will be changed, such as melting point reduction, color separation and discoloration, ultraviolet absorption, and shielding electromagnetic wave.
3. Quantum size effect: when the particle size is small enough, the electronic energy level near the Fermi level changes from quasi-continuous to discrete. At this time, the substance that was originally a conductor may become an insulator, and conversely, the insulator may become a superconductor.
4. The macro-quantum Yang tunnel effect tunnel effect refers to small particles that can pass through objects under certain conditions, just like there is a tunnel inside.

1.2 Research History of Electrospinning

Electrospinning method is to apply electric field force to polymer solution or melt. Under the action of electrostatic field force, polymer jet is ejected and stretched into nanofiber in the process of movement. Electrospinning equipment mainly includes

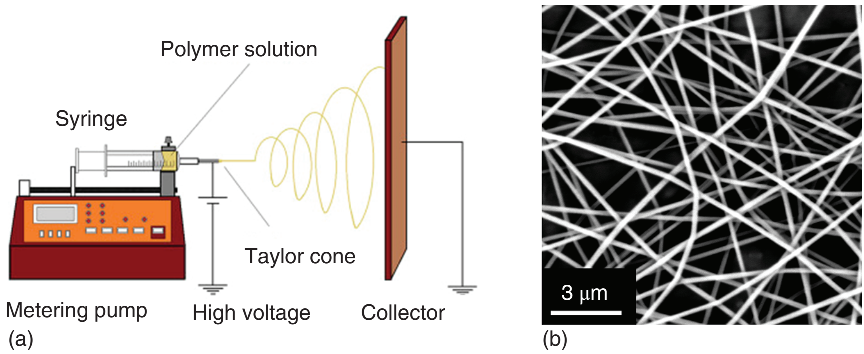

Figure 1.1 (a) Schematic of the electrospinning device. Source: Riazi et al. [1]. Reproduced with permission of Elsevier. (b) SEM image of nanofibers. Source: Xian Wen.

spinneret and solution supply device, high-voltage generator, and fiber collection device (Figure 1.1a). Apply high-voltage static electricity to the polymer solution or melt, and the charged droplets will deform under the action of electric field force and form Taylor cone at the top end of the spinneret. When the applied electric field force exceeds the surface tension of the solution, the polymer jet is ejected from the top of the Taylor cone. Under the action of electric field force and viscoelastic force of solution, the charged jet first moves along a straight line and then moves to a certain position. When it is disturbed by the outside world, it bends and deforms in a spiral curve. The jet is stretched and refined in the process of movement, and finally falls on the receiving device to form polymer nanofibers [2] (Figure 1.1b).

A brief history of the development of electrospinning and the number of publications of electrospinning in recent 2 decades are summarized in Figure 1.2. The predecessors invented the electrospinning technology on the basis of studying the water droplet morphology under the action of electric field force.

As early as 1600, William Gilbert [4] found that for a spherical water drop on a dry surface, a friction amber can be used to stretch it at an appropriate place to make the spherical water drop taper.

In 1882, Raleigh [5] theoretically studied the stability of independent droplets under the action of electrostatic field force. If the electric field force continues to increase, these droplets will become unstable; especially when the stable effect of charge and surface tension is broken, the droplets will split.

In 1899, Rayleigh [6] again studied the stability of liquid jet with and without electric field force, and proposed a linear theory of the stability of charged droplets. The theory shows that the spherical particle with uniform charge will keep its stable shape until the total charge on its surface exceeds the following critical value.

$$Q = (64\pi^2\varepsilon_0\gamma R)^{1/2}$$

where

Q is the total charge number of spherical droplets,
ε_0 is the vacuum dielectric constant,
γ is the surface tension of liquid, and
R is the radius of the droplet.

