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2 Green processes and green fibers

Abstract: "Green Electrospinning" not from only non-toxic solvents but also from biopolymer solutions has become popular in recent years. Green fibers are particularly interesting for biomedical applications such as tissue engineering, drug delivery, biocompatible scaffolds, biosensors, and for photovoltaics, supercapacitors, fuel cells, battery components as energy fields, and for filtration membranes as environmental applications. In this chapter, we classified green electrospinning into two groups: (i) green processes as polymer free, solvent free, solution, and colloid electrospinning, (ii) green fibers from natural polymers and blends.

Keywords: benign solvent, bioactive agents, clean electrospinning, colloid electrospinning, natural polymer, polymer-free, solvent-free

2.1 Green processes

2.1.1 Polymer-free electrospinning

Typically, electrospinning studies are carried out using high-molecular-weight polymers and high solution concentrations because of chain entanglements and the continuous stretching of the charged jet. Long et al. [1] reported that high-molecular-weight polymers are not the only the requirement, but the presence of sufficient intermolecular interactions can also act as chain entanglements for the continuous fiber formation. Therefore, besides easily electrospinnable polymers, globular proteins and low-molecular-weight compounds [such as Gemini surfactants, phospholipids, diphenylalanine peptides, and cyclodextrins (CDs)] have also been electrospun into fibers, since they exhibit a similar behavior to polymers in solution [2–5]. Electrospinnability of globular proteins (namely bovine serum albumin) was attributed to disruption of the tertiary structure and reduction of intramolecular disulfide bonds, allowing the

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reformation of intra- and intermolecular disulfide bonds [6]. Increasing the concentration of Gemini surfactants and phospholipids in the appropriate solvent results in the formation of entangled cylindrical or worm-like micelles. Furthermore, diphenylalanine peptides that self-aggregate into nanotubes were transformed into nanofibers due to the presence of π–π interactions. A cyclic oligosaccharide, CD, self-assembles in their concentrated solutions to form aggregates through intermolecular hydrogen bonding, which makes possible to obtain electrospun nanofibers. Polymer-free nanofibers of α-, β-, and γ-CDs have been fabricated by the selection of suitable solvents and concentrations ensuring sufficient viscosity and conductivity [7–10]. The morphology and the diameter of the resulting fibers are affected by not only the type of solvents but also the type of CDs. CD derivatives, such as hydroxypropyl-β-cyclodextrin (HPβCD) [11, 12], hydroxypropyl-γ-cyclodextrin (HPγCD) [11, 13], and methyl-β-cyclodextrin (MβCD) [11], have been obtained using water, *N*,*N*-dimethyl formamide (DMF), and dimethyl acetamide (DMAc) as solvents. Among them, DMF is found to produce bead-free fibers with the three derivatives [12].

Compared to other small molecules, CDs are advantageous because they can form host–guest inclusion complexes (ICs) with different compounds, such as drugs, volatile compounds, food or cosmetic additives, and antibacterial agents. The CD inclusion complexation enhances thermal stability and water solubility of the hydrophobic guest molecules, which provides a promising platform for drug delivery applications. Nanofibers of CD–ICs containing 4-amino benzene [14], spironolactone [15], triclosan [16], diclofenac [17], geraniol [18], vanillin, limonene [19], sulfobutyl ether$_7$-β-CD [20], vitamin E [21], camphor [22], and linalool [23] have successfully been produced without using a polymeric matrix. The most commonly used solvents for CD–ICs are water, ethanol, aqueous sodium hydroxide, DMF, DMAc, dimethyl sulfoxide, and ionic liquids (ILs, e.g., 1-ethyl-3-methyl imidazolium acetate). To obtain nanofibers as reconstitutable solids for drug-release applications, electrospinning IC is also used as an alternative to the freeze-drying process for the preparation of fast-dissolving CD-based solid complexes containing limited soluble drugs [17].

On the one hand, because of their hydrophobic cavity and the hydrophilic surface, CDs can form the host–guest ICs with various bioactive compounds to be used for the fast-dissolving, prolonged release, and long shelf-life of the active component, enhanced thermal stability, and water solubility. On the other hand, CDs are used as both reducing and stabilizing agents for the green synthesis of gold nanoparticles [24].

