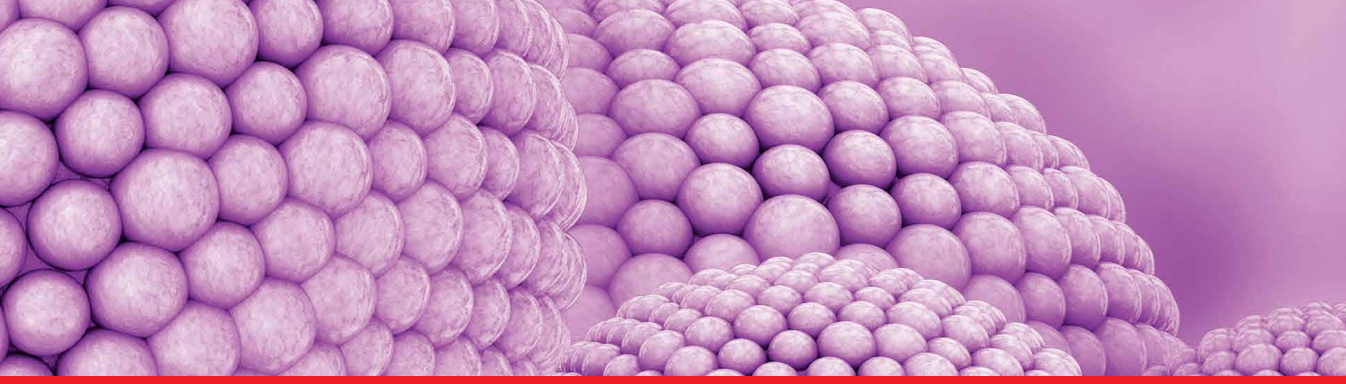


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Advances in Nanofiber Research

Properties and Uses

Edited by Sadia Ameen, M. Shaheer Akhtar and Ing Kong



Advances in Nanofiber Research - Properties and Uses

Edited by Sadia Ameen, M. Shaheer Akhtar and Ing Kong

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Nanotechnology and Nanomaterials

Volume 7

Aims and Scope of the Series

Humans face growing challenges in environmental sustainability. We need better materials, more effective devices, and higher computing power to conquer these challenges. Researchers have long explored the realm of science at the nanometer scale for developing novel problem-solving technologies. In the past decades, breakthroughs in multiscale computer simulation, novel hierarchical material and device structures, nanometrology, and new functionalities from interfacial and quantum size effects have enhanced our ability to utilize nanotechnology to solve real-world problems. This book series addresses important advancements in nanotechnology and nanomaterials. It showcases how nanotechnology is being continually developed and implemented in a variety of domains of science and technology. The two main topics this series covers are Nanotechnology and Nanodevices, and Nanomaterials and Nanostructures.

Meet the Series Editor



Jung Y. Huang, a university educator and researcher, has been working to unravel the structures and functional properties of materials and cellular events in living cells with varying optical methodologies. He has co-authored hundreds of journal papers and five book chapters. He also holds tens of patents in laser techniques and single-molecule/hyperspectral imaging and has developed architectural photonics based on hierarchically structured materials. Currently, his research focuses on the integration of artificial intelligence methodology with optics to automatically discover meaningful information from optical sensing/imaging data cubes. He has been an editorial board member and reviewer for several scientific journals. As a member of the global scientific community, he sincerely supports and endeavors to promote the spread of scientific knowledge.

Meet the Volume Editors



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Preface

The field of nanotechnology has made tremendous strides over the past few decades, reshaping industries ranging from medicine to electronics. Among the most promising and versatile materials to emerge from this field are nanofibers, which exhibit unique properties owing to their high surface area-to-volume ratio, exceptional mechanical strength, and remarkable electrical and thermal conductivity. As these fibers continue to be explored, their potential applications have expanded, offering innovative solutions to long-standing challenges in various sectors.

Advances in Nanofiber Research – Properties and Uses serves as a comprehensive resource that highlights the latest developments and findings in the field. It delves into the fundamental properties of nanofibers, the processes used for their fabrication, and the wide array of their applications. It brings together the work of leading researchers and industry experts to provide an in-depth understanding of how nanofibers can be engineered for specific functionalities, ranging from biological microlenses and nanofibers and engineering thermoset-containing electrospun nanofibers to characterization methods for nanofibers.

The chapters in this book are organized to offer both theoretical insights and practical applications, making it an invaluable reference for scientists, engineers, and students alike. It covers topics such as application of high-temperature superconductors in nanotechnology, characterization methods to assess their structural and functional properties, and real-world applications that demonstrate their potential impact. Special emphasis is given to emerging trends as well as advances in scalable production methods.

By bringing together this diverse work, we hope to provide readers with a deeper appreciation for the possibilities that nanofibers offer in driving future technological advances. We extend our gratitude to the contributors whose expertise and dedication have made this compilation possible. We extend our heartfelt gratitude to IntechOpen for their unwavering support and dedication in bringing this book to life. Their professionalism, expertise, and commitment to quality have been instrumental in transforming our vision into a published work. We hope that this book will inspire further innovation and collaboration in the exciting world of nanofiber research.

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Chapter 1

Architecting Performance: High-Performance Design Principles for Nanofibers

Ghazal Saki Norouzi

Abstract

This chapter offers a comprehensive overview of electrospinning, a versatile technique for fabricating nanofibers with exceptional properties. It delves into the intricate mechanisms governing fiber formation, emphasizing the importance of understanding these processes to optimize fiber characteristics. The chapter discusses the influence of polymer solution properties, processing variables, and environmental conditions on fiber morphology and performance. It explores innovative strategies for creating hierarchical nanostructures, primary and secondary structures, which significantly enhance the functional properties of nanofibers. Post-modification techniques, including chemical modification, physical modification, and nanoparticle incorporation, are also discussed further to tailor the properties of nanofibers for specific applications. The chapter highlights the significance of modeling and optimization methods in achieving desired nanofiber characteristics, ultimately enabling their use in various fields, such as tissue engineering, drug delivery, filtration, and energy applications.

Keywords: electrospinning, high-performance nanofibers, hierarchical structures, tailored properties, modeling

1. Introduction

Nanofibers have emerged as a focal point in nanotechnology with their extraordinary properties. Electrospinning, a technique that enables the creation of continuous fibers with diameters measured in nanometers, has revolutionized the production of these ultrafine structures. This method's simplicity and efficiency have made it a cornerstone in manufacturing nanomaterials [1, 2].

By carefully controlling the electrospinning process, researchers can create nanofibers with specific shapes, internal structures, and properties [3–5]. This precise control opens up a vast array of potential applications. Numerous factors influence the formation of these nanofibers, and by expertly managing these variables, scientists can produce nanofibers with desired characteristics, such as size and shape [2, 3]. **Figure 1** visually represents the various factors that influence the device's performance.

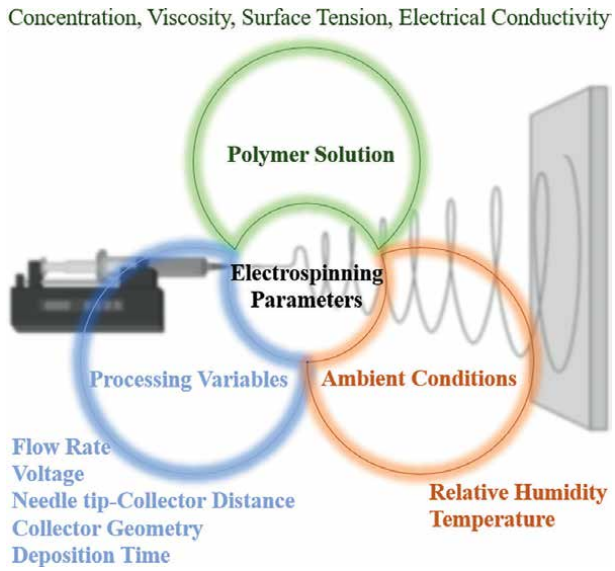


Figure 1.
Effective parameters on the Electrospinning process.

The properties of a polymer solution play a crucial role in determining the characteristics of the nanofibers produced from it. Concentration, viscosity, electrical conductivity, and surface tension significantly impact the final product.

Polymer concentration is crucial for production; insufficient concentration can lead to fiber breakage or particle formation. Each polymer's minimum concentration varies, which can affect other variables. Viscosity, influenced by concentration, surfactants, and polymer molecular weight, is essential for forming the Taylor cone. Too high viscosity hinders the spinning process. Polymers with lower molecular weight or a more linear structure can be used to reduce the viscosity. Surface tension, determined by polymer interactions, impacts fiber formation. Surfactants can reduce surface tension, aiding in fiber stability. Electrical conductivity is vital due to the electromagnetic forces involved. Organic solvents often require salt additives to enhance conductivity. Balancing salt concentration and solvent evaporation is crucial [6–8].

Various processing variables heavily influence the electrospinning process. These factors include the flow rate of the polymer solution, the applied voltage, the distance between the needle tip and collector, the collector's shape, and the deposition time. When using a rotating drum as the collector, its speed becomes a significant variable [2, 6].

The rate at which the polymer solution is dispensed, determined by the syringe pump, is critical. An insufficient flow rate can disrupt the formation of the Taylor cone, which is essential for jet formation. Conversely, excessive flow rate can lead to droplet formation, affecting fiber quality. The distance between the needle and collector directly impacts fiber diameter [9]. As this distance increases, the polymer jet experiences more stretching, resulting in thinner fibers. However, excessive distance can interfere with the electromagnetic field, hindering fiber deposition. The applied voltage is the primary force driving the electrospinning process [10]. A voltage, flow rate, and distance balance are necessary to achieve optimal results. Lower voltage and shorter distances generally produce thinner fibers, while higher voltage allows for increased distance and flow rate without droplet formation. Collector design also plays a crucial role in fiber morphology [11]. Stationary collectors produce randomly

oriented fibers while rotating collectors often yield aligned fibers. Various collector geometries have been explored to manipulate fiber orientation and pattern, including knife-edge, grid, and electrode-based designs. Deposition time is another influential factor. Shorter deposition times generally lead to longer and thinner fibers, but excessive time can cause jet instability [10].

Environmental factors play a significant role in the electrospinning process. Relative humidity and ambient temperature are particularly influential in determining the characteristics of the resulting nanofibers.

High relative humidity can hinder solvent evaporation from the polymer solution, leading to fiber deformation or re-dissolution due to the high surface area of nanofibers. Some biopolymers are susceptible to moisture absorption under high humidity, further complicating the process. While high humidity can create porous fibers by inducing water droplet condensation, it is generally advisable to maintain low humidity levels. Ambient temperature influences solvent evaporation rates. Organic solvents typically evaporate readily at room temperature, but aqueous solutions benefit from increased temperature to accelerate evaporation. Temperature also affects polymer solution viscosity. Lower viscosity enhances ion mobility, increasing electrical conductivity and potentially reducing fiber diameter [1, 12, 13].

Havlíček et al. [14] investigated the influence of the electrospinning technique on the morphology of polyvinyl butyral and polyurethane nanofibers. Five distinct methods were compared: AC electrospinning and four DC variants (rod, Nanospider™, needle, and centrifugal). Results indicated pronounced morphological disparities between nanofibers produced by different techniques. AC electrospinning yielded predominantly fine, disordered nanofibers with elevated porosity. Conversely, DC methods produced nanofibers with varying characteristics. Nanospider™ generated nanofibers of uniform thickness, while centrifugal spinning produced highly oriented fibers exhibiting significant surface roughness. Zhu et al. [15] investigated the determinants of polyvinylidene fluoride (PVDF) nanofiber. A systematic study was conducted by varying solution concentration, cartridge speed, voltage, electrode distance, rotating wire speed, winding speed, and slit diameter. Results indicated that solution concentration exerted the most significant influence on both nanofiber morphology and diameter. While cartridge speed, voltage, electrode distance, and slit diameter showed some correlation with fiber diameter, these effects were less pronounced due to substantial data variability. Rotating wire speed and winding speed exhibited negligible impact on fiber diameter. Yalcinkaya [16] conducted a comparative analysis of nine distinct polymers and their combinations to optimize nanofiber properties. Polymer blends demonstrated superior performance, enhanced adhesion properties, increased production efficiency, and reduced fiber diameter. Aghasiloo et al. [17] investigated the impact of relative humidity on the fabrication of electrospun TiO_2/ZnO/PVP (Polyvinylpyrrolidone) nanofibers. Results indicated that elevating relative humidity from 50 to 65% resulted in a decrease in fiber diameter attributed to polymer solution dilution during the spinning process and enhanced electrospinning jet elongation, consequently increasing specific surface area. Further increasing relative humidity to 80% led to a counterintuitive increase in fiber diameter due to excess water molecules between the needle tip and collector, attenuating the magnetic field. Additionally, elevated humidity impeded solvent evaporation, contributing to increased fiber diameter and specific surface area. Silva Vaz et al. [18] investigated the influence of process parameters on the fabrication of polyacrylonitrile (PAN) nanofibers using N, N-dimethylformamide (DMF) as a solvent. Capillary diameter emerged as a critical factor affecting fiber diameter, with

a statistically significant reduction observed from 472 ± 120 to 364 ± 63 nm as the capillary diameter decreased from 0.80 to 0.45 mm. These findings underscore the importance of precise capillary dimensions in controlling nanofiber morphology for tailored applications. Hajikhani et al. [19] found the incorporation of polylactic acid (PLA) into PVP/PEO (Poly(ethylene oxide)) nanofibers resulted in enhanced fiber porosity due to the differential evaporation rates of the polymers. This morphological modification was attributed to the disparate chemical properties of the polymers, which influenced surface tension and phase separation during the electrospinning process. Studies have shown that increasing the proportion of DMF in PLA solution containing dichloromethane (DCM) reduces fiber diameter. This effect is attributed to DMF's ability to enhance jet elongation through increased electrical conductivity, vapor pressure, and dielectric constant. However, excessive DMF can induce PLA precipitation due to its insolubility in this solvent, limiting its applicability [19, 20]. Selective removal of hydrophilic polymers from electrospun scaffolds offers a promising approach to enhancing porosity. By incorporating water-soluble polymers like PVP or Polyvinyl alcohol (PVA) into the electrospinning solution and subsequently subjecting the fibers to aqueous or alkaline treatments, researchers have successfully eliminated these components, resulting in highly porous structures. This technique demonstrates the potential for tailoring nanofiber architecture and optimizing material properties for specific applications [17, 21].

This study aims to investigate the intricate factors influencing nanofiber formation through electrospinning comprehensively. By systematically exploring the effective parameters, it seeks to understand the mechanisms governing nanofiber morphology and performance. This knowledge will optimize electrospinning techniques for fabricating nanofibers with tailored properties, opening up new avenues for applications in diverse fields.

2. Hierarchically electrospun nanofibers

Electrospinning, a versatile technique for fabricating continuous nanofibers, has garnered significant attention due to the unique properties imparted by its nanoscale dimensions. While conventional electrospun fibers exhibit promising characteristics, integrating hierarchical structures offers an avenue to enhance their performance further. Hierarchical morphologies, characterized by multiple structural levels, introduce additional complexity and functionality to nanofibers. These structures can be engineered through precise control of electrospinning parameters or by incorporating supplementary fabrication steps [22].

Incorporating hierarchical architectures has been shown to augment the properties of electrospun nanofibers in several ways. Notably, increased surface area, porosity, and structural stability are commonly observed in these materials. Moreover, the anisotropic nature of specific hierarchical structures enables the development of multifunctional materials with tailored properties [22, 23]. By understanding the underlying principles and fabrication methodologies, researchers can harness the potential of hierarchical nanofibers for a wide range of applications.

2.1 Hierarchical primary structures

Primary electrospun fibers are created in a single step using specialized techniques. These techniques involve manipulating the spinning solution or the

electrospinning process itself. Researchers can produce fibers with intricate structures by altering factors like the spinneret design, the type of solution, or the electrospinning conditions [23].

These structures range from simple to complex. The challenge often lies in controlling the behavior of multiple polymers within a single solution to achieve the desired structure. Combining different polymers offers advantages. For instance, it allows for incorporating materials with varying electrospinning properties [22, 23].

2.1.1 Core-shell fibers

Core-shell fibers, characterized by a distinct core surrounded by a shell, have emerged as a promising material with a wide range of applications (**Figure 2**). This structure provides unique properties and functionalities, making it a focus of significant research in various fields. The core and shell materials can vary widely, offering opportunities to tailor the fiber's properties.

Fabrication methods for core-shell fibers primarily include:

- Template Deposition: Involves coating a pre-existing fiber with the desired shell material [25].

- Immiscible Polymer Blends: Electrospinning a mixture of incompatible polymers can lead to phase separation, resulting in a core-shell structure. However, this method is relatively uncommon [26].

- Emulsion Electrospinning: A single nozzle is used to electrospinning a stable emulsion. The rapid solvent evaporation induces phase separation, forming the core-shell structure. This method is versatile and suitable for various applications [27, 28].

- Coaxial Electrospinning: This precise technique employs concentric spinnerets for the core and shell solutions, offering excellent control over the structure [28, 29].

Modifications to this method have led to further advancements, such as creation of hollow fibers. Removing the core fibers after calcination usually results in the desired hollow structure. Hollow core-shell fibers can be made using various methods. Zhang

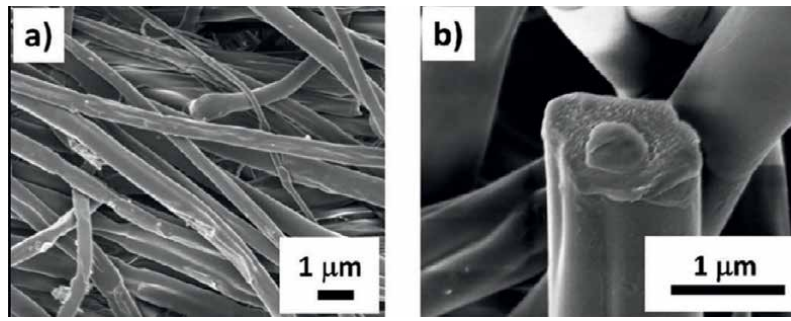


Figure 2.
Scanning electron microscopy (SEM) images of (a) electrospun oligothiophene-b-oligo phenylene vinylene (OT-b-OPV)/PVP core-shell nanofibers and (b) high magnification image of the same material showing a nanofiber edge exhibiting a core-shell morphology [24].

et al. [30] developed a technique involving coaxial electrospinning, using mineral oil as a temporary core and a combination of phosphomolybdic acid and polyacrylonitrile for the outer layer. This core is later removed. Alternatively, Huang et al. [31] used a phase separation approach, where specific solvents cause pores to form within the fiber structure. Hollow fibers have potential applications in sensing. Choi et al. [32] created hollow zinc oxide fibers more sensitive to nitrogen dioxide gas than traditional flat sensors. Gao et al. [33] produced hollow polyaniline fibers for enhanced trimethylamine gas detection. Park et al. [34] combined titanium dioxide and zinc oxide in core-shell fibers for oxygen sensing, while Zhan et al. [35] produced hollow titanium dioxide fibers with adjustable properties.

The core and shell materials need to be compatible with the electrospinning process, and the ratio between the core and shell material plays a crucial role in determining the fiber's final properties. Additionally, for methods like coaxial electrospinning, choosing suitable solvents for both materials is essential [36–38].

Core-shell fibers offer several advantages, including precise control over their structure, the ability to encapsulate materials, and the flexibility to choose from a wide range of core and shell materials. However, the complexity of fabrication methods presents a challenge that needs to be addressed [31, 32].

Despite these hurdles, core-shell fibers represent a dynamic area of research with immense potential. As fabrication techniques continue to advance and new material combinations are explored, core-shell fibers are expected to revolutionize applications across diverse industries [38].

2.1.2 Side-by-side fibers

Side-by-side (SBS) electrospun nanofibers offer an exciting alternative to coaxial core-shell fibers, particularly for multi-fluid applications [39–41]. As the name suggests, SBS fibers are essentially two fibers in one, with both surfaces exposed to the environment (**Figure 3**). They are fabricated similarly to conventional electrospinning but with a crucial twist—special side-by-side spinnerets are used for the different polymer solutions [41].

In contrast to some distinct advantages, SBS electrospinning remains a less explored territory compared to other nanofiber techniques. These advantages include [43, 44]:

- Tailored Surface Chemistry presents hydrophilic and hydrophobic surfaces within a single fiber structure. This dual functionality provides unprecedented opportunities for developing materials with tailored surface properties and advanced applications.

- Precise Drug Dosing: SBS allows easy control over the amount of drug loaded within the fiber.

- Co-culturing cells: Scientists can use SBS fibers to culture different cell types near, mimicking natural environments.

Despite the potential advantages of SBS electrospinning, the technique has faced significant challenges in widespread adoption. A primary obstacle is the inherent electrostatic repulsion between the two polymer solutions during the electrospinning process. This phenomenon, arising from the solutions' similar charge, hinders the formation of stable and uniform SBS fibers. Researchers have implemented various

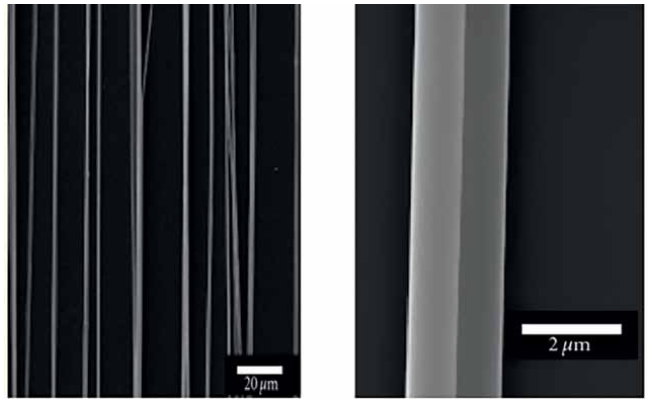


Figure 3.
Two-dimensional SEM images of well-aligned and uniform side-by-side TiO2/SnO2 nanofibers provide a comprehensive scheme of the detailed microstructure (the right-side picture) [42].

modifications to the electrospinning setup to address this issue, primarily focusing on strategies to counteract the electrostatic repulsion between the polymer jets [41, 45].

Despite the current challenges, SBS electrospinning presents a promising avenue for creating multifunctional fibers with tailored properties, and ongoing research efforts are expected to expand the capabilities of this technique [43]. Lee et al. [39] introduced a promising new type of luminescent sensor with potential applications in environmental monitoring and water quality control. They developed a dual-sensor system for detecting Fe^{3+} and Cu^{2+} ions in aqueous solutions. The core innovation lies in the fabrication of SBS nanofibrous membranes incorporating europium (Eu) and terbium (Tb) metal-organic frameworks (MOFs).

2.1.3 Honeycomb structure

Inspired by the architectural marvel of beehives, honeycomb structures have emerged as a promising concept in electrospinning. Unlike complex fabrication methods required for core-shell or side-by-side nanofibers, honeycomb fibers self-assemble into a highly ordered hexagonal network during electrospinning (**Figure 4**). This self-assembly phenomenon is attributed to the influence of electric field forces on the polymer solution jet, forming distinct thicker and thinner fiber populations within the structure [23, 47–49].

Several factors, including polymer concentration, working distance, deposition time, and electric field strength, influence the formation and characteristics of honeycomb fibers. While these structures exhibit potential for tissue engineering applications due to their resemblance to the extracellular matrix, challenges such as mechanical weakness and limited pore size must be addressed. To overcome these limitations, researchers are exploring strategies such as combining 3D printing, freeze-drying, and needleless electrospinning to enhance the properties of honeycomb scaffolds [50].

In addition to the honeycomb structure, another intriguing development is the formation of honeycomb-like fiber mats or "nano nets." These nano nets, composed of sub-50 nm fibers, exhibit a spider-web-like structure due to the interplay of various forces during the electrospinning process [23, 47]. Their potential applications extend beyond tissue engineering, with promising use as filters for removing nanometer-sized particles and viruses [48, 51].

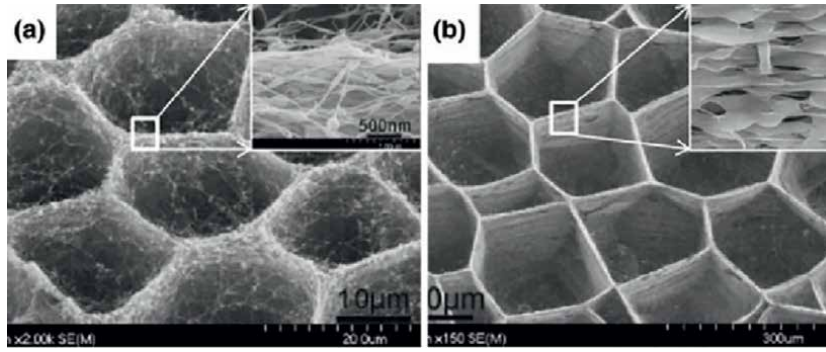


Figure 4.
SEM images of the honeycomb structure with different polymers. (a) honeycomb structure with 3 wt. % PAN from DMF solution at the voltage of 22 kV, (b) honeycomb structures with 6 wt. % PVA from aqueous solution at the voltage of 22 kV [46].

Honeycomb-based structures represent a versatile platform for fabricating functional materials with potential applications in diverse fields, including tissue engineering, filtration, and beyond [52].

2.1.4 Block copolymers

Block copolymers (BCPs) are macromolecules of distinct polymer blocks with varying properties (**Figure 5**). Their ability to self-assemble into well-defined nanoscale structures, such as spheres, cylinders, or lamellae, makes them attractive candidates for creating functional materials [23]. While BCPs have been extensively studied in bulk and thin film formats, their application in electrospun fibers is relatively recent [54, 55].

Electrospun BCP fibers offer the potential for tailoring fiber properties by carefully selecting polymer blocks. For example, the hydrophilic-hydrophobic balance of a BCP can be manipulated to create fibers with specific surface characteristics, making them suitable for biomedical applications [22, 56]. Additionally, researchers aim to achieve long-range order within BCP fibers to create materials with enhanced properties and functionalities [55, 57].

To address the inherent hydrophobicity of many BCPs, strategies such as blending with hydrophilic polymers, like PVP, have been employed to improve

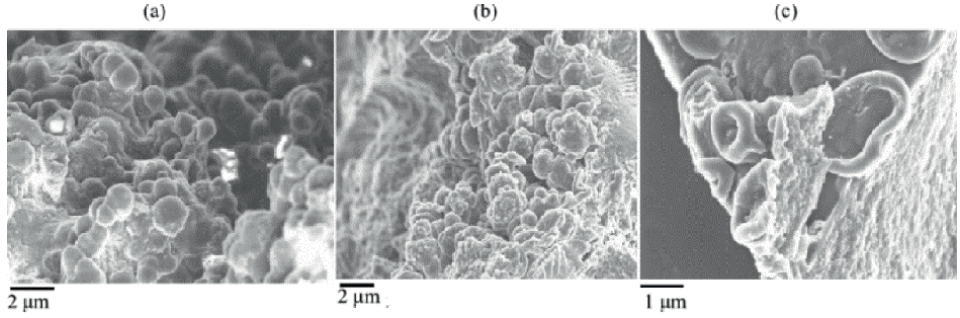


Figure 5.
SEM images of the surface of HEMA-EGDMA block copolymers. (a) Gel pores and mesopores in the mesoporous matrix of CP-1, (b) chips on the surface of CP-1, and (c) looser structure of the matrix of MIP-1 [53].

biocompatibility [56]. Furthermore, BCP fibers have demonstrated potential for creating porous structures with high permeability, as reported by Shi et al. [57]. Techniques like annealing, blending with more robust polymers, or metal coating have been explored to enhance the mechanical properties of BCP fibers [22].

Achieving long-range order in electrospun BCP fibers remains challenging due to the rapid solidification process during electrospinning. To overcome this, researchers have investigated annealing, substrate-guided self-assembly, and coaxial electrospinning as potential solutions [23].

While significant progress has been made, further research is necessary to unlock the full potential of BCPs in electrospun fibers. By addressing related challenges, BCP-based electrospun materials can be used in a wide range of fields, including biomedicine, filtration, and electronics, to achieve phase separation, fiber alignment, and mechanical properties.

Previous studies have demonstrated the formation of various nanoscale structures within electrospun BCP fibers, including lamellar, spherical, and cylindrical morphologies [23]. Ruotsalainen et al. observed lamellar-within-spherical structures in poly (styrene-b-vinyl pyridine) fibers [58], while Ma et al. reported gyroid-type structures in poly (styrene-b-dimethyl siloxane) fibers [59]. These internally structured fibers exhibit promising properties for applications in filtration, optics, and nanotechnology [54].

2.1.5 Beads-on-string

Beads-on-string structures, characterized by discrete bead-like formations along a continuous fiber, are a common outcome of electrospinning (**Figure 6**). While often considered an undesirable artifact, these structures possess unique properties that can be exploited for specific applications, such as drug delivery and water treatment [61].

The formation of beads is attributed to Rayleigh instability, which causes the polymer jet to break into droplets during electrospinning [22]. These droplets subsequently solidify into beads on the forming fiber. Various factors influence beads' size, shape, and distribution, including polymer concentration, solution viscosity, electrical voltage, and solvent choice [22, 62].

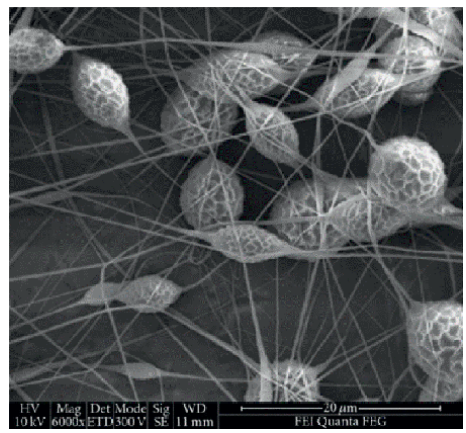


Figure 6.
SEM image of formed beads on electrospun fibers prepared using 10% PVP dissolved in DMF [60].

Beyond the basic beads-on-string structure, advanced electrospinning techniques can create more complex fiber morphologies. Core-shell nanofibers, side-by-side electrospun fibers, and honeycomb structures offer additional possibilities for tailoring fiber properties and functionalities [63].

It is important to note that the formation of beads can also be controlled through careful manipulation of electrospinning parameters.

The properties of the solution used in electrospinning can be manipulated to control the characteristics of the resulting fibers. Specifically, by altering the solution's concentration and thickness, researchers can influence the fibers' size, shape, and distribution of bead-like structures. This technique has been successfully applied to create superhydrophobic fibers from both polystyrene (PS) and polycaprolactone (PCL) materials [22].

In conclusion, beads-on-string structures, while often associated with process imperfections, can be harnessed for specific applications and serve as a foundation for creating more complex and functional electrospun fibers. A comprehensive understanding of the factors influencing bead formation is essential for tailoring fiber properties to meet desired specifications.

2.1.6 Helical nanofibers

Helical nanofibers, inspired by the helical structures found in nature, such as proteins and DNA, have garnered significant interest due to their potential applications in diverse fields (**Figure 7**). These fibers exhibit unique properties, including enhanced porosity, elasticity, and toughness, making them suitable for applications in textiles, filtration, and biomedical devices. Additionally, helical nanofibers composed of conjugated polymers offer promising opportunities for electronic and photonic devices [65, 66].

While early methods for producing helical structures involved the contraction of metal oxide sols or vapor-phase deposition of materials like zinc oxide and silicon

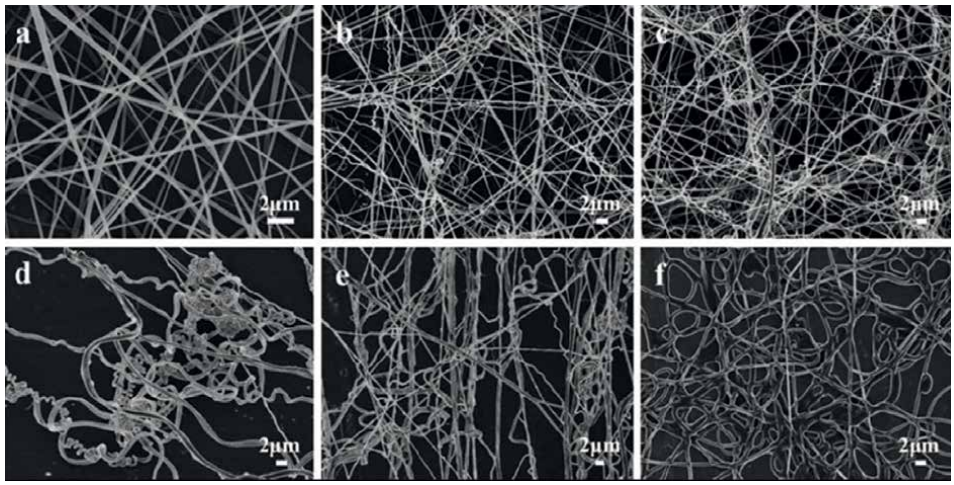


Figure 7.
SEM images of cellulose acetate (CA)/Thermoplastic Polyurethane (TPU) helical nanofibers obtained from (a) 18 wt% TPU in DMF and 16 wt% CA in DMAc/Acetone without LiCl, and from 18 wt% TPU in DMF and 15 wt% CA in DMAc/Acetone with the addition of (b) 1.3 wt% LiCl, (c) 1.6 wt% LiCl, (d) 1.8 wt% LiCl, (e) 2.0 wt% LiCl, (f) 2.5 wt% LiCl [64].

dioxide, electrospinning has emerged as a versatile and efficient technique for fabricating helical nanofibers. Researchers have employed various approaches to achieve helical morphology, including using bicomponent fibers composed of polymers with different properties. The formation of helices is often attributed to the viscoelastic properties of the polymers and the influence of the electric field during the electrospinning process.

Beyond bicomponent systems, helical nanofibers have been fabricated from single-component polymers by carefully controlling electrospinning parameters [67, 68]. Physical forces, such as bending instability and solvent effects, have been implicated in the formation of helical structures [69].

Moreover, near-field electrospinning techniques have been explored to induce helical patterns by manipulating the electrical field and jet dynamics [23].

The morphology of helical fibers can be influenced by various factors, including polymer composition, solution concentration, applied voltage, collector type, and solvent properties [69, 70]. While circular cross-sections are typical, ribbon-like structures have also been observed in some instances [70].

In conclusion, the fabrication of helical nanofibers through electrospinning represents a promising area of research with potential applications in various fields. A deeper understanding of the underlying mechanisms governing helical formation is essential for achieving precise control over fiber morphology and properties.

2.2 Hierarchical secondary structures

Electrospun fibers, renowned for their exceptional properties, can be further enhanced by incorporating secondary structures. Secondary structures are typically introduced through either a two-step or a single-step process. The former involves initial electrospinning followed by post-processing treatments, while the latter relies on precise control of humidity and solvent conditions during the electrospinning. These structures can be classified based on their dimensionality: zero-dimensional (nanopores), one-dimensional (nanorods and nanopillars), or two-dimensional (nanosheets). The presence of secondary structures bestows a wide range of properties upon the fibers. These include enhanced wettability (superhydrophobicity or superhydrophilicity), catalytic activity, sensing capabilities, and filtration efficiency. While electrospinning is a common platform for developing secondary structures, it is important to note that these structures can also be formed independently using methods like heat treatments or solvothermal reactions [22, 23].

2.2.1 Nanopores

The introduction of patterned arrays enhances surface roughness, which, according to the Cassie-Baxter law, influences wettability or water contact angle. Both surface energy and roughness determine this property. Consequently, roughened hydrophobic surfaces become more water-repellent, while hydrophilic surfaces exhibit increased water affinity [23].

Nanopores within electrospun fibers can be generated through various methods, including thermally induced phase separation and selective polymer removal. To create porous fibers, careful selection of polymer, solvent, temperature, and humidity is crucial [71, 72].

The role of polymer-solvent interactions in pore formation remains controversial. While some studies have reported pore formation using specific polymer-solvent

combinations, others have found no such effect. Polymer-solvent phase separation has been identified as a primary factor in pore creation. However, not all electrospinnable polymers exhibit porosity under these conditions and the underlying mechanisms that influence pore shape are not fully understood. While some polymers, such as polystyrene, poly (methyl methacrylate), and PLA, are more prone to porosity, the choice of solvent is often limited to volatile options like dichloromethane, chloroform, and tetrahydrofuran [31, 72, 73]. The formation of pores within electrospun fibers is primarily controlled by the solvent system used during electrospinning. The size and shape of these pores can be manipulated by adjusting the solvent composition and processing parameters. The presence of pores in fibers can significantly alter their properties, such as hydrophobicity, and can be exploited for various applications, including sensing [31, 74].

Phase diagrams can predict pore formation based on temperature and solvent composition. Different phase separation processes, including non-solvent-induced, vapor-induced, thermally induced, and breath figure, lead to distinct pore structures. The breath figure method, involving water droplet condensation and evaporation on the fiber surface, typically produces circular pores [31].

Humidity plays a significant role in pore formation, with optimal conditions ranging from 30 to 70%. The resulting pore size is influenced by humidity levels. Additionally, solvent evaporation and electrospinning temperature impact pore formation [31, 75].

Combining thermal-induced phase separation (TIPS) with non-solvent-induced phase separation (NIPS) can enhance pore formation. However, this process is energy-intensive due to the need for low-temperature collectors [31]. Thermal and solvent-based methods, when combined (N-TIPS), affect both the overall fiber structure and the creation of pores on the fiber's surface. While one method might primarily influence fiber shape, the other impacts pore formation, but these processes are interconnected. Precise temperature control is crucial for optimizing pore size and distribution, as it affects both the rate at which the solvent evaporates and the solidification of the polymer (**Figure 8**) [76, 77].

2.2.2 Nanopillars

Polymer properties can be tuned by manipulating their structure at the nanoscale. Electrospinning and the template-wetting method reign supreme for fabricating one-dimensional (1D) nanopillars (**Figure 9**). The electrospun fiber is the foundational

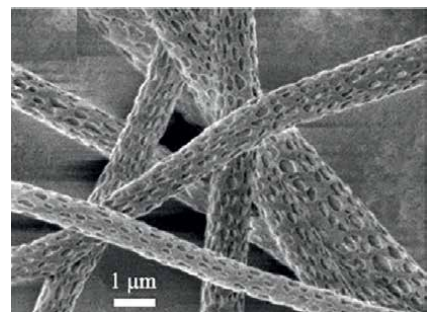


Figure 8.
SEM image of nanopore Poly-L-lactide (PLLA) in dichloromethane [77].

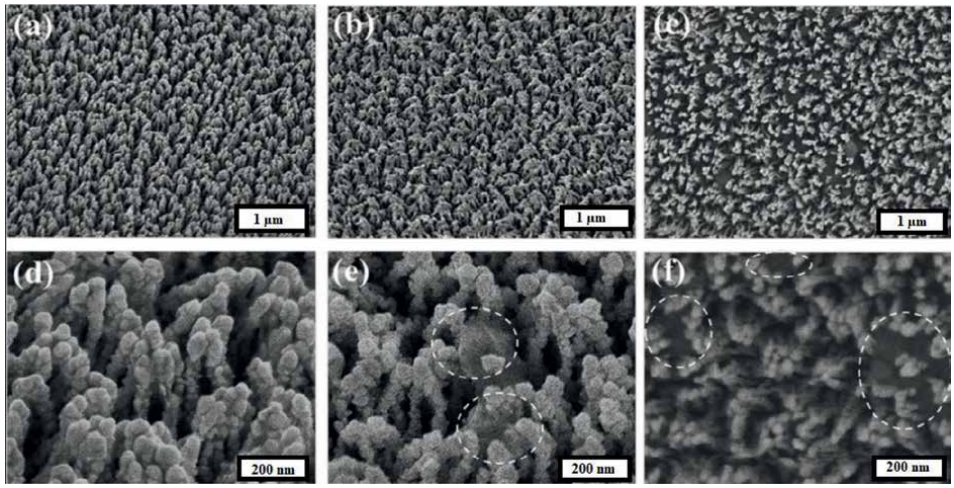


Figure 9.
SEM images of a methylene blue (MB)-deposited plasmonic nanopillar array after water evaporation in a, d) the Wenzel state, b, e) the partial wetting state, and c, f) the Cassie-Baxter state. The dotted circles in panel (e) indicate the MB molecules deposited in the nanogaps atop the nanopillar clusters. In contrast, the MB molecules in panels (c) and (f) were deposited mainly within the large interstitial areas among the nanopillars and the area surrounding the cluster perimeter (dotted region) [78].

layer in this process, requiring no particular setup. The magic truly unfolds at the second level—the nanopillars. Anodized aluminum oxide (AAO) templates are the preferred choice, although alternatives like nano machining and lithography exist. AAO templates stand out due to their simplicity, and AAO templates offer precise control over the dimensions of polymer nanostructures [60, 79].

The widespread use of AAO templates is attributed to several advantages: adjustable pore diameter, well-defined geometry, high density and aspect ratio, favorable surface interactions, and thermal stability [79].

Creating nanopillars using AAO templates involves several steps: AAO template preparation, sandwiching the electrospun nanofiber within the template, nanopillar nucleation through heat treatment, and finally, AAO template removal. This last step can be challenging to some extent, so various methods have been used [80, 81].

Resende et al. [82] demonstrated the fabrication of 3D AAO templates by directly applying a pulsed potential profile in a single anodization step. Limiting the number of anodization steps directly decreases total fabrication time while bypassing the need to remove the oxide typically grown in the two-step process chemically. Xu et al. [83] demonstrated a novel template for creating vertically aligned nanostructures developed using vertically oriented TANI nanocrystals grown on graphene. This new approach offers several advantages. Firstly, it is versatile, accommodating various materials and deposition techniques. Secondly, it can produce both solid and hollow nanostructures. Thirdly, the removal of the template after creating solid nanostructures is unnecessary. Finally, these vertical arrays' size, density, and placement can be precisely controlled. Nanopillars exhibit various shapes, from vertically aligned to those with round bases and hemispherical tops. Moon et al. [84] introduced a method to control nanopillar size by manipulating AAO template parameters and using polydimethylsiloxane (PDMS) to enhance surface area ratio and template reusability. Wang et al. [85] introduced an approach to fabricate ordered arrays of 10-nm-scale silica-filled apertures in a metal film. Additionally, nanopillars can be

further modified. Moon et al. [84] deposited metals on nanopillars, achieving asymmetric bending and enhanced properties. Mijangos et al. [79] delved into the methods for creating polymer nanostructures using AAO templates, the impact of nanoscale dimensions on polymer properties, and the potential applications of these materials. Zhu et al. [86] tailored nanopillar structures on surfaces.