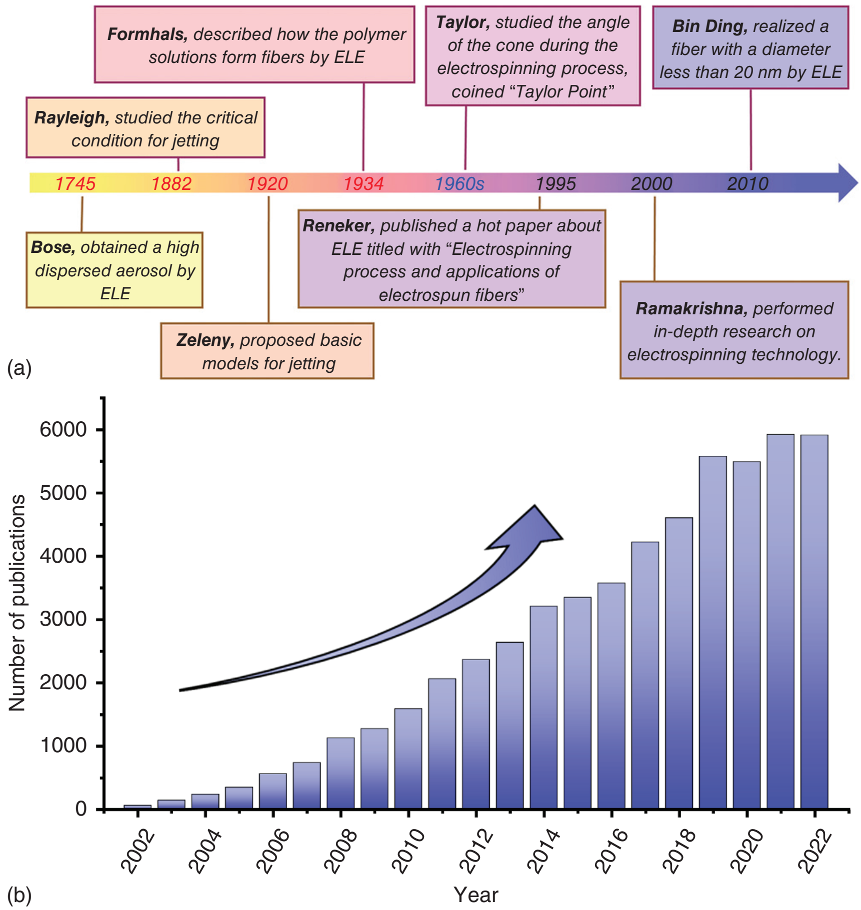

Figure 1.2 (a) Brief history and the development of electrospinning. Source: Shi et al. [3]. Reproduced with permission of John Wiley and Sons. (b) Number of publications in recent 2 decades (Data obtained from Web of Science by searching the keyword of "electrospinning"). Source: Xian Wen.

In 1917, Zeleny [7] studied experimentally the splitting phenomenon of the droplet at the end of the capillary and observed several different jet patterns. He also pointed out that the decomposition of the droplet was due to the instability of the droplet, not the formation of the surface charge of the droplet, and the critical voltage for the formation of the instability was:

$$V^2/r_0T = C$$

where

V is the applied voltage,
r_0 is the radius of liquid drop,
T is the surface tension of solution, and
C is the constant.

Taylor [4, 8] described in detail the morphological changes of conductive droplets under the action of electric field force from the theoretical and experimental aspects. The liquid drop at the top of the capillary tube will become a convex hemisphere. When a certain potential is applied to the surface of the droplet, the curvature of the conductive droplet surface will gradually change. When the potential reaches a certain critical value V_c, the conductive liquid can maintain a balanced cone shape under the action of the electric field force. The angle of its half cone top angle is 49.3°, and it is found that only when the angle of the half cone top angle is 49.3°, the conductive droplet can maintain a balanced cone shape. This charged cone is called Taylor cone. The critical potential value V_c is determined by the following formula:

$$V_c^2 = \frac{4H^2}{L^2}\left(\ln\frac{2L}{R} - \frac{3}{2}\right)(0.117\pi\gamma R)$$

where

H is the distance between capillary tube and receiving device,
L is the capillary length,
R is the capillary radius, and
γ is the surface tension of the liquid.