Apart from CDs, Allais et al. [25] enlarged the list of possible small molecules used for electrospinning using tannic acid without the addition of any polymer. They also reported the cross-linking of tannic acid nanofibrous membranes by the oxidation of galloyl groups with sodium iodate and ferric ions to obtain mechanical integrity.

2.1.2 Solvent-free electrospinning

Techniques associated with solvent-free electrospinning neither have a risk of residual solvents being present for biomedical applications, nor is there any solvent that may evaporate into the air [26]. To remove solvents, special conditions are needed, because electrospinning requires that the polymer chains are able to flow and extend in an electric field, so that the fibers are formed, which will be covered in detail in Chapters 4 and 5.

The most common techniques for solvent-free electrospinning are electrospinning from the melt state, supercritical carbon dioxide (CO_2)-assisted anion-curing, UV-curing, and thermocuring [27]. Two methods that we will focus on are the use of supercritical CO_2 as a "solvent" and melt electrospinning. In both systems, there is no traditional solvent used to dissolve the polymers. Supercritical CO_2 uses the semiliquid semigaseous properties of CO_2 under high pressure and temperatures to aid in the flow of polymer chains. Electrospinning of polymer melts, on the other hand, eliminates the use of toxic solvents by heating semicrystalline polymers or glassy polymers above their melting temperature (T_m) and glass transition temperature (T_g), so that a viscous solution in which polymer chains are capable of flowing can be formed.

Supercritical carbon dioxide

CO_2 is a gas at standard temperature and ambient pressure. When temperature and pressure are increased above a critical point, CO_2 behaves somewhat like both a gas and a liquid [28]. Electrospinning in the presence of supercritical CO_2 is similar to the one at ambient temperature, but the supercritical CO_2 liquid is used to alter the viscosity of the polymer, much like a solvent or a plasticizer. CO_2 is used because it is relatively nontoxic, nonflammable, inexpensive, easily available, odorless, tasteless, and relatively environmentally friendly. In addition, CO_2 evaporates at ambient conditions and is therefore easily released from the products [29].

The solvating power of supercritical CO_2 is connected to its density, which in turn depends on the pressure and the temperature [30]. The solubility of the polymer in supercritical CO_2 therefore may be controlled greatly by pressure and temperature, as a higher density generally translates to higher solubility.

Melt Electrospinning

In melt electrospinning, polymers are processed by using heat to melt the polymer. Unlike traditional electrospun fibers, which form due to the precipitation out of solution of the polymer as the solvent is evaporated, fibers are formed in melt electrospinning by the cooling down of the polymer melt while being collected [31]. Melt

electrospinning is like an extrusion technique with the addition of a high voltage to further stretch the fibers [32]. The greatest problem with melt electrospinning is that the absence of solvent greatly reduces the surface charge density of polymer melts, which results in instabilities of the fiber jet [33].

2.1.3 Solution electrospinning

In a classical electrospinning process, nanofibers are fabricated by dissolving the polymer in an appropriate solvent. However, except water, most of the electrospinning solvents used to dissolve polymers are toxic and harmful to the environment and human health. Thus, the use of green solvents is significant to reduce environmental and health impacts. The main alternatives to traditional organic solvents are (i) water, (ii) mild solvents, and (iii) ILs.

Water as a Solvent

Water is certainly a good solvent for water-soluble polymers. However, mechanical strength of nanofibers electrospun from water-based polymers is low, limiting their use in aqueous systems. To enhance their mechanical properties and make them water-insoluble, additional modifications such as cross-linking, UV, or plasma treatment are required. On the other hand, frequently used cross-linkers like gluteraldehyde, diethylene glycol, glyoxal, epichlorohydrin, and 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide together with *N*-hydroxysuccinimide are toxic. New trends are directed toward the use of nontoxic reagents such as citric acid, proanthocyanidin, epigallocatechin-3-gallate (EGCG), genipin to reduce the use of the aforementioned toxic cross-linkers. Table 2.1 reports a summary of nanofibers from aqueous precursors by green electrospinning, including the details about fiber diameters, cross-linking and/or reducing agents, and applications.