2.2.3 Nanorods

Nanorods, distinguished by their elongated one-dimensional structure, have emerged as a pivotal component in nanotechnology, as shown in **Figure 10**. Unlike their spherical or sheet-like counterparts, nanorods offer a unique combination of properties arising from their anisotropic shape. This characteristic, coupled with their ability to be constructed from various materials, including metals, semiconductors, and polymers, has made them a focal point of research and development [87, 88].

Fabrication methods for nanorods are diverse, with electrospinning standing out as a versatile technique. This process involves the generation of fine fibers that can be subsequently transformed into nanorods through thermal or chemical treatments [88]. Cherian et al. [87] synthesized nanorods through electrospinning and heat treatment. These nanorods exhibited a unique structure composed of interconnected nanoparticles. The material demonstrated exceptional electrochemical performance as an anode for lithium-ion batteries, characterized by high reversible capacity, excellent cycling stability, and impressive rate capability. The superior performance is attributed to the unique morphology of the nanorods, which facilitates efficient lithium-ion diffusion and storage.

The precise control over nanorod dimensions, such as length and diameter, is crucial for optimizing their performance in specific applications. Moreover, controlling temperature and duration during this process is essential, as it directly influences the nanorod's dimensions and properties [89]. Bai et al. [90] focused on understanding how the shape of gold nanorods changes when heated to determine the relationship between heating temperature and nanorod shape, identify the underlying mechanisms driving this shape change, and quantify the factors involved. By understanding these processes, the researchers aim to develop a framework for

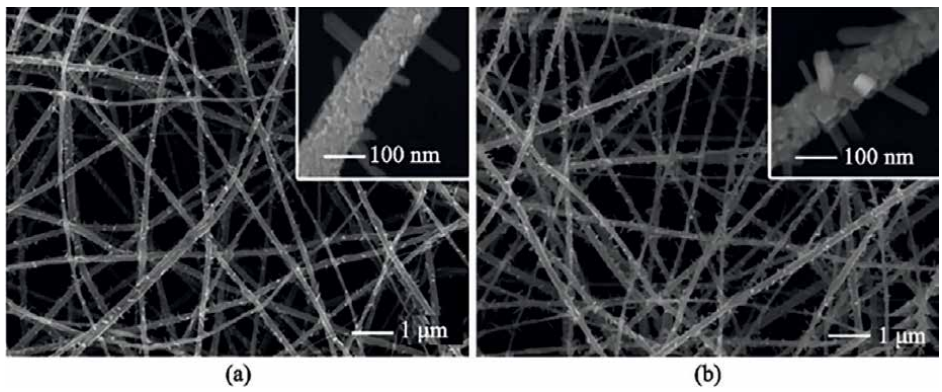


Figure 10.
SEM image of V_2O_5 nanorods on electrospun TiO_2 nanofibers. The calcination temperature is 475 °C for a different time: (a) 10 min and (b) 60 min. Insets show magnification at 100 nm. Reprinted with permission from Ostermann et al. [87].

creating nanostructures with specific shapes and sizes on surfaces. Ostermann et al. [91] developed a novel method for creating hierarchical nanostructures by combining electrospinning and calcination to grow single-crystal V_2O_5 nanorods on rutile nanofibers. This approach offered precise control over the size of the V_2O_5 nanorods by adjusting the composition of the starting materials and the calcination temperature. The researchers believe that this technique can be expanded to create even more complex nanostructures. In another study [92], TiN nanorods were synthesized through a multi-step process involving electrospinning, calcination, and nitridation. Starting with TiO_2 nanofibers, a subsequent nitridation process transformed them into TiN nanorods. The size of the resulting TiN nanocrystals increased with higher nitridation temperatures. The electrochemical performance of these TiN nanorods was found to be dependent on the nitridation temperature.

The functional properties of nanorods are intrinsically linked to their morphology. For instance, the aspect ratio, or the length-to-diameter ratio, significantly influences their optical, electrical, and mechanical behaviors. This anisotropic nature makes nanorods attractive for electronics, optics, and energy storage [89, 92]. Bai et al. [90] fabricated Nanorods on electrospun fibers subjected to thermal treatment, causing them to evolve from long rods to shorter, round shapes. This morphological change directly impacted the material's functionalities. Surface-enhanced Raman spectroscopy (SERS) activity initially improved but declined with increasing heat treatment, while catalytic performance steadily decreased. This study highlights the critical role of nanostructure in determining material properties and provides valuable insights for tailoring nanomaterials for specific applications. Burrows et al. [89] focused on the importance of understanding and controlling the surface chemistry of gold nanorods for their practical application, which highlights the need to characterize the surface composition and structure of these nanorods, including ligand type, density, and location to reach precise control over their properties and leading to the development of new applications.

As research progresses, a deeper understanding of the structure-property relationships in nanorods is emerging. This knowledge is essential for tailoring these materials to meet the demands of specific applications. By carefully controlling the synthesis process and post-treatment conditions, researchers are developing innovative nanorod-based materials with promising properties and functionalities [89].

2.2.4 Nanosheets

The remarkable geometry of nanosheets (**Figure 11**) makes them highly attractive for applications in photocatalysis, supercapacitors, sensors, and batteries [22]. Unlike some nanostructures, nanosheets can be fabricated independently without relying on electrospun templates. Various methods exist for their creation, and they [94] can be categorized into top-down and bottom-up strategies. Top-down methods include sonication exfoliation, and bottom-up techniques include interfacial, three-layer, and surfactant-assisted synthesis.

Transition-metal dichalcogenide (TMD or TMDC) and semiconducting metal oxides (SMOs) are popular choices for nanosheet fabrication [95]. These nanosheets are strategically grown on nanofibers to capitalize on the benefits of increased surface area, robust structural integrity, and good electrical conductivity. The concentration of the nanosheet material plays a crucial role in achieving optimal uniformity and structural stability [96]. Unfortunately, agglomeration is a recurring challenge in nanosheet fabrication.

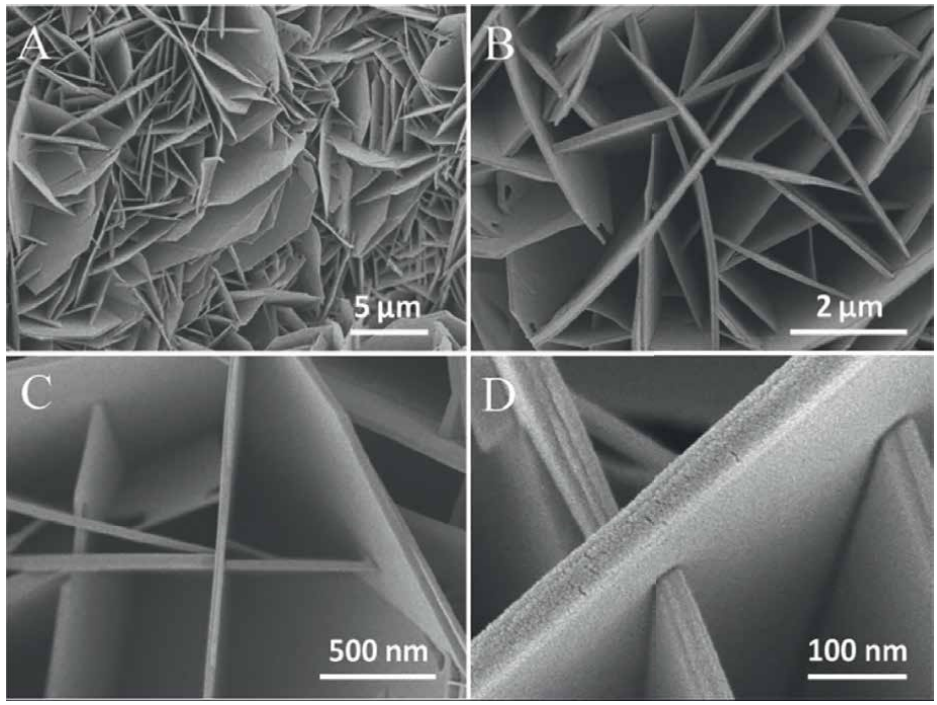


Figure 11.
SEM images of interconnected hierarchical Ni-Fe nanosheets (A), the corresponding different enlarged views (B, C), and a close-up view of a single nanosheet array showing the interconnected nano "wall block" nanostructure (D) [93].

To further enhance the properties and functionalities of TMD nanosheets, researchers have focused on creating hybrid nanoarchitectures by combining them with other materials. These composites offer the opportunity to engineer novel materials with tailored chemical, physical, and electronic characteristics [94]. Beyond the synthesis of standalone 2D MOF nanosheets, the creation of nanocomposites incorporating these materials has emerged as a fruitful area of research. Combining 2D MOF nanosheets with other materials makes it possible to engineer novel functional materials with tailored properties for specific applications [94]. Li et al. [97] synthesized 3D-structured photodetectors based on 2D materials. They identified and addressed the challenges and opportunities inherent in developing and practically implementing 3D-structured photodetectors based on 2D nanosheets.

Li et al. [96] integrated the SnO_2 nanosheets with polyaniline to create composite-based gas sensors to enhance gas sensing capabilities. Notably, these nanocomposite sensors demonstrated exceptional performance in detecting ammonia (NH_3), including a remarkably high response magnitude, an ultra-low detection limit, excellent repeatability, and impressive selectivity at room temperature. In another study, Fe_2O_3 nanosheets were directly grown on interdigitated gold electrodes through a hydrothermal process involving electrospun nanofibers containing $FeCl_3$ and poly (vinyl butyral); the resulting nanocomposite exhibited exceptional performance in detecting low concentrations of NH_3 [96]. Baqi et al. [98] created a highly efficient photocatalyst by combining two 2D materials, SnS_2 and g-CN, into a heterojunction. This combination significantly enhanced the material's ability to convert light into

energy, specifically for hydrogen production. Lin et al. [99] combined the advantages of small-molecule photochemistry with the ordered structure of MOFs, resulting in highly efficient and recyclable heterogeneous photocatalysts and presented a significant step forward in the design and development of advanced photocatalysts with improved performance and potential applications in various fields. A bottom-up synthesis method was developed to embed photoactive cores into 2D MOF nanosheets successfully.

3. Electrospinning fiber modifications

While electrospinning offers a versatile platform for creating micro- and nanofibers with a wide range of properties, post-modification techniques are often essential for tailoring these materials to specific applications. By introducing new functionalities or altering existing properties, post-modification processes enable the creation of advanced materials with enhanced performance [4, 100–102].

3.1 Chemical modification

Chemical modification involves altering the chemical composition of the fiber surface to impart desired properties. This can be achieved through various techniques:

- Grafting: Covalently attaching polymer chains or functional groups to the fiber surface to enhance properties like hydrophilicity, biocompatibility, and conductivity [101–103].
- Cross-linking: Creating covalent bonds between polymer chains to improve mechanical properties, stability, and resistance to degradation [103, 104].
- Wet Chemistry: Utilizing liquid-phase reactions to introduce functional groups or modify existing ones. Techniques include hydrolysis, oxidation, and esterification [4, 103].
- Click Chemistry: Employing highly specific and efficient reactions to introduce desired functionalities with high precision [101, 102].

3.2 Physical modification

Physical modification alters the fiber surface without changing its chemical composition. Common techniques include:

- Plasma Treatment: Exposing fibers to a gas plasma introduces functional groups, improves surface energy, and enhances biocompatibility [103, 105].
- UV Irradiation: Using ultraviolet light to induce chemical changes on the fiber surface, such as forming reactive groups [103].
- Physical Adsorption: Coating fibers with polymers, surfactants, or nanoparticles through physical interactions [103, 106, 107].

3.3 Structural modification

Structural modification involves altering the physical arrangement of the fibers to create new properties or functionalities. Techniques include:

- Rolling Up: Transforming 2D electrospun mats into 3D structures by rolling up multiple layers of fibers [108].
- Laser Ablation: Using a laser to modify its properties, creating patterned structures on the fiber surface [109].
- Gas Foaming: Introducing gas bubbles into the electrospun mat to create a porous 3D structure [110].

3.4 Nanoparticle incorporation

Incorporating nanoparticles into the fiber matrix or on the fiber surface can significantly enhance material properties. Techniques include [103]:

- Direct Blending: Dispersing nanoparticles within the polymer solution before electrospinning.
- Post-Electrospinning Deposition: Applying nanoparticles to the fiber surface through dip coating or spray coating.
- In Situ Formation: Generating nanoparticles within the polymer solution during electrospinning.

3.5 Reinforcement

Reinforcing fibers with additional materials can improve mechanical properties, such as strength and stiffness. Common reinforcement materials include carbon nanotubes, graphene, and cellulose nanocrystals [73, 111].

3.6 Challenges and opportunities

While post-modification techniques offer significant potential for enhancing fiber properties, challenges such as achieving uniform modification, maintaining fiber integrity, and ensuring long-term stability must be addressed. Future research should focus on developing novel modification techniques, optimizing process parameters, and understanding the underlying mechanisms to unlock the full potential of these approaches.

By carefully selecting and combining different post-modification techniques, highly functional and versatile nanofiber-based materials for various applications can be created.

4. Modeling

Optimizing electrospinning process parameters to achieve desired nanofiber characteristics is a critical yet complex task. The traditional one-factor-at-a-time (OFAT)

approach is time-consuming and inefficient, often failing to identify optimal parameter combinations. More sophisticated experimental design and statistical modeling techniques have emerged to address these limitations.

Design of Experiment (DOE) methodologies provide a structured approach to investigate multiple process parameters simultaneously. Researchers can identify significant factors and their interactions by carefully selecting experimental conditions. Response Surface Methodology (RSM) builds upon DOE by developing mathematical models to predict the relationship between process parameters and response variables. However, RSM may encounter limitations when dealing with complex nonlinear relationships, highlighting the need for alternative solutions. Machine learning techniques, such as Partial Least Squares (PLS) regression and Locally Weighted Kernel Partial Least Squares (LW-KPLSR), offer potential solutions for modeling these complex interactions [112, 113].

Empirical models based on experimental data have been developed to correlate process parameters with fiber diameter. While these models provide valuable insights, they often lack generality and predictive power. In the field of nanofiber technology, there is a growing need for more accurate and reliable predictions, which can be achieved through comprehensive mathematical models grounded in fundamental physical principles [114].

Pervez et al. [112] combined the RSM optimization model with a machine learning model to predict the diameter of electrospun nanofiber membranes using three case studies. Following this, the model's root mean squared error (RMSE), absolute mean error (MAE), and coefficient of determination (R^2) were calculated to assess its accuracy. The performance of the fuzzy model, principal component regression (PCR), and least square support vector regression (LSSVR) models were evaluated for comparison. RSM design was employed to identify optimal electrospinning parameters for minimizing nanofiber diameter. The LW-KPLSR model significantly outperformed other models, demonstrating superior prediction accuracy with lower error values and higher R^2 values (up to 0.9989). These findings suggested the potential of the LW-KPLSR model as a diameter prediction tool for electrospun nanofiber membranes. Tariq et al. [115] focused on optimizing electrospinning parameters to fabricate PVDF-based nanofiber mats with enhanced β-phase content. The Taguchi design methodology was employed to investigate the influence of key process variables, including PVDF concentration, applied voltage, flow rate, and drum speed, on the resulting fiber morphology and β-phase fraction. A statistical model was developed to establish a correlation between these process parameters and critical response variables: β-phase content and fiber diameter. Analysis of the results revealed that PVDF concentration significantly impacted fiber properties. Higher concentrations promoted the formation of the β-phase while also increasing fiber diameter. The optimal electrospinning conditions were determined to achieve the desired balance between β-phase content and fiber morphology. Kalantary et al. [116] developed and compared the performance of Multi-Layer Perceptron (MLP), Radial Basis Function (RBF) Neural Network, and Support Vector Machine (SVM) models for predicting the diameter of PCL/Gt nanofibers. Four key parameters—weight ratio, applied voltage, injection rate, and distance—were considered input variables for these models. A Prediction of the Nanofiber Diameter Model (PDNFM) was established using these techniques. The MLP-based model (PDNFMMLP) demonstrated superior accuracy, efficiency, and cost-effectiveness compared to the RBF and SVM counterparts. Sensitivity analysis revealed that the PCL/Gt weight ratio significantly influenced the PDNFMMLP output. To evaluate the performance

of the developed models, metrics such as Root Mean Squared Error (RMSE), Mean Absolute Error (MAE), Coefficient of Determination (R^2), Willmott's Index, and Taylor diagram were employed. The results consistently demonstrated the superior accuracy of the MLP model over RBF and SVM. Bian and Qing [117] studied three modeling techniques—common solid element, solid element embedded region (ER), and beam element ER—to simulate the mechanical behavior of Carbon nanotube/nanofiber reinforced composites (CNRCs). Geometric configurations were generated using MATLAB, and finite element models were created with MSC.Patran. The influence of nanofiber aspect ratio and load cases on mechanical properties was investigated. Results indicate that while all models showed linear relationships, solid element ER models exhibited higher stiffness due to volume sharing between the nanofibers and matrix. Standard solid element models tended to underestimate strength due to stress concentration. Beam element ER models were suitable only for nanofibers with large aspect ratios. Bagherzadeh et al. [118] presented a theoretical model for estimating the number of contacts in random multilayer nanofiber assemblies, considering fiber diameter and network porosity. Experimental validation confirmed the model's predictions. A novel technique was developed to measure fiber-to-fiber contacts in nanofiber mats directly. Results indicate that fiber diameter and network porosity significantly influence the number of contacts. The proposed model offers a more accurate and versatile approach to studying multilayer nanofiber assemblies compared to existing theories.

Despite advancements in modeling techniques, several challenges persist in electrospinning process optimization. Complex interactions between process parameters, scale-up issues, and accurate characterization of fiber properties remain significant hurdles. Future research should focus on developing hybrid modeling approaches, gathering comprehensive experimental data, and addressing scale-up challenges to enhance the predictive capabilities of electrospinning models.

5. Conclusion

Electrospinning has emerged as a versatile technique for fabricating nanofibers with exceptional properties. Researchers can create nanofibers with tailored characteristics for various applications by carefully controlling process parameters and incorporating innovative modifications. This chapter delved into the intricacies of electrospinning, emphasizing the influence of polymer solution properties, processing variables, and environmental conditions on fiber formation. Incorporating hierarchical structures, including nanopores, nanopillars, and nanosheets, was explored to enhance fiber functionality further. Post-modification techniques, such as chemical and physical modifications, nanoparticle incorporation, and reinforcement, were discussed as strategies for tailoring fiber properties. The chapter also highlighted the importance of modeling and optimization techniques for achieving desired nanofiber characteristics.

While significant progress has been made in electrospinning technology, there is still immense potential for further advancements. Future research should focus on developing novel modification techniques, optimizing process parameters, and understanding the underlying mechanisms governing fiber formation. By addressing these challenges, researchers can unlock the full potential of electrospun nanofibers and create innovative materials with transformative applications across various industries.

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Chapter 2

Perspective Chapter: Sustainable Nanofibers and Their Applications

Ghazal Tuhmaz

Abstract

This chapter introduces mixed topics between nanotechnology, especially nanofibers and environment. The growing awareness related to the environment has led to interest and scrutiny of everything related to environmental preservation and sustainable development of technological processes in general. With the emergence of nanotechnology and the demand for electrospinning technology specifically to obtain nanofibers with unique features, there was a need to highlight the so-called green electrospinning because of the use of chemicals and solvents that may sometimes be very dangerous in the traditional electrospinning process. In this section, the concept of development and sustainable materials were mentioned, and green electrospinning was shown, both in terms of the materials used in it and in terms of the different methods adopted in this technology. Despite the many advantages of this technology for producing sustainable nanofibers, there are a number of challenges that were mentioned in this section. Finally, various applications of green electrospinning in the fields of tissue engineering, wound dressings, facemasks, food, agriculture, environment, biomedicine and textiles were discussed.

Keywords: sustainable, nanofibers, green electrospinning, friendly environment, renewable polymers

1. Introduction

Nanotechnology has attracted great attention in recent years because of its wide applications and great importance in various fields, and nanofibers have received many studies because of their important features.

Nanofibers are fibers that have diameters in the nanometer range. Nanofibers prepared from biomaterials have dual properties of both nanofibers and renewable polymers such as high surface-to-volume ratio, superior cell adhesion, proliferation, and differentiation.

Methods of producing nanofibers have varied, such as melt blowing, centrifugal spinning, the mold method, various types of electrospinning, and others.

The use of electrospinning technology has been the most widespread among the previous methods because of its ease of use and flexibility in dealing with different

materials, as materials can be used that are added to polymeric materials to give the resulting fibers new properties.

Electrospinning is a simple, cost-effective, and versatile technique for forming nanofibers. The driving force behind the formation of nanofibers in electrospinning is the electrostatic force. It consists of three main components: a high-voltage power supply, a feed pump, and a ground collector. In a typical electrospinning process, the polymeric solution is loaded into a syringe and the solution is pumped through the syringe needle at a specific rate, thus collecting the fibers on the collector due to the electrostatic force. In electrospinning, there are two opposing forces, namely surface tension and electrostatic repulsive effects on the droplet coming from the tip of the needle. When the electrostatic force overcomes the surface tension of the droplet, the droplet begins to expand to form a cone, called a Taylor cone. Thus, the charged droplet is ejected from the needle tip, and the microfibers are collected on the grounded collector. There are several electrospinning parameters that affect the shape and homogeneity of nanofibers. These parameters include solution parameters (viscosity, dielectric constant, surface tension, and conductivity), process parameters (voltage, solution flow rate, needle diameter, distance between needle tip and collector), and ambient conditions (humidity, air velocity, temperature, pressure).

A different modification in the standard electrospinning setup has been made to overcome several limitations related to the shapes, sizes, and morphologies of nanofibers. These modifications include multi-jet electrospinning (single needle, multiple needles, needleless system), emulsion electrospinning, coaxial electrospinning, drum electrospinning, and porous hollow tube electrospinning.

However, the evaporation of solvents used in the electrospinning process, especially organic ones, in addition to the use of environmentally harmful polymers, has restricted this industry, especially since most of the polymers used at present are toxic and harmful and lead to environmental pollution, which limits their use in many fields, especially biomedicine [1].

The demand is for the discovery of sustainable, environmentally friendly, and biodegradable materials with superior and distinctive physical and mechanical properties. The aim of this chapter is to discuss the role of sustainable nanofibers in many fields, with reference to sustainable methods of manufacturing them.

Therefore, attention has been directed to developing this industry in line with sustainable green development [2].

2. The concept of sustainable development

The development meets the needs of the present time without negatively affecting the ability of generations to meet their needs. Therefore, sustainability requires the presence of materials that are sustainable without affecting the environment.

Given the importance of the topic, research in this field has increased due to increased awareness of global warming and the tangible effects of this phenomenon. Therefore, the application of sustainable development leads to reducing the risks associated with the depletion of materials, energy, and pollution, in addition to carrying out waste management. Hence, the need in industry to use sustainable materials has emerged [1].

3. The concept of sustainable materials

It includes materials extracted from natural resources, whether organic or inorganic. The term sustainable materials is associated with renewable materials, which are materials made from natural resources that can be renewed generation after generation. As an example, we mention wood products or materials derived from them, such as cellulose. Wood products are renewable materials because trees grow. Again, it can be planted to grow in the future and benefit future generations.

Through research on the topic of sustainable development, sustainable materials, and electrospinning, the concept of green electrospinning emerged due to the importance of membranes manufactured with this technology, which are composed of nanofibers, in addition to the increasing demand for green chemistry and applications in medicine, pharmacy, and biology [2].

4. The concept of green electrospinning

It has become possible to achieve a greener and more sustainable future in materials science, thanks to green electrospinning, because the nanofibers resulting from it have a number of useful properties such as biodegradability, less negative impact on the environment, and the possibility of renewing resources, in addition to their various applications in industry such as textile engineering, environment, energy and agriculture, and others. **Figure 1** shows the main parts of green electrospinning.

Using green electrospinning requires environmentally friendly products and environmentally friendly methods [3].

According to the above, green electrospinning is studied within three categories:

Green and decomposed materials, green solution, and solventless electrospinning methods.

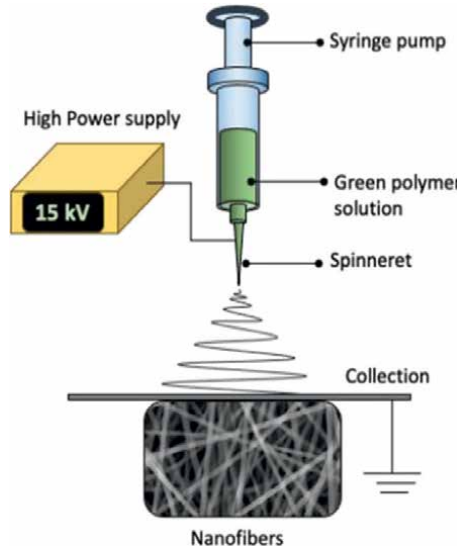


Figure 1.
Main parts of green electrospinning.

4.1 Green and decomposed materials

4.1.1 Natural polymeric materials

Natural polymeric materials, including cellulose, starch, chitosan, wool protein, and silk are biodegradable and easily degraded by microorganisms [4].

4.1.2 Bioindustrial polymeric materials

These materials are produced by fermentation of carbon sources and include polyesters and amino acids that have vital properties such as biodegradability and biocompatibility. Examples of these are polyhydroxyalcones (PHAs) and

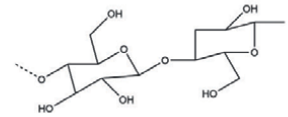
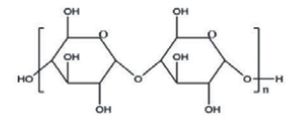
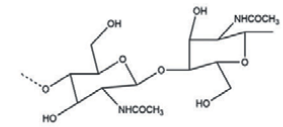
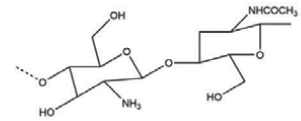
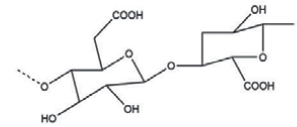
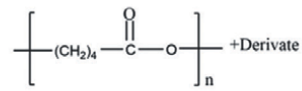
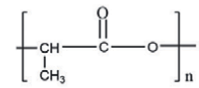
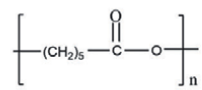
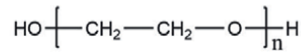
Polymer system	Chemical formula
Cellulose	
Starch	
Chitin	
Chitosan	
Alginate	
PHB	
PLA	
PCL	
PEG	

Table 1.
Biodegradable polymers.

polyhydroxybutyrolactone (PHB), which can be decomposed into CO_2 and H_2O by bacteria and can be mixed with other types of polymers to improve properties, as biodegradable polymers which are shown in **Table 1** [4].

4.1.3 Chemically manufactured polymers

Biodegradable macromolecules, which have a structure similar to natural macromolecules, can be manufactured chemically. Examples include:

PCL, PLA, and PEG, which can be widely used in medicine, pharmacy, and agriculture [4].

4.1.4 Natural additives

These substances include plant extracts, bioactive compounds, antimicrobials, antioxidants, and other required functions [4].

4.2 Green solvent solutions

The use of water as a solvent is one of the most important options in green electrospinning, with the presence of water-soluble polymers that contain polar groups such as −NHR, −SO3H, NH2−, −OH, and −COOH.

Solutions containing a polymer mixed with a water-soluble polymer can also be used, such as:

PVA, PEO, PAA, PVP, and HPC that form homogeneous solutions in water.

Non-toxic organic solvents such as ethanol can be used [4].

4.3 Electrospinning without using a solvent

Below are some examples of using the electrospinning process without the presence of a solvent, without going into detail about it [4]:

1. Melt electrospinning.
2. Electrospinning by anionic treatment.
3. Electrospinning using ultraviolet rays.
4. Electrospinning with the help of CO_2.
5. Electrospinning by heat treatment.

5. Features of green electrospinning

The use of sustainable and green materials whether polymeric, additive, or solvent is consistent with the concept of green electrospinning. This contributes to sustainable development and better environmental protection, especially with regard to the use of solvent-free electrospinning as an environmentally friendly process that does not suffer from the evaporation of the solvent, the presence of toxic substances, or the need to recover the solvent.

The use of recyclable materials in the production of nanofibers, such as biodegradable bioplastics, and their application in filtration membranes can be considered an important advantage in this process, as these materials can become an alternative to polymers and be used to manufacture products in various fields.

Recycling polymeric materials not only solves the problem of environmental pollution but also solves the problem of resource shortage, as recycling polymeric materials and using them to produce nanofibers using electrospinning technology is consistent with the concept of green electrospinning, which is understood in sustainable development [4].

6. Challenges facing green electrospinning

The most prominent challenges facing this process are the high requirements for preparing this process, especially with regard to solvent-free electrospinning, in addition to the larger diameters of fibers manufactured by the latter method compared to the traditional method. Research is still ongoing on these processes and requires greater effort and continued work and research in this field [4].

7. Applications of green electrospinning

7.1 Application in the field of tissue engineering

The distinctive properties of nanofibrous scaffolds, which are similar in structure to that of human tissue, make them an excellent environment for cell adhesion, proliferation, and differentiation. Sustainable nanofibers promote the regeneration of human tissues, including skin, bones, cartilage, and others.

This field includes providing biologically active scaffolds and manufacturing supporting materials to build or repair damaged tissues or organs [3].

In bone tissue engineering, researchers have recently used safe materials as an alternative to previously used organic materials, such as hydrosil apatite (HAP), as this material has specifications that make it an alternative to bone material because it has a structure similar to the structure of natural bones. Researchers also found that silk is biocompatible and has the ability to be degraded. It is biocompatible and has excellent mechanical strength, making it suitable for use in bone tissue engineering [5].

In the field of skin tissue engineering, skin grafting operations have spread as an effective treatment, where biological materials, including sugars, are used to renew skin tissue. Beetroot has been used to obtain nanofibers through the electrospinning process. A composite material was prepared from hydroxyethyl cellulose (HEC) with silver nanoparticles and showed excellent specifications for regenerating skin cells. Additionally, cellulose reinforced with keratin, polycaprolactone with polysaccharides, gelatin, electrospun glucosamine, and PLLA with silver were used. These mats showed suitable porosity, wetness angle, mechanical strength, ability to decompose in water, and suitable support for cell cultivation. Some of them also showed resistance to bacteria, especially for mats containing silver particles [5].

In the field of muscle tissue engineering, a functional support similar to muscles has been created that has the ability to contract and relax. The trend has been to produce supports from materials that have electrical conductivity to allow the transmission of electrical signals to the muscle. Several studies have appeared in which

PLLA/PANi and chitosan were used with gelatin. Biocompatible nanofibers of the "shell-core" type were also used, the core being polycaprolactone and the coating being gelatin [5].

The researchers used PLGA to form vascular stents, which showed compatibility and biocompatibility [5].

7.2 Application in the field of wound dressings

In this field, plant extracts are used and added to the electrospun polymeric solution. The meshes used in this field are made of chitosan and polyvinyl alcohol. These have shown resistance to inflammation and antioxidants, as well as promoting the rapid reshaping of epithelial tissue. They also exhibit elastic mechanical properties and biocompatibility.

When using chitosan and polyvinyl pyrolidone, the mats were resistant to *Staphylococcus aureus* and *Escherichia coli*, and were antioxidants, with good thermal stability and affinity for water [6]. There are many materials used in this field, which are shown in the following **Tables 2** and **3**. While **Table 2** shows some synthetic polymers used in manufacturing wound dressings and explains the advantages and disadvantages of each material used, **Table 3** shows the natural polymers used for the same purpose with the same explanation in **Table 2** for each material.

7.3 Application in the field of face masks

Masks containing nanofibers have recently gained great importance due to their high filtration efficiency and the possibility of incorporating other materials into the polymeric materials that make up the fibers.

Material	Advantages	Disadvantages
PVA	Flexibility, breathability, keeps wet environment	Non-biodegradable, low strength, poor thermal stability
PEG	Biocompatibility, sensitive to different physical and chemical stimuli, soluble in organic solvents and water, neutral in acidity and alkalinity	Non-biodegradable, lack of immunogenicity, may cause contact allergy
PCL	Flexibility, biocompatibility, fast crystallization rate, durability	Slow biodegradation rate, hydrophobic, lack of cell binding sites, low mechanical strength
PLA	Good mechanical strength, in vitro or in vivo mechanical sustainability, thermal stability, reproducibility, versatility, and case of processing	Hydrophobic, lack cell binding sites
PEO	Biocompatibility, non-cytotoxic, water-soluble, easy to prepare	Non-biodegradable, low strength, poor thermal stability
PVP	Simple and clean synthesis process, low cost, soft and able to store large amounts of water without loss of mechanical integrity	Non-biodegradability, weak mechanical strength, and poor thermal stability
PLGA	Adjustable wettability, biodegradation rate, good solubility in common solvents	Poor ductility, relatively low drug loading capacity, high synthesis cost, poor cell affinity
PU	Good moisture permeability and elasticity, mechanical strength similar to natural tissue, high porosity	Hydrophobic, poor hemocompatibility

Table 2.
Some synthetic polymers used in the preparation of wound dressings [7].

Categories	Material	Advantages	Disadvantages
Polysaccharide polymers	SA	Biocompatible, degradable, low cytotoxicity, non-immunogenic, good film-forming properties	Not easy to spin, lack of cell binding sites, poor mechanical properties, and heat resistance
	Cellulose	Non-cytotoxic, excellent mechanical properties, good stability	Not easy to dissolve, difficult to spin
	CS	Biocompatible, biodegradable, low immunogenicity, anti-microbial, antioxidant activity, promotes fibroblast migration and proliferation	Poor solubility, slow and uncontrolled biodegradation rate
	HA	Biocompatible, stimulates cell migration, differentiation, and proliferation, regulates metabolism and organization of ECM	High molecular weight, high viscosity of spinning solution
Peptide-based polymers	GE	Biodegradability, non-immunogenic, macrophage activation, efficient hemostasis	Limited water solubility, poor mechanical strength and elasticity, poor thermal stability
	Col	Low immunogenicity, biocompatibility, promotes cell proliferation, adhesion, and granulation tissue formation	Low mechanical strength, easy to degrade

Table 3.
Some natural polymers used in the preparation of wound dressings [7].

All of this opened a great field of research in the field of multifunctional filtration membranes [8].

7.4 Application in the field of food

The use of toxic organic solvents and polymers that are harmful to the environment limits the use of the electrospinning process in the food field. Therefore, green electrospinning has found great interest in food engineering, especially for solutions that rely on using water as a solvent, in addition to biocompatible and recyclable polymers that can be combined with... Water-soluble polymers, examples of which include: PVA, PEO, silk fibers, protein, soybeans, collagen, and chitosan.

Food packaging is one of the most widespread applications, as nanofibers are loaded with materials that give them functional properties such as moisture absorption, bacteria resistance, and resistance to oxidation.

In this field, the term smart packaging appeared, where fibers play the role of a sensor and give information about the quality of packaged foods during handling and storage, in addition to information about the quality of the food. Gelatin fibers were used with chitosan loaded with turmeric, for example, and these fibers had properties of resistance to bacteria and oxidation and sensitivity to coloring. For ammonia, these membranes were effective in monitoring the quality of protein-rich foods in addition to being antibacterial.

Nanofibers were also produced using green electrospinning to treat beverages, as they filtered suspensions that negatively affect the quality of beverages [9].

7.5 Application in agriculture

The agricultural field is one of the most important fields in the world, but it suffers from various problems such as diseases, low soil fertility, and other problems.

Recently, nanotechnology and sustainable nanofibers have been used to solve many of these problems and reduce losses in agricultural chemicals such as fertilizers and pesticides, in addition to increasing crop productivity by eliminating plant pathogens [10].

The main applications of nanofibers have been identified along the following axes [10]:

1. Agricultural waste for the production of nanofibers.

2. Nanofibers help seeds germinate well by covering them with them.

3. Nano fiber-based filters for irrigation systems.

4. Nanofibers for plant protection.

5. Nano silica fibers grafted.

6. Smart nano-textiles for sustainable agriculture.

7. Nanofibers for packaging agricultural chemicals.

Globally, a large number of discarded wastes are produced annually from agricultural operations, and these wastes are considered a major source of cellulose, such as grain hulls, garlic, soybeans, cane sugar bagasse, and others. It is possible to produce cellulose nanofibers and cellulose nanocrystals from these wastes. Films of PVA were produced with starch reinforced with cellulose nanofibers from bagasse, and bamboo waste was used to produce cellulose nanofibers, in addition to the use of waste resulting from orange juice to obtain a biodegradable film of cellulose fibers. And use pomegranate peel extract with PVP and PVA for cosmetic purposes. Cellulose nanocrystals from coconut and rice husks were also obtained through hydrolysis, in addition to the production of cellulose nanofibers from pineapple waste.

It is also worth noting the possibility of using nanofibers in agriculture as smart conductors to improve the germination and growth of seedlings in rice, cowpeas, peanuts, and sesame.

Many nanocomposites, such as graphene oxide, can be used as an antibacterial agent with strong adsorption properties and photocatalytic ability [11].

7.6 Application in the environmental field

7.6.1 Using sustainable nanofibers in water and wastewater treatment

In light of the global water crisis, treating water and obtaining it in a pure and safe condition is extremely important.

Many researches have emerged studying the production of nanofibers based on environmentally friendly bio-origin polysaccharides and cellulose nanofibers for use in water treatment and pollutant removal.

The basic processes that take place in wastewater treatment include filtration, adsorption, catalysis, centrifugation, and biological treatment.

Many natural, synthetic, and biopolymers have been spun and used in wastewater treatment.

However, in this section, we will focus on the use of sustainable nanofibers, such as cellulose nanofibers (CNFs), which can be bonded to surface carboxylic functional groups via oxidation, in addition to modified cellulose nanofibers incorporated with different metal or organic molecules.

Water purification using these fibers is a physical and chemical method where stable chemical bonds are formed between nanofibers and metal ions.

Physically, the specific area of nanofibers, the pore size of their component mats, and their interconnected structure are among the basic elements that determine the performance of water treatment [11].

7.6.2 Using sustainable nanofibers in the field of air filtration

Electrospun nanofiber filters were fabricated from biopolyesters and the performance of these filters was evaluated by measuring the extent of penetration of aerosol particles through the filters. It was observed that the penetration of the nanoparticles to be filtered was obviously reduced by increasing the thickness of the filter, and the penetration rate of the particles to be filtered reached 0.8% using bio-nanofiber filters [12].

Polyvinyl alcohol (PVA) filters with carbon nanoparticles (CNP) and tea leaf extract (TLE) manufactured using the electrospinning method successfully produced functional nanofilters (FNA) and these filters showed a filtration efficiency of 99.9% [13].

7.7 In the field of textile

Recently, some innovative technologies have emerged in the textile industry that have departed from traditional ones and added a high value to this industry, such as smart textiles that have the ability to respond to various physical stimuli and interact with the environment. This was done by integrating sensors, motors and other electronic elements. In addition, chemically treated textiles have emerged that have added functional properties such as protection from sunlight, resistance to heat, flames, chemicals, microorganisms, ultraviolet rays, protection from wind and water, etc.

With the development of science, nanotechnology has played a major role in the textile industry by producing fine particles of nano-sized diameters with which the fabric is processed to obtain unique properties. Also, by obtaining fibers with diameters less than a micron, and for the latter, electrospinning technology played a pivotal role in the manufacture of smart textiles [14]. All of this can be summarized in **Figure 2**:

The combination of electrospinning and biopolymers has had the greatest impact in creating a qualitative shift in the textile sector by reducing its harmful environmental impact and introducing a model of sustainable, high-performance fabrics.

In this field, a wide range of smart textiles have emerged that also contribute to preserving the environment by using electrospinning based on biopolymers [14]. Below we explain some types of these textiles:

7.7.1 Antibacterial fabrics

Mats of electrospun fibers from biopolymers have appeared, with materials resistant to bacteria, whether Gram-positive or Gram-negative bacteria, such as *Staphylococcus aureus* and *Escherichia coli*, which cause infections in wounds and burns.

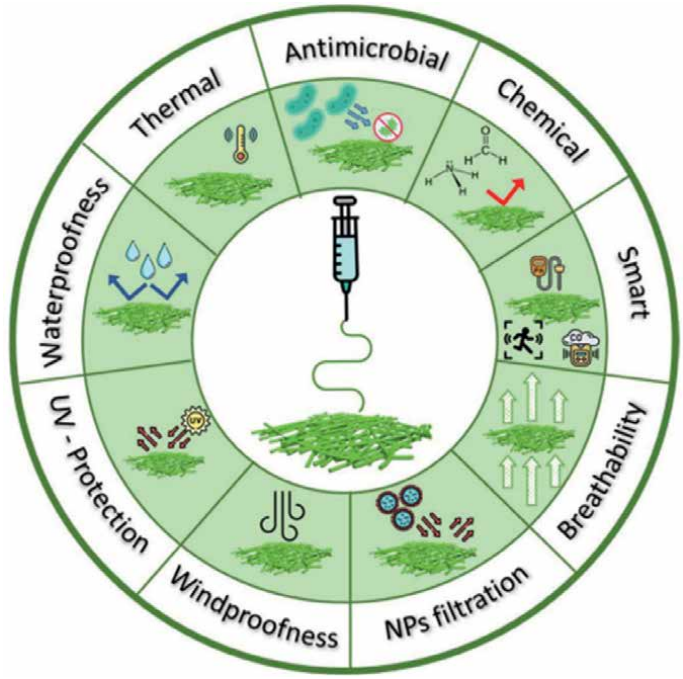


Figure 2.
Types of electrospun textiles prepared on bio-based and biodegradable basis [14].

One of the most commonly used polymers in this field is polyurethane (PU). Although this polymer is not resistant to bacteria, the addition of silver nanoparticles makes it antibacterial, in addition to chitosan, polyvinyl alcohol, polycaprolactone, and cellulose acetate, to which nanomagnesium and zinc oxides are added [14].

7.7.2 Air filters

Air filters can effectively filter fine particles from the air, such as particles with a diameter of 2.5 micrometers or less and smoke components. These textiles can be used in face masks and protective suits, as electrospinning technology is used to produce low-porosity nanonets capable of trapping nanoparticles.

Filtration efficiency depends on various factors such as mesh porosity, fiber diameters, and filter thickness.

Different types of polymers used in this field have emerged, such as chitosan, cellulose acetate, polylactic acid, and others [14].