Taylor used the formula to describe the conical state of the liquid drop before the jet generated. There is a mathematical singularity at the top of the cone of the jet. In Taylor's research, only the electrostatic field force and surface tension received by the liquid drop are considered, while the influence of external electric field and fluid pressure on the Taylor cone shape and the redistribution of charge in the process of Taylor cone shape change are not considered. Therefore, Taylor's theoretical model is not applicable to the study of the morphology of equilibrium droplets in the electrospinning process. Spivak and Dzenis [9] analyzed the electric field force, fluid pressure, surface tension, and coulomb repulsion force on polymer droplets during electrospinning. It is assumed that the top of Taylor cone is a parabola shape, and the conditions required for the meniscus droplet to maintain its equilibrium state are studied, namely

$$\sigma \geq 0.447378 * \delta\emptyset\varepsilon_0 E$$

among them

σ is the surface tension coefficient of solution,
$\emptyset$ is the electrostatic field energy,
δ is the charge density carried by the droplet,
ε_0 is the medium permeability of vacuum, and
E is the applied voltage.

This formula also determines a maximum voltage to maintain the equilibrium state of the meniscus droplet, that is, the state of the meniscus droplet is stable only when the applied voltage is less than a certain value, which is determined by the medium permeability, surface tension, and the charge density carried by the jet. This assumption can avoid the mathematical singularity of the cone top.

Yarin et al. [10] found that for a specific self-similar solution, it will form a cone shape similar to the Taylor cone under the electric field force, while for a nonself similar solution, it will form droplets of other shapes under the electric field force. Yarin et al. assumed that the shape of Taylor cone top of nonself similar solution is hyperbolic and calculated the equipotential line with high curvature that is close to the cone top. The cone top angle calculated by hyperboloid shape is very small, and the semi-cone top angle deduced theoretically is 33.5°. This theory applies to non-viscous Newtonian fluid and viscoelastic fluid after stress relaxation. For non-viscous Newtonian fluids, this specific half-cone apex angle does not depend on the performance of the fluid, but on the viscoelasticity and surface tension of the solution.

Liu et al. [11, 12] established the mechanism of bubble electrospinning under the action of electric field force by using Maxwell's current mechanics equation, charge conservation equation, and fluid constitutive equation. In the process of bubble electrospinning, the bubble is subject to the applied electric field force, the surface tension of the solution, and the internal and external pressures of the bubble. The bubble gradually increases and changes from a circle to a cone. When the applied voltage exceeds the critical voltage, multiple jets are ejected from the top of the bubble cone. Liu approximates the bubble in two-dimensional state as a parabola and theoretically analyzes that the critical voltage for jet formation is:

$$E_c = \frac{k\gamma\sqrt{1+(\tan\overline{\alpha})^2}}{q\alpha_0\sqrt{1+4(\tan\overline{\alpha})^2\tan\overline{\overline{\alpha}}}}$$

where

E_c is the critical electric field strength,
q is the electric quantity per unit area,
γ is the surface tension of solution, and
α is the angle between the tangent line of any point on the bubble surface and the x-axis.

Therefore, in bubble electrospinning, the critical voltage of jet formation is related to the charge density of the bubble surface, the surface tension of the solution, the height of the bubble, and the radius of the bottom. When the height of the bubble is very low, that is, close to 0, the radius of curvature of the bubble is infinite, the liquid surface is approximately flat, and an infinite voltage is required for jet ejection, so electrostatic spinning cannot be carried out. In addition, Liu et al. [13, 14] also measured by experimental method that the critical spinning half cone apex angle formed by jet is 23.4°.

In the process of electrospinning, the change of Taylor cone shape and the formation of jet are the basis of electrospun nanofibers, and the surface tension of solution has a great influence on the change of Taylor cone shape and the formation of jet. Therefore, researchers, based on the change of Taylor cone shape under different surface tension conditions, further studied the critical voltage of jet formation under different surface tension conditions, and established a theoretical model between the surface tension of solution and the critical voltage between jet formation.