A different water-based electrospinning approach, which is also considered as green, is used to fabricate inorganic fibers. The production of inorganic fibers is based on the simultaneous electrospinning of water-soluble polymer and metal precursor(s) blends followed by calcination. Table 2.2 lists works on metal oxide fibers electrospun from aqueous polymer/metals salt(s) solutions, heat treatment parameters, as well as their potential applications. The resulting metal oxide nanofibers have been mostly used in energy applications such as solar cells, electrodes for batteries, fuel cells, and supercapacitors.

By taking Tables 2.1 and 2.2 into consideration, except the studies involving only fabrication and characterization, these water-based electrospun fibers find applications mainly in biomedical and energy fields.

Table 2.1: A summary of green nanofibers from aqueous precursors including information about fiber diameters, cross-linking and/or reducing agents, and applications.

Polymer or blend	Fiber diameter (nm)	Reducing/stabilizing agent	Cross-linking agent	Application	Reference
PEO/PHA-*b*-PEO	350–550	–	–	–	34
PVA/soy protein isolate	600–4,500	–	–	–	35
PEG/polyindole	720	–	–	An electrode for supercapacitor	36
PEO/silk fibroin/honey	480–2,230	–	–	Wound dressing	37
PVA/zinc acetate	~500	–	–	NH_3 sensor	38
PEO/silk fibroin/grape seed extract	~420	–	–	Skin care, skin regeneration, wound healing	39
PEO/chitin nanofibrils/biolignin	<150	–	–	Tissue engineering	40
PVA/AgNPs	~150	–	–	Antiseptic dressing	41
Silk fibroin/vitamin E	384–722	–	–	Skin care	42
PVA/soy protein isolate	200–900	–	–	Biodegradation	43
Folic acid/dextran; folic acid/PVP; folic acid/ODA-MMT	304–379	–	Reactive electrospinning and thermal treatment	–	44
PVA/ODA-MMT/poly(maleic acid-*alt*-acrylic acid)	50–300	–	Thermal cross-linking	–	45

(continued)

Table 2.1 (continued)

Polymer or blend	Fiber diameter (nm)	Reducing/stabilizing agent	Cross-linking agent	Application	Reference
PVA/citric acid	125–255	–	Thermal cross-linking with citric acid	–	46
PVA/sodium alginate	140–350	–	Physical cross-linking with $CaCl_2$ in ethanol	–	47
PEO/PHA-*b*-MPEG PEO/PCL-*b*-MPEG	340–600 275–720	–	Physical cross-linking	–	48
PVA/PEO/ulvan	75–109	–	Physical cross-linking	–	49
Silk fibroin/vitamin B5	625	–	Ethanol vapor	Skin care	50
PVA/PAA blend	225–408	–	Thermal cross-linking	Antimicrobial activity	51
PVA/ODA-MMT/folic acid	~100	–	Reactive electrospinning	Anticancer activity	52
Silk fibroin	623–805	–	Ethanol vapor	Tissue engineering	53
PVA/CMC/$AgNO_3$	100–150	CMC	–	–	54
PVA/WPU+ PVA/$AgNO_3$	255–405	PVA	–	–	55
PVA/$AgNO_3$/cyclodextrin	290–500	PVA and HPβCD	–	–	56
PEO/CMC	50–200	Immersed in $AgNO_3$ solution reduced by UV irradiation	–	Antimicrobial dressing	57