7.7.3 Air-permeable and water-resistant fabrics

These types of fabrics have aroused interest in industry and sports as they can protect the human body from external factors while allowing water vapor to move from the body to the outside. Using electrospinning technology, it was possible to produce these types of fabrics due to the unique and distinctive properties of these mats [14].

7.7.4 Thermal protection fabrics

Fabrics with thermal protection, such as flame-retardant or thermoregulating, have attracted attention. Electrospinning has been used to design polymer fibers with thermal properties by incorporating phase change materials (PCMs) that can absorb heat and store it as potential energy within a limited temperature range.

These types of thermal protection fabrics have many problems that have been overcome by producing electrospun fibers with a shell-core structure of green polymeric materials [14].

7.7.5 Fabrics with chemical protection

The production of chemical protective textiles using electrospinning is of great importance as these textiles are used for personal protective equipment, military purposes, and medical and pharmaceutical industries.

These fabrics efficiently prevent dangerous chemicals from contacting the skin or exhibit anti-oxidation properties.

Mats were produced from biopolymer materials added to nano-metal oxides treated thermally or by irradiation with ultraviolet light [14].

7.7.6 Electronic textiles

Recently, there has been a notable boom in the market for wearable electronic devices integrated with clothing sensors for health monitoring, medical diagnosis, or entertainment. However, a common technical challenge these devices face is their reliance on rechargeable batteries. Hence, there is an urgent need to address this critical issue with flexible and sustainable energy sources that can consume the energy of body movement. Electrospun nanofibers are used as templates for the adsorption of conductive nanomaterials, such as silver, graphene, and carbon. Although the fields of e-textiles have seen tremendous developments, it is important to realize that a large proportion of these developments are based on non-biobased polymers, so there is a significant effort toward incorporating biopolymers into these technologies, highlighting how important it is to shift to friendlier alternatives. For the environment and sustainability [14].

8. Conclusion

The rapid progress in the field of nanotechnology and profound environmental awareness have directed researchers to pay attention to sustainable technology, including its inputs and outputs.

In this chapter, reference was made to the sustainable materials used and resulting from the green electrospinning process, and the basic principle of this process was explained, taking into account the difficulties faced by those responsible for it, in addition to explaining the applications of green electrospinning products represented by sustainable nanofibers, according to a brief explanation of these applications within different fields.

A quick overview of sustainable nanofibers was given, starting from the materials involved in their production, moving to the methods used in obtaining them and reaching the areas of their use, thus showing the possibility of using advanced technologies, taking into consideration the preservation of the environment.

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Chapter 3

High-Temperature Superconductors

Derejaw Gardew

Abstract

One of the main areas of focus in condensed matter physics over the past several years has been research on high-temperature superconductors and their physical characteristics. One of a metal's most crucial characteristics is its electrical resistivity, and scientists were especially curious about how this value varied with temperature. Because superconducting wires can carry enormous electrical currents without heating up or losing energy, superconductor-based electromagnets are incredibly strong. The current state of technology is predicted to alter with the development of a superconductor that runs at room temperature. Compared to existing technologies, a superconducting power grid would save a great deal of energy because it would not waste energy owing to very little resistance. High magnetic fields can be produced by high-temperature superconductors, which are advantageous for applications in medicine, such as magnetic resonance imaging (MRI) equipment. Superconductors are used in levitating trains, very accurate electromagnets, lighter and smaller engines, generators, transformers, and SQUIDs.

Keywords: condensed matter physics, high-temperature superconductors, Cooper pairs, Meissner effect, unconventional superconductors, BCS

1. Introduction

At low temperatures, a phenomenon in solid materials is known as superconductivity. It is remarkable and gives a clear explanation of the development in condensed matter physics, which could have a significant impact on society if we can transition to a low-carbon future. Mercury has no electrical resistance below the transitional temperature of 4.2 K, as Onnes observed in 1911 [1]. Certain materials have the ability to have strictly zero electrical resistance below a threshold temperature (T_C), a phenomenon known as superconductivity (SC). The scientific and engineering potential of superconductivity's uses has not yet been completely realized after more than a century of research on the phenomenon; the microscopic mechanics of superconductivity are still up for debate [2]. Even though superconductivity was discovered by accident, the experiment was part of a well-planned Leiden research program. It was fascinating to examine how metals, like gold and platinum, behave in terms of electrical resistance at extremely low temperatures, both theoretically and practically [3].

Merely a tiny portion of the electrons in a superconducting material are condensed into a molecule that fills the entire system and has the ability to move independently.

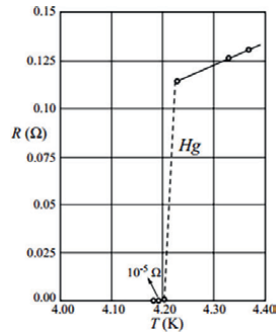


Figure 1.
Resistance versus temperature superconductivity in mercury [7].

Even while all electrons contribute to the formation of this superfluid at absolute zero temperature, only those electrons close to the Fermi surface have their movement critical understanding by the condensation. A weakly interacting gas of excitations is created when some of the condensates' electrons evaporate at a higher temperature. This gas permeates the superfluid and the full volume of the system. The system undergoes a second-order phase transition from the superconducting to the normal state when the temperature hits a critical point (T_C), at which time the fraction of remaining electrons in the superfluid approaches zero. This two-fluid model of a superconductor is conceptually comparable to that which represents superfluid He, despite dramatic differences between the two systems [4–6]. Research on high-temperature superconductors and their physical properties has been one of the major activities in condensed matter physics in the past several years, and still, there are some limitations on the superconductor material in general. The main goal of this chapter is to review high-temperature superconductors based on their different properties (**Figure 1**).

2. Discovery of superconductors

Following the discovery of superconductivity in mercury, it was discovered through measurement that a number of other metals were also superconducting, albeit at extremely low temperatures. Niobium has the highest elemental metal transition temperature (T_C), measuring 9.25 K. The so-called cuprate high-temperature superconductors are a novel type of ceramic superconductors that Bednorz and Müller discovered in 1986. Their highest T_C to date is 135 K at ambient pressure [8]. Since the 2008 discovery of the iron-based SC $LaFeAsO_{1-x}F_x$, researchers have been more interested in the phenomenon of high-temperature superconductivity [9].

3. Cooper pair and BCS theorem

One-particle states with time reversal typically yield Cooper pairs. As a result, any disruption that has a different effect on each member of a pair will cause the paired state to become unstable and subsequently collapse [10–13]. In iron-based superconductors, the pairing symmetry that is most often known is the

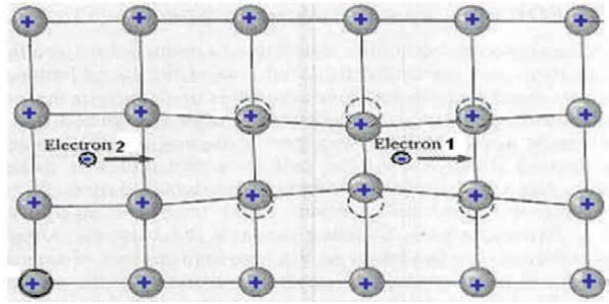


Figure 2.
Cooper pair formation in a lattice [16].

spin-fluctuation-mediated nodeless s ± model, where the superconducting gap between the hole and electron pockets moves in one direction [14].

In 1957, Bardeen, Cooper, and Schrieffer (BCS) introduced a remarkably successful microscopic quantum theory which is nowadays known as the BCS theory. Although experimentalists in the field do not yet have a reliable theoretical framework for determining high-T_C materials, the BCS theory provides the theoretical background for superconductivity at the microscopic level [5]. The remarkable accuracy of the BCS theory of the superconducting state increases the possibility that, in the future, researchers may be able to grasp and estimate specific numerical values for the superconducting transition temperature [15]. According to the BCS theory, electrons in a superconductor form Cooper pairs together while also condensing in a way that is analogous to the way bosons condense into a superfluid state. Two electrons with opposing momenta are drawn to one another in a superconducting state to create a bonded pair. The binding interaction is the connection of electrons with lattice vibration. The Boltzmann constant, k_B, and the pairing strength, which is expressed as an energy gap (Δ), are related as follows: $2\Delta = 3.53k_BT_C$. After electron-phonon interactions in fundamental superconductors were quantified and confirmed by experiments in the mid-1960s, the BCS theory became the accepted theory for typical superconductors (**Figure 2**) [16].

4. Meissner effect and types of superconductors

Unquestionably, the Meissner effect's discovery in 1933 marked a turning point in the development of superconductivity [17]. The Meissner effect occurs when a superconductor is maintained in a magnetic field. This phenomenon is associated with the elimination of magnetic fields upon cooling a superconductor to its critical temperature. This attribute, which is perhaps the most crucial for superconductors, indicates that their resistance is zero. Hence, superconductors exhibit zero, or almost zero, resistance to electrical currents in accordance with the Meissner effect; additionally, they are ideal diamagnetic [18]. We shall draw inspiration from another well-known electromagnetic interaction, namely the Coulomb repulsion between two electrons, to describe the Meissner effect, which is caused by an electromagnetic interaction [19]. Both the internal magnetic field of the superconductor and the external magnetic field are expelled in the superconducting state. Superconductivity's perfect diamagnetism allows the superconductor to hover above magnets (**Figure 3**) [20, 21].

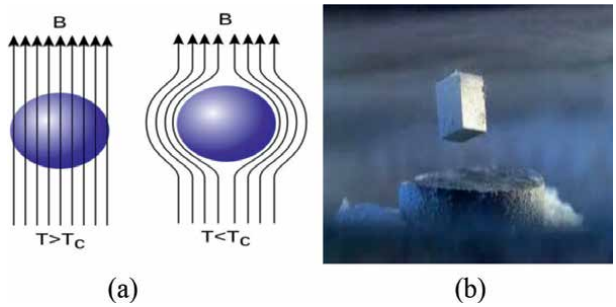


Figure 3.
Diagrams of the Meissner effect (a) and magnetic levitation (b) [20].

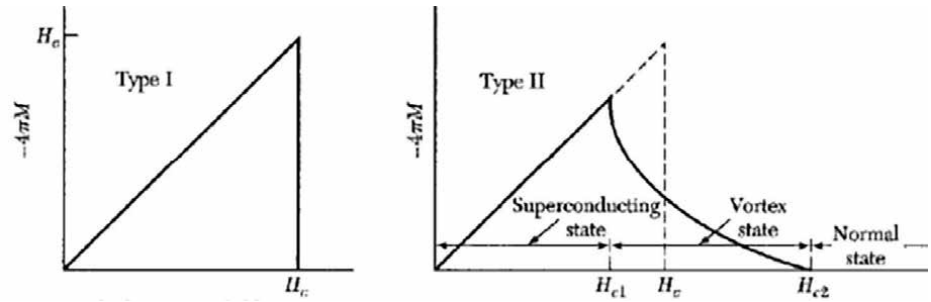


Figure 4.
Phase diagrams of type I and type II superconductors [22].

Superconductors are divided into two categories based on their response to applied magnetic fields. These are type I and type II superconductors. Meissner and normal states are supported by type I superconductors, whereas type II superconductors can generate magnetic vortices under sufficiently strong applied magnetic fields. Among the most amazing occurrences in crystalline materials is the development of vortices in type II superconductors when they are exposed to magnetic fields. These vortices are found in all type II superconductors, or superconductors where the GL characteristic parameter (the ratio of the magnetic field penetration length to the coherence length), $\kappa > 1/\sqrt{2}$ (**Figure 4**) [22–24].

5. High-temperature superconductors

High-critical temperature cuprates and the iron-based superconductors have common properties. Each of them has a layered crystal structure. (i) It is built on a planar layer of iron atoms connected by tetrahedral coordinated chalcogenide (S, Se, Te) or pnictogen (P, As) anions arranged in a stacking sequence and separated by alkali, alkaline-earth, or rare-earth blocking layers. These typical iron layers, which resemble the typical copper-oxygen layers in high-critical temperature cuprates, are where the superconductivity is observed. (ii) In high-critical temperature cuprates and the majority of iron-based superconductors, the superconductivity arises from the parent compounds by doping electrons or holes into them. The parent compounds possess a magnetic ground state. (iii) Many experimental and theoretical findings point to the existence of spin-singlet pairing in both cuprates and iron-based superconductors,

which are both unconventional superconductors. (iv) It is believed that an essential component of understanding superconductivity and its associated features is the Coulomb interaction between electrons, primarily because the superconducting state of the parent material is close to its magnetic state. Yet, there are several more differences between iron-based superconductors and cuprates. That is, cuprate parent compounds are mostly Mott insulators. On the other hand, the iron-based superconductors are mostly metals. Iron-based superconductors are typically thought to be in the intermediate correlation domain. The magnetic order in iron-based superconductors is spin density wave, as opposed to the localized magnetism of the Heisenberg antiferromagnetic [25–29].

5.1 Copper-based superconductor

Several families of cuprate HTS superconductors have now been identified. They are all composed of layers of CuO_2 divided by layers of insulating material. The insulating layers act as charge reservoirs, feeding the charge carriers to the CuO_2 layers, where superconductivity is found [8]. Superconductivity was later found at a transition temperature exceeding 105 K in the Bi-Sr-Ca-Cu-O system. Thallium-based cuprates were discovered to exhibit superconductivity at 120 K that same year [30].

Due to the weak conductivity of these compounds, their conclusion was unexpected and required attention. The highest confirmed critical temperature at the time was 33 K, and dozens of high-temperature compounds have been identified in subsequent years. As nitrogen boils around 77 K, it is highly plausible that new technologies in the realm of high T_C such as superconducting quantum interference device (SQUID) magnetometers and Josephson integrated circuits may developed. While this is interesting, early reports of levitating trains appear improbable. Although the essential current densities are still too low for the majority of engineering fields, this area is moving forward rapidly [31, 32].

5.2 Iron-based superconductors

The study of iron-based superconductors is currently one of the most interesting fields of condensed matter physics since the discovery of superconductivity in fluorine-doped LaFeAsO. Two significant subgroups of the numerous families of components contained in the iron-based superconductors are the iron pnictides and iron chalcogenides. The highest superconducting transition temperature (T_C) for iron pnictides is 56 K, but it is 65 K or even higher for single-layer iron chalcogenides. High-temperature superconductivity in iron-based superconductors is considered to arise from interactions between electrons. This makes it easier to compare and contrast cuprates and other related superconductors with iron-based superconductors. Superconductivity only appears within a narrow spectrum of chemical doping, and the majority of parent compounds of iron-based superconductors, like cuprates, have an antiferromagnetically ordered ground state. These properties cause substantial questions regarding the role of electron correlations in iron-based superconductors and how they interplay with the superconductivity in these materials [33]. Understanding the process of unconventional superconductivity has shown to be one of the most challenging problems in condensed matter physics. $LaFeAsO_{1-x}F_x$, an iron-based superconductor, has recently been found to be superconducting, which has brightened interest and might provide a creative viewpoint on this issue. Supposedly, it has been discovered that LaFePO,

LaFeAsO, and similar compounds possess first-principles band structures. These band structures, in contrast to the outcome for undoped LaFeAsO, have five Fermi surface components and are metallic [34].

An early theory about the physics of the pnictides is comparable with that of the cuprates and involves insulating properties driven by the proximity of antiferromagnetism and superconductivity [35–37]. Iron-based superconductors have several unique properties such as robustness to impurity, high upper critical magnetic field, and excellent grain boundary nature. These properties are advantageous for applications. Recent progress in the performance of superconducting wires of iron-based superconductors are wide eyed, that is, the maximal critical current has reached the level of commercial metal-based superconducting wires [38].

6. Families of iron-based superconductors

Iron-based superconductors can be classified into numerous different families, such as 11, 111, 1111, and 122, depending on their crystal structures and stoichiometry [39–41]. These four families have a variety of features similar to cuprate superconductors, including layered structure, the presence of competing orders, low carrier density, small coherence lengths, and unconventional pairing mechanisms. All of these behaviors could be a drawback in real-world applications, particularly given how they can cause significant thermal fluctuations and depressed grain boundary superconductivity. The iron-based superconductors have metallic parent compounds, their anisotropy is typically smaller and does not firmly rely on the amount of doping, and their order parameter symmetry is generally assumed s-wave, which is, in theory, not detrimental to current transport across grain boundaries [42, 43]. The 111 family has received significantly less attention than the 11, 1111, and 122 families of iron-based superconductors. It is still unclear where superconductivity has emerged in this group of compounds and, more broadly, in all iron-based superconductors. When the substance is doped or is under pressure, some families such as the 122 and 1111 possess long-range magnetic order, which is reduced in favor of a superconducting phase (**Figure 5**) [45].

FeSe, FeTe, and their variations, which have a square lattice of Fe-atoms with Se/Te above and below the Fe-plane, are among the families of Fe-based superconducting materials that are known to exist. Even though the 11 family members

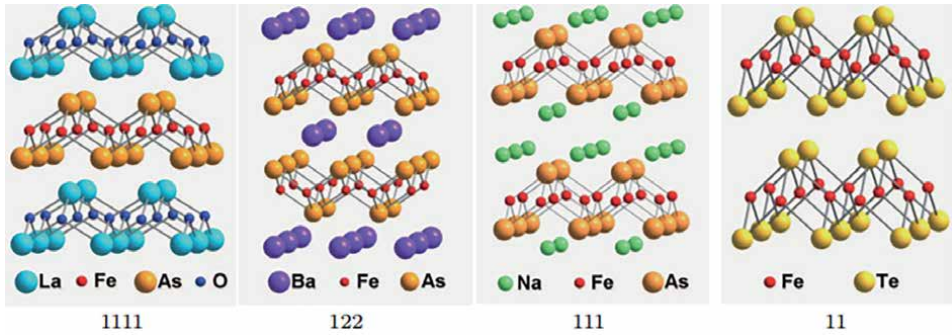


Figure 5.
Schematic crystal structure of four representative iron-based superconductors [44].

have the simplest structure compared to all the other families, they possess the highest (T_C = 100 K) when grown on single-layered $SrTiO_3$ [46, 47]. Since there are no intermediate layers in the 11 family, which is distinctive, it is thought that this family will enable the isolation of the intrinsic features of iron-containing planes [48, 49]. The magnetic correlations in the 111 family of iron-based superconductors have less attention than the other families. The origin of superconductivity in these series of compounds and in general in all IBSCs has still to be well clarified. An Angle-resolved photoelectron spectroscopy (ARPES) study on the LiFeAs system has addressed the consistency of the ARPES measurements on several families of IBSCs (111) [50].

The 1111 family of superconductors are the first family of the IBSCs to be discovered and one of the well-researched classes of high-temperature IBSCs [51–53]. The great resolution of these compounds was highlighted in the preliminary work of the critical current density (J_C) of 1111 polycrystalline samples, which prevented global J_C levels to very low values [54]. Another contentious issue is whether or not all families can be accurately depicted by a single image, specifically, the iron pnictides 1111 phase, whose parent molecule has the generic chemical formula REFeAsO (RE = La, Sm, Nd, Ce) [55, 56].

A mother compound of the 122 family of iron-based arsenide unconventional superconductors is $BaFe_2As_2$. It makes a tetragonal $ThCr_2Si_2$ crystal with the space group I4/mm as it crystallizes [57]. The crystal structure of the AFe_2As_2 (A = Ca, Sr, Ba) compound is simpler and consists of Fe_2As_2 layers that are similar to those present in LnFeAsO and are separated by single elemental layers [58]. The main members of the subclass of 122-type compounds are $CaFe_2As_2$, $SrFe_2As_2$, $BaFe_2As_2$, $EuFe_2As_2$, and their derivatives. Furthermore, in the Ba-based 122 systems, methodical replacement of Ba, Fe, or As atoms with a different element can, in some instances, motivate the antiferromagnetic state of the parent compound to a superconducting ground state. These factors made the parent $BaFe_2As_2$ composition the subject of most studies [59–62]. Since there are so many high-quality single crystals in this family, ARPES experiments have been done on iron-based superconductors, particularly for 122 families, to expose the Fermiology of iron-based superconductors. The basis of iron-based superconductors is the 122 family, which includes $BaFe_2As_2$, $CaFe_2As_2$, $SrFe_2As_2$, and other compounds of the highest quality single crystals [63, 64].

7. Crystal structure of high-temperature superconductors

These and related materials have been thoroughly investigated since the discovery of potential high-T superconductivity in a mixture of molecules in the La-Ba-Cu-O chemical system [65]. It has been stated that the structure of high-T_C copper oxide or cuprate superconductors is a deformed, oxygen-deficient, multi-layered perovskite structure. These compound structures are frequently closely similar to perovskite structures. Superconductivity occurs between alternating layers of CuO_2 planes, which is one of the characteristics of oxide superconductor crystal structures. Due to the holes created in the oxygen sites of the CuO_2 sheets, this structure results in a significant anisotropy in normal conducting and superconducting properties. With a far higher conductivity parallel to the CuO_2 plane than in the perpendicular direction, the electrical conduction is highly anisotropic. Critical temperatures are typically influenced by the oxygen level, cations substituted, and chemical compositions. They can be categorized as super stripes, which are specific implementations of

superlattices at the atomic limit consisting of wires, dots, and superconducting atomic layers divided by spacer layers, providing multiband and multi-gap superconductivity (**Figure 6**) [66].

A vigorous hunt for novel superconductors has been sparked by the recent discovery of superconductivity in $LaFeAsO_{1-x}F_x$ at the superconducting temperature of T_C = 26 K. The discovery of a high-T_C value of 55 K in $SmFeAsO_{1-x}F_x$ later on shows that Fe-based superconductors have promise that goes beyond that of traditional BCS superconductors. The large range of compounds is also helpful in the search for novel high-T_C superconductors. Two-dimensional stacking layers make up the typical crystal structure of Fe-based superconductors. The superconducting current is often conducted by Fe Pn- or Fe-Ch-layers (Pn = pnictogen, Ch = chalcogen) (**Figure 7**) [67].

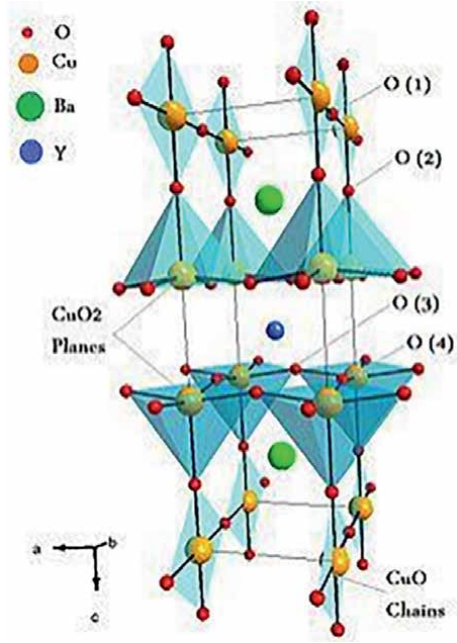


Figure 6.
YBCO unit cell [66].

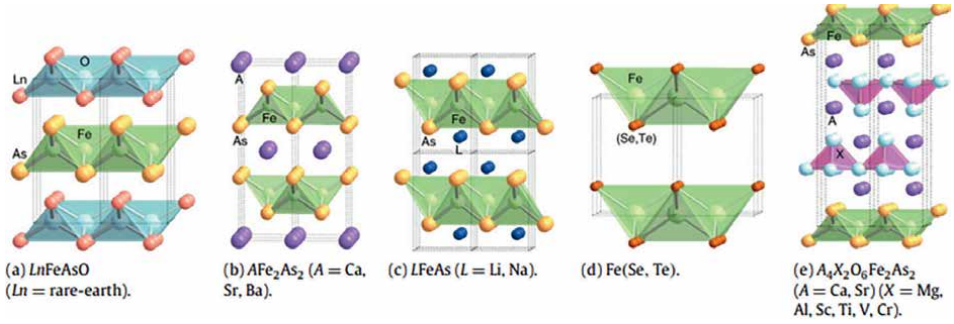


Figure 7.
Crystal structures of iron-based superconductors. (a) LnFeAsO, (b) AFe_2As_2, (c) LFeAs, (d) Fe(Se,Te), and (e) $A_4X_2O_6Fe_2As_2$ [67].

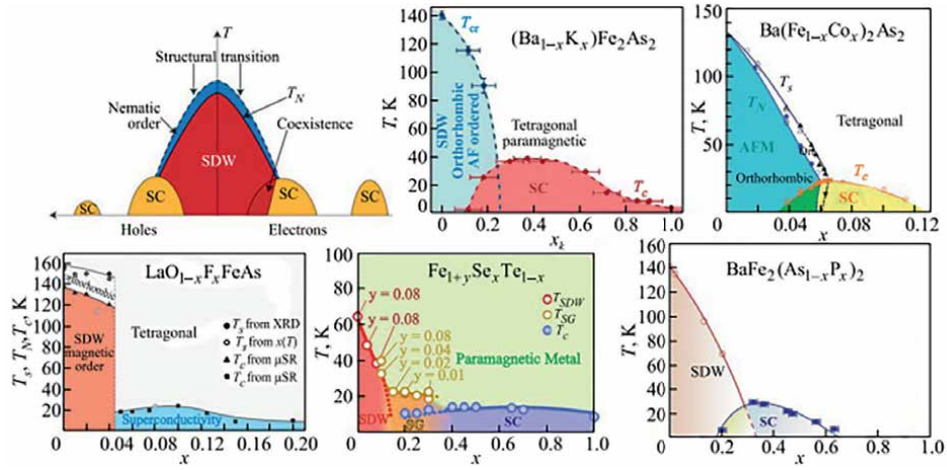


Figure 8.
Examples of the iron-based superconductors phase diagrams: a schematic one, and the diagrams measured for $(Ba_{1-x}K_x)Fe_2As_2$, $Ba(Fe_{1-x}Co_x)_2As_2$, $La(O_{1-x}F_x)FeAs$, $Fe_{1+x}Se_xTe_{1-x}$, $BaFe_2(As_{1-x}P_x)_2$ *[68].*

As a consequence of good crystal quality and the variety of its compounds, the 122 family is the most studied by ARPES (**Figure 8**) [68].

The phase diagrams in **Figure 9**, show the coexistence of the iron-based superconductors [69–71].

8. Pairing mechanisms of high-temperature superconductors

In iron-based superconductors, superconductivity can be produced by hole or electron doping, as well as pressure or the isovalent substitution of one pnictide element by another, such as As by P [72, 73]. In type II superconductors, the transitional current frequently exhibits an unusual peak right before it drops to zero at the upper critical field (H_{C2}). Since the early 1960s, this peak effect has been well-known and has received ongoing interest. Yet the exact mechanism is still unknown [74].

By using tunneling and neutron scattering experiments (TNSE) on conventional superconductors, the electron-phonon interaction, which is responsible for electron pairing and the existence of superconductivity, was proven. It is essential to comprehend the electron-boson coupling and how it relates to superconductivity in order to collect the equivalent data for high-transition temperature superconductors [75–77].

A framework for investigating the process of high-temperature superconductivity is provided by iron-based superconductors. Since superconductivity is practically realized at a spin density wave phase, the significance of spin fluctuations as a potential superconductor process is well acknowledged. An electronic nematic phase, which is also near the superconducting phase, is present, as can be seen by carefully examining the phase diagram of an iron-based superconductor. Although the cause of the nematic phase is still debatable, it has been demonstrated that orbital nematic fluctuations result in strong coupling superconductors with an observation compatible with the onset temperature. The electrical structure of iron-based superconductors is distinguished by several bands that emerge from five of the Fe ion's third-order orbitals. As a result, orbital fluctuations are also investigated as a potential superconducting mechanism. The transition temperature is thought to be

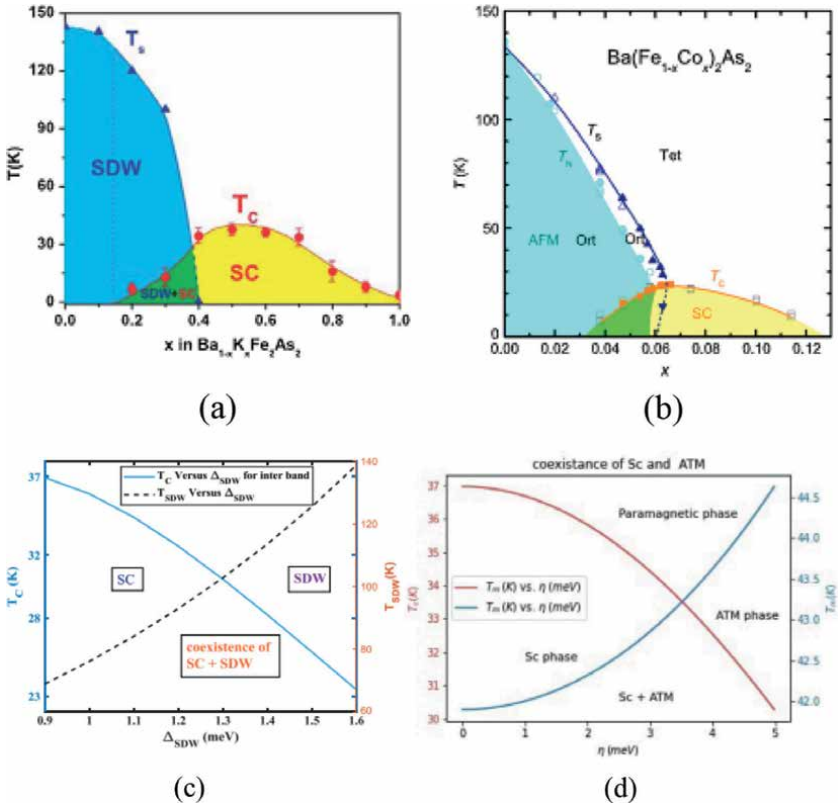


Figure 9.
Coexistence of iron-based superconductors $Ba_{1-x}K_xFe_2As_2$ (a), $Ba(Fe_{1-x}Co_x)_2As_2$ (b), $Ba_{1-x}Rb_xFe_2As_2$ (c), $Sr_{1-x}N_xFe_2As_2$ (d) [69–71].

too low compared to the observation despite the fact that electron-phonon coupling is present in real materials and is predicted to lead to superconductivity. One of the main problems with iron-based superconductors is distinguishing between various superconducting mechanisms [78]. It is important to clarify the pairing symmetry of the superconducting wave functions for the recently discovered high-temperature iron-based superconductors, for which the multi-gapped s-wave is often acceptable. The conventional s ± wave without sign-reversal and the unconventional s ± wave with sign-reversal are examples of potential candidates [79]. Spin fluctuation, charge fluctuation, and electron excitation-based non-BCS mechanisms have recently received considerable interest, especially in light of the potential for high-transition temperature superconductivity. These methods share the fact that superconductivity is the result of the electron-electron (e-e) interactions [80].

8.1 Phonon-mediated pairing mechanism

It has been demonstrated that certain dense metal hydrides have high-critical temperatures (T_C) and are conventional superconductors. Nevertheless, computing the T_C of such materials from first principles via electron-phonon calculations is still quite costly. Prior to these computations, it was not always evident which specific systems and structures could display high-temperature superconductivity [81].

The creation of Cooper pairs is based on an attractive contact controlled by phonons in most conventional superconductors. Furthermore, due to its inherent interest, Cooper pairing driven by other bosonic excitations have also received a lot of attention. Many compounds found during the past few decades have exhibited superconducting property which has been explained by some of the suggested mechanisms. But, in this discipline, detailed microscopic knowledge is really essential. In the boro-carbide materials, where new investigations have revealed that the issue with the pairing interaction, which was formerly assumed to be typical electron-phonon coupling, is far from being understood, the situation is especially perplexing. These substances exhibit highly intriguing phase diagrams where superconductivity coexists with antiferromagnetic order in modest critical temperatures ranging from 6 K to 16.5 K [82–84].

It is essential to acquire a thorough knowledge of electron-phonon physics in these recently discovered superconductors since it is thought that the electron-phonon interaction will play a significant role in these observations. ARPES has been extensively used to study the strength of the electron-phonon coupling in graphite and graphene, although the interpretation of the data is not always simple. For instance, the anisotropy of the electron-phonon interaction in graphene adorned with alkali metals led to intense discussion regarding the function of van Hove singularities [85]. Several theories have been proposed to explain the occurrence of superconductivity in mercury and other metals since its discovery. The Bardeen-Cooper-Schrieffer theory, which is still among the most effective theories to date was ultimately shown to be the accurate explanation. The bosonic Cooper pairs formed by the electrons and an effective attraction between them are the two fundamental components of the BCS theory [5]. The Bose-Einstein condensate is then formed by the Cooper pairs in the superconducting state. Nevertheless, electrons should have repelled one another rather than attracted one another in accordance with Coulomb's law. The attraction mediated by phonons was used by the BCS theory to resolve this somewhat conflict (the quanta of atomic ion vibration). But how can the Coulomb repulsion countered by a phonon-mediated attraction that is powerful enough? The response to this question requires a unique metal characteristic: A metal acts as though its electrons were quasi-particles, which are non-interacting objects despite the strong Coulomb repulsion at low energy (or low temperature). Any source of attractive interaction between the quasi-particles will now serve. In typical materials like mercury, phonons are essential imaginary excitations that convert the repulsive Coulomb attraction into a weak attraction. However, it is generally believed that the electron-phonon interaction alone is insufficient to overcome the remaining Coulomb repulsion in high-transition temperature superconductors, such as cuprates and iron-based superconductors [86].

Due to their special qualities and strong connection to the mysterious occurrence of high-transitional temperature superconductivity, superconductors with atypical or non-s-wave gap symmetries continue to be of significant interest [87]. The degree of electron-phonon coupling and the specific energy range of phonon excitations are the key indicators of the superconducting transition temperatures of phonon-mediated superconductors [88].

8.2 Pairing mechanism by spin fluctuations

Fundamentally, a phonon mechanism was demonstrated to be impossible for this system, and from the very beginning of the work, a spin-fluctuation-mediated pairing was suggested [89]. The gap function possesses s-wave symmetry, but its

sign is changed between the electron and hole Fermi surfaces, where the spin fluctuation mediates s-wave pairing [90]. The adjacent phase's order parameter fluctuations could be a superconducting mediator. With a scientific study of the order parameters and order parameter fluctuations of the adjacent phases, this idea could contribute to the identification of novel types of superconductors. There have been two hypotheses for the superconducting mechanism of iron-based superconductors, which are based on magnetic and orbital characteristics. Iron-based superconductors exhibit magnetic and structural phases close to the superconducting phase. Two novel potential candidates for the superconductivity mechanism of iron-based superconductors are spin fluctuations and orbital fluctuations [91]. It has been suggested that the pairing in iron-based superconductors is likewise mediated by spin fluctuations with a sign change in the superconducting order parameter based on the similarities of the phase diagrams for iron-based superconductors and cuprates. Hence, in order to obtain the Fermi surface geometry of the iron-based superconductors, the sign changes symmetry must differ from the cuprates d-wave symmetry. This can be met by an extended s-wave pairing between various Fermi surface pockets [92].

It is important to note that the unconnected electron and hole Fermi surfaces are present in this system because they cause spin fluctuations around the wave vector that joins the Fermi surfaces. The corresponding pairing state, known as the s ± state, occurs when the gap function between the electron and hole Fermi surfaces change direction [93–95]. The relationship between magnetic spin fluctuations and superconductivity has a historical background. It was first discovered as a way to explain why the transition temperatures of some transition metals were suppressed, then it provided a mechanism for p-wave pairing in ^{3}He, and finally, it was proposed as a d-wave pairing mechanism for some organic superconductors, heavy-fermion systems, and perhaps the high-transition temperature cuprates [96].

Large isotope effects pointing at the involvement of phonons have also been reported; however, resonances shown in inelastic neutron scattering experiments suggest the presence of spin fluctuations in the pairing mechanism. The typical energy of these phonons is of the same order of magnitude as the corresponding spin fluctuations. The symmetry of the superconducting gap itself has shown to be important in this situation for identifying the pairing mechanism that drives it [92]. Another benefit of this pairing mechanism is that if the gaps in the various bands have the same or opposite signs, the inter-band pair interaction can increase superconducting regardless of whether it is attractive or repulsive [97–99].

9. Factors affecting superconductors

9.1 Effect of hole-electron doping

Currently, condensed matter researchers are debating on the symmetry, structure, evolution, and potential effects of doping on the superconducting gap of the iron-based superconductors. According to the researchers, superconductivity in iron-based superconductors is of electronic origin and occurs from the standardized assessment of Coulomb interaction, which is accelerated at specific moments by significant magnetic fluctuations or orbital fluctuations [100]. Several theoretical investigations and experimental findings strongly support the significance of inelastic inter-band

scattering between hole and electron Fermi surface pockets connected via the antiferromagnetic wave vector, despite the fact that the specifics of the pairing mechanism in the recently discovered iron-based superconductors are still one of the hotly debated topics in physics [100, 101]. The structural phase of $BaFe_2As_2$ (122-family) is especially well suited for a systematic investigation of the chemical potential shift since it can be doped with either electrons or holes as a result of partial substitutions by Fe^{2+} Co^{3+} or Ba^{2+} K^{+}, respectively [101].

Superconductivity cases with electron and hole doping are undergoing extensive experimental investigations. For instance, $LnFeAsO_{1-x}F_x$ provides an illustration of how electron doping successfully induces superconductivity in the 1111 phase (where Ln: lanthanide). It is crucial to have knowledge of both the electron- and hole-doped superconductivity instances when analyzing how carrier doping affects the Fermi surface [63].

Generally, the electronic structure of Fe pnictides near the Fermi level consists of several bands mainly from Fe 3d orbitals hybridized with Arsenic (phosphorus) orbitals. The undoped compounds show two different types of Fermi surfaces: hole pockets at the Γ-point and electron pockets at the M-point in the Brillion zone. They are almost the same size, leading to spin-density-wave type instability through strong inter-band nesting effects. Upon hole-doping, for example, the hole pockets grow while the electron pockets shrink, which in turn spoils the nesting condition for the spin density wave phase. Strong inter-band scattering is thought to offer a key pairing channel for superconductivity after the spin density wave phase is adequately suppressed [102]. More doping, however, causes the electron bands to entirely empty and lose the inter-band pairing channel, which suppresses superconductivity and causes the T_C to fall. The electron-hole symmetry in the phase diagram may result from a similar mechanism working in the electron doping regime as well [103, 104].

9.2 Effect of external pressure on iron-based superconductors

The $AeFe_2As_2$ (Ae = Ba, Sr, Ca) family of iron-based superconductors show a comparable behavior under external hydrostatic pressure as under chemical ion substitution. They work as a control parameter that inhibits the parent compound's static magnetic order and causes superconductivity [105]. From the initial study on iron-based superconductors, it has been clear that one of the most common ways to raise the critical temperature of superconducting is by introducing external pressure. $BaFe_2As_2$, which has not been doped, is an antiferromagnetic metal that does not show a superconducting transition in the presence of atmospheric pressure. Yet, applying external pressures of 1–3 GPa exhibits superconductivity with a T_C up to 34 K, and the suppression of antiferromagnetism is exhibited [106].

The key to determining the normal and superconducting properties of iron-based superconductors is thought to be their magnetic properties [107]. By changing bond lengths and angles, the pressure modifies the lattice constants, which in turn affects the electronic and magnetic correlations and has an impact on variables like the electron-transfer integral and exchange coupling. Both cuprates and iron-based superconductors exhibit the dome-shaped pressure dependency of T_C, where pressure initially improves T_C to an ideal value, but later T_C decreases. This implies that, the Cooper pair coupling, strength fluctuates, and the electronic state with the highest T_C under high pressure is regarded as the ideal doped state [108].

9.3 Effect of isovalent substitution on superconductivity

When the antiferromagnetic phase is inhibited by pressure or chemical doping of the parent material, superconductivity appears in iron pnictides. It is now commonly recognized that charge doping and an isovalent chemical dilution (such as the partial replacement of As by P or Fe by Ru) exhibit distinct distinctions. Charged dopants provide robust scattering potentials that have an impact on superconducting characteristics including vortex pinning, the upper critical field, and even the superconducting gap symmetry while isovalently diluted chemicals are closer to the clean limit [109, 110].

Superconductors formed via isovalent doping have completely different gap structures than those created by hole or electron doping while being close to an identical ground state of the parent material and having similar T_C values. At the ideal doping, the superconducting gap in charge-doped BaK-122 and BaCo-122 is full and isotropic. Moving away from the appropriate doping level toward either end of the superconducting dome becomes anisotropic and even produces nodes. Regardless of the doping amount, the superconducting gap of isovalently substituted BaP-122 shows line nodes [111, 112]. Another way to look at how the transport characteristics of the 122 family have changed is through the isovalent replacement of Fe by Ru. Ru has recently been demonstrated to suppress spin density wave magnetic order and generate superconductivity in polycrystalline materials [109, 113].

10. Applications of high-temperature superconductors

In fact, such wires already exist, but they are still relatively expensive and therefore they are only used in applications where the best performance prevails over costs [3]. The discovery of higher T_C materials extends the feasible applications of superconductors [81]. A very high upper critical magnetic fields, low anisotropy, and large J_C values of iron-based superconductors, which are only weakly lowered by magnetic fields at low temperatures, recommended significant potential in large-scale applications shortly after the exploration of superconductivity in these materials. This is especially the case at low temperatures and high fields. The 122 compounds with the chemical formula AFe_2As_2 (A = alkaline-earth metal) are considered to be the most promising among the various families of the IBSCs since they are the least anisotropic, have a fairly high T_C of up to 38 K, which is comparable to MgB_2, and have high-critical current densities [114]. Ferromagnetic interactions also play a significant role in the hole- and electron-doped $BaFe_2As_2$ families of iron-pnictide superconductors, according to recent nuclear magnetic resonance observations [115].

The extremely low operating temperature of conventional superconductors inhibits their use in practical applications. The practical uses of superconductors are increased by the discovery of materials with larger T_C. Lossless power transfer is another potential used for superconductivity. A superconducting wire has the ability to transmit an AC with a very small loss or a DC with no losses. The potential of superconducting Maglev trains is made possible by the extreme type II feature of high-temperature superconductors, which is characterized by extremely high upper critical magnetic field values. Japan railways (JR) is working on a novel project to build a superconducting magnetically levitated (maglev) train called the JR Maglev, which travels at a high speed without touching the tracks due to

persistent-current-mode superconducting magnets. Superconductivity has three outstanding properties: persistent current, perfect diamagnetism, and the Josephson Effect [116, 117].