During electrospinning, after leaving the top of Taylor cone, the jet is accelerated due to the coulomb repulsion force. The coulomb repulsion force generated by the charge carried by the front part of the jet pushes the jet to move toward the receiver. Even though there is radial disturbance in practice, the liquid jet still maintains a straight line motion, which is called the stable section jet. Spivak et al. [15] proposed a model of steady-state jet in the electrospinning process. They assumed that the jet flow was axisymmetric along the direction of the external magnetic field, simplified the equation into a one-dimensional problem, and selected the dimensionless Weber number (Weber number) N_W, Reynolds number N_R, and Euler number (Euler number) N_E. The reciprocal of E takes the potential at a point in the jet as the sum of the potential generated by surface charges and external charges. The differential equation of jet radius R with respect to coordinate axis Z is obtained by solving one-dimensional differential equation:

$$\frac{\mathrm{d}}{\mathrm{d}Z}\left[R^{-4} + (N_W R)^{-1} - {N_E}^{-1}R^2 - {N_R}^{-1}\left(\frac{\mathrm{d}R^{-2}}{\mathrm{d}Z}\right)^m\right] = 1$$

where

$N_w = \frac{\rho Q^2}{2\pi^2 {R_0}^3 \sigma}$, which is the ratio function of inertial force and solution surface tension,

$N_E = \frac{4\varepsilon_0 \rho Q^4}{\pi^2 J^2 {R_0}^6}$, which is the ratio function of inertial force and pressure generated by electric field force,

$N_R = \frac{Q^2 \rho}{2\pi^2 {R_0}^4 \mu}\left[\frac{6\pi E J {R_0}^2}{Q^2 \rho}\right]^{-m}$, which is the ratio function of inertial force and viscous resistance of solution.

Holman et al. [16, 17], using the concept of leakage medium model, considered the free charge when establishing the jet dynamics model, established a slender body theory involving electric field force, charge transport and jet stretching, and further elaborated the influence of surrounding electric fields on the jet surface charge through the model. Taking the solution as Newtonian viscous fluid, the stretching model of electrospun jet was established and the stability mechanism of the jet in the stable section was studied. The stability of a charged jet in a tangential electric field with all fluid parameters is comprehensively analyzed by coupling the stretching of the jet, the transfer of polarized charges, and the external electric field. The axisymmetric instability and whip instability of the charged jet are further analyzed. However, he used the Newtonian fluid constitutive equation, so he could not predict the effect of solution viscoelasticity on the stability of the jet.

Feng [18] improved and extended the slender body model of Hohman, and adopted Giesekus viscoelasticity model, the solution of the model is extended to non-newtonian fluid, and a one-dimensional non-newtonian fluid stretching model is established. He also studied the changes of charge density and applied electric field intensity on the jet surface with the axial distance of the jet. The equation of the applied electric field intensity changing with the axial distance of the jet is:

$$E(z) = E\infty(\mathrm{z}) - \left[\frac{1}{\varepsilon_0}(\delta R)' - \left(\frac{\varepsilon}{\varepsilon_0} - 1\right)\frac{(ER^2)''}{2}\right]\ln\left(\frac{d}{R_0}\right)$$

where

E is the electric field strength,
Δ is the charge density,
R_0 is the initial jet diameter,
D is the spinning distance, and
E_∞ is the electric field strength.

Carroll et al. [19, 20], based on Feng's slender body theory, modeled and analyzed the stretching of isothermal polymer melt jet, introduced Oldroyd-B and FENE-P models related to fluid momentum equation and Gaussian law, and established the momentum equation of jet stretching:

$$\rho v v' = \rho g + \frac{F_{\mathrm{T}}'}{\pi R^2} + \frac{\gamma R'}{R^2} + \frac{\sigma\sigma'}{\varepsilon_0} + (\varepsilon - \varepsilon_0)EE' + \frac{2\sigma E}{R}$$

where

F_{T} is the tensile force,
E is the electric field strength,
Σ is the charge density,
R is the initial jet diameter, and
γ is the surface tension.