PVA/Au nanoparticles	50–450	*Couroupita guianensis* leaves extract	–	Antibacterial wound dressing	58
PVA/$AgNO_3$ (PVA-Ag) PVA/$AgNO_3$ (PVA-Ag@heat) PVA/$AgNO_3$ (PVA-Ag@UV)	300–500 150–350 150–350	PVA Heating UV irradiation	–	Biocidal activity and cytotoxicity	59
PVA/PAA/SiO_2 nanoparticle/$AgNO_3$	200–800	UV reduction	Thermal cross-linking	Antibacterial air filtration	60
PVA/*O*-carboxymethyl chitosan/$AgNO_3$	100–200	Ascorbic acid	Glutaraldehyde vapor	Antimicrobial activity	61
PVA/Triton X-100/$AgNO_3$	150–250	PVA	Thermal cross-linking	Catalytic hydrogenation	62
PVA/chitosan/$AgNO_3$	150	Glucose	Glutaraldehyde	Antimicrobial wound dressing	63
PVA/$AgNO_3$/EGCG PVA/PEI	630 508	EGCG	Glutaraldehyde vapor	Biosensor	64
PVA/$HAuCl_4$ PVA/$AgNO_3$ PVA/H_2PtCl_6	275–355 295–385 260–360	EGCG	Glutaraldehyde vapor	Biosensor for H_2O_2 detection	65

CMC, carboxymethyl cellulose; HPβCD, hydroxypropyl-β-cyclodextrin; ODA-MMT, octadecyl amine-montromorillonite; EGCG, epigallocatechin-3-gallate; AgNP, silver nanoparticles.

Table 2.2: Metal oxide nanofibers fabricated by electrospinning an aqueous solution containing metal salt(s) or metal alkoxide and a template polymer.

Electrospinning solution	Heat treatment parameters	Metal oxide(s) nanofibers	Application	Reference
PVP/TTIP/EtOH/AA/ $Zn(NO_3)_2{\cdot}6H_2O$	500 °C for 2 h	Zn-doped TiO_2 hollow fibers	Dye-sensitized solar cells	[66]
PVA/polymerized sucrose	350 °C (stabilization) 650 and 950 °C (carbonization in Ar/H_2)	Porous carbon nanofibers	Electrode for supercapacitor	[67]
PVP/$Cu(CH_3COO)_2{\cdot}H_2O$/ $Ce(NO_3)_3{\cdot}6H_2O$	500 °C for 3 h	CuO/CeO_2 nanofibers	–	[68]
PVA/$Zn(CH_3COO)_2{\cdot}2H_2O/AlCl_3{\cdot}6H_2O$ + PVA/$Zn(CH_3COO)_2{\cdot}2H_2O$/ $Co(CH_3COO)_2{\cdot}4H_2O$	450, 500, 550, 600 °C for 2 h	Co- and Al-doped ZnO nanofibers	Nanogenerator	[69]
PVA/$SnCl_2{\cdot}2H_2O$	1,000 °C for 2 h	SnO_2 nanofibers	–	[70]
PVA/$Zn(CH_3COO)_2{\cdot}2H_2O$	550 °C	ZnO nanofibers	Nanogenerator	[71]
PVA/$Mn(CH_3COO)_2{\cdot}4H_2O$	1,000 °C for 1 h	Mn_3O_4 nanofibers	–	[72]
PVA/$Al(NO_3)_3{\cdot}9H_2O$/ $Al[OCH(CH_3)_2]_3$	1,200 °C	Al_2O_3 nanofibers	–	[73]

Table 2.3: Mild solvents and solvent mixtures that belong to Class 3 used in green electrospinning.

Mild solvents	
Water	Ethanol
Acetic acid	Ethanol/water
Acetic acid/formic acid	Ethanol/acetic acid
Acetic acid/water	2-Methoxy ethanol
Acetic acid/2-methoxy ethanol	Ethyl acetate
Acetone	Formic acid
Dimethyl sulfoxide	Limonene

Mild Solvents

Many types of polymers, both synthetic and natural, have been used for the fabrication of "green fibers" using mild solvents. Table 2.3 summarizes the solvents used

for green electrospinning that belongs to Class 3 (solvents with low toxic potential) according to the classification in ICH (International Conference on Harmonisation of Technical Requirements for Registration of Pharmaceuticals for Human Use) [74].

Literature documents the ability to electrospin synthetic polymers by using mild solvents instead of commonly used toxic ones. The investigated synthetic polymer/mild solvent systems are PVP/ethanol [75, 76], PVP/2-methoxy ethanol and acetic acid [77], PVA/ethanol/water [78], poly[*N*-isopropylacrylamide-*co*-(maleic acid)]/ethanol [79], PCL/glacial acetic acid [80], PCL/acetone [81], and nylon-6/formic acid [82].