11. Conclusions

Research on high-temperature superconductors and their physical properties has been one of the major activities in condensed matter physics in the past several years. The electrical resistivity of a metal is one of its most important properties, and scientists were particularly interested in how this number changes with temperature. As is well-known, Maglev trains, magnetic resonance imaging, magnetic confinement fusion reactors, and particle accelerator beam-steering and focusing magnets all require powerful superconducting electromagnets. Lossless wires and cables are made possible by superconductor technology, which increases the grid's dependability and efficiency. Nowadays, it has been found that iron-based superconductors (IBSCs) have high upper-critical magnetic fields (H_{C2}), low electromagnetic anisotropy, relatively high superconducting transition temperatures (T_C) and a large critical current density (J_C). Because of these, iron pnictides have attracted enormous attention since the discovery of iron-based superconductivity in LaOFeAs with a superconductor transition temperature of 26 K. Shortly after this discovery, higher T_C values IBSCs were discovered in these oxypnictides.

Furthermore, there are various compelling reasons to be interested in IBSCs. First, they yield intriguing physics due to the coexistence of superconductivity and magnetism in these systems. Secondly, by giving a considerably wider range of compounds for research and having a multiband electronic structure, they raise the prospect of resolving the process of high-temperature superconductivity and discovering a technique to increase T_C . They are desirable for electrical power and magnet applications since they have higher H_{C2} than cuprates and high isotropic critical currents, while the coexistence of magnetism and superconductivity makes them fascinating for spintronics.

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Conflict of interest

The authors declare no competing interests.

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Chapter 4

Perspective Chapter: Applications of Biological Microlenses and Nanofibers

Yuchao Li, Heng Li, Xixi Chen, Tianli Wu and Baojun Li

Abstract

In recent years, with the rapid development of micro/nano optics, biophotonics, and biomedicine, micro/nano optical devices have been widely used in biosensing, medical imaging, molecular diagnosis, and other fields due to their advantages of miniaturization and integration. However, micro/nano optical devices composed of semiconductor and precious metal materials are prone to irreversible physical damage to biological cells and tissues and require chemical synthesis, which cannot be naturally degraded *in vivo*. In addition, due to the limitation of solid materials, micro/nano optical devices are difficult to deform and move in practical applications such as optical imaging and signal detection. Therefore, it is necessary to find a natural, biocompatible, biodegradable, and controllable micro/nano optical device. During the evolution of nature, some organisms have formed bio-optical devices that can manipulate light beams. For example, algal cells have the ability to concentrate light, which can improve the efficiency of photosynthesis. Visual nerve cells have the ability to direct light and transmit images to the retina with low loss and distortion. These natural materials capable of light regulation bring new opportunities for biological micro/nano optical devices, which have potential applications in the assembly of biological cells, detection of biological signals, imaging *in vivo*, and single-cell diagnosis.

Keywords: optical micro/nanofibers, microlenses, bioimaging, optical detection, biomedicine

1. Introduction

High-sensitivity detection of biological signals and magnified imaging *in vivo* is of great significance in the diagnosis, prevention, and treatment of biological diseases. Among them, the application of micro/nano optical devices in the fields of life science research, environmental monitoring, and medical applications is growing, providing the possibility to detect and observe biological signals in complex environment [1]. At present, micro/nano optical devices based on surface plasmon resonance, optical micro/nanofibers, optical resonators, and photonic crystals have made significant progress in optical detection [2–4]. Surface plasmon-based optical detection uses metal film and medium surface to excite surface plasmon, and its resonant transmission changes with the change of medium refractive index. By observing the change of the

characteristic parameters of the interaction between the incident wave and the surface plasmon, the change of the surface plasmon resonance transmission constant can be obtained [5]. With the continuous development of surface plasmon resonance, optical detection technology utilizing plasmon resonance of single metal nanoparticles or particle arrays can effectively improve the sensitivity and integrability of devices. When surface plasmons are confined in nanoparticles, and the size of particle is comparable to the incident wavelength, the free electrons of the particles can oscillate to generate localized surface plasmons [6]. The local surface plasmon resonance can be tuned by particle size, shape, and composition [7], but surface plasmon resonance is sensitive to interference factors such as sample composition and temperature. Unlike those based on surface plasmon resonance, optical micro/nanofibers are usually fabricated on thin films and low-refractive-index substrates using silicon or polymers [8]. When the light propagates in the micro/nanofiber film, the evanescent wave passes through the cladding covering the target molecule to sense the refractive index change of the sample, which can detect the optical signal [9]. In addition to optical detection using surface plasmon resonance and optical micro/nanofibers, optical detection can also be performed using optical resonators. When the incident beam is totally reflected in the cavity, the generated evanescent field can interact with the material on the surface of the resonant cavity, thereby realizing the detection of the signal in the cavity [10]. Most of the existing optical resonators are made of glass, semiconductor, and other materials [11]. Their surfaces can be functionalized, but the change of external factors will cause the shift of the resonant wavelength, thus affecting the performance of the optical resonator. Photonic crystals can be periodically arranged in space by two or more materials with different refractive indices, while introducing defects to break the problem that light cannot propagate in the crystal, providing new possibilities for controlling and manipulating light [12]. This structure has the advantages of low cost and integrability. These micro/nano optics have become powerful tools for applications such as optical imaging, signal detection, and disease diagnosis. With the rapid development of science, especially the advancement of biomedicine and nanophotonics, more and more studies have been conducted on micro/nano optical devices with high biocompatibility and good biodegradation for nano manipulation, optical imaging, and biosensing in a biological environment [13–16]. The existing micro/nano optical devices are mainly composed of inorganic materials or metal materials. These materials have the advantages of high transparency and high refractive index, but they need to be chemically synthesized and cannot naturally degraded *in vivo*. Therefore, biological cells and tissues are susceptible to irreversible physical damage, thereby greatly limiting their applications in biological environments such as optical imaging and detection.

In the process of evolution of nature, some organisms have formed bio-optical devices that can regulate light beams, which provides inspiration for the construction of biocompatible and biodegradable micro/nano optical devices. These bio-optics are expected to realize biological signal detection, *in vivo* imaging, and disease diagnosis in biomedical applications. Based on the interaction between biological cells and light, biological microlens, biological optical micro/nanofiber, biological laser, and other micro/nano optical devices can be constructed [17–19]. Biolasers can be constructed through Fabry-Perot cavities and whispering gallery resonators to achieve cell labeling, diagnosis, and imaging. Bio-optical devices designed and assembled by biological cells or biological macromolecules have the advantages of biocompatibility, noninvasiveness, biodegradability, and resorbability, so they can perform optical detection and imaging in biological environments, offering great potential for bioimaging and therapy [20, 21].

This book chapter aims to review the fundamental principles and essential technologies of microlenses and nanofibers, along with their applications in biomedicine including optical devices, signal detection, and biological imaging. Specifically, it summarizes the fabrication of microlenses and nanofibers from natural biological materials like biological cells, spider silk, and diatoms. Furthermore, we have highlighted the issues and challenges faced by bio-inspired microlenses and nanofibers while exploring the future development directions of these technologies.

2. Biological microlens

2.1 Biological microlenses in nature

In nature, we are able to observe different types of microlens structures in animals and plants. In the optic nervous system, when a light beam enters the eye through the cornea, the refractive power of the cornea bends the light, so the light can pass freely through the pupil, and then, the light passes through the microsphere lens, where it converges into a focal point on the retina, and the image conversion is formed on the retina. The electrical impulses are transmitted by the optic nerve to the brain, as shown in **Figure 1a**. In addition, mitochondria in the eye's photoreceptor cells also act as microlenses, helping

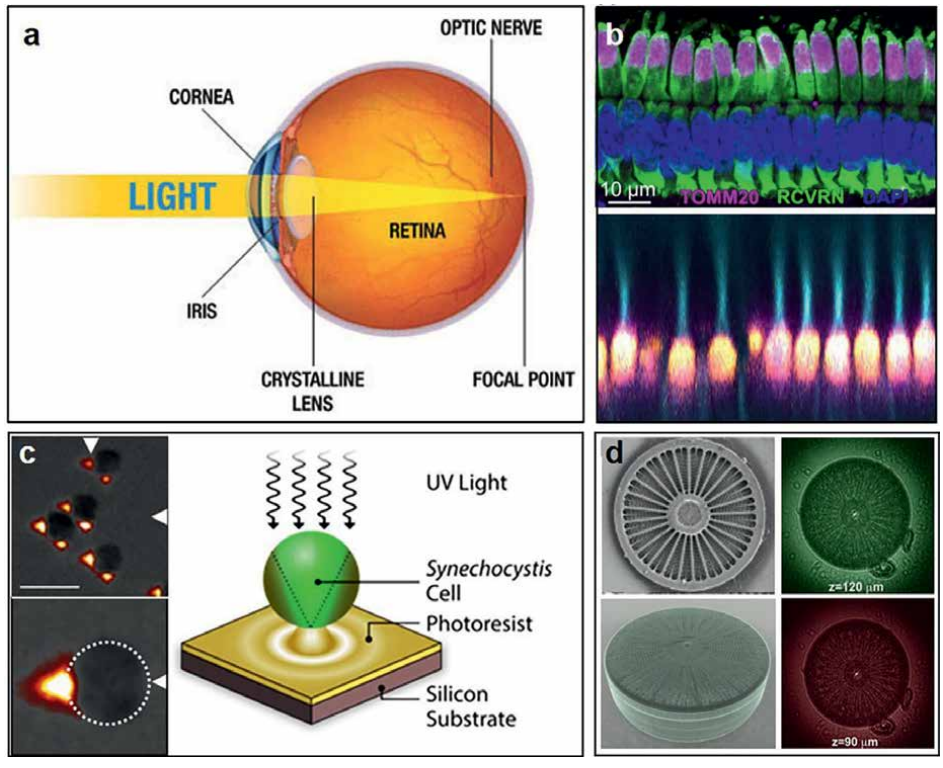


Figure 1.
Biological microlenses in nature. (a) Schematic diagram of the eye focusing on light. (b) Vertical anatomy of a cone photoreceptor and optical properties of mitochondria under light [22]. (c) Schematic diagram of the near-field optical effects of cyanobacterial cells and cyanobacterial cells illuminated by light from different directions [23]. (d) Scanning electron micrographs and 3D renderings of diatoms [24] and the transmitted intensity of illumination light on the XY plane of diatoms [25].

transport light from the inside of the cell to the outside of the cell and converting it into neural signals. Ball et al. used confocal microscopy to observe the optical properties of mitochondria [22]. When the light from the light-emitting diode passes through the mitochondria, the side of the mitochondria can be focused into a beam with high brightness, and the half-width of the focused beam is 1 μm, as shown in **Figure 1b**. In plants, organisms such as algal cells convert solar energy into chemical energy through photosynthesis and store it in the plant. The stored chemical energy can be released to provide fuel for the activities of the organism and its life energy [26, 27]. Schuegers et al. discovered that cyanobacteria have phototaxis, which can directly and accurately perceive the location of light sources. This is because cyanobacterial cells act as microlenses, confining incident light to a focal point near the plasma membrane on the back of the light source. Photoreceptors on the cell membrane sense the light and drive the pili to pull towards the side close to the light source, thereby driving the cell to move towards the light source (as shown in **Figure 1c**) [23]. To measure the perturbations of light by cyanobacterial cells in the near field with high resolution, the team used a photolithographic method in which cyanobacterial cells were adsorbed on the surface of a photopolymer and UV light was projected vertically onto the cells so that each cell produces a sharp peak beneath the center. In addition, diatoms in plants also have the ability to focus light [24]. When the 100 μm red laser spot is reduced to 10 μm after passing through the diatom with regular geometrical structure, this focusing effect is produced by the superposition of the scattered waves from the holes on the surface of the diatom flap, which proves that the diatom has the function of microlens [28]. As shown in **Figure 1d**, as the illumination wavelength increases, the position of the focus of diatoms along the optical axis gradually decreases [25]. Researchers have gradually demonstrated that spherical or cylindrical objects with an index of refraction less than 2 can produce light-focusing effects.

Most living organisms are composed of proteins, water, and carbohydrates in nature. These biological materials have a refractive index of less than 2 under visible light conditions and can confine the light beam in the living organism, which makes the use of biological materials as Concentrators become possible [29, 30]. Monks et al. found that natural spider silk can act as a microlens [31]. When the spider silk is in close contact with the imaging object, the near-field evanescent waves of the imaging object can be transferred to the far-field by the spider silk, thereby realizing super-resolution imaging. This imaging process from near-field to far-field conversion is extremely sensitive to the gap distance between the spider silk and the object. Through experiments and simulations, it was found that when the gap between the spider silk and the imaging object is less than 100 nm, the micro/nano structure is magnified and imaged, thereby realizing super-resolution imaging. Therefore, under dry conditions, spider silk cannot achieve super-resolution imaging. Mammalian cells also exhibit lensing behavior. Miccio et al. demonstrated that suspended red blood cells can act as microlenses at the microscale. Due to the inherent elastic properties of erythrocytes, by changing the osmotic pressure, the shape of erythrocytes can be expanded from a disc shape to a spherical shape, and the focal length adjustment from negative to positive values is realized [32], thus demonstrating the imaging ability of erythrocytes and the advantages of adjustable focal length.

2.2 Application of biological microlenses in optical imaging

Synthetic microsphere lenses combined with optical microscopy imaging devices can be used in the field of biological imaging, but polymer microspheres are easy to cause damage to biological cells and tissues. When observing the structure of biological cells *in vitro*, it is necessary to stain the specific observation object, and

then, the specific structure of biological cells can be observed through a fluorescence microscope [33]. However, biological cells, as a naturally occurring material, can focus light and guide light propagation in biological systems, acting as optical microlenses. Therefore, natural biomaterials with high biocompatibility and strong biodegradability have great potential in the field of bioimaging. In 2018, Liu et al. introduced a laser with a wavelength of 980 nm into a tapered fiber optic probe. The tip of the fiber optic probe was immersed in the red blood cell suspension. Under the action of the optical gradient force, the red blood cells could be stably trapped on the tip of the optical fiber. The structure can realize three-dimensional optical scanning imaging of single-cell membrane with a magnification of 1.7 times (as shown in **Figure 2a**) [34].

Using the same method, the tip of the optical fiber can trap yeast under the action of light force, and the trapped yeast can be used as a biological amplifier, which can realize real-time magnification imaging of nanostructures and any position of biological samples under an optical microscope [35]. As shown in **Figure 2b**, under the ordinary light microscope, the intracellular fibrous cytoskeleton and the double-layered structure of the cell membrane are difficult to distinguish. When the yeast trapped by the optical fiber is placed above the epithelial cells, the incident light and the reflected light are passed through. The interference effect of the cytoskeleton can enhance the interaction between light and matter, and the double-layer structure of the cytoskeleton and the cell membrane can be clearly seen. This process does not require the use of specific fluorescent molecules to label cells, providing a direct imaging method for biomedicine. In order to increase the imaging field and imaging efficiency, Jiang et al. used fiber-optic tweezers to trap yeast [36]. Because of the spherical shape of the yeast, the capture laser can be focused to a very small area through the yeast and can exert a strong light force on the connected yeast, which are arranged in an orderly cell chain under the action of light superposition. When the cell chain is in the vicinity of the imaged sample, the biological chain can act as a near-field magnifying glass, capable of capturing sub-diffraction information of nanoscale objects under each cell and projecting them into the far field (as shown in **Figure 2c**). This label-free, real-time nanoimaging device lays a solid foundation for the development of super-resolution imaging devices and systems for biomedicine.

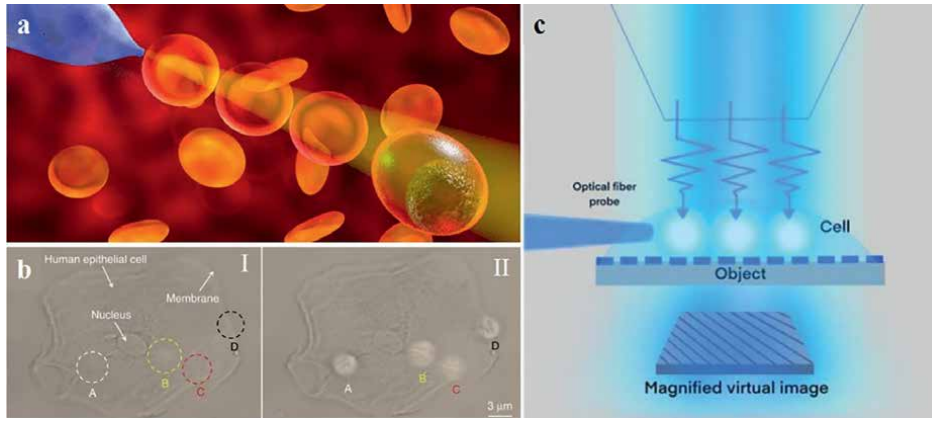


Figure 2.
Optical imaging of biological microlenses. (a) Schematic illustration of the assembled erythrocyte microlenses imaging the cell membrane [34]. (b) Using biological microlenses to image the intracellular cytoskeleton and two-layer structure on the cell membrane [35]. (c) Schematic diagram of the imaging of nanostructures by yeast cell chains [36].

2.3 Application of biological microlenses in signal detection

Due to the advantages of high biocompatibility and good bioabsorbability of biological cells, biological cells have great potential in biosensing and signal detection. When a highly focused beam irradiates a biological microlens, the microlens can generate a subwavelength-sized focused spot in its near-field region, resulting in a high localized light intensity. This is due to the interaction between the scattering field of the microsphere lens and the incident light beam passing through the microsphere, which allows the microsphere lens to enhance the interaction between photons and substances under the illumination of the incident light, enhance the fluorescence signal, and realize the control of the object signal. Using biological microlenses to detect and enhance fluorescence signals provides a convenient and low-cost method for nanomaterial characterization and biomolecular diagnosis [37–39]. In 2017, Li et al. used spherical yeast and biological cells as natural biological microlenses to enhance upconversion fluorescence [40]. First, the optical fiber is placed in the upconversion nanoparticle suspension, and near-infrared light with a wavelength of 980 nm and an optical power of 3 mW is passed into the optical fiber probe. The upconversion nanoparticle suspension can be directly affected by the output laser of the optical fiber probe. As shown in **Figure 3a** and **b** when the optical force is used to trap biological cells at the tip of the fiber probe, the fluorescence intensity of the upconversion nanoparticle suspension excited by the incident laser is significantly enhanced. Under the action of the optical gradient force, the photon jet generated by the biological microlens can trap a single *Escherichia coli* and *Staphylococcus aureus*, which indicates that the existence of the biomicrolens can significantly enhance *Escherichia coli* and *Staphylococcus aureus* (as shown in **Figure 3c-f**). In addition, *Staphylococcus aureus* and *Escherichia coli* can be trapped and linked together under the action of light gradient force, and their upconverted fluorescence signals can be

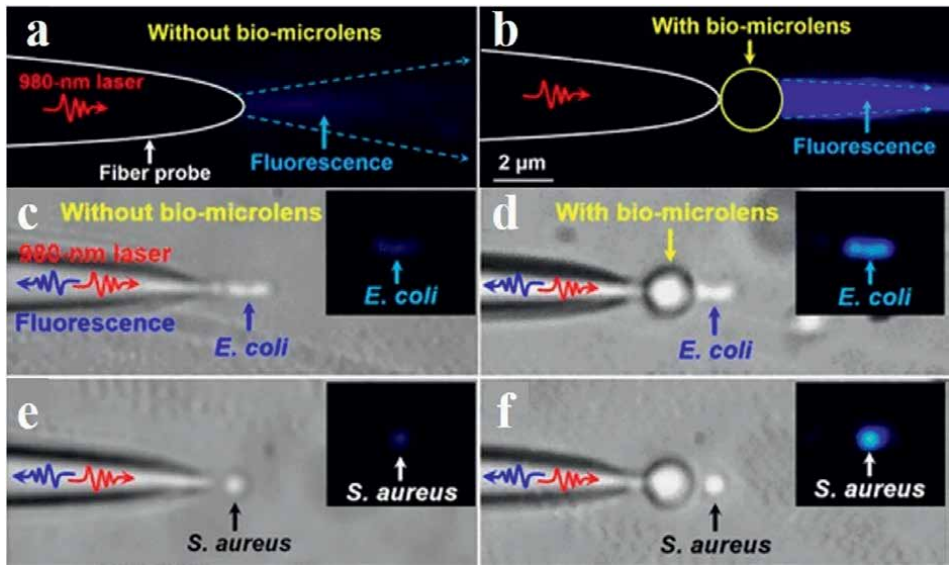


Figure 3.
Optical detection of biological microlenses. (a and b) Biomicrolenses enhance the fluorescence of upconverting nanoparticles. (c–f) Enhanced fluorescence of single Escherichia coli and Staphylococcus aureus using biological microlenses [40].

simultaneously enhanced by biological microlenses. In addition to yeast, mammalian cells can also be used as biological microlenses to enhance fluorescent signals. Since red blood cells do not have nucleus and organelles, they are disc-shaped with uniform refractive index distribution at pH 7.4. A single *Escherichia coli* can be trapped by red blood cells under the action of optical gradient force, and the fluorescence enhancement of single cells to nanoparticles can be realized. By lowering the pH of the erythrocyte solution, the morphology of the erythrocytes is swollen from the disk shape to a spherical shape under hypotonic conditions. When light passes through spherical red blood cells, a focused photon jet can be generated at the end of the red blood cells to excite strong fluorescence intensity of the upconverting nanoparticles modified on *E. coli*. Spherical human cells with larger diameters can also achieve enhanced fluorescence signals. For example, both K562 cells with a diameter of 10 μm and Raji cells with a diameter of 12 μm can enhance the upconversion fluorescence intensity of modified *E. coli*. This is because the surface of Raji cells and K562 cells is relatively rough and is a sphere with a nonuniform refractive index distribution. The experimental method can also be used to detect the capture process of single fluorescent polystyrene nanoparticles in real time [35].

Due to the diffraction limit of light, individual nanoparticles cannot be directly observed and detected under an optical microscope until the particles are trapped, although faint fluorescent spots from the nanoparticles can be seen in fluorescence mode. When a single nanoparticle was captured in the yeast's focus, the nanoparticle was able to be seen clearly in both optical and fluorescence images due to the magnification mechanism of the microlens. When a single nanoparticle is released, the nanoparticle moves away from the focus of the biomagnifier due to the strong Brownian motion in the aqueous environment, and the size and fluorescence intensity of the nanoparticle observed under the microscope are compared with the case of the trapped nanoparticle somewhat reduced. In addition, since the highly focused beam generated by the microlens is illuminated on the nanoparticles, the backscattered signal of the trapped nanoparticles is enhanced. By analyzing the intensity and angular distribution of enhanced backscattering from nanoparticles located within the nanojet, one can derive the nanoparticle's size and relative position with nanometer precision. In order to improve the sensitivity and biocompatibility of optical detection, Li et al. used yeast as a biological lens and used optical fiber probes to capture yeast to enhance the backscattering signal of *E. coli* chains [40], which provides applications for single-cell analysis and nanosensors.

3. Bio-optical micro/nanofiber

3.1 Bio-optical micro/nanofibers in nature

Signal detection and optical imaging of biological samples are of great significance in the research of biomedical applications such as biosensing and medical diagnosis. Because the biological optical micro/nanofiber can manipulate the light beam in a controlled manner, it will not cause irreversible physical damage to the biological sample during biological imaging and signal detection in the biological environment. Therefore, it is of great significance to construct biocompatible and movable biological optical micro/nanofibers. In the natural environment, natural optical micro/nanofibers can be widely observed in animals and plants. In the optic nervous system, the retina of vertebrates has specific optical properties, and light must penetrate multiple

cell layers to reach the photoreceptor cells. As shown in **Figure 4a**, the geometry of Müller cells is similar to that of optical fibers so that Müller cells in their physiological environment can act as optical micro/nanofibers, allowing light to pass through the channels of the retina with low scattering. At the same time, the Müller cells arranged in parallel can direct light directly to their respective cone photoreceptors, thus maintaining the original image resolution and minimizing image distortion [41]. Furthermore, Müller cells enhance the signal-to-noise ratio by minimizing scattering and preserving the spatial distribution of light patterns in the propagating image [46]. Photons produced by bioluminescent organs are usually guided by some micro/nanofiber structure and emitted in a specific pattern. The jellyfish, for example, is mainly made of a translucent gel-like substance, the tunica media, which has a water content of up to 95–98%, and its bioluminescence is guided by fiber-like antennae to be used as bait to attract prey [47]; when the floating silkworm is stimulated externally, it can produce yellow light on its feet (as shown in **Figure 4b** and **c**) [42].

In plants, some photoreceptors exist at the root of the plant, allowing the roots to sense different wavelengths of light [43]. As shown in **Figure 4d**, there are photoreceptors activated by light in root cells, which can act as natural optical micro/

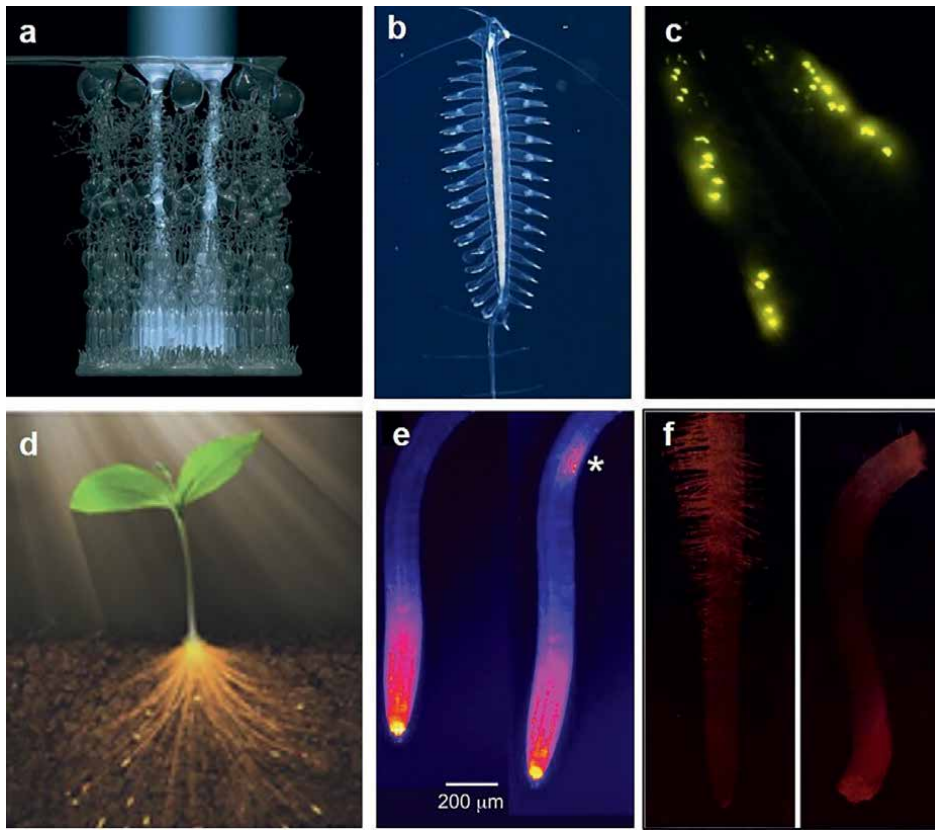


Figure 4.
Types of bio-optical micro/nanofibers. (a) Müller cells guide light from retina to photoreceptor cells [41]. (b) Optical microscope image of floating silkworm [42]. (c) Floating silkworms produce yellow light when stimulated [42]. (d) Light is directed through the stem of the plant to the roots [43]. (e) Fluorescence images of HY5 and protein fusions in wild-type seedling roots under light conditions [44]. (f) Optical image of a red laser guided by corn roots and oat seedlings [45].

nanofiber devices to receive the stimulation of light beams. In addition, the roots of plants can receive information on light conditions through signal molecules and respond to the signal molecules, allowing the light beam to travel from the stem to the root. At the same time, the roots of the plant can also directly sense the light transmitted by the plant tissue. As shown in **Figure 4e**, the stem of the plant acts as an optical fiber that can conduct light to the phytochrome receptors in the root, triggering the production of the HY5 protein, which promotes healthy root growth [44]. As shown in **Figure 4f**, when the laser irradiated the corn roots and oat seedlings, the light energy was extended in a curve to the tips of the roots, which promoted the germination of the plants [45]. In addition, various optical micro/nanofibers can also be fabricated from naturally derived materials such as spider silk [48], cellulose [49], and bacterial cells [50] in nature. Spider silk has been shown to be an efficient optical fiber in various environments with an optical loss of 10.5 dB/cm. Natural spider silk fibers are also capable of delivering light in physiological fluids and integrated photonic chips. Through genetic engineering, large-scale production of spider silk proteins has become possible. Recently, researchers have created optical micro/nanofibers by using genetically engineered spider silk proteins [51]. In addition, the recombinant spider silk optical micro/nanofiber contributes to the efficient propagation of light due to its smoother surface, higher refractive index, and lower optical loss, which can deliver light to deep tissues with lower optical loss. Based on the light-guiding ability of a single cell, using optical power to assemble cellular micro/nanofibers can transmit light. *Escherichia coli* can be assembled into a biological micro/nanofiber using a tapered optical fiber by a laser with a wavelength of 980 nm. By changing the intensity of the laser light, the number of captured cells can be changed, thereby changing the length of the biophotonic micro/nanofiber.

3.2 Application of bio-optical micro/nanofiber in biomedicine

Natural biomaterials with high biocompatibility and biodegradability have been extensively studied for various medical applications such as drug delivery, biosensing, and optical imaging [52–55]. Silk fibroin films were prepared from aqueous solutions of silk fibroin polymers, and crystallinity was induced and controlled by methanol treatment. In the process of processing into thin films, dextran and proteins of different molecular weights are encapsulated in the drug delivery device, and the drug release effect of silk fibroin can be evaluated by drug release kinetics [56]. In terms of biosensing and detection, light-capturing force is used to capture yeast cells and lactobacillus chains at the tip of an optical fiber. When light propagates through this biological chain, a beam with a half-width of 190 nm can be formed (as shown in **Figure 5a**) [57]. This biological micro/nanofiber can be used to detect the local fluorescence of leukemia cells in human blood; that is, when the biological micro/nanofiber is far away from the leukemia cells, no fluorescence can be detected. When the optical micro/nanofiber is in close contact with the cell membrane, a distinct fluorescent spot appears in leukemia cells stained with green fluorescent protein, which enables real-time detection of optical signals in the near field with subwavelength resolution. This biological cell-based optical micro/nanofiber can bend without damaging the cell membrane when it contacts the cell, and is highly flexible and deformable. At the same time, Wu et al. used the light-harvesting force to assemble a photofluidic cell chain of *Enterococcus faecalis* with a length of up to 360 μm and a propagation loss of 0.03 dB/μm [59]. When the incident light irradiates the cell chain, a beam with a focused beam waist radius of 400 nm can be obtained, which proves

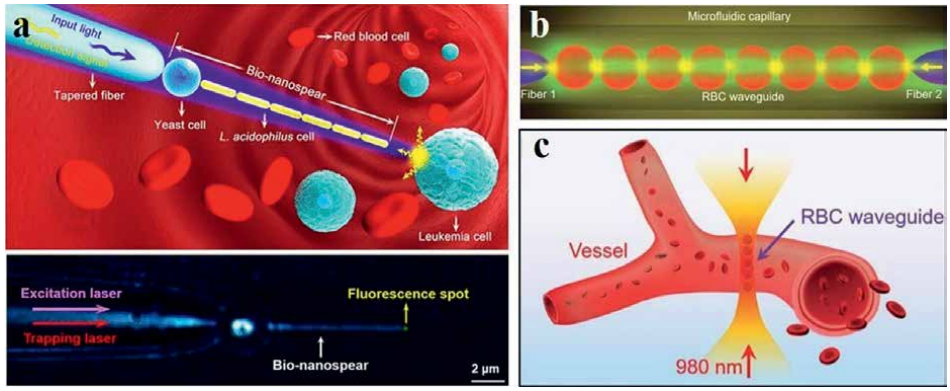


Figure 5.
Applications of bio-optical micro/nanofibers. (a) Bio-optical micro/nanofibers for single-cell detection [57]. (b) Schematic diagram of red blood cell optical micro/nanofiber for pH sensing [58]. (c) Schematic illustration of the assembly of red blood cell optical micro/nanofibers within zebrafish blood vessels [58].

the micro/nanofiber performance of the *E. faecalis* cell chain. This biological micro/nanofiber can be used to detect backscattered signals from normal and pathological human red blood cells, providing a new approach for biocompatible devices for real-time biomedical sensing and single-cell diagnostics. In addition, the red blood cell micro/nanofiber can also be constructed using two tapered optical fibers. As shown in **Figure 5b**, red blood cells can be confined within the optical axis of two tapered optical fibers. By monitoring the light propagation of the red blood cell micro/nanofibers, the pH value of blood solutions can be detected in real time. This method can be used to diagnose pH-related issues. After diagnosis, optical torque is applied on the red blood cell micro/nanofiber, which rotates like a micromotor to transport the particles to the target area, and the red blood cell micro/nanofiber can be constructed in the zebrafish blood vessels (as shown in **Figure 5c**), providing a powerful tool for diagnosis and drug delivery [58]. In addition, spider silk as a natural optical micro/nanofiber enables the construction of biocompatible thermometers [60]. The spider silk used in the experiment was taken from the abdomen of the spider, and core-shell structured upconversion nanoparticles were assembled on the surface of the spider silk by photophoresis. Membrane temperatures of individual breast cancer cells were obtained by measuring the fluorescence spectra of upconverting nanoparticles on spider silk. This biomaterial-based thermometer enables real-time monitoring of temperature changes during apoptosis, providing a biocompatible tool for precise biosensing and single-cell analysis.

4. Conclusions

To sum up, the noncontact and noninvasive signal detection and optical imaging of nanoparticles and biomolecules in the microenvironment has important research value and potential application prospects in the fields of biomedicine and nanophotonics. Biophotonic devices such as bio-microlenses and bio-optical micro/nanofibers have played an important role in scientific research. However, in the process of rapid scientific development, due to the good flexibility and membrane elasticity of biological materials, biological microlenses and biological optical micro/nanofibers have an important impact on the detection of microbial environment and the manipulation of

living bodies. Using the radiation force generated by the focused optical field of the optical tweezers, operations such as capturing, stretching, and rotating the object can be achieved without contact and damage.

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Conflict of interest

The authors declare no competing financial interests.

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Chapter 5

Synthesis and Characterization of Thermosetting Polymers: An Electrospinning Approach

Alexander Fainleib, Olga Grigoryeva, Olga Starostenko and Daniel Grande

Abstract

This chapter is devoted to the description of the main recent approaches and technical solutions for the creation of polymer micro- and nanofibers engineered by electrospinning precursors of thermosetting polymers. Structure-property relationships have been analyzed for miscellaneous complex systems, including epoxy electrospun micro and nanofibers; submicron carbon nanotube–epoxy nanocomposite filaments, rigid fibers based on functional polynorbornenes with epoxy or carboxylic pendant groups, and core-shell nanofibers with a structure of semi-interpenetrating polymer network (semi-IPN) based on thermoplastic polyamide and thermosetting epoxy resin. The first experimental results for creating electrospun nanofibers with a semi-IPN structure based on polysulfone/polycyanurate or polysulfone/polycyanurate/polybenzoxazine are discussed. Due to the unique properties of polycyanurates, namely high heat- and chemical resistance to aggressive substances, low dielectric losses, and low water absorption, the resulting electrospun fibers could potentially become useful as components in different advanced materials with improved properties to operate in aggressive environments over a range of temperatures.

Keywords: electrospinning, micro–/nanofibers, polysulfone, cyanate ester resins, thermosets

1. Introduction

Electrospinning has attracted significant attention as a manufacturing process for producing fiber materials with diameters ranging from the micro- to nanometer scales [1]. One such technique provides a promising and straightforward way to fabricate infinite and continuous fibers applied as nanostructured and biomedical materials [2, 3]. Electrospun nanofibers exhibit exceptional characteristics, including a high surface-to-volume ratio, permeability, absorbance capacity, porosity, interconnected ultrafine fibrous structure, and low weight. Electrospun fibers in nanoscale diameters have enticed wide consideration from researchers, who are ready to move toward a practice-based

interdisciplinary approach in a variety of fields [4–9]. A wide range of materials, such as engineered and biological polymers, ceramics, and composites, have been successfully electrospun into one-dimensional materials having many different microstructures [10]. Due to their remarkable characteristics, such as very small fiber diameters, large surface area per mass ratio, high porosity along with small pore sizes [3, 11], flexibility, and superior mechanical properties [12], nanofiber mats have found numerous applications in biomedicine (drug delivery, tissue engineering, and wound dressing), filtration, reinforcement in composite materials, protective clothing, and microelectronics (sensors, batteries, display devices, supercapacitors, and transistors) [13–18].

Most of the polymers are dissolved in a solvent before electrospinning, as the processing conditions involved are simple and straightforward. Moreover, polymers that melt at high temperatures can be fabricated into nanofibers through electrospinning; nevertheless, much fewer polymers have been electrospun from such a state [9, 19]. Importantly, all the polymers used for electrospinning were thermoplastics, which means soluble and meltable. Electrospinning of thermally reactive oligomers that result in significant thermosets constitutes a real challenge. This contribution addresses the fabrication of micro- and nanofibers containing thermosetting polymers with a special focus on polysulfone/polycyanurate (PSF/PCN) fibrous materials.

2. Principles of electrospinning

Continuous nanofibers and fibers of desired dimensions can be produced by the electrospinning technique [18]. This process also offers the possibility to form various fiber assemblies, including nonwoven, aligned or patterned fiber meshes, haphazard 3D structures, and tangled fibers [19]. Both natural and synthetic polymers, liquid crystals, suspensions of solid particles, ceramics and emulsions can be used to produce ultrafine fibers with diameters ranging from 2 nm to 10 μm by the electrospinning approach [20, 21].

Figure 1a, **b** shows a typical electrospinning setup that consists of a capillary tube with a needle, a high-voltage supplier, and a collecting screen [23]. Taylor [24] found that 6 kV applied voltage is required for the initiation of the electrospinning process to take place.

A syringe pump with a feeding rate of 0.1–60 mL/h is required for the electrospinning process [25] as well as a spinneret (clip spinneret, tube-less spinneret, coaxial spinneret, or heating spinneret) and collector (plate collector, rotary drum collector,

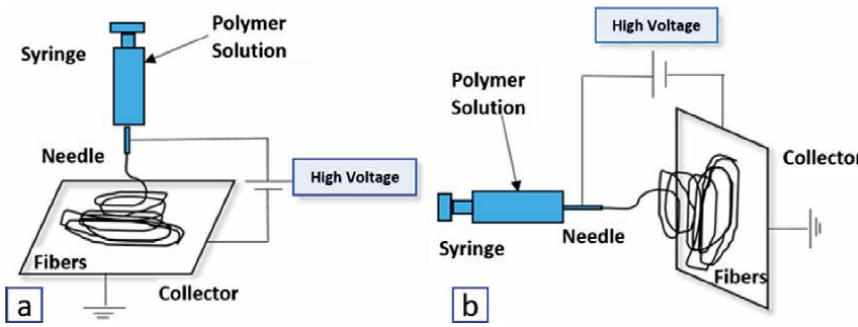


Figure 1.
Schematic diagram of set-up of electrospinning apparatus (a) vertical set-up and (b) horizontal setup [22].

grid type collector, edge type collector, mandrel collector, steel sheet collector, or dual rings collector) [21]. Solution parameters (effect of dielectric property of solvent, surface charge density, surface tension, solution viscosity, polymer molar mass, and concentration), process parameters (effect of voltage, flow rate, collector type, and distance between needle tip and collector), type of spinning setup, and ambient parameters (effect of temperature and humidity) are responsible for the variations in morphology and diameters of the nanofibers produced by electrospinning [26]. Almost all soluble or fusible natural or synthetic polymers, as well as their blends, can be used for electrospinning [27]. This broad variety of polymers belongs to thermoplastics and comprises nucleic acids, proteins, and polysaccharides [28].

3. Nanofibers fabricated by electrospinning epoxy-based polymers

Recently, some publications have appeared on thermoset-containing nanofibers fabricated by electrospinning [29].

Epoxy is a thermosetting precursor that chemically reacts to form a crosslinked matrix when combined with a curing agent. Shneider et al. [29] demonstrated that to produce continuous micro- and nanofibers through electrospun epoxy, one must control the curing reaction with precision. For this purpose, epoxy and triamine curing agent were dissolved in a tetrahydrofuran-dimethylformamide (THF-DMF) binary solvent. The authors determined a narrow working window at which a proper solution for electrospinning is close to the gel point, right before the transition from liquid to solid gel state. The solution was characterized by means of (i) Fourier-transform infrared spectroscopy (FTIR) to control the curing process, (ii) steady shear viscosity to detect the divergence near the gel point, and (iii) oscillatory loss and storage shear moduli to identify the liquid-to-gel transition. The chemical transformations of epoxy solution, including chemical interconnections and gelation, could be monitored through these measurements, allowing for a working window to be defined for electrospinning.

Aliahmad et al. [30] described the electrospinning fabrication process (**Figure 2**) and characterization of submicron carbon nanotube (CNT) – epoxy nanocomposite

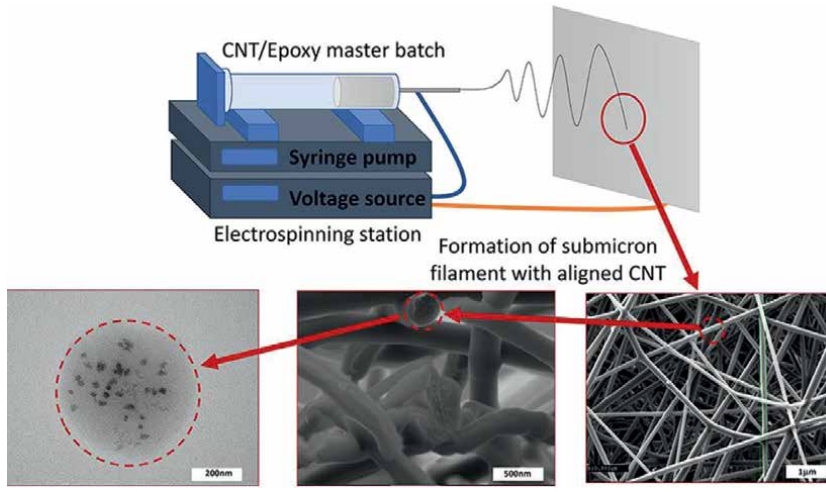


Figure 2.
Schematic process for electrospinning a submicron CNT/epoxy filament [30].

filaments. Electrospinning of submicron epoxy filaments was carried out by partial thermal curing of the epoxy resin with the hardener without any thermoplastic component. This semi-curing approach made the epoxy solution viscous enough for the electrospinning process. The electrospun filaments were fabricated using a CNT/epoxy solution in DMF with a viscosity of 65 cP using 16 kV and a collector distance of 10 cm. The diameter of these filaments could be tuned to as low as 100 nm with the adjustment of electrospinning parameters. Better structural, electrical, and thermal stabilities were achieved by incorporating a low amount of CNT into epoxy. As a result of the electrostatic field during the electrospinning process, the CNT fibers were aligned within the epoxy filaments. The improvement of modulus up to 49% for CNT/epoxy filaments compared to epoxy was found. The authors concluded that the developed filament was suitable for many applications due to the use of a commercial epoxy suitable for industrial composite productions.