Wan et al. [21, 22] improved Spivak's model by adding charges due to strong electric fields. The three improved conservation equations for the influence of polarization on electrospinning are as follows:

Charge conservation equation:

$$\frac{\partial q_{\mathrm{e}}}{\partial t} + \nabla \cdot j = 0$$

Force balance equation:

$$\rho\frac{\mathrm{d}u}{\mathrm{d}t} = \nabla \cdot t + \rho f + q_{\mathrm{e}}E + (\nabla E) \cdot p$$

Energy conservation equation:

$$\rho c_p \frac{\mathrm{d}T}{\mathrm{d}t} = Q_{\mathrm{h}} + \nabla \cdot q + J \cdot E + E \cdot \frac{\mathrm{d}p}{\mathrm{d}t}$$

where

$\nabla \cdot t$ is the viscous resistance,
Pf is the gravity,
$q_{\mathrm{e}}E$ is the electric field force, and
$(\nabla E) \cdot p$ is the polarization force.

He et al. [23] studied the critical length and critical radius of the electrospun straight section jet according to the force on the jet in the stable section during the electrospinning process, using the mass conservation equation, the charge conservation equation, the N–S momentum conservation equation, and the famous Cauchy inequality:

$$L = \frac{4kQ^3}{\pi\rho^2 I^2}\left(R_0{}^{-2} - r_0{}^{-2}\right)$$

$$R_c = \frac{r_0}{\sqrt{\beta r_0{}^2 L + 1}}$$

where

$$R_0 = \left(\frac{2\sigma Q}{\pi k\rho E}\right)^{1/3}$$

L is the critical length of straight section jet and
R_c is the critical radius of jet in straight section.

1.3 Development Prospect of Electrospinning

1.3.1 Application of Electrostatic Spinning Technology

With the development of nanotechnology, electrospinning, as a simple and effective new processing technology that can produce nanofibers, will play a huge role in biomedical materials, filtration and protection, catalysis, energy, photoelectric, food engineering, cosmetics, and other fields (Figure 1.3)[24].

1. In the biomedical field, the diameter of nanofibers is smaller than cells, which can simulate the structure and biological function of natural extracellular matrix; most human tissues and organs are similar to nanofibers in form and structure, which makes it possible for nanofibers to be used for tissue and organ repair; some electrospun materials have good biocompatibility and degradability, which can be used as carriers to enter the human body and be easily absorbed; in addition, electrospun nanofibers also have large specific surface area, porosity, and other excellent characteristics. Therefore, they have attracted continuous attention of researchers in the biomedical field, and have been well applied in drug-controlled release, wound repair, biological tissue engineering, and other aspects.
2. The filtration efficiency of fiber filter materials will increase with the decrease of fiber diameter. Therefore, reducing the fiber diameter becomes an effective method to improve the filtration performance of fiber filter materials. In addition to small diameter, electrospun fibers also have the advantages of small pore size, high porosity, and good fiber uniformity, which makes them show great application potential in the fields of gas filtration, liquid filtration, and personal protection.
3. Electrospun fiber can effectively control the fine structure of fiber. Combined with materials with low surface energy, it can obtain materials with super