Although the fibers from synthetic polymers are often a more economic and versatile alternative, biofibers are especially interesting because of their biocompatibility or biodegradability. Awal et al. [83] fabricated cellulose-based nanocomposite fibers electrospun from wood pulp and nylon 6,6 solution in formic acid. The same research group also obtained green composite fibers prepared by electrospinning PEO/wood pulp solution with a mixed solvent of ethanol and water [84]. Another wood component, lignin, is being used as an eco-friendly precursor [85]. The fabrication of lignin fibers through green electrospinning is problematic because of its low viscoelastic properties and water insolubility. The former case can be solved by using additional synthetic polymers to improve the spinnability. The water-soluble derivatives of lignin such as lignosulfonates can be an alternative for the latter case. Misra et al. [86] prepared sulfur-free anionic sodium carbonate lignin, chitosan, and PEO blend solution in aqueous acetic acid. The inherent negative charge of the lignin was neutralized by cationic chitosan. The free-sodium ions and PEO were removed from the fibers through a water-soaking process to obtain pure carbon fiber.

Chitosan, which is derived from chitin (the second most abundant natural polymer after cellulose), has also been used as a reducing and stabilizing agent in nanoparticle synthesis. For instance, Zhuang et al. [87] demonstrated the use of chitosan as a reducing agent and stabilizer for the fabrication of chitosan/gelatin nanofibers containing silver nanoparticles (AgNPs). A mixed solution of AgNPs–chitosan composite with gelatin containing acetic acid was used to produce nanofibers.

Green synthesis of metal nanoparticles on the electrospun nanofibers is also based on the addition of metallic salt(s) into the polymer solutions and further treating the nanofibers by using nontoxic and safe chemicals from plant extracts as reducing agents, annealing, and irradiation techniques (i.e., UV or plasma). As an example, Arvand et al. [88] described the synthesis process for antibacterial AgNP-modified chitosan-based fibers using Eucalyptus extract. Aqueous acetic acid solution of chitosan, PEO, and silver nitrate was electrospun, and the composite fibers were immersed in ethanolic extract of Eucalyptus as a reducing agent instead of using $NaBH_4$ to form AgNPs on the nanofibers. Similarly, use of *Falcaria vulgaris* extract as a green reductant was reported by Kohsari et al. [89]. Using a different approach, ex situ prepared AgNPs were added into the electrospinning solution of chitosan and PEO in aqueous acetic acid.

In addition to functionalization with metal nanoparticles, by combining chitosan with natural biocomponents leads to potential usage in biomedical applications. A new nanofibrous wound dressing containing green tea extract was proposed by Sadri and collaborators [90]. They prepared electrospinning solutions that dissolved chitosan/PEO/green tea extract in aqueous acetic acid solution. Chitosan and PEO were used as matrix, and green tea extract was used to improve the wound healing performance of the chitosan-based nanofibers. Li et al. [91] reported fabrication of electrospun zein fibers as carriers to stabilize green tea polyphenol, (−)-epigallocatechin gallate (EGCG). The fiber-forming solution was prepared by dissolving EGCG in an aqueous ethanol solution of zein. Encapsulating EGCG in zein fibers resulted in the enhanced stability of the bioactive compound.

Collagen scaffolds are preferred for use in tissue engineering, artificial skin, and drug delivery. Zhang et al. [92] prepared PVA/collagen micro-nanofibers from water/acetic acid mixtures. Zhou et al. [93] proposed a greener synthesis of electrospun collagen/hydroxyapatite (HAP) composite fibers from phosphate buffer saline/ethanol/water system. The desalination of collagen solution and the use of HAP sol led to a good dispersion of HAP particles in the electrospinning solution and the alignment of HAP particles in the collagen/HAP composites in the meantime improved the mechanical properties of the resulting composites. The use of acetic acid and water as a green solvent for the fabrication of a collagen/HAP composite fibers was also highlighted by Castilla-Casadiego et al. [94].