Two functional polynorbornenes (PBNEs), one bearing an epoxy pendant group and another one bearing carboxylic pendant groups, were electrospun in the presence of a variety of cross-linkers [31], resulting in long (several mm), rigid fibers with diameters that can be tuned in the range of 300 nm–10 μm and glass transition temperatures (T_gs) higher than 300°C. A 20 kV positive voltage was applied to the needle tip. Despite being brittle, these fibers formed a continuous nonwoven mat, which was similar to any textile mat. Remarkably, injection rates as high as 1 mL/min could be used to prepare the fibers. Commarieu et al. [31] explained such a surprising result by the low propensity of entanglement for solutions of rigid-rod polymers. Curing of the fibers was performed by leaving the fibers 2 hours at 200°C. Once cross-linked, the fibers did not swell and became insoluble. The electrospinning process was used to encapsulate dyes or graphitic dots (GDs) (**Figure 3**). Due to the high cross-linking density of the fiber, dye leaching was prevented. In contrast with other rigid-rod polymers, electrospinning of PNBE is facile and can be performed at injection rates

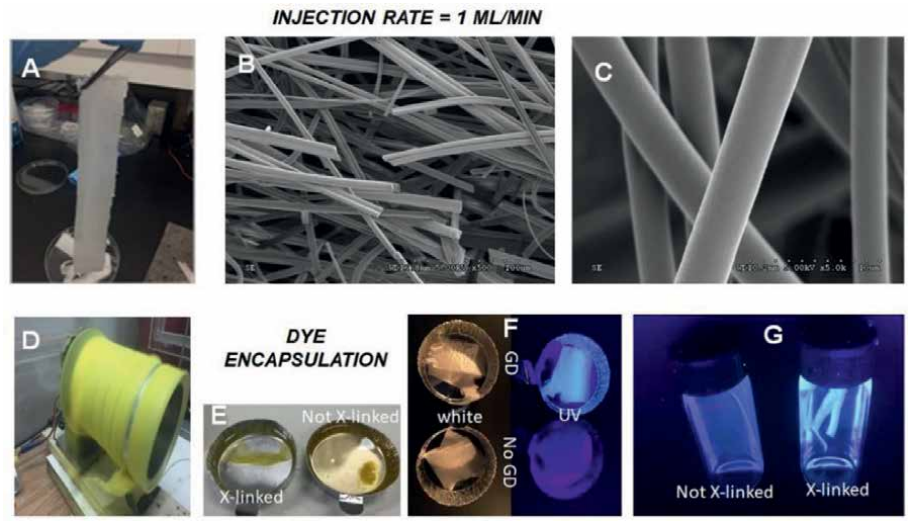


Figure 3.
Mat of fibers obtained at high injection rate (1 mL/min) (a) and SEM pictures of the fibers (b, c). Cylindrical collector with fibers containing a yellow dye after 5 minutes of electrospinning (d) and same fibers immersed in THF, (e) before and after cross-linking. Pictures of fibers containing GDs under white and UV light (f) compared with fibers devoid of GDs. GD-containing fibers before and after cross-linking immersed in THF, under UV light (g) [31].

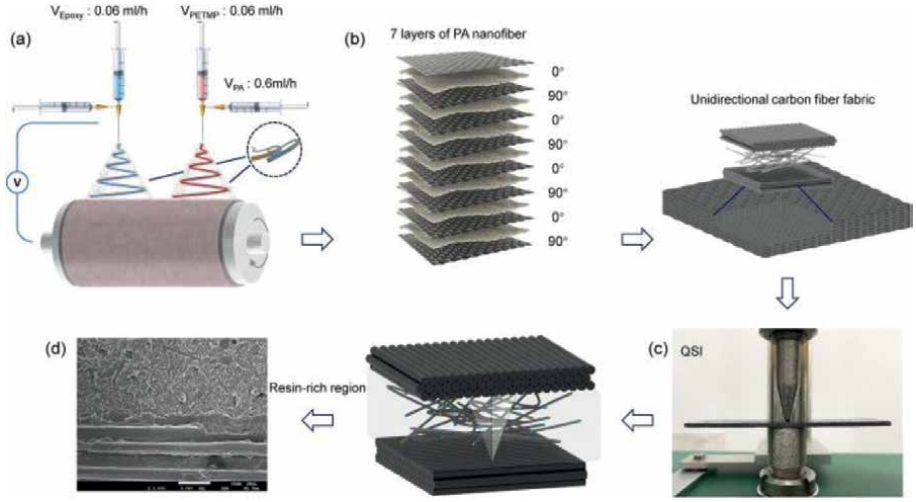


Figure 4.
Schematic diagram of self-healing composites based on PA core-shell nanofiber: (a) preparation of PA core-shell nanofiber, (b) self-healing composites, (c) quasi-static indentation test, and (d) the PA core-shell nanofiber healed interlaminar damage [33].

as high as 1 mL/min. Due to their unique photophysical properties, GD-containing PNBE fibers could potentially become useful photocatalysts or fluorescent materials and as components in antibacterial textiles [32].

Core-shell nanofibers were prepared by coaxial electrospinning of a mixture of thermoplastic polyamide (PA) and thermosetting epoxy resin (ER) on the premise that the melting temperature of PA matches the curing temperature of the ER healing agent [33] (**Figure 4**). Such a combination without any chemical interactions between both polymeric components is called semi-interpenetrating polymer network (semi-IPN) [34]. The morphological and chemical study proved the core-shell structure of the nanofibers without any chemical reactions. The PA shell and the two-component epoxy core acted together as the healing agent to fill microcracks. Using this producing method allowed for obtaining self-healing composites possessing enhanced healing efficiency and responsiveness as well as the toughened interlaminar region (the bending strength of the composite having the embedded core-shell nanofiber was increased by 16%). Heating the composite samples at 130°C for 20 minutes after damage provided the healing effect for the composite containing neat PA nanofiber around at 62% and for the one with PA core-shell nanofiber at around 90%. The authors noted that the thermoplastic PA nanofiber had a good healing function and rapid healing responsiveness. It was found that the encapsulated thermosetting healing agent improved the healing efficiency of thermoplastic PA. Thus, electrospun core-shell nanofibers obtained from polymer combinations as semi-IPN-type are promising for healing agent encapsulation systems.

4. Polysulfone-based electrospun thermoplastic nanofibers

Scientific specialists in the field of membranes pay special attention to polysulfone, namely a widely used thermoplastic polymer in microfiltration [35]. Polysulfone has a unique set of useful physical properties: high mechanical strength, destruction temperature (> 400°C), glass transition temperature (150–180°C), and resistance to aggressive

environments with different pH values. Polysulfone can be dissolved in aprotic polar solvents, such as dimethylsulfoxide, dimethylacetamide, dimethylformamide, nitrobenzene, and their halogen-containing derivatives [36–39], which is very important in the manufacture of membranes. As a rule, polysulfone membranes are obtained by the phase inversion method [40–43], the disadvantages of which are the limited range of pore sizes in the membrane and residual solvent, which affect the quality of the membranes [44].

The electrospinning technique is free from the abovementioned disadvantages and is effectively used to produce highly porous nonwoven mats with interconnected pores and a highly effective surface area suitable for large-scale production [45–48]. Membranes obtained by the traditional method are inferior to those produced by the electrospinning technique in flux performance at equal rejection efficiency [49]. The effect of the solution concentration (15, 20, and 25 wt.%) of polysulfone in dimethylformamide, voltage (12.5, 15, and 18 kV), and solution feed rate (1.5, 2.5, and 3.5 ml/h) on the morphology of the obtained fiber samples was systematically studied [35]. The SEM results showed that the samples obtained from a solution with a concentration of 15 wt.%, possessed a bead-on-a-string type morphology, and the homogeneous fiber samples without compactions and with an average diameter from 0.876 to 2.078 μm depending on the voltage and feed rate were produced from solutions with a higher polysulfone concentration. The authors recorded the optimal combination of electrospinning parameters (solution concentration of 25 wt.%, feed rate of 3.5 ml/h and voltage of 18 kV) for obtaining high-quality fibers [35].

The THF-DMF binary solvent system with different ratios and dielectric constants (ε) from 16.33 to 27.97 was used for preparing smooth surface electrospun polysulfone nanofibers, while THF alone (ε = 7.60) and DMF alone (ε = 36.70) led

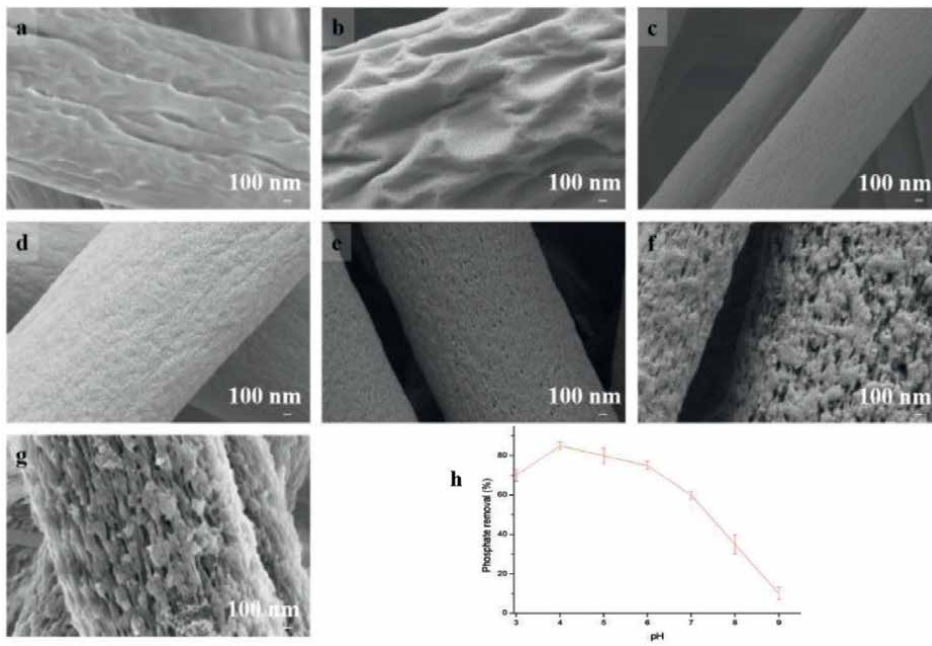


Figure 5.
The FESEM micrographs of the fabricated electrospun nanofibrous membranes (a) PD0T10, (b) PD1T9, (c) PD3T7, (d) PD5T5, (e) PD7T3, (f) PD9T1, and (g) PD10T0 at 25 kV magnification, and phosphate adsorption (h) by fabricated electrospun PSF membrane [50].

to the formation of electrospun PSF nanofibers with a rough and porous surface (**Figure 5a-g**). Field emission scanning electron microscopy (FESEM), tensile strength technique, and contact angle measurements were applied for the investigation of the nanofibers obtained [50]. The results obtained did not correlate with the common conclusion that only a binary solvent provided formation of electrospun nanofibers with a rough or grooved surface [50]. The authors concluded that the dielectric constant was an important factor, besides boiling point and polymer solubility for binary solvent systems, which affected the phase separation in the solution and formation of nanofibers with non-smooth surfaces (**Figure 5h**).

Mazoochi et al. [3] studied the effect of processing parameters such as voltage applied and tip-to-collector distance (TCD) on the morphological features of electrospun PSF fibers. The formation of different morphologies, such as dry, bead-free, and continuous fibers, was observed without any breaking up to droplets. It was noted that jet flight time and electric field strength were directly affected by TCD. Decreasing TCD value decreased flight times and solvent evaporation time and increased electric field strength. This led to increasing bead generation. Rising in spinning distance provided the higher homogeneity of PSF fibers. The higher influence of the spinning distances was observed at higher applied voltages; the higher the applied voltage, the higher the surface charge of the jet and lower the beads formation.

5. Heat-resistant nanoporous thermosetting materials based on polycyanurates from cyanate ester resins

Cyanate ether resins (CERs) are high-performance heat-resistant polymers that, unlike other polymers, have a highly regular polymer network structure. These polymers are also called polycyanurates (PCNs). They are obtained by polycyclotrimerization of dicyanate ethers of bisphenols. At present, many monomers of dicyanate ethers of bisphenols with different chemical structures have been synthesized [51–54]. CERs (PCNs) have attracted the attention of scientists and industrialists due to a unique set of physical and chemical properties: high thermal and thermooxidative stability (up to ~400–450°C), high glass transition temperatures (up to ~270–400°C), radiation resistance, chemical resistance to aggressive environments (alkalis, acids, alcohols, sea water, *etc*.) and adhesion to various metals (aluminum, titanium and their alloys, iron, *etc.*), carbon and glass plastics or fibers, as well as low dielectric losses ($\varepsilon \approx 2.64–3.11$), water absorption and absence of gas release during their synthesis, *etc*. [51–53]. As a result, CERs are currently used as structural and functional materials in microelectronics, aeronautics, and space structures as adhesives, binders, composite strips, nose radar domes, keels, heat shields, printed circuit boards, *etc*. [55].

Recently, a number of new methods of creating nanoporous film-forming materials based on thermostable PCNs have been developed [56]. Some of them were based on the modification of PCN networks by using a reactive porogen such as poly(ε-caprolactone) (PCL), which was first partially chemically incorporated into the PCN network, and then the nanoporous structures were formed by partial hydrolysis or extraction of unincorporated PCL chains. Another method for creating nanoporous structures used partial thermal degradation of PCN chains (at $T \approx 250$°C) with subsequent separation of destroyed chains. Four other suitable methods were developed to create a nanoporous structure in PCNs: (1) by using some high-boiling liquids (phthalates) as porogens; (2) by controlling the final degree of conversion of cyanate groups during the synthesis of PCN networks (with the subsequent selection of

unreacted chain fragments); (3) by using different ionic liquids as porogens during *in situ* synthesis of PCN networks followed by their removal during extraction; and 4) by irradiating PCN-based thin films with α-particles to obtain track-etched membranes.

All the abovementioned nanomaterials based on PCNs are promising for use as high-tech thermally stable nanoporous membranes, demonstrating after complete extraction of the porogen a significant increase in He permeability and higher sorption abilities of CO_2 and O_2, as well as approximately 1.5–1.8 times increased He/O_2 and He/CO_2 selectivity factors [57]. It was shown that before extraction of the porogen, both the mobility of the chains and the free volume determine the gas transport properties of nanoporous membranes based on PCNs. Whereas after extraction of the porogen, the volume content of pores and the pore size distribution become the predominant factors. Additional annealing of these nanomaterials in a rubber-like state (at $T < T_g$) leads to an obvious modification of their nanoporous structure, which makes it possible to increase the separation capacity between large and small gas molecules in the glassy state (at $T \geq T_g$) of the samples.

Nuclear technologies have also been used to create heat-resistant track-etched nanoporous membranes and filters based on PCNs. First, thermosetting grafted semi-IPNs based on PCN and linear polyurethane (LPU) were synthesized by stepwise *in situ* thermal curing, then the PCN/LPU thin films were irradiated with α-particles followed by chemical etching of the created tracks [58]. SEM studies confirmed the formation of well-defined highly regular nanoporous structures in the films. FTIR spectroscopy measurements showed no significant changes in the chemical structure of the polymer components of the samples after the α-irradiation and chemical etching procedures. The nanoporous PCN/LPU-grafted semi-IPNs had a narrow pore diameter distribution with an average pore diameter of about 12 nm. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) measurements showed that the nanoporous PCN/LPU films exhibited high thermal performance: T_g ~ 167°C to ~199°C, $T_{d5\%}$ ~ 293–359°C and T_{dmax} ~ 429–457°C. The thermosetting nanoporous PCN/LPU track membranes also demonstrated effective gas transport properties with various gases such as O_2, CO_2, N_2, and CH_4 (**Figure 6**). It was also found that the use of an additional γ-ray sensitization step and an increase in the chemical etching time improved the nanoporous structure and other properties of the

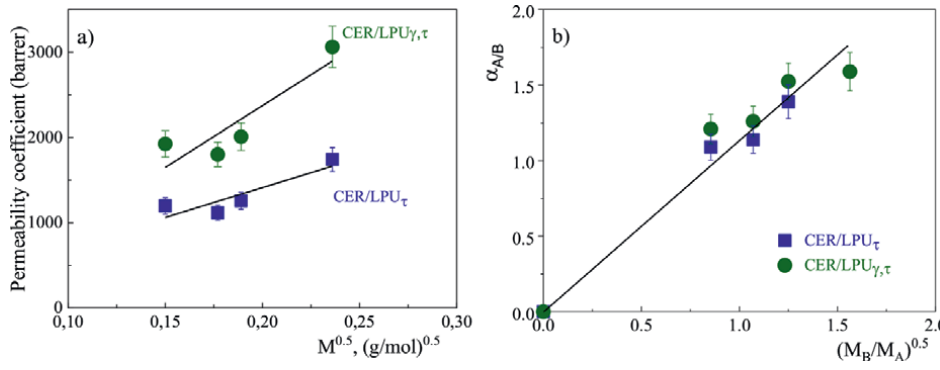


Figure 6.
Transport properties of different gases for nanoporous CER-based hybrid networks treated in different conditions: (a) permeability coefficients as a function of the inverse of the square root of the molar mass of the diffusing molecule; (b) gas selectivity values as a function of the ratio of the molecular weights of the diffusing molecules for the gas pairs CO_2/O_2, CH_4/N_2, N_2/O_2, CH_4/CO_2 [58].

developed nanoporous track membranes. Such materials open the way to numerous applications, for example, as ultrafiltration thermo- and chemically stable membranes for advanced technologies that can operate even under extreme conditions, that is, in aggressive environments, at high (> 250–400°C) or very low (up to −250°C) temperatures in a vacuum, under high radiation in space, *etc*.

6. Polysulfone/polycyanurate-based electrospun nanofibers with semi-IPN structure

Recently, we have implemented the electrospinning technique for preparing thermostable nanofibers with a structure of polysulfone/polycyanurate, polysulfone/polycyanurate/polybenzoxazine or polysulfone/poly(cyanurate-co-benzoxazine) semi-IPNs, and their structure-property relationships have been studied [59, 60]. The chemical structures of the components used for these studies are shown in **Figure 7**.

In this work, the nanofibers with a structure of polysulfone/polycyanurate semi-IPNs were prepared by electrospinning a mixed solution of PSF and dicyanate ester of bisphenol E (DCBE) (PSF/DCBE ratio was varied from 75/25 to 95/5) or 1–10 wt.% of the oligomer of dicyanate ester of bisphenol A (DCBA) (with cyanate groups conversion of about 25%) in DMF. The concentration of the polymer solution was equal to 21%. The electrospun nanofibers were fabricated using standard electrospinning equipment with a feeding rate of 8 mL/h, an accelerating voltage of 20 kV, and a distance between the needle tip and collector of 15 cm. The obtained nanofibers were dried at 25°C for 1.5 hours in a vacuum.

Figure 8 shows the typical SEM micrograph for PSF/DCBA = 95/5 wt.%, PSF/DCBA = 90/10 wt.%, and PSF/DCBE = 75/25 wt.% electrospun nanofibers and the corresponding fiber diameter distributions. Electrospinning of PSF/DCBA = 95/5 wt.% and PSF/DCBA = 90/10 wt.% solutions resulted in very similar morphology with fiber diameter distributions of 350–1800 and 360–1600 nm, respectively (**Figure 8a**,**b**). The nanofibers were not entirely uniform, as they contained some oval-shaped structures, and some single fibers displayed diameters larger than 2 microns. With the increase in cyanate monomer DCBE (up to 25%) in the spinning dope, the nanofibers become thinner in diameters with narrower size distributions (**Figure 8c**). This was due to the substantial decrease in the viscosity of the spinning

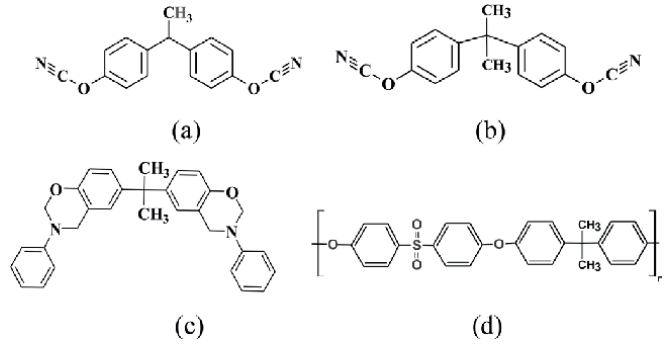


Figure 7.
Chemical structure of (a) dicyanate ester of bisphenol E (DCBE), (b) dicyanate ester of bisphenol A (DCBA), (c) bisphenol-A based benzoxazine (BA-a), and (d) polysulfone (PSF, M_w = 35,000 g·mol^{-1}).

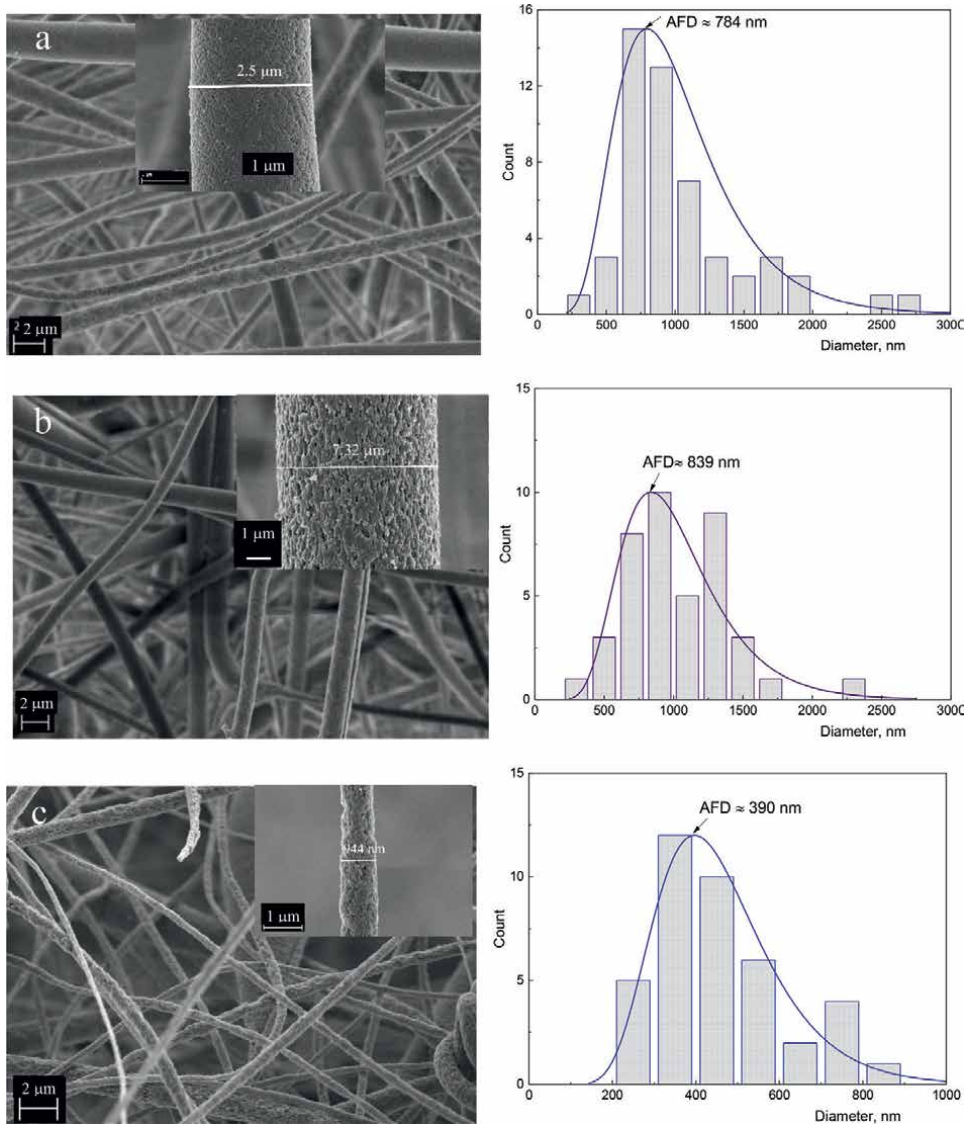


Figure 8.
Typical SEM micrograph for electrospun nanofibers of different compositions (wt.%): (a) PSF/DCBA = 95/5, (b) PSF/DCBA = 90/10, and (c) PSF/DCBE = 75/25. The corresponding fiber diameter distributions are displayed on the right side.

solution with the increase in the content of the low-molecular DCBE monomer. It could also be seen that all the electrospun nanofibers showed high internal porosity.

The electrospun nanofibers with a structure of polysulfone/polycyanurate/polybenzoxazine semi-IPN were prepared by electrospinning from a mixed solution of PSF and PCN-BA prepolymer in DMF. The prepolymer poly(cyanurate-*co*-benzoxazine) (PCN/BA) was synthesized as follows: BA-a in the amount of 25 wt.% was added to liquid dicyanate ester of bisphenol E (DCBE) at 100°C, then the blend was heated to 120°C and vigorously stirred during 60 minutes. The prepolymer synthesized was a viscous liquid at room temperature. According to the calculations from the FTIR data, the conversion of cyanate groups of DCBE in the PCN/BA prepolymer

obtained was estimated as $\alpha \approx 30\%$. Electrospinning operational parameters such as applied voltage, working distance between the spinneret and the planar collector, flow rate, and temperature were 20 kV, 150 mm, 2.5 ml/h, and 20°C, respectively. The collected mat was placed under vacuum at 30°C to remove residual solvent.

To produce polysulfone/polycyanurate/polybenzoxazine semi-IPNs, the PSF/PCN-BA fiber meshes were cured using astep-by-step schedule: 1 hour at 80°C, 1 hour at 120°C, 1 hour at 150°C, 1 hour at 180°C, 1 hour at 220°C, and 1 hour at 250°C. After each heating step, the morphology of nanofibers was monitored using SEM, and the formation of triazine cycles of PCN and the oxazine ring opening of BA-a in PSF/PCN-BA were analyzed using FTIR.

Initially, four different polymer solutions were selected in an attempt to prepare PSF/PCN-BA nanofibers with two different PSF/PCN-BA ratios (60/40 and 70/30 wt.%) and two different concentrations (20% (wt/vol) and 30% (wt/vol)). **Figure 9** represents the typical SEM micrograph for nonwoven PSF/PCN-BA fiber mesh obtained. At lower concentrations (**Figure 9a**,**b**), the fibers were not completely uniform and contained some beads, twists, and ovoid structures. The fiber diameter of the electrospun fibers was varied from 110 to 270 nm for PSF/PCN-BA = 60/40 wt.% (20 wt/vol) and from 180 to 360 nm for PSF/PCN-BA = 70/30 wt.% (20 wt/vol) nanofibers. With the increase in concentration, the electrospun nanofibers became more uniform with larger diameters (ranging from 350 to 800 nm with some individual fibers measuring about 1.5 microns). At the same time, the PSF/PCN-BA = 60/40 wt.% (30 wt/vol) nanofibers exhibited some twists and were conglutinated and distorted at their cross-points (**Figure 9c**). **Figure 9d** shows that better-defined and more uniform nanofibers (fiber diameter distribution: 500–900 nm) were obtained from the PSF/PCN-BA = 70/30 wt.% (30 wt/vol) polymer solution.

To investigate the morphology of PSF/PCN-BA = 70/30 wt.% (30 wt/vol) electrospun nanofibers during the thermal curing of a cyanate/benzoxazine prepolymer and to control the formation of the cross-linked structure, respectively, SEM and IR spectroscopy studies were conducted. **Figure 10a** shows the FTIR spectra of individual DCBE, BA-a, and PSF. In the spectrum for neat DCBE, the characteristic bands at 2237 and 2266 cm^{-1} denote C ≡ N stretching vibrations, and the characteristic absorption bands at 1500 and 1601 cm^{-1} are due to the vibration of benzene ring. The FTIR spectrum of BA-a monomer includes the absorption bands at 1232 and 1159 cm^{-1} assigned, respectively, to the antisymmetric stretching vibration of C–O–C and C–N–C of oxazine functionality. There is also the characteristic band at 947 cm^{-1} which corresponds to the benzene ring mode that is attached to the oxazine ring. The spectrum of individual PSF contains bands with maxima at 1150 and 1324 cm^{-1} corresponded to symmetric and asymmetric S=O stretching vibrations, band at 1244 cm^{-1} assigned to C–O–C stretching vibrations, and bands at 874 and 833 cm^{-1} are due to aromatic CH deformation vibration.

Figure 10b shows the evolution of FTIR spectra for PSF/PCN-BA electrospun nanofibers with thermal treatment. The absorption bands distinctive for all the individual components of unheated nanofibers are present in the spectrum. The intensities of the absorption bands with maxima at 2266–2235 and 947 cm^{-1} start to decrease gradually with increasing the curing temperature from 120 to 150°C. After raising the curing temperature to 180°C, the aforementioned absorption bands disappear, demonstrating that cyanate and oxazine groups have been completely exhausted under the applied conditions. Typically, for the complete curing of the individual monomers DCBE and BA-a, temperatures of 177–200°C and ~ 200°C are required,

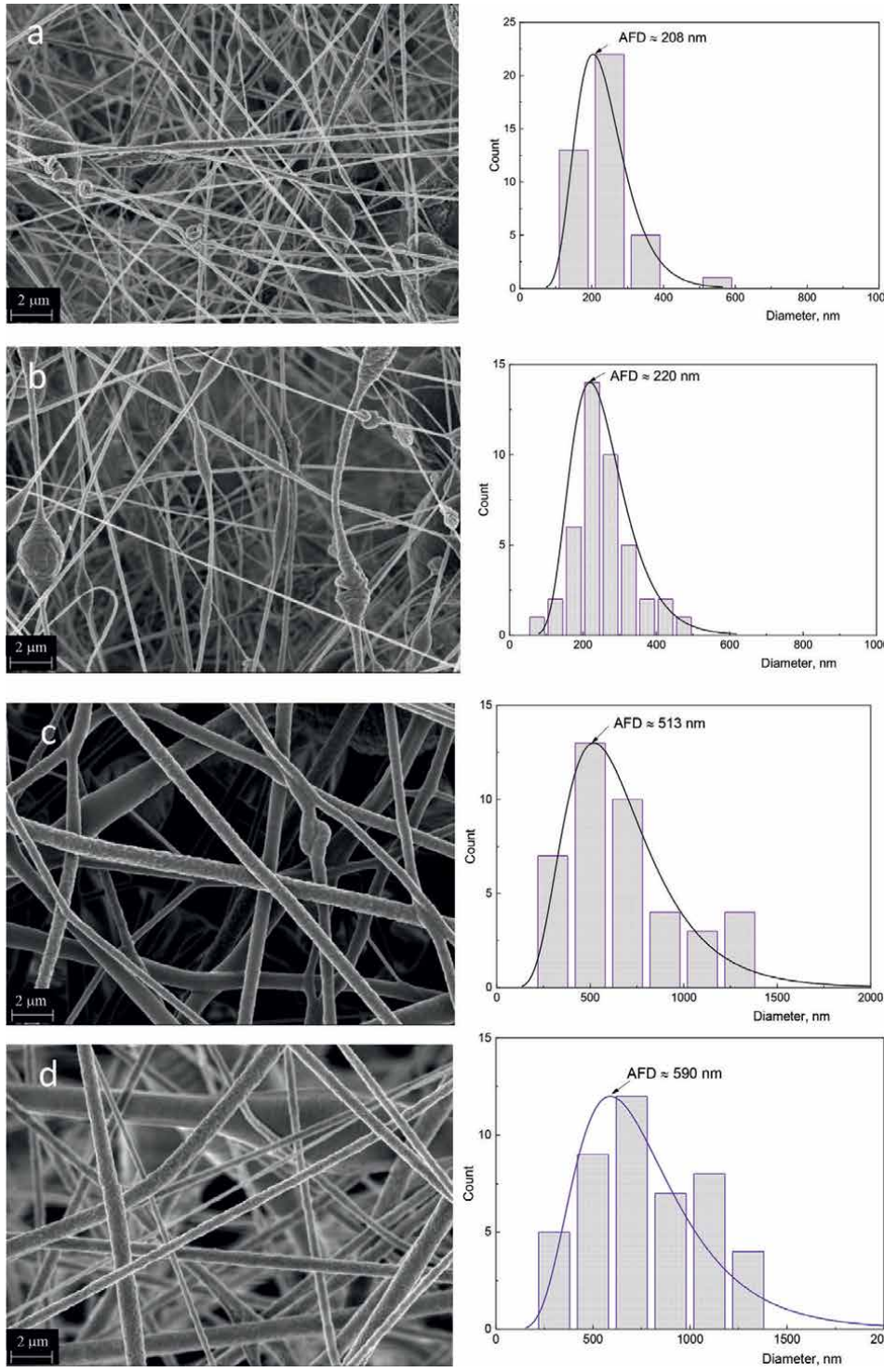


Figure 9.
Typical SEM micrographs for the samples (wt.%): (a) PSF/PCN-BA = 60/40 (20 wt/vol), (b) PSF/PCN-BA = 70/30 (20 wt/vol), (c) PSF/PCN-BA = 60/40 (30 wt/vol), and (d) PSF/PCN-BA = 70/30 (30 wt/vol) electrospun nanofibers. The corresponding fiber diameter distributions are displayed on the right side.

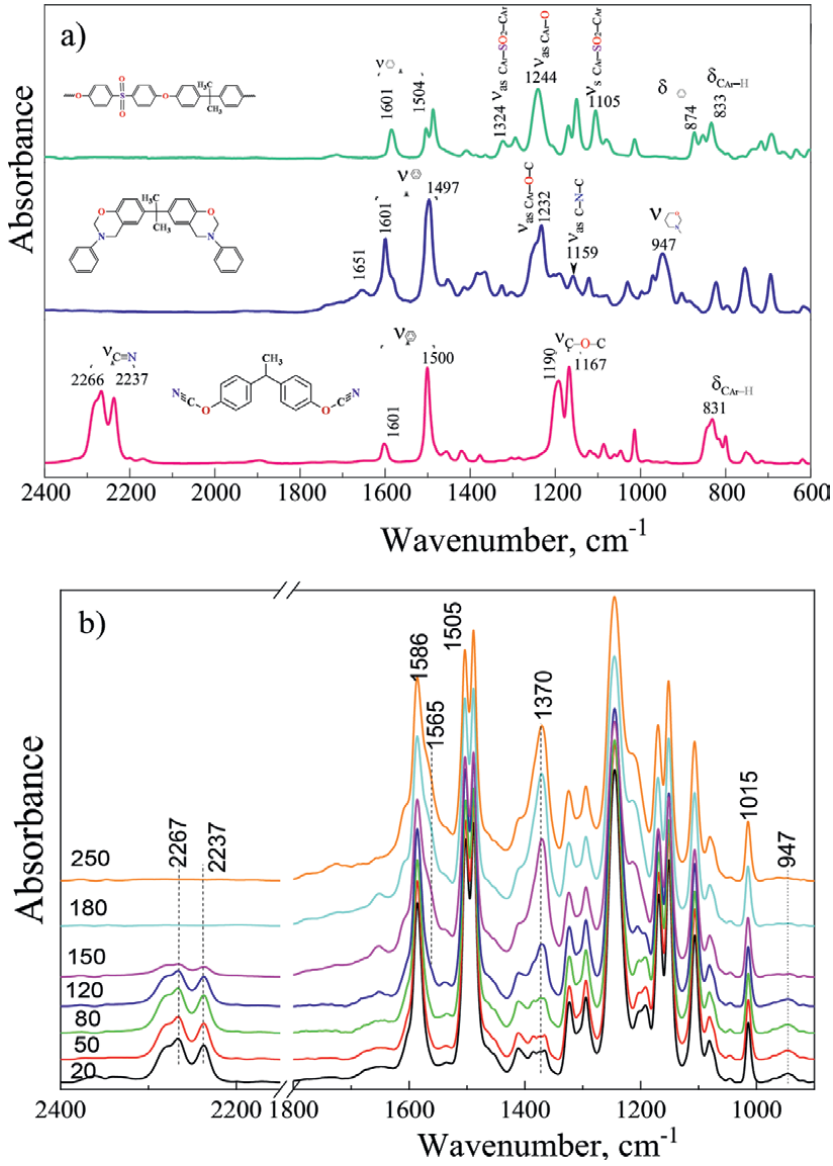


Figure 10.
FTIR spectra of typical samples: (a) individual DCBE, BA-a, and PSF; (b) PSF/PCN-BA = 70/30 wt.% electrospun nanofibers cured at different temperatures (indicated in the spectra).

respectively [51–55]. Therefore, it can be concluded that in this ternary composition, the curing of DCBE and BA-a is completed at a lower temperature, probably due to a synergetic effect.

Figure 11 shows the change in fiber morphology for PSF/PCN-BA = 70/30 (wt.%) electrospun fibers after their heat treatment during the synthesis of the PCN-BA component within the PSF fibers. It is obvious that the electrospun fibers retain their shape when heated to 80°C (**Figure 11a**). With a further increase in the synthesis temperature of PCN-BA as an IPN component to 120°C, a noticeable thickening of the fibers occurs (**Figure 11b**), apparently due to swelling of the PSF matrix of the

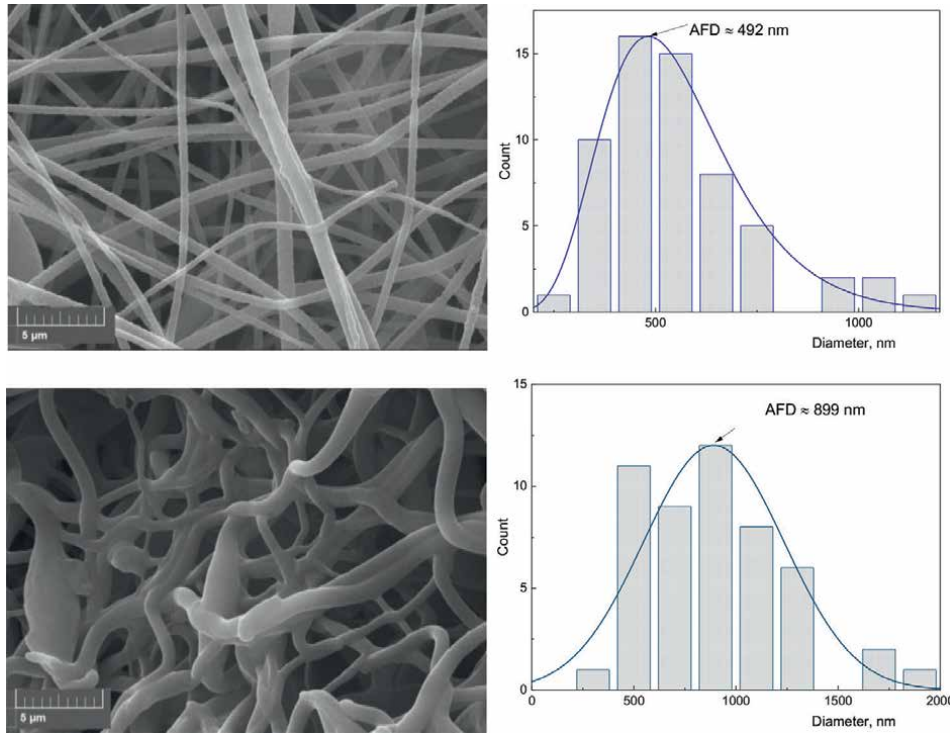


Figure 11.
Typical SEM micrograph for PSF/PCN-BA = 70/30 (wt.%) electrospun nanofibers cured at different temperatures: (a) 80°C and (b) 120°C. The corresponding fiber diameter distributions are displayed in the right side.

electrospun fibers with the low-viscosity PCN-BA prepolymer. It can be concluded that the synthesis of the PCN-BA component of PSF/PCN-BA electrospun nanofibers should be carried out at temperatures below 80–100°C. Our new studies have shown that it is possible to significantly reduce the synthesis temperature (even up to room temperature) of polycyanurates or their copolymers by introducing various nanofillers, for example, amino-functionalized POSS.

The thermal stability of cured PSF/PCN-BA semi-IPNs was examined by TGA, and the thermogravimetric curve (TG) and corresponding derivative curve (DTG) are shown in **Figure 12**. For comparison, the TG and DTG curves for the hybrid PCN-BA thermoset obtained under the same curing conditions and for the pure PSF are presented in the plot. Two mass loss stages were observed in DTG curves for the PCN-BA hybrid thermoset and neat PSF. During the first stage of degradation (at a temperature of maximum degradation rate $T_{d\,max1}$ ~ 421 and 535°C, respectively for PCN/BA and PSF), thermal degradation of the polymers occurs, and during the second mass loss stages at $T_{d\,max2}$ ~ 628 and 622°C, respectively, the oxidation of decomposed residues derived from the first stage takes place. Cured PSF/PCN-BA film sample possess good heat resistance with the initial thermal-oxidative degradation temperature ($T_{d5\%}$) above 390°C. The thermooxidative degradation in air of PSF/PCN-BA semi-IPN occurs in four stages: at $T_{d\,max1}$ ~ 421°C, $T_{d\,max2}$ ~ 542°C, $T_{d\,max3}$ ~ 589°C, and $T_{d\,max4}$ ~ 658°C. The mass loss of PSF/PCN-BA at the first low-intensity stage can be related to the thermal decomposition of the mixed PSF/PCN-BA microphase formed as a result of the interpenetrating of macromochains of PCN-BA

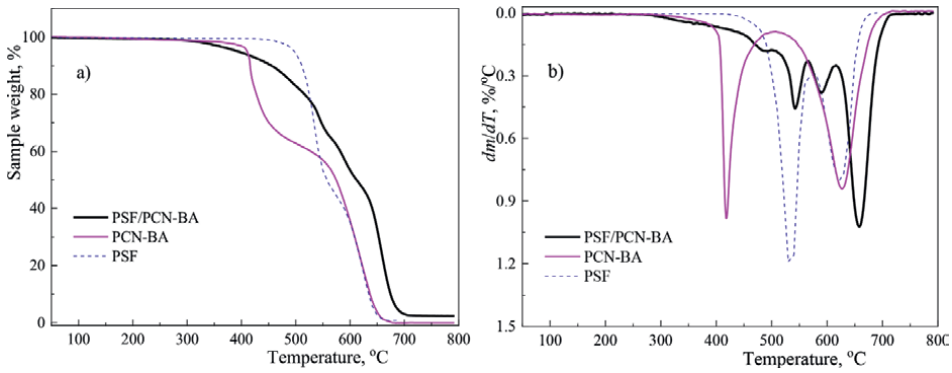


Figure 12.
TG (a) and DTG (b) curves for cured PSF/PCN-BA = 70/30 (wt.%) film sample with a structure of semi-IPNs, PCN-BA hybrid thermoset, and neat PSF (indicated in the plot).

and PSF during *in situ* synthesis. The mass loss at $T_{d\ max2}$ ~ 542°C can be attributed to the thermal decomposition of the PSF component. The mass losses with maxima rates at 589 and 658°C are caused essentially by oxidative processes in the composition. These results indicate that the PSF/PCN-BA semi-IPN has better thermal oxidative stability at temperatures higher than 600°C compared to their constitutive components.