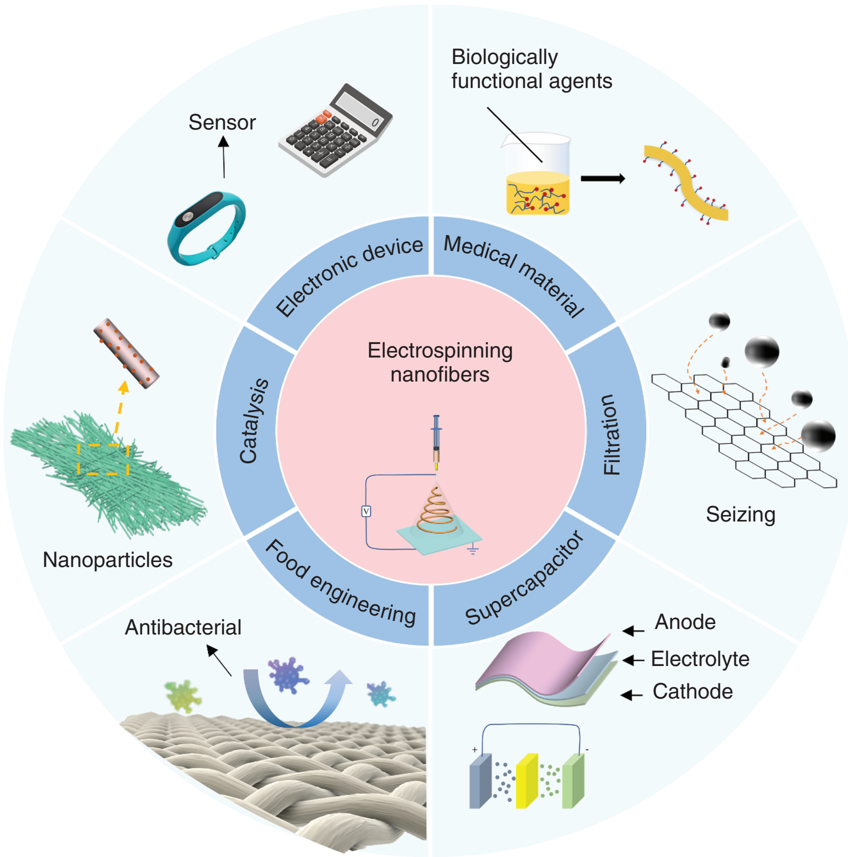

Figure 1.3 Applications of electrospinning nanofibers. Source: Xian Wen.

hydrophobic properties, and is expected to be applied to the shell of ships, the inner wall of oil pipelines, high-rise glass, automotive glass, etc. However, in order to realize the application of electrospun fiber materials in the above self-cleaning field, it is necessary to improve its strength, abrasion resistance, and the combination fastness of fiber membrane materials and matrix materials.

4. Catalyst particles with nanostructures are easy to agglomerate, thus affecting their dispersibility and utilization. Therefore, electrospun fiber materials can be used as templates to uniformly disperse, and also play a role in flexibility and operability of polymer carriers. In addition, strong synergistic effects can be generated by the composite of catalytic materials and polymer micro nanosize surfaces to improve catalytic efficiency.
5. Electrospun nanofibers have high specific surface area and porosity, which can increase the interaction area between the sensing material and the detected object, and are expected to greatly improve the sensor performance. In addition, electrospun nanofibers can also be used in energy, photoelectric, food engineering, and other fields.

1.3.2 Development Direction of Electrospinning Technology

Electrospinning technology has played a very important role in the field of building one-dimensional nanostructural materials. The application of electrospinning technology has successfully prepared nanofiber materials with various structures. Through different preparation methods, such as changing the nozzle structure and controlling the experimental conditions, the solid, hollow, core shell structure of microfiber or spider web structure of two-dimensional fiber membrane can be obtained; by designing different collecting devices, single-fiber, fiber bundle, highly oriented fiber, or randomly oriented fiber membrane can be obtained. However, electrospinning technology still faces some challenges in fiber structure regulation: first, in order to achieve the industrial application of electrospun fibers, it is necessary to obtain nanofiber bundles similar to staple fibers or continuous fibers. The preparation of oriented fibers provides an effective way to solve this problem, but there is still a lot of gap from the goal. Future work should try to improve the nozzle, the receiving device, and the addition of auxiliary electrodes to make the fibers as straight and oriented as possible to obtain an array of oriented fibers with excellent comprehensive performance. Secondly, as a new research field of electrospun nanofibers, the research of nanocobweb is still in its early stage, and the theoretical analysis and model establishment of the formation process of nanocobweb need to be further studied. In addition, in order to improve the application performance of electrospun fiber membrane in the field of ultra fine filtration, it is necessary to reduce the fiber diameter. How to reduce the average fiber diameter to less than 20 nm is a challenge for electrospun technology. In order to improve the application performance of fibers in sensor, catalyst, and other fields, it is an effective method to improve the specific surface area of fibers by preparing nanofibers with porous or hollow structures, but further research is still needed.

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