Nieuwland et al. [95] showed for the first time electrospinning of globular proteins under food-grade conditions. In the case of zein-based fibers, ethanol was used; while in gelatin-based fibers, warm water was used as a solvent. Uniform protein nanofibers were prepared by electrospinning of gelatin for air-filtration purposes. Instead of commonly used toxic solvents (e.g., TFA, TFE, or HFIP), a nontoxic solvent mixture of acetic acid/water mixture was used to fabricate uniform gelatin nanofiber mats as air-filtering material. In addition, the diameter of gelatin nanofibers was also reduced to be around 70 nm, which results in high surface area and high filtration efficiencies [96].

Another application is to use natural fibers as a skeleton material in gel polymer electrolyte (GPE) of lithium-ion batteries. Zhu et al. [97] fabricated soy protein isolate/PVA nanofibrous mats using acetic acid/water mixture as solvent. The biodegradable composite mat was then activated in liquid electrolytes providing high-performance and green skeleton material in GPEs.

Natural solvents can also be used as an alternative for electrospinning. For instance, limonene, which is the main component of lemon and orange oils, is a cyclic monoterpene and started to be used to obtain polystyrene [98–100], poly(vinyl alcohol) [101, 102], asolectin phospholipid [103], and CD nanofibers [104].

Ionic Liquids

ILs consist of ions influenced by the presence of Coulombic interactions and have different physicochemical properties compared to traditional VOCs. Many ILs are nonvolatile, more viscous than VOCs, highly conductive and they can provide dissolution of biopolymers not soluble in VOCs. Therefore, electrospinning of biopolymers, such as cellulose [105–110], heparin [106, 111], agarose [112], silk [113, 114], and chitin [115, 116], has attracted much attention. Unlike traditional solution process, using ILs requires dissolution of a polymer in the ILs as solvent followed by electrospinning into the coagulation bath which can be water, ethanol, or water/ethanol mixture as antisolvents. The most often used ILs are 1-butyl-3-methylimidazolium chloride ($[C_4mim]Cl$), bromide ($[C_4mim]Br$), and acetate ($[C_4mim][OAc]$), 1-ethyl-3-methylimidazolium benzoate ($[C_2mim][BA]$), acetate ($[C_2mim][OAc]$), and lactate ($[C_2mim][OLac]$). ILs containing basic acetate $[OAc^-]$ or chloride $[Cl^-]$ anions and alkyl imidazolium cations have been identified as the appropriate solvent for biopolymer dissolution [116, 117].

2.1.4 Colloid electrospinning

An improved method for the combination of complex nanostructures with fibers is referred to as colloid electrospinning, which leads to the development of physical and biological properties that encompass both materials [118]. This technique allows for the electrospinning of dispersions of polymer and/or inorganic nanoparticles/nanocapsules with a solution of an electrospinnable polymer. Colloid electrospinning can be classified into two main groups: as suspensions and as emulsions [118, 119]. The examples of organic/inorganic nanostructures used in suspension and emulsion electrospinning are summarized in Table 2.4. Inorganic nanostructures constitute the largest fraction of the suspension electrospinning category. They are advantageous when compared to organic colloids because inorganic nanostructures have a higher density than the polymer templates, and their location in the fiber is usually easily identified by electron microscopy [118, 120]. Metals, metalloids, and their oxides, fluorescent markers, and other minerals fall into the inorganic nanostructure groups used in suspension electrospinning. Besides inorganic nanostructures, hydrophilic/hydrophobic polymer particles and natural systems such as viruses and bacteria may be used in suspension electrospinning. In the case of emulsion electrospinning, water-in-oil, oil-in-water, and polymer/polymer (nonaqueous) emulsions can be electrospun to obtain core–sheath fibers, which are generally fabricated by coaxial electrospinning. Although this method allows for control of the nanofibers structure, it is relatively difficult to scale up. Conversely, emulsion electrospinning is advantageous in recent years as it allows the production of two-component nanofibers of controlled morphology. Furthermore, this approach is environmentally friendly, reducing the amount of organic solvent used during the process [119].