The thermophysical properties of cured PSF/PCN-BA film sample with a structure of semi-IPNs compared to PCN-BA hybrid thermoset and neat PSF were studied by DSC (**Figure 13**).

Both PCN-BA and individual PSF have DSC traces typical for amorphous polymers with single glass transition temperatures, T_g, at ~196 and ~ 184°C, respectively. The DSC thermogram of the PSF/PCN-BA film sample shows one main endothermic transition corresponding to T_g ~ 185°C of the PSF component in semi-IPNs. The presence of two other small endothermic transitions in the region of lower temperatures (at T ~ 130–160°C) may indicate the formation of some microphase heterogeneity in the sample.

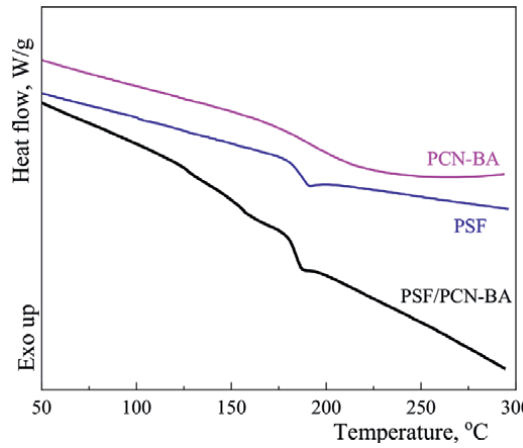


Figure 13.
DSC thermograms for PCN-BA hybrid thermoset and neat PSF, as well as for cured PSF/PCN-BA = 70/30 wt.% film sample with a structure of semi-IPNs (indicated in the plot).

Thus, our studies have shown the fundamental possibility of obtaining electrospun PSF/PCN-BA nanofibers with acceptable thermal properties. In addition, it is known that the polycyanurate component of these nanofibers, in addition to heat resistance, is characterized by high chemical resistance to various aggressive environments, as well as low dielectric losses and water absorption. Therefore, resulting electrospun fibers can potentially become useful components of various textile materials with an improved set of properties suitable for operation in aggressive environments over a wide temperature range.

7. Conclusions

Over recent years, significant attention has been paid to the development of new directions toward the engineering of heat-resistant micro- and nanofibers based on thermosets and their nanocomposites, as well as thermoplastic/thermosetting polymers with a semi-IPN structure. Thus, this chapter addresses the main recent approaches and technical solutions for the creation of polymer micro or nanofibers by electrospinning. Miscellaneous systems have been investigated, including epoxy electrospun continuous micro- and nanofibers, submicron carbon nanotube—epoxy nanocomposite filaments, rigid fibers based on functional polynorbornenes with epoxy or carboxylic pendant groups, and core-shell nanofibers with a structure of semi-IPNs based on thermoplastic polyamide (PA) and thermosetting epoxy resin. The first experimental results for the creation of electrospun nanofibers with a semi-IPN structure based on polysulfone/polycyanurate (PSF/PCN) or polysulfone/polycyanurate-benzoxazine (PSF/PCN-BA) are discussed. The possibility of controlling the average diameter from ~220 nm up to ~900 nm for the PSF/PCN or PSF/PCN-BA based electrospun nanofibers is fixed by changing the composition as well as processing parameters. It was found that the synthesis of the PCN-BA component of PSF/PCN-BA electrospun nanofibers should be carried out at temperatures below 80–100°C, but our recent studies have shown the possibility to reduce the synthesis temperature (even up to room temperature) for PCN by using various nanofillers, for example, amino-functionalized POSS. Due to the unique properties of PCNs, that is, high heat- and chemical resistance to aggressive substances, low dielectric losses and low water absorption, PCN-containing electrospun fibers could potentially become useful as components in advanced materials with improved properties to work in aggressive environment over a range of temperatures.

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Conflict of interest

The authors declare no conflict of interest.

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Chapter 6

Perspective Chapter: Characterization Methods for Nanofibers

Sahar Akhavan-Mahdavi and Mahnaz Sadat Mirbagheri

Abstract

Nanofibers represent a class of materials with diverse applications across fields such as materials science, biotechnology, and nanomedicine. Characterizing these materials comprehensively is essential for understanding their properties and behaviors. This chapter explores various characterization methods for nanofibers, including structural analysis using microscopy techniques, spectroscopic methods for chemical composition elucidation, mechanical testing for assessing mechanical properties, surface characterization techniques, and the integration of experimental techniques with computational modeling. By employing multimodal characterization strategies, researchers can gain insights into the structural, chemical, mechanical, and surface properties of nanofibers, enabling tailored applications in diverse fields. Continuous innovation in characterization methods is crucial to keep pace with the evolving demands of nanotechnology and unlock new insights into nanofiber behavior and potential applications.

Keywords: nanofibers, characterization, microscopy, spectroscopy, mechanical properties, computational modeling

1. Introduction

Nanofibers have garnered considerable attention across multiple scientific disciplines due to their exceptional properties and versatile applications. Characterization methods are essential for unveiling the intricate structure–property relationships inherent in these nanostructures [1]. Fundamental properties such as morphology, structure, surface chemistry, and mechanical behavior are pivotal for optimizing nanofiber design and fabrication processes. Techniques such as electron microscopy, spectroscopy, and mechanical testing provide invaluable insights into nanofiber characteristics with high resolution and precision. Despite notable advancements, challenges persist in achieving comprehensive and accurate nanofiber characterization [2]. Issues such as sample preparation techniques, instrument limitations, and data interpretation complexities pose hurdles in obtaining reliable characterization results. Overcoming these challenges necessitates innovative approaches and interdisciplinary collaborations to develop robust characterization protocols and techniques [3].

Future directions in nanofiber characterization research hold promise for continued innovation and advancement. Emerging trends include the development of multimodal characterization approaches that integrate multiple techniques to provide comprehensive insights into nanofiber properties and behavior [4]. *In situ* and real-time characterization techniques are anticipated to revolutionize nanofiber research by enabling dynamic observation of nanofiber formation processes and behavior under various environmental conditions. Furthermore, the integration of artificial intelligence and machine learning algorithms holds potential for automating data analysis and interpretation, facilitating the extraction of meaningful insights from complex characterization datasets [5].

Overall, nanofiber characterization plays a pivotal role in advancing nanofiber research and enabling the development of tailored nanofiber materials with enhanced performance and functionality for diverse applications. By addressing existing challenges and leveraging emerging trends, researchers can unlock new opportunities for innovation in this burgeoning field, driving transformative advancements in nanotechnology [6].

This chapter aims to thoroughly explore the methods used to characterize nanofibers, providing insights into the complex relationship between their structure and properties. By exploring key characteristics such as morphology, mechanical behavior, surface chemistry, and porosity, coupled with an analysis of advanced techniques such as microscopy, spectroscopy, mechanical testing, and thermal analysis, researchers gain valuable tools to assess and enhance nanofiber materials for various applications.

2. Analytical techniques for nanofiber assessment

Characterization of nanofibers is crucial for correlating test metrics with the practical characteristics of materials and verifying product quality during synthesis. Through single-fiber characterization, fundamental data are obtained to understand the relationship between the structure and properties of nanofibers [7]. Although theoretically, several characterization techniques can be utilized for nanofibers, a principal limitation is the lack of confirmed appropriate techniques. Challenges in characterizing nanofibers include manipulating small fibers, finding appropriate observation modes, identifying suitable sources for force transducers and actuators, and preparing single nanofiber samples. However, many of these limitations have been addressed with advancements such as high-resolution nanomanipulators and simplified sample preparation procedures [8]. The various analytical techniques for nanofiber assessment and mechanical properties characterization are summarized in **Table 1**.

The type of characterization approach used for testing nanofibers depends on the ultimate application of the prepared materials. For example, nanofibers intended for air filtration in gas masks require testing for air permeability, particle penetration, porosity distribution, and residual electrical charge on fibers [25]. For sensors, electrical conductivity, optical characteristics, equilibrium swelling by organic vapors, and chemical reactivity are important parameters to test. Although characterization techniques are still evolving, the demand for efficient methods is increasing. Therefore, this section reviews basic information about selected characterization methodologies used for nanofibers, including those adopted as standard test methods by organizations such as the American Society for Testing and Materials (ASTM) or the International Organization for Standardization (ISO) and provides information on limited single-fiber assessment techniques [26].

Analytical technique		Application	Information provided	Refs.
Morphological characterization	Scanning electron microscopy (SEM)	Visualization of nanofiber morphology	Fiber diameter, surface structure, and alignment	[9]
	Diameter and size distribution of nanofibers	Determination of fiber diameter and size distribution	Fiber diameter, size distribution, and uniformity	[10]
	Confocal laser scanning microscopy (CLSM)	3D imaging of nanofiber structures	Spatial distribution, layer thickness	[11]
Structural analysis	Transmission electron microscopy (TEM)	High-resolution imaging of nanofibers	Internal structure, crystallinity, and defects	[12]
	X-ray diffraction (XRD)	Crystallinity and phase identification	Crystal structure, degree of crystallinity	[13]
Mechanical characterization	Atomic force microscopy (AFM)	Surface topography and mechanical properties	Surface roughness, mechanical stiffness, and elasticity	[14]
	Dynamic mechanical analysis (DMA)	Mechanical properties characterization	Viscoelastic behavior, storage, and loss modulus	[15]
	Universal tensile machine	Mechanical properties characterization	Tensile strength and Young's modulus	[16]
Thermal properties	Thermogravimetric analysis (TGA)	Thermal stability and composition analysis	Decomposition temperature and compositional analysis	[17]
	Differential scanning calorimetry (DSC)	Thermal transitions and stability	Glass transition, melting point, and crystallization	[18]
Chemical characterization	Fourier-transform infrared spectroscopy (FTIR)	Chemical composition analysis	Functional groups and molecular structure	[19]
	Raman spectroscopy	Chemical and structural characterization	Molecular vibrations and chemical bonding	[20]
	Nuclear magnetic resonance (NMR)	Molecular structure and dynamics	Chemical environment and molecular interactions	[21]
Surface characterization	X-ray photoelectron spectroscopy (XPS)	Surface chemical analysis	Elemental composition, oxidation states, and functional groups	[22]
	Surface plasmon resonance (SPR)	Surface binding interactions analysis	Biomolecular interactions and affinity measurements	[23]
	Contact angle measurement	Wettability characterization	Surface energy and contact angle	[3]
	Dynamic light scattering (DLS)	Particle size and size distribution analysis	Hydrodynamic diameter and aggregation state	[24]

Table 1.
Analytical techniques for nanofiber assessment and mechanical properties characterization.

Analytical techniques play a pivotal role in the assessment of nanofibers, offering researchers valuable insights into their structure, properties, and performance. These techniques encompass a wide range of methodologies tailored to the unique challenges posed by nanoscale materials. Advanced microscopy techniques, such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM), are at the forefront of nanofiber assessment, providing high-resolution imaging of nanofiber morphology and structure. By visualizing nanofibers at the atomic and molecular levels, researchers can discern features such as fiber diameter, alignment, and surface morphology, laying the foundation for comprehensive characterization [27].

In addition to microscopy, spectroscopic techniques are instrumental in probing the chemical composition and surface properties of nanofibers. Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy enable researchers to identify functional groups, chemical bonds, and molecular species present on nanofiber surfaces. These techniques offer valuable insights into surface chemistry, surface functionalization, and interactions with surrounding environments, providing crucial information for tailoring nanofiber properties to specific applications [28].

Mechanical testing techniques are also essential for evaluating the mechanical properties of nanofibers, such as tensile strength, modulus, and flexibility. Techniques such as tensile testing, atomic force microscopy (AFM), and nanoindentation allow researchers to assess the mechanical behavior of nanofibers at the nanoscale level. By subjecting nanofiber samples to controlled mechanical forces, researchers can characterize their elastic and plastic deformation behavior, as well as their resistance to mechanical stresses, guiding the design of robust and durable nanofiber materials [29]. Furthermore, thermal analysis techniques are employed to investigate the thermal stability, crystallinity, and thermal conductivity of nanofibers. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) provide insights into the thermal properties and degradation behavior of nanofiber materials under controlled temperature and atmosphere conditions. These techniques offer valuable information for understanding the thermal stability, phase transitions, and degradation kinetics of nanofibers, guiding their application in high-temperature environments [30].

3. Morphological characterization of nanofibers

Nanofibers exhibit a diverse range of morphologies, influenced by factors such as processing conditions, polymer composition, and post-treatment methods. Morphological insights into nanofibers delve deep into their physical dimensions, surface features, and overall structural organization. Advanced microscopy techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), and atomic force microscopy (AFM) reveal intricate details about nanofiber morphology, including diameter distribution, length, alignment, and surface roughness. These insights are crucial for understanding the behavior and properties of nanofibers in various applications, from tissue engineering to filtration membranes [31].

SEM images can showcase aligned polymeric nanofibers fabricated through electrospinning, where fibers exhibit uniform morphology with consistent diameter and orientation. For instance, in a study by Smith et al., aligned polycaprolactone nanofibers were fabricated for tissue engineering applications. The aligned morphology enhances cell attachment and alignment, making these nanofibers suitable for guiding cell growth in tissue regeneration therapies [32].

TEM images reveal branched nanofibers synthesized *via* the self-assembly of block copolymers, similar to structures observed in the work of Jiang et al. on poly(ethylene glycol)-block-poly(caprolactone) copolymers. The branched morphology enhances the surface area and porosity of the nanofibers, making them promising candidates for applications in gas adsorption and catalysis [33].

AFM images can depict core-shell structured nanofibers composed of a polymer core surrounded by a shell of functionalized nanoparticles. This morphology is reminiscent of the core-shell nanofibers synthesized by Mamun et al. using a coaxial electrospinning technique. The core-shell structure provides controlled release properties for encapsulated drugs and enhances mechanical strength, suggesting potential applications in drug delivery and tissue engineering (**Figure 1**) [34].

These examples highlight the diverse morphologies achievable in nanofiber structures and underscore the importance of morphological characterization in tailoring their properties for specific applications. Incorporating such visuals enhances the understanding of nanofiber morphology and its implications in various fields of research and development.

The study on the morphological characteristics of electrospun poly(vinyl alcohol) (PVA) and chitosan (CS) nanofibers found that fiber quality and morphology were highly dependent on polymer concentration and electrospinning conditions. Under optimized conditions, uniform fibers with diameters ranging from 77 to 292 nm, averaging 153 nm, were produced (**Figure 2**) [35].

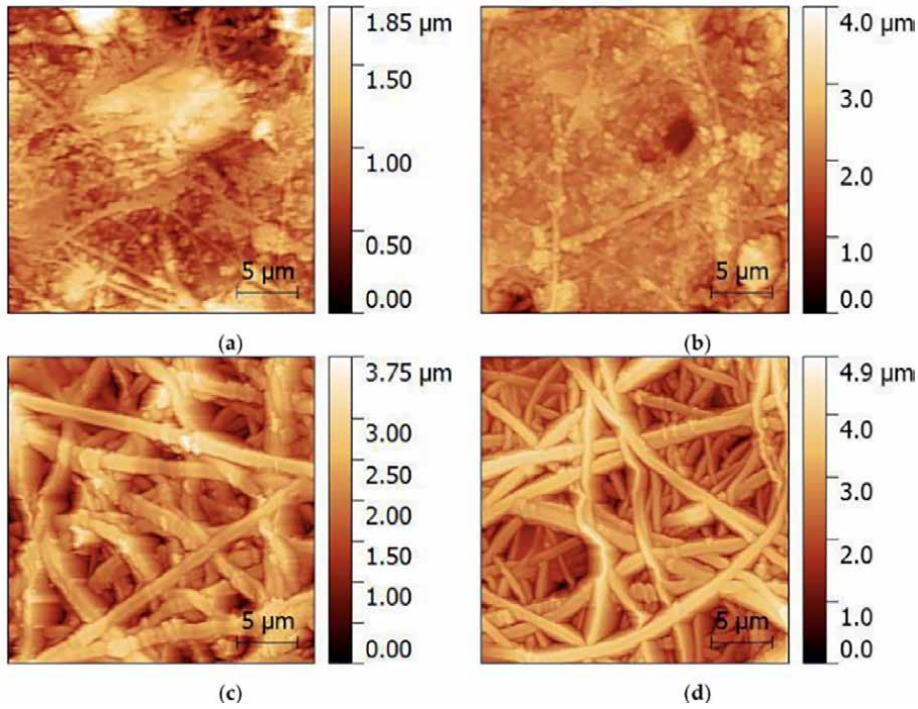


Figure 1.
Atomic force microscopy (AFM) images of the magnetic nanofiber mats: (a) magnetic fibers (MF); (b) magnetic fibers with rotating collector (MR); (c) core-shell fibers (CSF); and (d) core-shell fibers with rotating collector (CSR) [34].

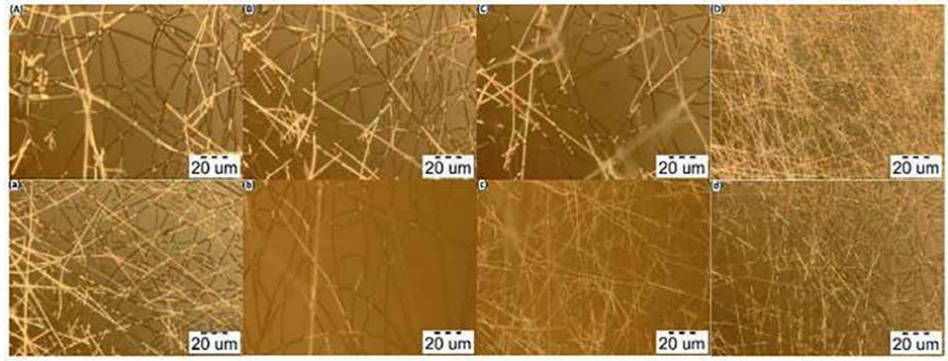


Figure 2.
Micrographs of the electrospun fibers from different concentrations of poly(vinyl alcohol)/chitosan (1000× magnification) [35].

Morphological analysis using field emission scanning electron microscopy (FESEM) revealed that activated carbon nanofibers (ACNFs) had more porous and compact fibers with a smaller average diameter of 263 ± 11 nm compared to carbon nanofibers (CNFs), which had a larger average diameter of 323 ± 6 nm (**Figure 3**) [36].

SEM images showed that coaxial nanofiber mats were uniformly distributed with smooth surfaces and a distinct porous structure. TEM confirmed the core-sheath structure, with diameters of 222 ± 44 nm for drug-free PCL/GEL nanofibers, 204 ± 21 nm for PCL-Cur/GEL, 173 ± 43 nm for PCL/GEL-TH, and 178 ± 25 nm for PCL-Cur/GEL-TH nanofibers (**Figure 4**) [37].

Morphological assessment of nanofibers requires a diverse toolkit of techniques tailored to probe their structural intricacies with precision. Techniques such as SEM, TEM, and AFM provide high-resolution imaging to visualize nanofiber morphology at different length scales. Image analysis software facilitates quantitative assessment of parameters such as fiber diameter and alignment. Complementary methods such as X-ray diffraction (XRD) and small-angle X-ray scattering (SAXS) offer insights into the hierarchical structure and crystallinity of nanofibers, guiding material design and optimization [38].

Advanced techniques for morphological assessment include innovative approaches that enhance precision and sensitivity. Techniques such as super-resolution microscopy and cryo-electron microscopy (cryo-EM) allow visualization of nanofiber morphology at unprecedented resolutions, revealing intricate internal structures and surface features. Spectroscopic techniques such as surface-enhanced Raman spectroscopy (SERS) and tip-enhanced Raman spectroscopy (TERS) provide exceptional sensitivity in examining the chemical composition and molecular interactions of nanofibers. Computational modeling techniques also aid in predicting and interpreting nanofiber structures, pushing the boundaries of nanofiber characterization and unlocking new opportunities for diverse applications [39].

Characterizing the porosity and pore size distribution of nanofibers is essential for applications such as scaffolding and filtration. Porous materials are categorized into micropores, mesopores, macropores, capillaries, and macrocapillaries based on their sizes. Techniques used for porosity characterization include: (1) mercury intrusion porosimetry, which measures accessible voids by forcing mercury into the sample under pressure, though it can distort soft polymers; (2) liquid extrusion porosimetry,

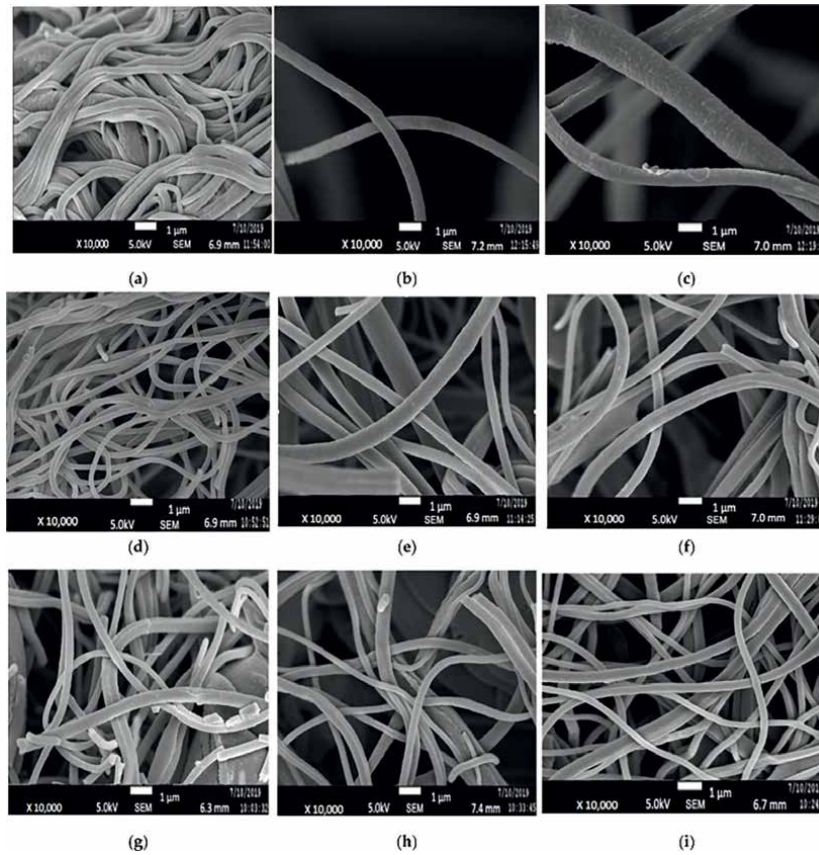


Figure 3.
Field emission scanning electron microscopy images of polyacrylonitrile/lignin/poly(ethylene glycol) nanofibers with different ratios and polyethylene glycol contents [36].

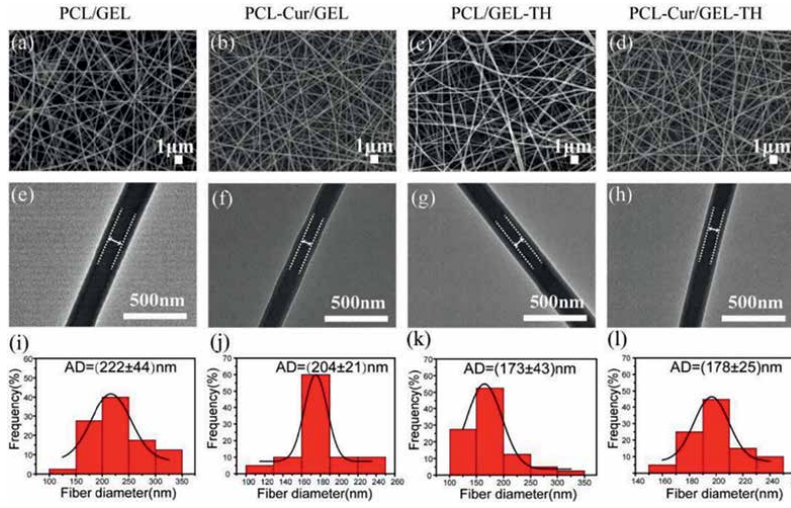


Figure 4.
(a–d) SEM, (e–h) TEM images and (i–l) the corresponding diameter distribution of different coaxial nanofibers. The white dash line in (e–h) indicates the border between the PCL core and GEL shell [37].

which uses lower pressure to intrude liquid into voids, suitable for softer materials; (3) capillary flow porometry, which monitors gas flow rate under differential pressure to measure pore size and volume without distorting the structure; and (4) nuclear magnetic resonance (NMR), which uses proton relaxation times to provide insights into pore size distribution and polymer structural characteristics [8].

Microscopic characterizations, such as optical and electron microscopy, are routinely used to monitor diameter distributions of nanofibers. However, due to the irregular geometry of most pores, these methods only determine surface voids, and the porosity data obtained may differ from other methods [40].

4. Structural analysis methods for nanofibers

Structural analysis methods for nanofibers encompass a wide array of experimental and computational techniques to understand these minute structures' architecture and composition. Researchers employ a combination of microscopy, spectroscopy, diffraction, and computational modeling to gain comprehensive insights into nanofiber properties. Microscopy allows for the visualization of nanofiber morphology, while spectroscopy provides details about chemical composition. Diffraction techniques, such as X-ray diffraction (XRD), are crucial for assessing crystallinity and atomic structure. Additionally, computational modeling aids in simulating nanofiber structures and behaviors, further enhancing understanding [41].

Exploring nanofiber structures through analysis involves systematically examining their structural attributes using various analytical techniques. This approach enables researchers to uncover details about nanofiber morphology, alignment, and organization, providing a deeper understanding of their properties and potential applications. Methods for structural analysis encompass a diverse set of experimental and computational techniques tailored specifically for characterizing nanofiber structures. These methods include microscopy for visualizing nanofiber morphology, spectroscopy for analyzing chemical composition, and mechanical testing for assessing material properties [42].

Analytical techniques for nanofiber structural analysis focus on specialized tools and methodologies designed to probe the structural properties of nanofibers in detail. These techniques include electron microscopy, X-ray diffraction, and atomic force microscopy, offering high-resolution imaging and precise measurement capabilities for analyzing nanofiber architecture and behavior. Advanced methods for nanofiber structure analysis utilize cutting-edge technologies such as advanced microscopy techniques, tomography, and molecular modeling to investigate the complex hierarchical organization of nanofibers [28].

Tools and methods for structural characterization encompass a diverse set of experimental tools and techniques for characterizing nanofiber structures. These tools include microscopy, spectroscopy, chromatography, and diffraction methods, which provide complementary information about nanofiber morphology, composition, and properties [43]. Approaches to nanofiber structural analysis involve systematic strategies for studying nanofiber structure and behavior. These approaches integrate experimental techniques with data analysis and modeling to understand how factors such as processing conditions and material composition influence nanofiber structure and performance. Methods for structural examination focus on the systematic investigation of nanofiber structure using analytical techniques, enabling researchers to assess parameters such as fiber diameter, length, alignment, surface

properties, and chemical composition, providing valuable insights into nanofiber structure and behavior [44]. Diverse range of techniques used for structural analysis of nanofibers, each providing unique insights into their properties and behavior for various applications. XRD can identify the crystalline phases present in nanofibers, providing information about their atomic arrangement and crystal structure. For example, XRD can be used to determine whether electrospun polymer nanofibers exhibit an amorphous or crystalline structure [45].

5. Mechanical characterization techniques

Mechanical characterization techniques play a crucial role in understanding the mechanical behavior and properties of nanofibers, which are essential for various applications ranging from tissue engineering to reinforcement materials. Various mechanical characterization techniques were employed to evaluate the properties of nanofibers [46]. A summary of these techniques, their key features, measured properties, and applications are provided in **Table 2**.

One commonly used technique is tensile testing, which involves applying a controlled force to a nanofiber sample and measuring its deformation response. Samples are prepared into rectangular or dumbbell-shaped specimens and tested using a universal testing machine. During the test, the sample is slowly extended until it fractures, and data on gauge elongation and applied force are recorded. These data

Technique	Key features	Measured properties	Applications	Refs.
Tensile testing	Involves applying a controlled force and measuring deformation; samples prepared in rectangular or dumbbell shapes	Tensile strength, Young's modulus, and elongation at break	Assessing mechanical performance and durability	[47]
Atomic force microscopy (AFM)	High-resolution imaging and mechanical mapping; measures stiffness, adhesion forces, and surface roughness	Nanoscale mechanical properties and surface topography	Surface characterization and nanometer-scale analysis	[48]
Nanoindentation	Presses a sharp indenter into the surface; analyzes force-displacement curve	Hardness and elastic modulus	Characterizing composites and coatings	[49]
Dynamic mechanical analysis (DMA)	Measures viscoelastic properties under controlled mechanical oscillations; uses temperature and frequency changes	Storage modulus, loss modulus, tan δ	Evaluating viscoelastic behavior and temperature dependence	[50]
Rheological testing	Assesses response to deformations under various conditions	Viscoelastic properties	Understanding behavior under frequency, temperature, and humidity variations	[51]

Table 2.
Summary of mechanical characterization techniques for nanofibers.

are then used to calculate engineering strain and stress, providing valuable information about the tensile strength, Young's modulus, and elongation at break of nanofibers. This allows researchers to assess their mechanical performance and durability. However, the fragility of nanofibers poses challenges during sample preparation and handling. Another important mechanical characterization technique is atomic force microscopy (AFM), which enables high-resolution imaging and mechanical mapping of nanofiber surfaces. AFM can be used to measure nanofiber stiffness, adhesion forces, and surface roughness with nanometer-scale resolution, providing insights into the nanoscale mechanical properties and surface topography of individual nanofibers. Nanoindentation is also widely used for assessing the hardness and elastic modulus of nanofibers. In this technique, a sharp indenter tip is pressed into the surface of a nanofiber sample, and the resulting force-displacement curve is used to calculate mechanical properties such as hardness and modulus [52]. This technique is particularly useful for characterizing nanofiber composites and coatings. Dynamic mechanical analysis (DMA) and rheological testing are employed to evaluate the viscoelastic properties and dynamic mechanical behavior of nanofibers. These techniques involve subjecting nanofiber samples to controlled mechanical oscillations or deformations and measuring their response, providing insights into their behavior under varying mechanical conditions such as frequency, temperature, and humidity. DMA, for instance, measures properties such as storage modulus, loss modulus, and tan δ master curves. The Arrhenius equation is often used to calculate the activation energy of nanofibers, which increases with the addition of reinforcing materials like nanotubes. However, the reinforcing effect may decrease at higher concentrations of nanotubes [53].

The mechanical properties of nanofibers are influenced by various factors, including polymer chemistry and processing conditions. Nanofibers electrospun from the same polymer but using different solvents can exhibit diverse mechanical properties due to variations in solvent evaporation rates and phase morphologies. Additionally, factors such as fiber density, fusion degree, diameter distribution, and fiber defects can affect tensile properties. Mechanical characterization techniques such as tensile testing, AFM, nanoindentation, DMA, and rheological testing offer valuable insights into the mechanical properties and behavior of nanofibers. These techniques facilitate the optimization and customization of nanofibers for specific applications, enabling researchers to gain a comprehensive understanding of the mechanical performance of nanofiber materials and design them for enhanced functionality and durability in various applications [54].

6. Thermal property evaluation of nanofibers

Understanding the thermal behavior of nanofibers is crucial for optimizing their performance in diverse applications. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) are commonly used techniques for this purpose. DSC measures heat flow associated with phase transitions and thermal events, providing information on melting temperature and crystallization behavior. On the other hand, TGA measures changes in sample weight as a function of temperature, offering insights into thermal stability and decomposition kinetics. These techniques help assess the suitability of nanofibers for high-temperature applications and their degradation behavior over time [55].

DSC has been employed to study the thermal behavior and mechanical performance of these nanofiber-reinforced composites under different temperature conditions. Understanding the thermal properties of these composites is essential for applications in aerospace, automotive, and structural engineering where thermal stability is critical.

TGA is frequently used to study the thermal degradation behavior of nanofiber materials. For example, researchers have investigated the thermal stability and decomposition kinetics of electrospun polymer nanofibers for biomedical applications. By analyzing the weight loss and decomposition temperatures using TGA, researchers can determine the thermal stability and degradation characteristics of these nanofiber materials, ensuring their suitability for specific biomedical applications [56].

In addition to DSC and TGA, thermal conductivity measurements are essential for evaluating the heat transfer capabilities of nanofiber materials. Techniques such as laser flash analysis and thermal conductivity meters provide valuable information about heat conduction and dissipation, and critical for applications such as thermal insulation and heat management [57]. Dynamic mechanical analysis (DMA) and thermo-mechanical analysis (TMA) techniques further enhance our understanding by studying the coupled mechanical and thermal behavior of nanofibers. These techniques subject nanofiber samples to controlled mechanical and thermal loading conditions, providing insights into parameters such as thermal expansion and viscoelastic behavior. This knowledge is vital for designing nanofiber materials that can withstand thermal cycling and mechanical stress in various applications. These techniques collectively provide comprehensive insights into the thermal behavior and performance of nanofiber materials, allowing researchers to tailor their design and composition for specific applications requiring thermal stability, efficient heat transfer, and thermal management capabilities [58].

7. Chemical characterization approaches

Chemical characterization approaches are crucial for understanding the composition, structure, and surface chemistry of nanofibers, influencing their properties across various applications. Fourier-transform infrared spectroscopy (FTIR) stands out as a common technique, offering insights into functional groups, chemical bonds, and molecular composition. By monitoring chemical changes due to processing or environmental exposure, FTIR aids in identifying specific species in nanofiber materials [59]. In a study involving polyvinyl alcohol (PVA) nanofibers, FTIR spectroscopy was used to analyze the functional groups present in the nanofibers. By comparing FTIR spectra before and after surface modification, researchers identified changes in peak intensities corresponding to new functional groups, indicating successful surface functionalization for specific applications such as drug delivery or tissue engineering [60].

X-ray photoelectron spectroscopy (XPS) complements FTIR, providing details on elemental composition and chemical states of surface atoms. Employing XPS, researchers can discern elements on nanofiber surfaces and quantify their concentrations, essential for studying surface modifications and interfacial interactions. In a research project focused on graphene oxide (GO)-modified polymeric nanofibers, XPS analysis was employed to investigate the elemental composition and chemical

states of surface atoms. By analyzing the binding energies of carbon, oxygen, and other elements, researchers determined the extent of GO incorporation and its effects on surface chemistry, providing insights into the nanofiber interface for applications in sensors or membranes [61].

Nuclear magnetic resonance (NMR) spectroscopy is also employed to probe molecular structure and chemical environments within nanofibers. Offering insights into molecular conformation and bonding, NMR is particularly useful for characterizing nanofiber-based polymers and composites. In a study investigating the molecular structure of electrospun cellulose acetate (CA) nanofibers, NMR spectroscopy was used to analyze chain conformation and polymer crystallinity. By measuring relaxation times and chemical shifts in the NMR spectra, researchers characterized the polymer chain dynamics and crystalline phases present in the nanofibers, guiding the optimization of electrospinning parameters for enhanced fiber properties [62].

Chromatography techniques such as gas chromatography–mass spectrometry (GC–MS) and liquid chromatography-mass spectrometry (LC–MS) further analyze nanofiber chemical composition and molecular weight distribution. Separating and identifying individual components, including monomers and additives, these techniques offer valuable data on nanofiber purity and composition. In an analysis of polymeric nanofibers containing antimicrobial agents, gas chromatography–mass spectrometry (GC–MS) was employed to identify and quantify volatile compounds released during degradation. By separating and analyzing individual compounds, researchers determined the release kinetics of antimicrobial agents from the nanofibers, providing insights into their long-term efficacy and potential applications in wound dressings or air filtration [63].

Raman spectroscopy, another technique, delves into low-frequency modes in nanofibers, providing insights into polymer distribution and the presence of carbonaceous materials. It has been used to study stress transfer in polystyrene nanofibers loaded with multi-walled carbon nanotubes (MWCNTs) and to determine crystallite sizes in PAN-based carbon nanofibers [64].

8. Surface characterization of nanofibers

Surface characterization of nanofibers involves the examination of their surface properties, interactions, and functionalities, which are crucial for determining their performance in various applications. In addition to techniques such as scanning electron microscopy (SEM) and atomic force microscopy (AFM) for visualizing surface morphology and texture, spectroscopic methods such as X-ray photoelectron spectroscopy (XPS) and Fourier-transform infrared spectroscopy (FTIR) are utilized to analyze the chemical composition and surface chemistry of nanofibers. These techniques provide valuable insights into surface modifications, chemical reactions, and functionalization on nanofiber surfaces [65]. Moreover, contact angle measurements and surface energy analysis play a significant role in understanding the surface characteristics of nanofibers. By measuring the angle formed by a liquid droplet on the nanofiber surface, contact angle analysis evaluates surface hydrophilicity, hydrophobicity, and surface energy. This information is crucial for applications such as filtration, membrane separation, and biomedical devices, where surface interactions with liquids are essential [7]. Contact angle analysis, in particular, provides insights into the wettability and surface energetics of nanofiber materials, complementing

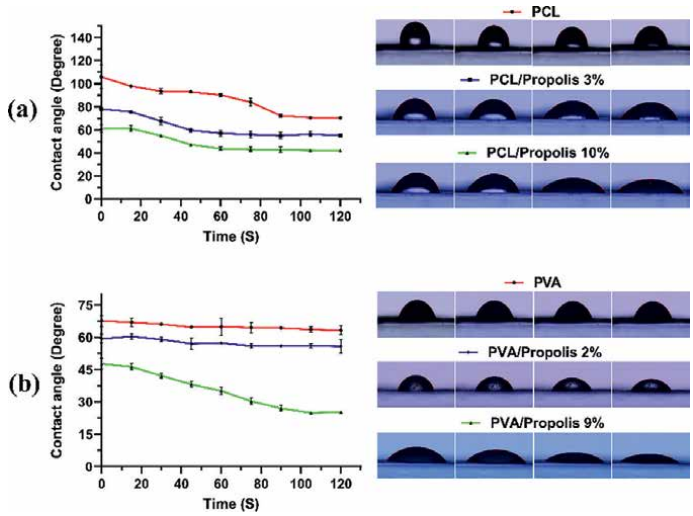


Figure 5.
Water contact angle of electrospun nanofibers: (a) PCL and PCL/PRP blend, and (b) PVA and PVA/PRP blend. Reproduced with permission [3].

other surface characterization techniques. Understanding the surface wettability and energy of nanofibers enables researchers to tailor their surface properties for specific applications, enhancing biocompatibility, promoting adhesion, or improving functionality in fields ranging from materials science to biotechnology and nanomedicine [53]. Contact angle analysis was employed to evaluate the effectiveness of surface functionalization techniques on nanofibers. Electrospun nanofiber mats were functionalized with biomolecules such as proteins or peptides, and the contact angle of biological fluids on the modified surfaces was measured. Changes in contact angle indicated successful surface functionalization, enabling tailored interactions with biological environments for applications in drug delivery, wound healing, and tissue engineering (**Figure 5**) [3].

9. Electrical and optical characterization methods

Electrical and optical characterization methods are crucial for assessing the electrical conductivity, optical properties, and performance of nanofibers in various applications. One commonly used technique for electrical characterization is electrical impedance spectroscopy (EIS), which measures the impedance of nanofiber materials as a function of frequency. EIS allows researchers to analyze the electrical properties of nanofibers, including conductivity, resistivity, and capacitance, providing insights into their charge transport mechanisms and electrical behavior [26].
In addition to EIS, conductivity measurements using techniques such as four-point probe and Hall effect measurements are employed to quantify the electrical conductivity of nanofiber materials. These techniques enable researchers to determine the intrinsic and extrinsic conductivity of nanofibers, assess their doping level, and study the effect of processing parameters on electrical properties. Understanding the electrical conductivity of nanofibers is crucial for applications such as electronic devices, sensors, and energy storage devices [28].

Furthermore, optical characterization techniques such as UV–visible spectroscopy and fluorescence spectroscopy are utilized to analyze the optical properties and photophysical behavior of nanofiber materials. UV–visible spectroscopy provides information about the absorption and transmission of light by nanofibers, allowing researchers to study their optical absorption spectra, bandgap energy, and optical transparency [29]. Fluorescence spectroscopy, on the other hand, enables the detection and analysis of fluorescent properties of nanofiber-based materials, which is useful for applications such as biosensing, imaging, and photonics. Additionally, techniques such as photoluminescence (PL) spectroscopy are employed to investigate the photoluminescent and vibrational properties of nanofibers, respectively. PL spectroscopy allows researchers to study the emission of light by nanofibers upon excitation with photons, providing insights into their optical properties and potential applications in optoelectronic devices [13].

Electrical and optical characterization methods play a crucial role in assessing the electrical conductivity, optical properties, and performance of nanofibers in various applications. By employing techniques such as EIS, conductivity measurements, UV–visible spectroscopy, fluorescence spectroscopy, and PL spectroscopy, researchers can gain comprehensive insights into the electrical and optical behavior of nanofiber materials, facilitating their design, optimization, and integration into electronic, photonic, and optoelectronic devices [7].

10. Integrative approaches for comprehensive nanofiber characterization

Integrative approaches for nanofiber characterization involve combining multiple analytical techniques to provide thorough insights into their properties and behavior. By merging complementary methods, researchers can surpass the limitations of individual techniques, gaining a more holistic understanding of nanofiber materials. One such approach involves integrating imaging techniques such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) with spectroscopic methods such as Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy. This integration allows simultaneous visualization of nanofiber morphology and structure at high resolution alongside analysis of their chemical composition and bonding characteristics [26].

In addition to imaging and spectroscopy, mechanical testing techniques such as tensile testing and atomic force microscopy (AFM) can be incorporated into integrative characterization strategies to evaluate nanofiber mechanical properties. By correlating structural information from microscopy with mechanical data, researchers can elucidate structure–property relationships and understand how structural features impact mechanical behavior. Furthermore, surface characterization techniques such as X-ray photoelectron spectroscopy (XPS) and contact angle measurements can be integrated to analyze nanofiber surface chemistry and wettability, offering insights into their surface properties and interactions [54].

Computer modeling plays a crucial role in the characterization of nanofibers by providing insights into their structural, mechanical, and thermal properties at the atomic and molecular level. Through computational simulations and modeling techniques, researchers can predict the behavior of nanofiber materials under different conditions, complementing experimental observations and providing deeper insights into their properties. One area where computer modeling excels is in predicting the structural properties of nanofibers. Molecular dynamics (MD) simulations,

for example, allow researchers to simulate the movement and interactions of atoms and molecules within nanofiber materials over time. By modeling the atomic structure of nanofibers and applying appropriate force fields, MD simulations can predict properties such as mechanical strength, flexibility, and stability, helping researchers understand how nanofibers respond to external forces and environmental conditions. Furthermore, computational modeling is invaluable for studying the mechanical properties of nanofibers. Finite element analysis (FEA), for instance, enables researchers to simulate the mechanical behavior of nanofiber structures under different loading conditions. By modeling the nanofiber geometry, material properties, and boundary conditions, FEA can predict parameters such as stress distribution, strain response, and deformation behavior, providing valuable insights into the mechanical performance of nanofibers and their suitability for various applications. In addition to structural and mechanical properties, computer modeling also aids in the study of the thermal behavior of nanofibers. Through techniques such as molecular dynamics simulations and Monte Carlo simulations, researchers can investigate thermal transport mechanisms, heat transfer processes, and thermal stability of nanofiber materials. Computational models allow researchers to explore how factors such as nanofiber morphology, surface chemistry, and intermolecular interactions influence thermal conductivity, diffusivity, and thermal expansion, facilitating the design of nanofibers for thermal management applications [8].

11. Conclusions

The characterization of nanofibers represents a multifaceted endeavor necessitating a comprehensive approach that integrates various analytical techniques. Throughout this chapter, we have delved into a plethora of methods utilized to probe the properties and behaviors of nanofiber materials. It is apparent that no single technique can furnish a comprehensive understanding of nanofiber materials. Instead, a confluence of complementary methods is imperative to surmount the limitations of individual techniques and attain a holistic perspective of nanofiber properties. By employing multimodal characterization strategies, researchers can delve into the structural, chemical, mechanical, and surface properties of nanofibers in depth, paving the way for their tailored application across diverse fields such as materials science, biotechnology, and nanomedicine. As the field of nanofiber research continues to evolve, it is essential to further refine and develop characterization methods to align with the evolving demands of nanotechnology. Through continuous innovation in characterization techniques and the assimilation of emerging technologies, researchers can unearth fresh insights into the behavior and potential applications of nanofiber materials, propelling progress in this dynamic field.

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Chapter 7

Perspective Chapter: Nanotechnology – A Sustainable Delivery System for Environmental Remediation

Iheoma Chigoziri Nwuzor

Abstract

There has been an emerging trend in using nanotechnology to transform modern-day technology completely. Advanced nanomaterials are greatly used in engineering applications, including environmental remediation, due to their excellent performance properties like high surface area, which always results in high performance efficiencies. The global environment is exposed to various types of pollution, including air, soil, and water, due to modernization and urbanization driven by industrialization. As a result, it is critical to explore novel technology to help address the problem of environmental contamination. Nano-remediation is a growing approach that uses an engineered material to clean and restore contaminated environments. This technology is a fast, efficient, and effective approach for handling persistent substances, such as heavy metals, pathogens, pesticides, halogenated chemicals, and chlorinated solvents. However, the traditional techniques of producing nanoparticles present dangerous environmental risks. To address these issues, scientists have shifted their attention to the more beneficial green synthesis approach, which is both cost-effective and environmentally friendly as it does not require any hazardous precursors or demanding processing conditions. Nanoparticles have great potential in water/wastewater treatments and environmental pollution control. This chapter is focused on some possible green synthesis and applications of nanomaterials for environmental remediation, challenges, and solutions.

Keywords: nanotechnology, environmental remediation, sustainable nanomaterials, eco-friendly materials, wastewater

1. Introduction

One of the most crucial challenges faced by the modern world today is environmental pollution resulting from ever-growing urbanization, deforestation, and industrialization. Different pollutants, like fertilizers, toxic dyes, heavy metals, inorganic and organic compounds, and industrial effluents, affect all major environmental components, including water, soil, and air [1–3]. Several strategies like adsorption, filtration,

aerobic/anaerobic digestion, coagulation, biological filters, activated sludge, chemical precipitation, photocatalysis, and advanced oxidation processes with diverse strategies are being investigated for the redress of these contaminants from the environment [4, 5]. Nanotechnology-based techniques have been reported to be more effective and efficient in environmental remediation [6]. Research into physical and biological chemistry modification by engineers and scientists to create new materials with desired morphology that can eliminate a variety of environmentally hazardous pollutants is still ongoing. These materials aim to utilize minimal energy, reduce cost, and achieve optimal performance [7, 8].

A variety of materials are sourced from various plant-based and other minerals and materials to redress the contaminants from the environment, including nanomaterials, nanoparticles (NPs), graphene oxide (GO), ceramics, zeolites, metal-organic frameworks, biochar, carbon nanostructures, and synthetic natural polymer membranes [9–12]. Due to their remarkable properties, including their distinct physical and chemical properties, regeneration efficiency, chemical reactivity, lower mass, greater surface area, and affordability, these materials are garnering a lot of research interest [13]. They are reported to be excellent materials for polymer grafting with different functional groups, both from inorganic and organic sources [11, 14]. However, the physical structures of nanomaterials, like size, porosity, shape, and other chemical factors, have a direct impact on their efficiency in performance [15]. Thus, by focusing on particular pollutants, nanomaterials' functionalization capacity boosts their efficacy and aptitude in environmental remediation. The elimination of environmental pollutants is accomplished by the application of nanomaterials through a variety of processes, including chemical reaction, adsorption, photocatalysis, filtration, and absorption [6, 16, 17].

For effective environmental remediation, adequate care is needed to avoid using materials that produce secondary or intermediate pollutants. This has led to the widespread use of nanomaterials derived from sustainable and biodegradable sources as an efficient environmental remediation in the field of nanoscience [17]. In addition, there is growing interest in using agricultural waste materials, such as fallen plant leaves and rice husks, for nanomaterials synthesis and low-cost manufacturing. These materials include biodegradable industrial and agricultural waste [18, 19].

The development of new nanomaterials for environmental remediation faces the key challenges of the sustainable synthesis method, nontoxicity, recycling ability, regeneration competence, biodegradability, and more essentially, sustainability. Understanding various types of nanomaterials used for soil, water, or air reclamation as well as their manufacturing process with reference source materials like plants, is essential to identifying sustainable nanotechnology-based approaches for environmental remediation [12]. This chapter aims to investigate and assess the capacity of nanotechnology as a viable and efficient method for delivering environmental remediation, targeting different types of environmental contaminants, and enhancing the cleanliness and safety of ecosystems.

2. Fundamentals of nanotechnology

Nanomaterials such as nanorods, nanotubes, nanofibers, nanoparticles, nanoplates, and carbon-based nanomaterials have excellent mechanical, optical, thermal, and electrical properties and performance for diverse applications, including environmental remediation, due to their nanoscale size. Nanocomposites are a combination

of nanoparticles (reinforcements or fillers) and a matrix (a structure with a continuous phase in which the nanoparticles are immersed) of bulk materials like polymers and ceramics, which improves the base material's property performance [20]. The nanoparticles are a dispersed phase that gives the nanocomposite unique characteristics. These nanocomposite materials exhibit enhanced mechanical performance, improved durability and hardness with better heat resistance, barrier properties, and thermal conductivity. Composite materials have a wide range of engineering applications. Nanostructured materials have a larger density of reactive sites on their particle surfaces due to their high surface area, which increases their overall efficiency and high rate of decontamination [20]. Subject to the area of application and requirements, nanomaterials' performance properties through a design, selective, and controlled synthesis approach can be improved [21]. The surface of engineered nanomaterials is functionalized to enable targeted interactions with target molecules or contaminants for effective environmental remediation.

Hybrid composites with desired performance properties present possible and more efficient and stable nanomaterials for environmental remediation than single-type nanoparticles approach. Based on their mode of action, the nanoparticles (NPs) utilized in environmental remediation can be categorized as reactive nanomaterials and nano-adsorbents. These types of nanomaterials work through chemical processes like redox, acid-base, precipitation, photocatalysis, and catalysis. In real-world applications, pressure or gravity can be used to inject or spray an aqueous or colloidal suspension of NPs into the contaminated environment [22].

3. Applications of nanotechnology for environmental remediation

3.1 Water/wastewater remediation

One of the most remarkable features of the world is water, which is highly significant to the existence of life. Although the globe is surrounded by water, only 2.5% of the world's water is freshwater [23]. In polar regions, over 70% of freshwater is frozen, and the remaining significant amount is present either in deep aquifers or as moisture that is inaccessible to humans [24]. However, only 1% of all freshwater is fit for human use, highlighting how vital water is to life. A few decades ago, water, was a natural resource that was free and accessible, but owing to uncontrolled withdrawals, overuse, and excessive contamination of water resources, the world is currently experiencing a severe water crisis. Remediation of water from various hazardous substances, like dyes, heavy metals, and pharmaceuticals, is greatly needed to control this contamination of the water environment [25]. Many researchers have employed traditional methods with various materials, including biochar derived from various sources, activated carbon, and various low-cost adsorbents [18, 26] for water remediation.

Nanostructured materials have received increasing interest in the field of water and wastewater reclamation because of their exceptional qualities and improved performance [27]. Several researchers have investigated and reported that nanomaterials hold great potential in adsorbing and/or degrading a variety of contaminants from the air, soil, water, and wastewater, including heavy metals, pharmaceuticals, and dyes [12]. Various nanomaterials have been identified as promising options for environmental remediation, including nanomembranes, nanocatalysts, and nano-adsorbents [27, 28].

3.2 Nano-adsorbents

Nanoparticles are significantly utilized as viable adsorbents because of their larger surface area and comparatively reduced mass. Varieties of nano-adsorbents are used, including manganese oxide, activated carbon, graphene, titanium and ferric oxides, carbon nanotubes (CNTs), and activated carbon [29]. Nanomaterials have dynamic properties due to their innate surface and varied external functionalization. The nanoparticles have proven to be perfect adsorbents because of their inherent composition, extrinsic structure, and apparent size. Various characteristics may have an impact on the nanomaterials employed to remove dyes, heavy metals, and other contaminants from the aquatic environment.

It is important to note that the nanomaterials should be nontoxic, have a higher adsorption capacity, be effective even at low concentrations of pollutants, and allow for easy separation of the pollutants from their surface following treatment. Depending on their adsorption capacity, NPs can take many different forms. These include magnetic, metallic, and nanostructured mixed oxide NPs. In recent times, researchers have been in the production and utilization of carbonaceous nanomaterials, such as carbon nanoparticles, carbon nanosheets, and carbon nanoparticles, as adsorbents. It has also been reported that silicon nanomaterials, such as silicon NPs, silicon nanotubes, and silicon nanosheets, are being used as adsorbents for environmental remediation [30].

Aerogels, nanofibers, and nanoclays are among other nano-adsorbents that are used for the removal of heavy metals from aqueous solutions and dye adsorption [31]. The primary factors influencing the characteristics of nano-adsorbents are their morphology, dimensions, agglomeration state, solubility, crystal structure, and chemical compositions. Nanomaterials stand out from conventional adsorbents due to their fine sizes and strong chemical activity. Additionally, the characteristics and efficiencies of the nanomaterials can be improved by modifying them with various chemicals [31].

3.3 Nanocatalysts

Inorganic nanomaterials like semiconductors and metal oxides are gaining significant interest for use as catalysts in wastewater treatments. For use in wastewater reclamation, different types of nanocatalysts have been reported, including electrocatalysts, photocatalysts, and Fenton-based catalysts for chemical oxidation and antibacterial activities [32]. Nanocatalysts are NPs that exhibit a photocatalytic response when exposed to light. They can be used in wastewater to reduce a variety of toxins in a wide range of applications. To breakdown contaminants including heavy metals, detergents, pesticide dyes, and other substances found in wastewater, these photocatalysts are made of semiconductor metal [33].

3.4 Nanomembranes

One of the best techniques for wastewater treatment is nanomembrane filtration processes. The catalytic reactivity, economic viability, high permeability, high efficiency, water quality, fouling resistance, effective disinfection, and ease of design due to the potable filtration plant size make the application of nanomembrane filtration an excellent approach for environmental remediation [34]. Jun et al. [28, 35] reported successful and efficient removal of inorganic and organic contaminants through membrane filtration including dyes and heavy metals from wastewater for a cleaner

environment. A similar investigation on the use of one-dimensional nanomaterials like nanotubes, nanoribbons, and nanofibers in membrane filtration for wastewater treatment was reported by Liu et al. [36].

3.5 Nanotechnology-assisted soil pollution remediation

Another essential component of the environment, needed to sustain life on Earth is the soil which is the only means of food production and plant growth. Maintaining healthy soil is essential for preserving environmental balance and protecting agricultural productivity. The world is faced with the increasing challenge of environmental deforestation, soil erosion, urbanization, etc., resulting in soil contamination and degradation which need urgent attention [37]. This soil contamination is also caused by the presence of a variety of chemical contaminants in the soil. Approximately 30% of the global land has been deteriorated or contaminated due to human factors and the rate of deterioration increases by the day as urbanization increases [38]. Therefore, environmental remediation is urgently needed for healthy living. Nanotechnology holds enormous potential for use in soil remediation, similar to the treatment and recovery of water and wastewater. Several nanomaterials like CNTs, nano-zero-valent iron (nZVI), and titanium dioxide (TiO_2) have been reported as excellent nanomaterials for environmental soil remediation. The success history of nanotechnology-based techniques is attributed to their adjustable nature and the unique properties of certain contaminants, and this makes them far more suited for soil remediation [39].

Nano-zero-valent iron has been reported as an excellent nanomaterial for the reduction of Zn, Pb, Cr, and Cd in alloy-contaminated soil alongside sustainable stabilizers like carboxymethyl cellulose (CMC) and starch as soil remediation [40, 41]. Nanocomposites are excellent materials for arsenic removal from contaminated soil [42]. However, the combination of biochar with nZVI has proven to successfully remove 100% Cr [43]. By raising the pH of the soil, NPs with biochar increases soil fertility, which in turn promotes plant development and inhibits excessive iron release into the environment [44].

The release of Hg and Cr from the soil is significantly impacted by the use of water-treatment residual plant nanoparticles for soil remediation. Additionally, it was discovered that Na-zeolite nanomaterial was an effective agent for the adsorption of Cd from soil [45]. According to Ghrair et al. [45], NPs greatly reduced the amount of different hazardous substances discharged by various kinds of pesticides into the soil. Pesticide breakdown in the soil has been seen to occur through the process of photocatalysis. The nanoparticles undergo a reaction process where chemical pollutants are converted to harmless substances. The chemical pollution response in this case changes the compounds in the dangerous pesticides into safe pollutants. According to Fujishima et al. [46], titanium dioxide and zinc oxide are regarded as effective soil remediation photocatalysts. Soil quality is improved by the use of nanomaterials-based sensors or nanosensors resulting in increased crop productivity. In industrial units, nanosensors identify and monitor hazardous pollutants, herbicides, etc. Both the detection of pesticide residue and the wide range of soil quality can be greatly enhanced by nanosensors.

3.6 Air pollution remediation through nanotechnology

Air pollution is a global challenge involving dust, heavy metals, gases, and other particles due to various human-caused factors. Air pollution remediation and control

are extremely important and are seen as modern-day necessities. Accordingly, methods based on nanotechnology are essential for the discovery, treatment and control/prevention of air pollution [47]. These methods include the use of nano-adsorbents for the adsorption of air pollutants, nanocatalysts to degrade contaminants, and nanomembranes for pollutant separation. Several dimensional nano-adsorbents, including carbon nanotubes (CNTs), fullerene, graphite, and graphene have been reported for use as air pollution remediation [48].

Carbon dioxide and other greenhouse gases are constantly emitted into the atmosphere in a variety of ways, and they have harmful impacts on the ecosystem. Nanotechnology-based approaches have proven excellent and easy solutions to air pollution remediation. According to Ren et al. [49], nano-adsorbents have a high degree of hydrophobic, hydrogen bond, pie-pie, electrostatic, and van der Waals forces with organic molecules. The cylindrical pores possessed by CNTs make them excellent adsorbents over activated carbon. According to Shen et al. [50], titanium oxide nanoparticles (NPs) possess a self-cleaning coating that helps remove gaseous pollutants and other air pollutants including nitrogen oxides. Nanofiber-coated filter medium has proven an excellent air purifier for industrial plant air intake and turbine inlet air [51]. The identification of different air pollutants in an environment is a critical first step in the control of air pollution. When compared to nanosensors, the traditional techniques (chemical, biological, and physical) are much more expensive, time-consuming, and labor-intensive. Besides, it has been shown that these nanosensors can identify many hazardous and toxic substances at ppm and ppb levels [52, 53].

3.7 Sources of nanoparticles for environmental remediation

The use of nanomaterials for environmental remediation faces the key challenges of high elimination and transformation capability of the set compounds, adequate cost, smooth material synthesis, and sustainability. These challenges need adequate care to be controlled and overcome for successful environmental remediation applications. Based on particle morphology and physicochemical properties, NPs can be grouped into various categories like nano zero-valent iron, ceramic, metal, semiconductor, polymer, lipid and carbon-based, dendrimers, and composites [54]. Nanomaterials have been developed from a variety of materials from various sources. These nanoparticles can be categorized according to the materials from which they originate, including carbon-based, polymer-based, microbe-based (biogenic nanomaterials), and plant-based (phytogenic nanomaterials). In this chapter, we will focus on the following seven major types of nanoparticles, nano zero-valent iron, phytogenic-based, biogenic-based, carbon-based, polymer-based, metal-based, and silica-based nanoparticles (**Figure 1**).

3.7.1 Nano zero-valent iron

It has been demonstrated that a broad variety of contaminants, including metals, polychlorinated biphenyls, chlorinated insecticides, nitroaromatic compounds, and nitrates, can be effectively degraded by nano-zero-valent iron (nZVI) [55]. Since nZVI typically has a diameter of less than 100 nm, *in situ* remediation can be carried out by injecting the material into the soil. The nucleus of zero-valent or metallic iron is contained in the core-shell structure generated by H_2O and dissolved O_2, while the iron oxides and hydroxides created by oxidizing nZVI make up the shell [56]. By making use of its reducing characteristics, nZVI can be made from natural resources

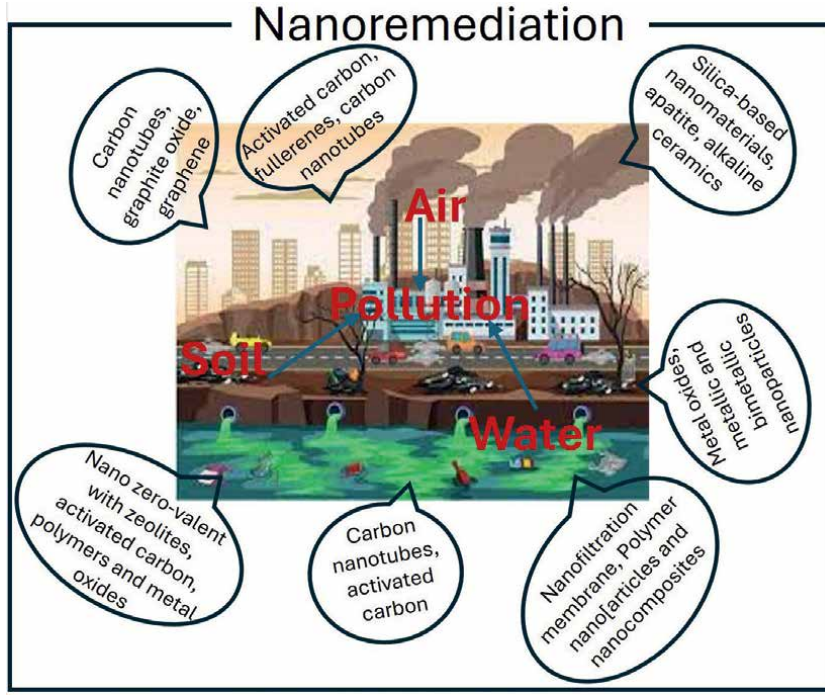


Figure 1.
Schematic elucidation of environmental remediation using various nanomaterials.

including extracts from tree leaves, green mango husks, and food industry residues. In this approach, antioxidant extracts combine with Fe^{3+} and Fe^{2+} to generate nZVI. Emulsified vegetable oil droplet encapsulation is an additional technique for stabilizing nZVI [57]. **Figure 2** shows the core-shell model of zero-valent iron nanoparticles and their reactivity with various pollutants. Research into enhancing the reactivity for pollutant degradation through the addition of a secondary metal, like Ag, Cu, Ni, and Pd, to the nZVI structure to generate bimetallic particles has also been investigated. Metals create a galvanic pair when they are deposited on nZVI, which promotes electron transmission from the Fe core to the outside surface. Furthermore, the secondary metal catalyzes the degradation reaction in the presence of contaminants that have been chlorinated [59].

3.7.2 Phytogenic-based nanoparticles

Plant materials like leaves, roots, shoots, or other plant parts, are used to synthesize phytogenic (plant) nanomaterials for environmental remediation. Due to their unique properties and environmentally friendly production processes that require no additional energy (high temperature and pressure), these nanomaterials are gaining significant attention for various engineering and industrial applications. **Figure 3** illustrates the method of synthesizing plant-based nanomaterials. The outstanding properties exhibited by plant extract, including several functional groups and chemical substances like proteins, polysaccharides, and enzymes make them a good candidate as a stabilizing and/or capping agent [60]. The chemical constituents present in each plant material determine the morphology of the synthesized nanomaterials [27].

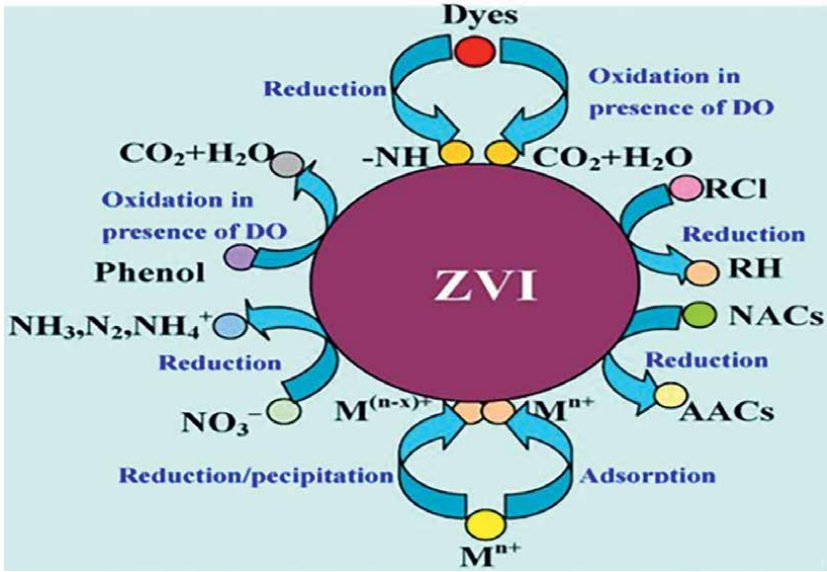


Figure 2.
The core-shell model of zero-valent iron nanoparticles and reaction mechanisms with various contaminants [58].

The functional groups contained in plant-based nanomaterials make it easy to synthesize at a minimal time compared to other nanomaterials sources like biogenic-based nanomaterials. Finding appropriate plant species for the synthesis of nanomaterials for particular targeted contaminants is a crucial step to optimal results. The use of plant-based nanomaterials for environmental remediation like water and wastewater treatment is rapidly gaining significant attention as a result of its ease of fabrication, environmentally friendliness, quick removal efficiency and cost-effectiveness. Tree ferns come in various varieties and can be purchased for a low price anywhere in the world. Plant extracts, on the other hand, are ideal nanomaterials because they contain a variety of functional groups, including carboxyl, hydroxyls, aminos, polyphenols, carbonyls, and aromatics. The general manufacturing procedure of phytogenic nanoparticles is shown in **Figure 3**. In addition to using a photocatalyst to aid in environmental remediation, the green phytogenic adsorbents essentially eliminate the contaminants through polar ion exchange [12].

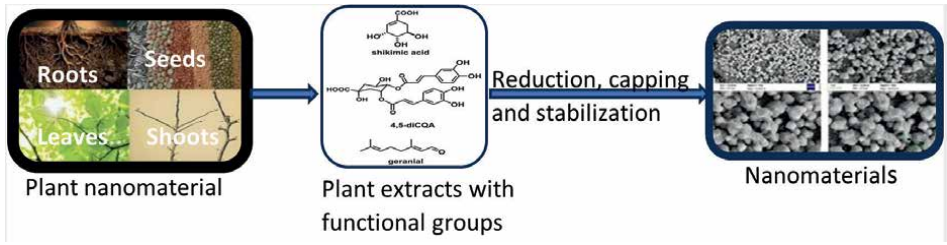


Figure 3.
Plant nanomaterials synthesis.

3.7.3 Biogenic-based nanoparticles

Bioprocesses using bacterial cells are employed for metal ions to solubilization through oxidation and/or reduction. The use of bacterial cells as nano-factories to produce biogenic nanomaterials is gaining attention from several researchers due to its unique biotransformation properties. Sahu et al. [61] reported the use of bioleaching, biomineralization and bioaccumulation processes for solubilization of metal ions. Through biomineralization, certain bacteria's innate defense mechanisms against naturally soluble metal ions enable them to form bio-nanocomposites [16, 62]. Various microbes are more easily manipulated, handled and separated, and therefore considered better candidates for intracellular and/or extracellular biosynthesis of bionanomaterials with great potential for environmental remediation. The synthesis of bacterial nanomaterials takes longer time than the chemical or physical processes. However, combining it with microwave and ultrasonic technology will help speed up the process. **Figure 4** elucidates the schematic presentation of the biogenic nanomaterials synthesis process.

3.7.4 Nanoparticles based on carbon

Many carbon-based nanomaterial types are employed for environmental cleanup, depending on their shape. Thus, the nanomaterials are called zero-dimensional (0D) nanomaterials when all three dimensions are less than 100 nm, such as fullerene and quantum dots [64], or 1-dimensional (1D) nanomaterials when only one dimension is greater than 100 nm but the other two are less than 100 nm, such as carbon and titanium nanotubes [65]. Furthermore, a nanomaterial is referred to as 2-dimensional (2D) when its two dimensions, such as those of graphene, are greater than 100 nm. Graphite nanocomposite is another type of materials which have all three dimensions greater than 100 nm and these materials are referred to as 3-dimensional (3D) [66]. Because of its high surface volume ratio, hydrophobic nature and strong electron affinity, fullerenes are considered an ideal material for environmental remediation. According to Gokhale and Somani [67], they have a wide range of applications, including surface coating, semiconductors, solar, harmful bacteria eradication, and water treatment [68]. The unique properties of carbon nanotubes, including larger surface area, porous structure, ease of surface functionalization, and adjustable nanosize, makes different dimensional carbon nanotubes (CNTs) extensively used in many pollution control applications with great promise in other environmental remediation.

Additionally, because of their increased surface area resulting from multiple graphene layers, CNTs have demonstrated excellent environmental remediation materials [68]. Wu et al. [69] reported a study investigating graphene and its modified form as an environmental remediation material for various pollutants. The authors suggested that graphene effectively adsorbed fluoride from aqueous solutions at a higher adsorption capacity of 35.59 mg/g. According to An and Yu [70], pristine graphene and its modified form have been deemed a promising approach for the remediation of other substances in the environment as their study demonstrated how pristine graphene was effectively used to remove fluoride from water. Several researchers have reported graphene oxide (GO) as a promising nanomaterial for environmental remediations due to its ability to adsorb various gaseous and volatile organic compounds (VOCs) such

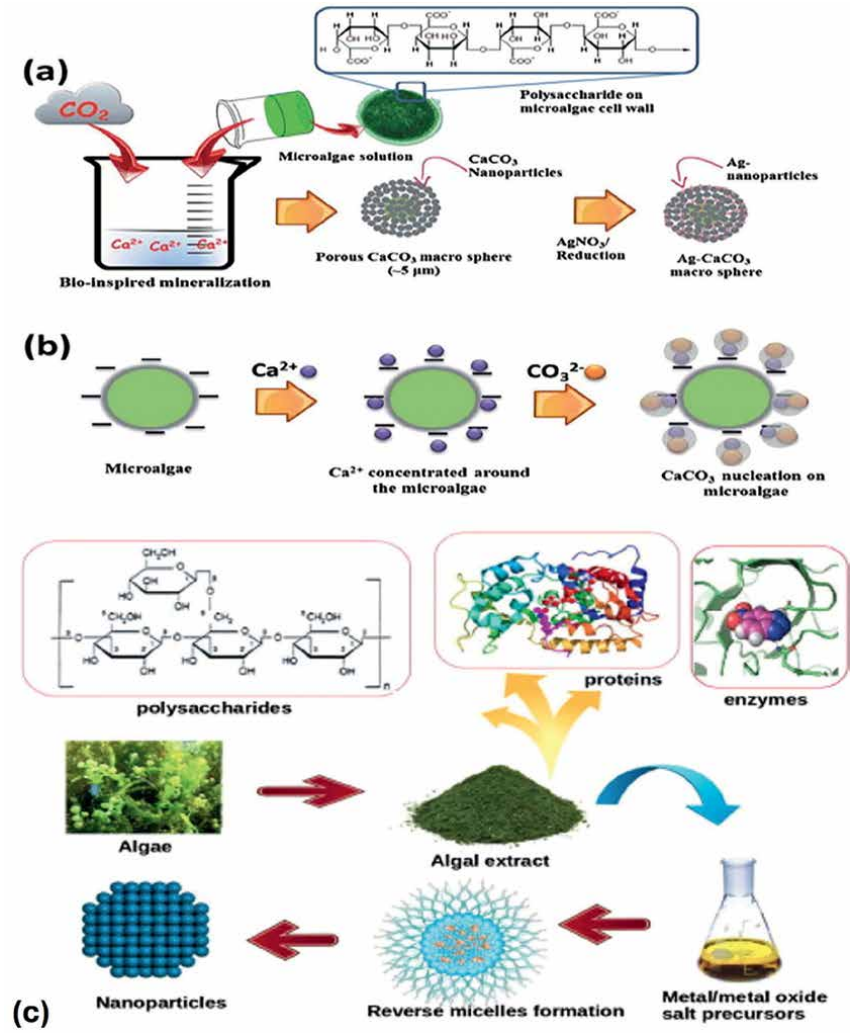


Figure 4.
(a) Schematic illustration of the synthetic steps for porous $CaCO_3$ microsphere by CO_2 mineralization pathway in the presence of microalgae. (b) Mechanism for the surface-directed mineralization of $CaCO_3$ and (c) biosynthesis of NPs with algae [63].

as hydrogen sulfide and sulfur dioxide and water-based compounds, such as heavy metals, pesticides, and pollutants from pharmaceuticals [71, 72]. The GO's surface has a variety of functional groups, including hydroxyl, carboxylic acids, and epoxy, which make them effective adsorbents for a wide range of environmental contaminants.

Although GO has demonstrated an effective material in adsorbing cationic metals. However, additional modification with organic or metal oxides could boost its capacity in anionic metal adsorption. According to Zhang et al. [15], titanium oxide and graphene nanocomposites have demonstrated great potential in adsorbing benzene from the environment. Nonetheless, there is a strong correlation between the material's photolytic activity and the ratio of titanium oxide to graphene. The composite loses efficiency after a certain graphene ratio [73]. Moreover, it has been found that ZnO- and CdS-graphene work well as adsorbents to remove heavy metals from water [74].

Modifying graphene with various metal oxides can improve their stability and efficiency in degrading contaminants from aqueous solutions or other substances for a cleaner environment. CNTs, on the other hand, are a different kind of carbon-based nanotechnology that is currently extensively utilized for environmental cleanup. According to Lithoxoos et al. [75], open-end CNTs have primarily been created to increase adsorption capacity. CNTs are typically bonded into a hexagonal configuration using porous and heterogeneous tube bundles. The adsorption on the nanotube surface reaches equilibrium before it does on the tube's interior surface [49]. Closed-end CNTs have more binding sites, and as such they can be more effective as an adsorbent for environmental remediation. With the appropriate modifications, carbon-based nanomaterials offer a lot of potential applications in environmental remediation.

3.7.5 Nanoparticles based on polymeric materials

Polymeric nanomaterials serve to improve materials' stability with enhanced performance for effective environmental remediation applications. The use of these polymeric materials is an excellent alternative for increasing the materials' durability and aiding the host materials to achieve a targeted specific purpose. Numerous polymer-based nanomaterials, including amphiphilic polyurethane NPs, polyamidoamine (PAMAM) dendrimers, polyamine-modified cellulose volatile organic compounds (VOCs), and polymer nanocomposites (metal ions, microorganisms), have been reported to be used in the remediation of contaminants [17]. According to reports, amphiphilic polyurethane nanoparticles (NPs) can eliminate up to 80% of phenanthrene while also increasing soil mobility [76]. However, PAMAM dendrimers were reported to be used in the treatment of wastewater because of their functional groups, which include carboxylates, hydroxamates, and amines, among others making it easier for a variety of cations to be encapsulated [77]. According to reports, polymer-based nanomaterials can also lower various forms of heavy metals from soil and water, as well as gaseous pollutants [78]. Although these nanomaterials have been used for environmental cleanup, there are significant drawbacks because of their non-biodegradable nature, which might result in the creation of new contaminants [17].

3.7.6 Nanoparticles based on metals

Because of the excellent performance properties of metal oxide nanoparticles, such as photolytic properties, high reactivity, and adsorbent properties resulting from their affinity to various chemical groups and enormous surface area, titanium dioxide (TiO_2), zinc oxide (ZnO), and iron oxide (Fe_2O_3/Fe_3O_4) are being used for water purification [79]. For example, iron nanoparticles have been used to remove colours in wastewater from the textile, paint, and paper industries due to their high adsorption capacity and stability in suspension media. According to Mashkoor and Nasar [80], these nanoparticles have demonstrated remarkable efficacy in the adsorption of dyes, including methylene blue and methyl orange. These dyes are widely used in industry and have been found to have adverse impacts on both the environment and human health. The effectiveness of magnetic iron oxide NPs in removing phenol and methyl orange when combined with carbon has been studied [81]. The results show that the nanocomposites have stronger interactions with the dye and carbon concentration, a key factor in the adsorbent behaviour of the NPs. Heavy metals, such as chromium (VI), are another important class of water pollution in addition to dyes. According

to recent studies, organic acids (like citric acid) and iron oxide or zero-valent iron nanoparticles may reduce the environmental danger of chromium (VI) [82, 83]. Titanium dioxide nanoparticles (NPs) are commonly utilized as photocatalysts in the water treatment industry to remove micropollutants. They are a viable substitute for developing contaminants like pharmaceuticals.

3.7.7 Silica-based nanoparticles

Mesoporous silica nanoparticles feature a large pore volume, adjustable pore size, and increased surface area. Their adaptable characteristics set them apart in environmental applications as catalysts and adsorbents [84]. There have been reports on the use of these silica nanoparticles for gaseous phase pollutant elimination [85]. Nonetheless, reports for the removal of gaseous pollutants (CO_2, H_2S, aldehydes, and ketones) also mention amine-modified xerogels, aluminosilicates, and porous silica as good materials for environmental remediation [17]. Additionally, it has been reported that mesoporous silica functionalized with thiol, amino acid, and carboxylic acid can remove heavy metal pollutants and dyes from water [17]. Because of the presence of hydroxyl groups on their surface, silica materials are easily modifiable to alter their surface phenomena.

Grafting of functional groups onto the pore walls is an excellent technical strategy for designing novel materials, such as catalysts and adsorbents for a cleaner environment. According to Huang et al. [86], amine surface-modified silica xerogels and mesoporous silica are effective materials that can remove up to 80% CO_2 in 30 minutes, while 80% H_2S adsorption takes 35 minutes. Reversible adsorption has been reported to be used by amine-modified aluminosilicates to absorb carbon dioxide and other carbonyl chemicals [87]. This substance is far more effective than the other costly silica-supported compounds which may be a simple and easy substitute [88]. Furthermore, mesoporous silica with carboxyl functionalization can behave as dynamic adsorbents for dyes and heavy metals in water.

4. Nanomaterials green synthesis approaches for environmental remediation

Chemical synthesis procedures have often been the key to preparing nanomaterials specially designed for efficient environmental remediation. The techniques are based on chemical reactions that allow for the transformation of starting materials into nanoparticles with desired properties for the reduction of environmental pollutants. These techniques are highly useful and versatile including some sol-gel methods and microemulsion methods which are topped with success rate.

4.1 Chemical reduction

Among these synthesizing methods, the chemical reduction processes are successfully used for the preparation of metallic nanoparticles (like nano-zero-valent iron [nZVI]) which shows high capability to remediate contaminated water and soil [1]. This method consists of the reduction of metal salts (e.g., ferric chloride for iron nanoparticles) with reducing agents such as sodium borohydride, hydrazine, or ascorbic acid. By adding the reducing agent, the process of reduction to convert metal ions to nanoparticles takes place followed by precipitate from the solution due to reduction

in electrons redistribution. The simplicity and effectiveness of chemical reduction is one reason that we have limited our attention to this method for large-scale applications. Nevertheless, obtaining a uniform size distribution could be problematic and particles will tend to agglomerate meaning that some capping agents are added to prevent this and stabilize the nanoparticles.

$$FeCl_3 + 3NaBH_4 + 9H_2O \rightarrow Fe(s) + 3NaCl + 3H_3BO_3 + 10.5H_2 \quad (1)$$

$$Fe^{3+} + 3e^- \rightarrow Fe(s) \quad (2)$$

4.2 Sol-gel method

Sol-gel is another widely used technology for producing nanomaterials, particularly metal oxides (silicon dioxide [SiO_2], zinc oxide [ZnO], and titanium dioxide [TiO_2]). It is a process that uses polycondensation and hydrolysis to change a sol (solution) into a gel (solid network). The precursors used are generally metal alkoxides or chlorides that hydrolyze to form a colloidal suspension. More condensation reactions followed, resulting in a gelled network. Afterward, the gel is dried and calcined to obtain nearly pure and uniform nanomaterials. Low-cost and environmentally beneficial applications of nanotechnologies have consequently been the primary focus of the sol-gel method due to it enabling controlled preparation (in terms of composition, size, and shape) and facilitation toward modifying surface properties in optimized state for effective remediation materials preparation. However, the process is relatively slow (time-consuming) and necessitates control of reaction conditions for optimal results.

4.3 Microemulsion method

The combination of surfactant, water, and oil mixture in the microemulsion approach produces a stable emulsion containing nanoscale droplet that acts as nanoreactors for the synthesis of nanoparticles [89]. In this strategy, the precursor materials are dissolved within a water/oil phase, and surfactants stabilize the interface to produce the emulsion. These nano-droplets serve as vessels for chemical reactions, such as reduction or precipitation, which result in the creation of nanoparticles. The method of microemulsion is established for a sophisticated control of particle size and morphology because of their consistent droplet sizes. This method applies to a large variety of nanomaterials in the forms of metals, oxides, and sulfides. The use of surfactants may, however, complicate the purification procedure, though, and not all nanoparticle types are suitable for using this technique.

The particle morphology allows the possibility to remove contaminants from water and clean up a polluted environment, which makes chemical reduction, sol-gel method, and microemulsion technique sustainable for the fabrication of nanomaterials for specific applications on environmental remediation [90, 91]. These approaches have their own peculiar merits such as being straightforward and effective in chemical reduction, good purity level, and manipulation of the size via sol-gel route or particle dimensions using the microemulsion method. While these offer advantages, they come with their downfalls such as the requirement of stabilizers and precise reaction control for purification steps. This knowledge is indispensable for the design of nanomaterials to remediate a broad range of releases that could make ecosystems dirty and unhealthy.

5. Potential environmental risks with the use of nanomaterials in environmental remediation and recommendations for mitigation and safe responsible use

5.1 Potential risks

The technology of nanomaterials is highly desirable for use in environmental remediation because of their high reactivity and owing to properties, such as large surface area, unique side effects, and the possibility of modification routes. However, the use of nanomaterials also introduces new environmental risks that must be effectively characterized and mitigated.

5.1.1 Effect of nanomaterials' toxicity on aquatic life

The first and main point is the possible toxicity at low concentrations of nanomaterials to aquatic and terrestrial organisms. Because they are small and reactive, these tiny particles can produce noxious effects; making contact with biological membranes and cellular elements more directly than harmless larger particles. As an example, in the case of nano zero-valent iron (nZVI) particles widely used as sorbents for contaminant degradation, reaction oxygen species (ROS) can be formed and cause oxidative stress to aquatic organisms [55]. Research has demonstrated that, at certain concentrations, silver nanoparticles which are commonly employed for their antibacterial qualities can be harmful to fish, crustaceans, and algae [92, 93]. Oxidative stress could be harmful to the growth, reproduction, and survival of aquatic life. Likewise, in soil, nanoparticle exposure can also affect the functions of microorganisms and plants responsible for nutrient cycles leading to a reduction or increase in plant growth (**Figure 5**) [94].

5.1.2 Bioaccumulation and biomagnification

Bioaccumulation and biomagnification are two other factors that could make nanomaterials highly hazardous. This could lead to high risks for humans, aquatic organisms and the entire ecosystem through food chains. Primary producers like algae

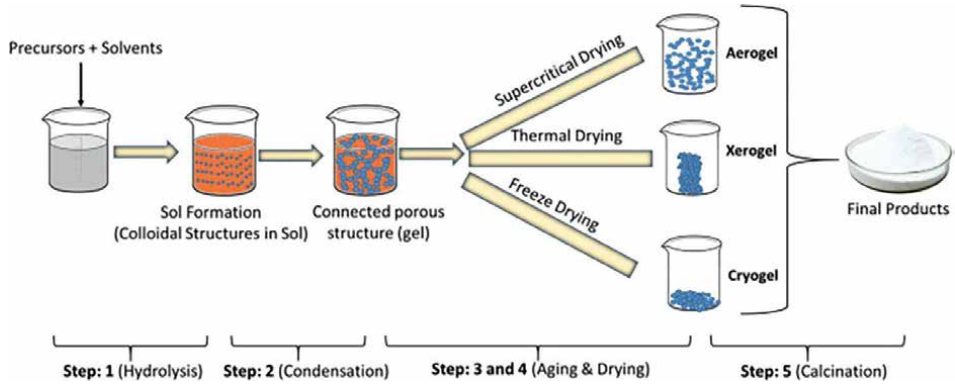


Figure 5.
Schematic elucidation of sol-gel method of metal oxide nanoparticles synthesis [94].

and plants can ingest nanoparticles, which are later on ingested by herbivores through the food chain to higher trophic levels [95]. This accumulation can allow a higher level of nanoparticles to trickle down through the food chain, potentially impacting human health and threatening ecosystem stability. It is, for example, known that silver nanoparticles can build up in fish tissues and hence raise concerns regarding their fate of action over the long-term effects on the overall health of fish and the predatory animals that eat them.

5.1.3 Persistence and mobility of the environment

Nanomaterials are also at risk of being persistent and mobile in soils and water bodies [96]. Nanoparticles, once exposed to the environment, can be extremely stable so that they may stay there for a long time or suffer considerable displacement from their point of origin. Small soil particles can move through soil and water systems faster than larger-sized particles, resulting in the potential of reaching remote or sensitive ecosystems with contaminating elements. For instance, water treatment nanoparticles can escape from the facilities that are intended to use them and perhaps impact aquatic ecosystems of natural bodies of such things as water. Nanomaterials also exhibit poor long-term stability, and the metabolic products of their degradation are generally poorly understood, raising questions concerning their fate in the environment. Although the reactivity of nanomaterials is significant for remediation, it can also be a problem if they are involved through side reactions with other species.

5.1.4 Possible interactions and byproducts

Interactions between nanomaterials and other environmental elements may occur, resulting in unexpected outcomes. For instance, there are unknown reactions that nanoparticles can have with metals, organic materials found in nature, and other contaminants. These reactions could result in the creation of new, potentially more hazardous byproducts [97]. Moreover, the physicochemical characteristics of pollutants may change as a result of the use of nanomaterials in remediation, influencing their toxicity and bioavailability.

5.2 Risk mitigation for safe responsible use

A multimodal approach is necessary to ensure the safe and responsible use of nanotechnology in remedial applications while also mitigating the environmental concerns connected with it. This strategy ought to encompass driving scientific and technology advancements, supporting regulatory structures, strengthening public outreach, engagement, awareness, and applying technological remedies [98–102]. To reduce the environmental concerns associated with nanotechnology in remediation applications, a proactive strategy and thoroughness are needed. Safe and ethical use of nanotechnology will be guaranteed by supporting and employing regulatory frameworks, expanding focused research, advancing creative and practice technological solutions, and raising public awareness. These will harness the benefits of nanomaterials while protecting the environment and public health if there is a strike in the balance between their enormous potential and aggressive risk management techniques. The sustainable development and use of nanotechnology in environmental remediation are contingent upon this integrated approach.

6. Conclusion and future direction

6.1 Conclusion

Utilizing nanotechnology to address some of the most difficult problems in the world of environmental remediation is gaining significant interest among researchers. Because of their large surface area, reactivity, and the possibility to engineer them at atomic scale; nanomaterials were well suited for pollution removal from water, soil, or air. This technology makes the design of more efficient, cheaper, and sustainable remediation methods possible compared to traditional techniques.

The targeting of some particular pollutants with high accuracy is one of the significant benefits that nanotechnology provides for environmental remediation. For example, nanoparticles like nano zero-valent iron (nZVI) can degrade/transform toxic heavy metals and organic contaminants in groundwater to a less harmful state. The above examples clearly show the promise of nanotechnology not only in cleaning existing pollution but also in preventing new environmental hazards by using improved waste treatment and more effective methods for controlling toxic substances.

The scalable and interchangeable websites of nanotechnology are what make it so valuable for use in supporting every part of the environment. Nanomaterials are highly versatile materials in that they can be designed to prevent and neutralize contaminant availability, no matter if applied *in situ* or *ex-situ* environments. An improved version of nanotechnology, combined with other new technologies such as biotech agencies and information technology (IT), may improve further efficacy in use. An interdisciplinary solution is necessary for an all-encompassing development in addressing complicated environmental issues that will lead to a cleaner and healthier planet.

Although the promise of nanotechnology is vast, it also introduces risks and challenges. The health and environmental effects of nanomaterials must be thoroughly evaluated to verify that their utilization is safe. The regulatory frameworks need to follow the changes in technological developments, guaranteeing that nanotechnology deployment for environmental remediation is technically effective and not harmful. Public awareness and acceptance of public perceptions of risks associated with nanotechnology are just as important for determining its overall impact on society as public knowledge.

Nanotechnology serves as a sustainable and powerful delivery system for environmental remediation. The fact that it can increase pollution control and measure both its efficiency and effectiveness makes climate change an important driver of future environmental sustainability. Through furthering research, tackling safety concerns, and promoting interdisciplinary collaboration, nanotechnology will become a key asset in restoring our planet to its natural state so that all future life may flourish.

6.2 Future directions

The creation of sophisticated nanomaterials intended for targeted remediation is one of the most growing developments in nanotechnology for environmental remediation. These substances, which include magnetic nanomaterials, nanocatalysts, and functionalized nanoparticles, are designed to bind and breakdown particular pollutants only. For example, highly efficient and selective nanoparticles functionalized with ligands or polymers can be used to absorb organic contaminants or heavy metals.

By utilizing external magnetic fields, pollutants can be easily separated from the environment through external magnetic fields. By using less energy and chemicals, this strategy not only improves the efficacy of remediation measures but also minimizes their negative environmental impacts.

The exploration of biogenic synthesis that creates nanoparticles from fungi, bacteria, and plants is gaining wider awareness with much interest due to their environmental friendliness. More interest is also focused on biodegradable nanomaterials, which, once their remedial purpose is fulfilled, can breakdown into nontoxic byproducts. These strategies seek to advance a circular economy and lessen the negative impacts of nanotechnology uses on the environment.

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Chapter 8

Perspective Chapter: Functionalization of Nanofibers – Applications in Food Technology

Ayomipo M. Ojo, Oluwatoyin I. Omotunde and Joseph Otugboyega

Abstract

The potentials of nanofiber in food technology cannot be over-emphasized. Nanomaterials have become a versatile resource of multiple applications. The various adaptability of nanofibers is attributable to its special qualities including controlled shape, high porosity, large surface area, high encapsulation efficiency, and high chemical and thermal stability. Functionalization adds more value to a nanofiber through the introduction of additional moieties to it. Food technology is a wide field of research due to its importance in the society. In recent years, new measures are being introduced to produce new smart food resources. This chapter provides a comprehensive overview of how nanofibers are being prepared, functionalized, and applied within the realm of food technology. The various techniques and reagents used for nanofiber functionalization suitable for food technology are discussed. The advantages inherent in functionalized nanofibers are also elucidated. The chapter closes with future recommendations on functionalized nanofibers for food technology.

Keywords: nanofiber, functionalisation, encapsulation, bio active compounds, food packaging

1. Introduction

Nanotechnology is a field of research in science and technology that deals with small-sized particles with a size of approximately 1 to 100 nm at least in one dimension [1]. Nanotechnology is a fast-rising area of research building on the unique properties of materials in the nano-range. Nanotechnology has fast-gaining importance in food technology, and this has been on the rise for some time now. This area of research engineered the production and synthesis of nanomaterials. These materials have great potential in food technology. Its utmost importance has birthed unique innovations in food quality monitoring, food packaging, and encapsulation and sensory devices for food monitoring [2].

The demand for high-quality functional materials with various applications keeps increasing. New and smart materials are being synthesized to replace oil-based materials that are expensive to come by and non-biodegradable [2]. This has led to the fabrication of various materials that are green, inexpensive, and environmentally friendly. Miniaturization of large molecule materials has resulted in nanodimension materials with superior modifiable material shapes, surfaces, characteristics, and functions [3]. Nanotechnology has found applications in almost all areas of human endeavors, namely, electronics, energy, medicine, environment, textile, and food. Research studies on these noble materials have led to many discoveries. It has opened a new field of research that has addressed so many challenges. It has greatly contributed to the human race in terms of health, welfare, and economy.

The increasing world population necessitates greater research to produce good quality and durable foods. This is being addressed through various technologies utilizing nanofibers for crop enhancement, food preservation, and security. Nanotechnology has led to the production of nanotubes, nanoparticles, nanofibers, nanosheets, and nanowhiskers with various application in food biotechnology, science, processing and packaging, development of functional foods, food safety, pathogen detection in food, and prolonged food and food product shelf lives [4].

The field of food nanotechnology involves the utilization of nanocarrier methodologies to enhance the bioactive components' biological accessibility and create a barrier against various chemical or environmental changes [5].

The goal of this chapter is to describe nanofibers and its applications in food industry with focus on its food packaging potentials. Various functionalization processes and encapsulation of active ingredients with nanofiber were also elucidated before capping it up with edible nanofiber for food sustainability.

1.1 Nanofibers and their applications

Nanofibers are classified as fibers having diameter in the range of 1–100 nm [6]. Nanomaterials are one-dimensional materials with diameter less than 100 nm. They are ultrafine fibers with diameters in the nanometer range [7]. According to Harish et al., 2022, "Nanofibers are flexible one-dimensional nanomaterial (1D-NMs) with a diameter of 100 nm or less" [8]. As the fiber diameters is being shrunk from the micro- to nanoscale, important properties are obtained making it a high-valued component. Research studies has shown that materials undergo a significant transformation in their characteristics as they go from bulk to nanoscale as well as almost atomic level [9]. Excellent surface area to volume ratio, high aspect ratio, high porosity, superior surface properties, quantum confinement effects, high mechanical strength, and flexibility in functionalization are the unique properties that make nanofibers adaptable to wide applications. The various nanostructured fibers which include hollow, mesoporous, core-shell, and surface-decorated fibers possess these distinctive characteristics which makes it appropriate for various fields. This also makes nanofibers better than other materials lacking these qualities. These materials can be generated from various polymer sources (natural and synthetic). Natural sources of nanofibers include collagen, cellulose, and polysaccharides, while synthetic sources include metals, poly(lactic acid) (PLA), polycaprolactone (PCL), and polyurethane (PU) [10]. The origin of the nanofibers dictates their different physical properties and application potentials. Nanofibers derived from natural sources are ecofriendly and are appropriate for food and water applications.

The applications of nanofibers spread across various fields, such as biomedicine [11], filtration of water/wastewater [12], textiles [13], additive manufacturing applications (Adapa and Jagadish, 2023), the automotive industry [14], the construction industry [15], thermal structure engineering (Sekhar et al. [17]), harvesting, printing technology [16], storage of energy [17], and food technology [18].

In addition to this, nanotechnology has been successfully incorporated into air filtration devices [19], wastewater treatment [20], tissue engineering [21], microsensors [22], fuel cells [23], lithium-ion batteries [24], photocatalytics [25], and nanostructured piezoelectric materials [26]. Thus, nanofiber through its numerous properties has gained importance in industry and academia. Diverse preparation methods being exploited for the synthesis of nanofibers are drawing, template synthesis, phase separation, self-assembly, and electrospinning [27]. Electrospinning has shown great potential in the synthesis of structural nanofibers, including mesoporous, core-shell, hollow, multicomponent, and surface-decorated nanofibers to mention a few [28].

Nanofiber dated back pre-historic era. The phenomenon of silk worm and spider web led human to the discovery and study of nanofibers [29]. The spindle was developed in the fourteenth century to prepare fibers from materials such as wool and cotton, and it became widely utilized in the textile industry in about 1880 [30]. Nanofibers has been utilized for a long time before its study became popular (**Figure 1**).

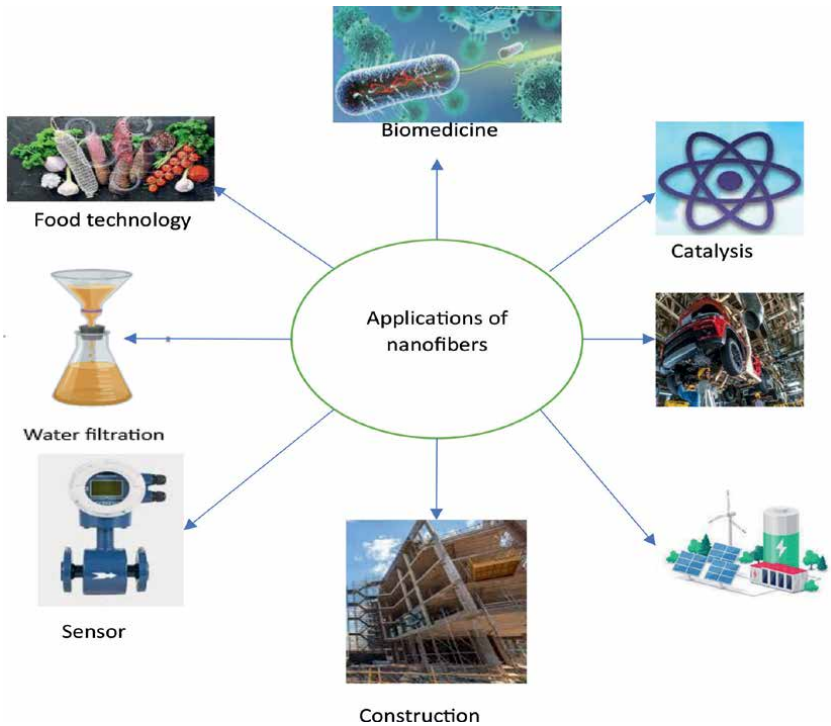


Figure 1.
Applications of nanofibers.

1.2 Sources of nanofibers

1.2.1 Natural sources

1.2.1.1 Cellulose nanofibers

Cellulose is a major component of plant cell walls giving them rigidity and strength. It is a complex polysaccharide consisting of a linear chain of several hundred to many thousands of β(1→4)-linked D-glucose units [31]. Cellulose has drawn great awareness in recent years due to its sustainability and ecofriendly nature. It is derived from green plant biomass. Cellulose can also be derived from other plant biomass among which are algae, acetobacter, and rhizobium [32]. Cellulose sources are renewable and can be assessed easily from plant biomass. The cellulose content of plants varies with cotton, fiber (90%), and wood (40–50%) [32]. Plants make cellulose fibers utilizing highly crystalline entities known as microfibrils. Cellulose nanofibers are obtained through mechanical fibrillation or chemical treatments of wood pulp, cotton, or other cellulose-rich materials [33].

1. *Chitin and Chitosan Nanofibers:* Extracted from the exoskeletons of crustaceans like crabs and shrimp, these nanofibers are produced through processes involving deacetylation and mechanical shearing. Chitin and chitosan are both biocompatible, biodegradable, and non-toxic biopolymers which possess antimicrobial and hydrating characteristics [34]. These properties have geared significant attention toward its utilization in food technology.

2. *Silk Fibroin Nanofibers:* Silk as can be described as fibrous protein is produced by a variety of insects including silkworm [35]. The natural protein known as fibroin gives silkworm silk its distinct physical and chemical characteristics and acts as a filament and support for the silk. There are sticky proteins known as sericin and are responsible for binding and gumming fibroin filaments. This material can be prepared in different forms such as gels, powders, fibers, or membranes, depending on the end use. The major source is from silkworm cocoons. Silk fibroin nanofibers are created through electrospinning or other fiber formation techniques after extracting and dissolving the fibroin protein [36]. SF is a promising resource for divers' applications due to its numerous beneficial properties, such as excellent biocompatibility, good permeability to oxygen and water vapor, biodegradability, and minimal inflammatory response. SF has enjoyed applications in a variety of fields, including cosmetics, medical materials for human health, and recently food additives.

3. *Collagen Nanofibers:* Collagen is a major component of the extracellular matrix contained in tissues of animals. Its proportion varies between 70 and 80% of dermis dry matter [37]. The physicochemical properties of various collagen samples depend on the sources [38]. Collagen obtained from sea animals possesses differing molecular structure to those obtained from domestic and land animals [39]. Collagen, primarily obtained from animal connective tissues, can be processed into nanofibers through electrospinning or self-assembly methods. In order to obtain collagen nanofiber, extracted collagen are sized down to a nanometer scale [40]. Collagen nanofibers has received interest in food industry, nutraceutical, cosmetics, tissue engineering, and food packaging of the last 3 years since introduced [41].

1.2.2 Synthetic sources

1. *Polymer Nanofibers*: Polymer nanofibers are derived mostly from oil-based polymers. Various synthetic polymers such as polyvinyl alcohol (PVA), polyacrylonitrile (PAN), polylactic acid (PLA), and polyurethane (PU) are used to create nanofibers via electrospinning, phase separation, or self-assembly techniques.

2. *Carbon Nanofibers*: Carbon nanofibers are made up of graphene layers stacked together in a cylindrical shape [42]. Derived from the carbonization of polymer precursors like PAN, these nanofibers are produced through electrospinning followed by thermal treatments. Properties of carbon nanofibers, namely, density (1.3–2 g/cm^3), electrical resistivity (1×10^{-3} to 1×10^{-4}), and thermal conductivity (1950–6000 W/mK) bestow outstanding qualities on it, making it relevant for divers' applications [43]. These applications include selective adsorption, polymer reinforcement, electrochemical catalysis, and hydrogen storage [44].

3. *Inorganic Nanofibers*: This includes metal and metal oxides nanofiber. They are created through electrospinning and subsequent calcination, materials like titanium dioxide (TiO_2), zinc oxide (ZnO), and silver nanofibers are synthesized for applications in catalysis, sensing, and antimicrobial coatings [45].

4. *Ceramic Nanofibers*: Ceramic nanofibers are produced from a composite solution of ceramic precursor and a compatible polymer solution of proper viscosity. Ceramic nanofibers possess the functional properties of the precursor ceramics as well as intrinsic properties of nanofibers such as high porosity, low density, high surface area to volume ratio, and high aspect ratio of nanofibers [46].

1.3 Types of nanofibers

Nanofibers can be fabricated in different structures. The structures can be used to classify nanofibers by types. These structures have different morphologies and properties. The shapes of the nanofibers depend on the starting materials and the method of production. By carefully selecting these parameters, the shape, structure, and arrangement of the nanofibers can be monitored to give the desired outcome. Prominent among the different shapes are (i) hollow, (ii) porous, (iii) non porous, and (iv) core shell nanofiber although herringbone, platelet, and ribbon-structured nanofibers has been reported in literature [47]. Arrangement of molecules in the nanofibers occur through non-covalent forces such as such as intra- and inter-hydrogen bonding, hydrophobic forces, and electrostatic forces of attractions [47].

1.3.1 Hollow nanofiber

The high specific surface area offered by inner and outer walls of hollow nanofibers has made it a delight for many applications in the fields of environment, food, energy, sensing, and health. Hollow nanofiber can be fabricated from both organic and inorganic materials such as ceramics, polymer, metal oxide, or their composites. Its prominent property is the porous structure which gives it an edge over other types of nanofibers [48]. Polymer molecular weight viscosity, conductivity, dielectric constant, dipole moment, and surface tension are some of the factors that determine the structure of HNFs [49].

1.3.2 Porous nanofiber

Pores or voids are the major characteristic of porous materials. Any material containing either cavities, channels, or interstices is porous. The size, shape, and arrangement of the pores and composition of the starting material dictates the characteristics of the nanofiber. Divisions of porous materials include microporous, mesoporous, and microporous materials depending on their sizes. Macroporous materials have pore diameters greater than 50 nm, while microporous materials have pore diameters lesser than 2 nm. In between the two ranges is mesoporous materials having pore diameter sizes between 2 and 50 nm. Nonporous nanofibers are plain shaped with no pores. These materials have less surface area compared to porous fibers. Although nanofibers have larger surface area, specialized porosity, more active sites, heterogeneous interfaces, and interior spaces—all critical for its functionality and performance—electrospun porous nanofibers combine the advantages of porous structures with nanofibers [50].

1.3.3 Core shell nanofiber

This is usually prepared from two independent materials with each complementing one another or acting independently. Until it is time for its release, the core remains protected [47]. The core shell nanofiber has notable core and shell component with average diameter varying from 20 to 100 nm. The notable advantage of core shell nanofiber is drawn from its flexible lightweight coupled with high mechanical strength and chemical stability [51]. This type of nanofiber operates by bringing the functional properties onto the surface of the nanofiber (shell) while keeping the intrinsic properties in the core [47].

2. Food industry before and after nanofiber introduction

The world population is increasing every year, and it has been projected to reach between 8.9 and 10.6 billion by 2050 [52]. This increment comes with greater demand in food resources. In order to avoid a looming large-scale food scarcity, new methods and resources needs to be incorporated in food manufacturing and preservation. There is a need to increase the present food production measures by 70% to meet the anticipated food demand in year 2050 [53]. Poor seedings, climatic change, soil degradation, and biodiversity losses are part of the challenges being faced in food production [54, 55]. These constraints have been militating food production over the years. The world's growing population and impending climate change worsened food supply and availability; however, reports suggest that the arable land available will not maintain the weight of these needs in the future years [56, 57]. The losses recorded due to these challenges cannot be overemphasized, and great measures are being devised to control it.

Conventional agrochemicals, such as pesticides and fertilizers, have long been used in traditional farming, leading to numerous detrimental effects on ecosystem and human health [58].

There is great need to optimize the available resources of land and water for maximum benefit. There are pressing need to introduce new food production and preservation measures. Sustainability of available resources to meet the food demand of increasing population is needed in order to avert food crises.

The many benefits of nanotechnology inspired researchers to exploit it in food production. The incorporation of nanotechnology has proven successful in food quality and preservation [59], active ingredient encapsulation [60], improved seedlings [61], effective agrochemical delivery [62] monitoring abiotic/biotic stresses in plants [63], controlling stresses in plants [64], and enhancing the uptake of nutrients from soil [65], all these are geared to enhance food production. Nanotechnology has proved to be a necessary step in modernizing agronomic techniques, resulting in lower losses and higher input efficiency.

Nanotechnology has found application in pre-farming to post-farming and eventually produced food. Nano-orchestrated system has been utilized in food production through cropping systems and animal production systems [66, 67].

Introduction of nanotechnology has increased yield of food products as well as suppressing losses encountered in food production. This has created good economic value for farmers and food processors, thus a great initiative for food production. Efforts has been made to ascertain the safety of nanotechnology in food production so as to safe guard the health of consumers. Various publications on the safety of nanomaterials in *food processing* and packaging with special attention to their reflection on food quality and safety are available [68, 69].

3. Food packaging

Nanofibers has been harnessed greatly in food packaging. These nano scale fibers tend to enhance the mechanical, barrier, and optical properties of packaging. Electrospun fibers has the potentials of regulating the release of functional agents into food products [70]. Incorporating additives into nanofibers allows them to respond to environmental changes. The use of nanofibers leads to better control over food properties. Food packaging technologies based on nanofibers can be used to program the release of active ingredients in a regulated manner [71] The biosafety and bioavailability of packaging materials were enhanced using biopolymer nanofibers. The stand out properties of nanofiber materials are high porosity, low density, good breathability, large specific surface area, and large area of pores [72]. Nanofiber composites have gained relevance leading to the fabrication of materials with wider potentials for applications in industries such as textiles, health care, energy storage, and more.

Food sources, namely, proteins and carbohydrates have been successfully fabricated through electrospinning to nanofibers of exact spacing among fibers, structural stability, and full biocompatibility. In addition, they can easily bind to bioactive food ingredients through interaction with their functional groups leading to entrapment of the active components and subsequent controlled release in the stomach. They are also used to maintain the integrity of food by safe guarding food from oxidation, moisture, and light penetration through food encapsulation. Additionally, nanofiber suppresses the flavor or odor of nutrients, aids in the immobilization of cells and enzymes, releases bioactive substances in a controlled manner, boosts the viability of probiotics, and makes handling them easier, and creates functional foods [73].

Increasing food production and its security must be achieved, and notable technological advancements in the field of food system can only be accomplished by the use of nanotechnology (**Figure 2**) [74].

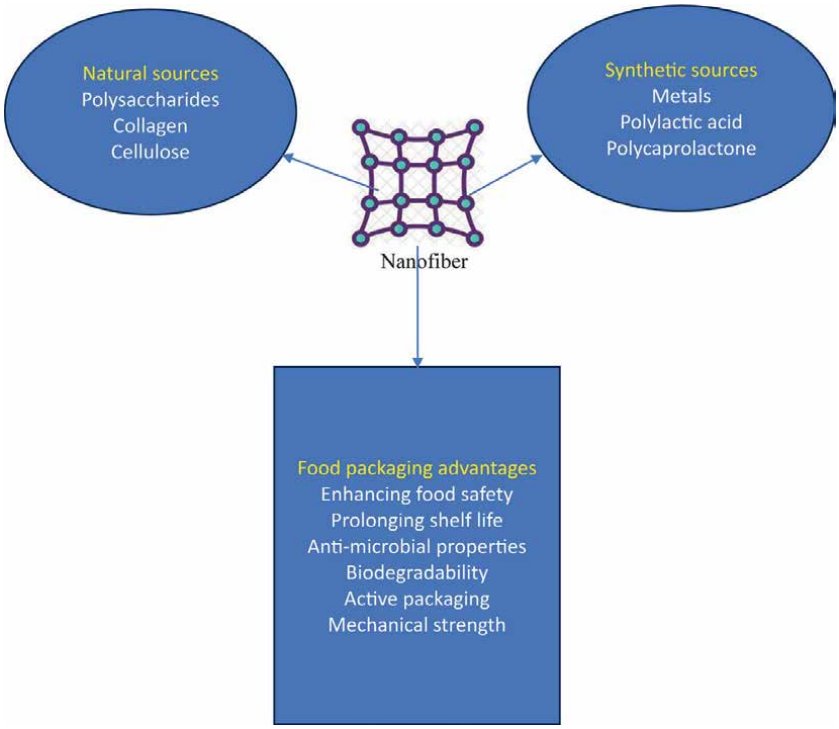


Figure 2.
Sources and advantages of nanofibers in food packaging.

4. Functionalization of nanofiber

A food can be considered "functional" if it has beneficial effects on specific bodily functions in addition to its nutritional value, with the goal of promoting overall health and well-being and/or lowering the incidence of chronic diseases [75].

Functional nanofibers are nanofibers that are structured, designed, and engineered to possess specific properties or functions, making them useful for specific purposes. This is done in order to introduce specific properties into the nanofiber. This will give them extra properties that are absent in the native nanofibers. This process makes the nanofiber desirable for a wide range of applications. Many methods have been explored to create functional nanofibers (NFs) with highly desired functional characteristics for advanced prospective applications.

4.1 Fabrication of structural functional nanofiber

Using electrospinning technique for nanofiber fabrication, specific properties such as pre-size, gradient parameters, specific surface area, and specific mechanical properties can be successfully introduced via fiber orientation and loading capacity [76]. Fabrication of core-shell fabric structures has been made possible through selective incorporation of miscible solvents into a polymer matrix [77]. Successful fabrication of porous NF has been made possible through the introduction of highly volatile solvents like methylene chloride during electrospinning through either bicomponent spinning or thermal treatment under different atmospheric pressure [78].

4.2 Composite functional nanofiber

Composite functional nanofiber are blends of two or more distinct materials, including metals, ceramics, or polymers, to produce a new material with special qualities. Examples include polymer/polymer, polymer/nanoparticles, and polymer/inorganic salt composite NFs. These blend of materials produces new nanofibers with advanced functional properties by incorporating the unique properties of the two materials.

4.3 Functionalization through surface modification

There is a need to preserve the intrinsic properties of nanofiber while adding other properties to it in order to meet specific purposes. Different technologies can be utilized for this purpose. Physical, chemical, and biotechnological treatments can be used to introduce specific surface functionalities on the nanofibers [79]. Modifying a surface's characteristics is often a necessary part of making the material suitable for industrial applications.

4.3.1 Physical technology treatment

In this technique, plasma ions are generated by ionizing gasses such as hydrogen, nitrogen, argon, oxygen, or a combination with an electric charge under low pressure in a chamber. The generated plasma ions react with the surface of the material and modify it at molecular level. This process either changes in ways of either etching, coating, sputtering, patterning, oxidizing, or chemical activation. The reaction leads to the generation of active species (free radicals, ions) without damaging the nanofiber. This process is ecofriendly. Using plasma technology, the surface wettability of PA6 NFs was significantly enhanced by cold plasma surface modification [80]. In a related development, plasma-induced grafting was utilized to improve the hydrophilicity of poly(lactic-co-glycolic acid), PLGA-NFs, using O_2 or ammonia gas resulting in the formation of polar groups onto NFs [81].

4.3.2 Chemical technology treatment

Chemical treatment involves surface grafting, chemical cross linking, chelation, and chemical vapor deposition processes for the purpose of functionalizing nanofibers.

PVOH NFs were cross-linked in acetone with glutaraldehyde as the cross-linker, and their mechanical characteristics and water resistance are both noticeably improved [66].

4.3.3 Nanoparticle technology treatment

This process incorporates nanoparticles through different measures in functionalizing the nanofibers. One of the measures is sol-gel measure in which coatings of nanoparticles are selectively deposited on the surface of the materials [82]. ZnO was coated on PVAC-NFs using the sol-gel technique prior to carbonization which resulted in the formation of functional carbon NFs [83]. In related research, the wettability of PET fabrics was greatly improved upon coating with Al_2O_3 through the sol-gel method [84]. Another chemical procedure is in situ reduction; Ag ions were reduced in N_2H_5OH solution, and the resultant nanoparticles were doped into

PAN-NF film resulting in better surface functionalities [80]. Also related is the use of layer by layer deposition technology for the fabrication of homogeneous and ultrathin carbon NF films with chitosan as binder [85].

4.3.4 Biotechnology approach

This process involves the immobilization of bioactive molecules on the surface of NFs. The fabrication is done to specially imbibe certain biological agents on the nanofiber. Enzyme immobilization can occur through either physical adsorption, chemical binding, or by entrapment [86, 87]. Catalase immobilization on AOPANNF membrane improved its reusability and storage stability for a variety of uses, including the production of pharmaceutical compounds, bioremediation, biosensors, and food processing [88].

5. Functional nanofibers for food packaging: Barrier properties and preservation

Food packaging is one of the many industries that nanotechnology has the potential to improve because of its capacity to control materials at the molecular and atomic levels [89]. The major aim of active food packaging is to increase the shelf life of the food and retaining its freshness for a period of time. The overall objectives of food packaging are to (i) inhibit the growth of microorganisms, maintain their stability against environmental hazards, and resist oxidation; (ii) mask offensive odors while retaining flavor; (iii) provide sustained nutrient delivery; (iv) assist in element filtration and accumulation; and (v) function as a sensor carrier [90, 91].

In the changing world, more food needs to be produced for the ever-increasing population. Incorporating nanotechnology into food production, food storage is a right step to take by the manufacturers in order to maintain food quality before consumption [69]. Focus is on incorporating active nanofibers in the packaging materials in order to boost food safety and quality. Integrating antioxidant in the packaging materials will preserve the physical properties of the food such as flavor and color [73]. Biodegradable functional films are made from materials that have a specific capacity to degrade spontaneously over time, hence reducing their detrimental impact on the environment [92]. These materials were developed especially with conservation in mind, and the goal is to reduce the environmental impact of traditional packaging [93, 94].

Nanofibers can be designed to improve barrier properties in packaging materials. In this regard, packaging materials having effective barrier against oxygen [95], water vapor [96], and carbon dioxide [97].

Food packaging can protect food by adding naturally occurring bioactive compounds (BCs) with antibacterial and antioxidant properties, such as essential oils and certain polysaccharides, such additives can guide food against microbial alterations and have its shelf life extended [98, 99] and natural inorganic particles (metals, metal and non-metal oxides, and nanoclays, such as silver) [100]. This reduces financial losses and health problems brought on by food-borne pathogens.

6. Encapsulation techniques using nanofibers for controlled release of food

Nano/microencapsulation systems is a technique of insulating bioactive components from severe processing environments and, where necessary, triggering

regulated release using innovative food-related colloidal delivery systems (CDS) [101]. Encapsulation involves the process of coating core (active ingredients) within particle matrix or the "encapsulant" [102]. The encapsulant will act as barrier protecting the active material for a specific period of time. The effectiveness of the encapsulant can be measured in terms of bioaccessibility and bioavailability. Bioaccessibility describes the portion of the active material that is being released from its original location while bioactivity describes the portion of the active material that reaches the intended or proposed site of action [101].

Release is the exposure of the encapsulated material (bioactives) and subsequent transportation in the delivery system. There are two types of release, immediate, and modified; in the case of the former, it happens soon after usage, while in the case of the latter, it either occurs at a specific site inside the body or for a longer amount of time following usage [103]. A unique type of modified release is described as controlled release. This involves consistency in the mode of release of the active ingredients, and they are released over a specific period of time in a pre-programmed manner.

Diverse methods can be used for the encapsulation of a particular core material in the nanofiber matrix, among them are spray drying, freeze drying, extrusion, coacervation, adsorption, spray-coating, and gel-particle technologies [104]. The stability of the active materials depends wholly on the stability of the active ingredient and the nanofiber properties. The release potentials of the active ingredients can be manipulated by the nanofiber used and the production process utilized.

Electrospun nanofiber has been successfully harnessed for the encapsulation of sensitive bioactive compounds with the intention of protecting it against adverse conditions that can affect its wholesomeness as well as safe release as at when due. This approach has been used to encapsulate probiotic microorganisms, antioxidants, antibacterial substances, and vitamin [105]. It is imperative to take into account that these active compounds exhibit low thermal stability, as high temperatures utilized in food processing can lead to the loss of their functions [106].

6.1 Encapsulation by electrospinning

Electrospinning technique has been exploited for the production of continuous nanofiber nonwovens. The dimensional and morphological characteristics of the electrospun materials in the food industry do not suggest any possible internal applications in humans through ingestion; however, they do provide opportunities for the development of innovative bioactive packaging strategies which utilize the use of these nanofibrous matrices solely or in combination with other packaging materials [73].

A standard electrospinning setup contains a metallic nozzle and a metallic collector connected to a high voltage power supply. A drop of polymer solution ejected at the nozzle tip will transform into a conical droplet known as a Taylor cone when a high voltage is applied between the polymer solution and a metallic collector. This is because the electrostatic repulsive force acting on the drop surface counteracts the surface tension, resulting in the ejection of a liquid jet that is deposited onto the collector in the form of nanofiber mesh [107]. It is possible to produce dry micro- and nanostructures using electrospinning and electrospraying in a single phase at ambient conditions, such as room temperature and air pressure [108].

The resulting electrospun nanofibers and electro-sprayed nanoparticles have the potentials of protecting the bioactive compounds from harsh environments (such as

high pressures and/or temperatures encountered during food processing or packaging, storage, light, or oxidation), maintaining the active compound's functionality within the electrospun nanofibers and enabling the controlled release and delivery of bioactive compounds at expected time [109].

7. Nanofiber based sensors for food quality monitoring

Food industry is a huge investment that requires a lot of money. Seeding, planting, and harvesting require a lot of resources as the investor is anticipating great gain on his investment over time. In some cases, this is not the case, the yield is less compared to the investment. Many factors could be responsible for this such as light, oxygen, heat, humidity, temperature, and spoilage bacteria [110].

Many scientific research has been deployed to curtail this occurrence as well as increase productivity, coupled with improvement in food quality and safety. One of the noble steps developed to improve the yield is to monitor food quality and safety potentials. Several indicators can be employed to monitor changes or declines in food quality [111]. These include the number of microorganisms present in food [112], pH variations [113], plant or microbial toxins [114], heavy metals [115], and pesticide and herbicide residues [116].

In recent times, biosensor deployment for food safety monitoring has yielded a great deal of results. It has positively affected the food industry in such a way that early detection of spoilage as well as accurate prediction of spoilage is now possible. A biosensor is made up of three parts which are transducer, amplifier, and processor. These work together to detect a recognizable response [117]. In a biosensor, the transducer's job is to transform the recognized event into quantifiable signals. The amplifier magnifies the signal received from the transducer and transmits it to the processor where it will be processed. The final stage involves the transmission of the signals into visible responses which will thereafter be made available for interpretation in the display unit [117].

Nanosensors can be used for the detection of moisture level, soil properties, plant diseases, nutritional needs, and pesticide residues in various environmental settings [118]. Sustainable development-based smart agriculture has been made possible by nanosensors, identifying plant diseases and tracking the development of plant diseases.

8. Bioactive nanofibers in functional foods

Bioactive nanofibers hold great promise for use in functional food packaging: To give food packaging functional qualities, electrospun nanofibers can be loaded with a variety of bioactive substances, including antioxidants, antimicrobials, and pH indicators. These bioactive substances can be efficiently encapsulated and released under regulated conditions because of the high surface area and porous structure of nanofibers. For instance, gallic acid, a substance with strong bioactivity, antibacterial, and antioxidant qualities but a high susceptibility to environmental influences, has been encapsulated in nanofibers. Gallic acid can be better protected, and the shelf life of packaged foods can be increased by encasing it in nanofibers. In addition, encapsulation of antimicrobial and antioxidant compounds can delay food spoilage by controlling the release of bioactive substances [119].

Incorporation of bioactive nanofibers in functional foods offer a novel way to enhancing the health benefits of food products. The purpose of these nanofibers is to deliver bioactive compounds, such as vitamins, minerals, antioxidants, and probiotics, in a controlled and efficient manner. Bioactive nanofibers can be described as fine fibers, typically with diameters in the nanometer range, that contain or are coated with bioactive compounds. The bioactive compounds include nutrients (vitamins, minerals, and amino acids), antioxidants (polyphenols, flavonoids, and probiotics), valuable bacteria, enzymes, and phytochemicals. Fortified beverages fortified with nanofibers has experienced enhanced nutritional profile without affecting taste or texture [120, 121].

8.1 Edible nanofibers for food sustainability

Nanofibers can be prepared to be edible and mostly used for food packaging. Conventional food packaging materials are non-edible and mostly discarded into landfills thereby generating environmental menace over time. Among these types of packaging materials are paper, plastic, glass, steel, aluminum, and different alloys. There is great concern of consumers on the results of nutrition to their health. There are cases of seepage of harmful chemicals in the packaging materials into the bioactive compounds denaturing it and making it less active to its intended purpose [122].

The food industry has a challenge as a result of current consumer demands and wants, which are focused on more natural, high-quality, convenient, and safe foods, coupled with growing environmental concern over the rate of waste from packaging materials. This is where edible nanofiber comes in handy to enable safe delivery of food and bioactive materials [123]. Edible nanofibers can be incorporated as edible dispensers of food ingredients in the form of encapsulants, nanofibers, nanoparticles, and nanoemulsions. In this way, edible packaging serves as an active form of packaging loaded with numerous advantages.

Quite a large number of these bioactive compounds lose their inherent functionality through food processing and storage. Digestive enzymes and various environmental conditions are also capable of degrading these materials [124]. Edible nanofiber has high holding capacity and wide specific surface area of active substances making it suitable for food packaging applications [125]. Fiber and nanofiber-based edible packaging materials can be produced in such a way it can easily inhibit the growth of pathogenic bacteria and lipid oxidation.

Varieties of edible polymers including polysaccharides (starch, gums, pectin, cellulose, alginate, agar, and chitosan), proteins (collagen, casein, corn zein, gelatin, soy protein, pea protein, and wheat gluten), lipids (paraffin wax, beeswax, mineral oil, fatty acids, and vegetable oils) as well as composites (bilayers, multi stacks, conglomerates, and blends) offer economic benefits that can be integrated into edible packaging, such as biocompatibility, the ability to withstand moisture and/or gas, non-toxicity, and non-polluting properties [122, 124, 125]. These materials retain their properties upon fabrication into nanofiber coupled with the added advantage of wide surface area. These materials are made up of complex substances that are not metabolized by endogenous enzymes and can easily provide safety for the active substances till the time of use. Various researches have shown that fiber and nanofiber derived from the cells of several plants make excellent food packaging materials that contain excellent antimicrobial and antioxidant properties [126]. It has also shown inhibitory properties against bacteria and lipid oxidation.

Coatings made of nanofibers can carry flavors, colors, antioxidants, enzymes, and anti-browning agents while also lengthening the shelf life of the ingredients.

The data available to date indicate that edible packaging has been successfully used for meat, grains, nuts, cheese, baked goods, confections, fruits, and vegetables (intact or fresh-cut) [126, 127]. The choice of edible packaging is mostly influenced by the type of food being packed as well as the conditions and duration of storage.

In all nanofiber utilized for food packaging should also guarantee that there are no adverse effects to the food's quality (color, taste, lipid oxidation, or nutrient deterioration).

9. Conclusion

It is anticipated that future advancements in fiber processing technologies will alleviate the current financial limitations related to the extraction and purification of fibers. Fiber-based packaging will probably become more scalable with the application of creative and economical techniques, opening up new avenues for its extensive commercial use. As the market for environmentally friendly packaging expands, more research and development will go into finding alternative fiber sources beside cellulose. Nanofiber development is progressing, and new applications in food system will continue to soar. Many industries are gradually shifting to use nanofiber for the scalable applications based on its unique properties.

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Advances in Nanofiber Research – Properties and Uses is an essential resource for scientists, engineers, and industry professionals looking to explore cutting-edge developments in the world of nanofibers. This book delves into the unique properties of nanofibers, their advanced fabrication techniques, and their diverse applications across fields such as biomedical engineering, energy storage, environmental solutions, and more. Featuring contributions from leading researchers, this book provides in-depth insights into the synthesis, characterization, and potential uses of nanofibers, highlighting emerging trends and future directions in the field. This book offers the knowledge to discover how the unique qualities of nanofibers are shaping the future of materials science.

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