Polymers that are commonly used in colloid electrospinning are polystyrene [121–123], poly(ε-caprolactone) [124–126], poly(L-lactide) [127], poly(vinyl alcohol) [120, 124, 128–137], poly(vinyl acetate) [124, 138], poly(vinyl alcohol)/poly(acrylic acid) [139], poly(*N*-vinylpyrrolidone) [140–145], poly(vinyl formamide) [130], silk fibroin [146], poly(ethylene oxide) [147–150], *N*-carboxyethylchitosan [151], polyacrylonitrile [152, 153], polymethyl methacrylate [154–156], polyamide 6 [157–159], chitosan/gelatin [160], polyurethane [124, 161, 162], poly(ethylene terephthalate) [163], dextran [164], polyacrylamide [165–167], poly(L-lactide-*co*-glycolide) [168], methylcellulose [169], collagen [170], polyacrylic acid [171], polyvinylidene fluoride [172, 173], which act as a structural network for the organic/inorganic nanostructures. Since colloid electrospinning allows the use of almost any polymers and nanostructures, the resulting hierarchically structured or compartmented nanofibers pave the way for various applications such as catalysis [138], adsorption [143], membrane distillation [172, 173], optics [174–176], energy conversion and production [123, 177], drug delivery [131, 146], tissue engineering [113, 127, 178], and wound dressing [51, 134].

2.2 Green fibers

2.2.1 Natural material-based electrospinning

The use of eco-friendly products to reduce the consumption of synthetic plastics that appear in groundwater and soil is an obvious potential area for green electrospinning. Natural materials suggest that the material comes from a source in nature and therefore can be acquired from something that grows, for example, chitin, lignin, and silk. Many natural materials have the added benefit that they are more easily biodegradable than synthetic materials [251].

Some natural materials could be used as a solvent or a plasticizer to induce flow within the polymer fibers during the electrospinning process. For instance, D-limonene, which is a natural solvent made of monoterpene hydrocarbon and is the main component of orange or lemon peel oil, has been used as an electrospinning solvent [99, 252]. In addition, other natural molecules such as hyaluronic acid can be used in the formation of nanofibers without the difficulty of dissolution of much higher molecular weight polymers [253]. Natural polymers can roughly be made up of two classes of biomacromolecules; proteins like silk, collagen, and gelatin or polysaccharides like cellulose, chitosan, and dextran. [86, 254, 255]. Renewable resource-based molecules such as lignin have the potential for green electrospinning as they are abundantly available and cheap [256].

Again we are brought to the question of Chapter 1, “how green is green?,” as many natural polymers do not necessarily dissolve with ecofriendly solvents, and other natural materials might only be a fraction of the final materials found in the nanofibers and do not necessarily biodegrade any faster a than synthetic polymers.

Table 2.4: Classification of the colloid electrospinning and the nanoparticles employed in the literature.

Colloid Electrospinning	
Electrospinning of suspensions	
Electrospinning of inorganic nanostructures	
Metals and metalloids	Ag [133,159,179], Au [121,180], Si [152], Ti, Zn [124], Co, Cu [181], Pt [144]
Metals and metalloid oxides	SiO_2 [122,134,135,139,143,147,154,165,172,173,182–188], TiO_2 [189–192], TiO_2/grapheme [123], Fe_3O_4 [193–198], ZnO [199], $BaTiO_3$ [200], ITO (In_2O_3/SnO_2) [148], MgO/ ZrO_2/Al_2O_3 [201], $Ni_{0.5}Zn_{0.5}Fe_2O_4$ [202], Boehmite (Al(O)-OH) [125, 158], $LiCoO_2/CeO_2$ [188], SnO_2/CeO_2 [135], WO_3 [145], graphene oxide [203, 204]
Fluorescent markers	Tris(8-hydroxyquinoline) aluminum(III) (Alq3) [164], CdS/CdSe/PdS [155, 205], ZnS/PdS [138], CoS [206]
Other minerals	Clay minerals (mica, zeolite, montmorillonite, bentonite) [207–210], boron nitride[124, 211], $CaCO_3$ [212, 213], calcium phosphate [214, 215], hydroxyapatite [124,155,161,168,170,216–219], hydroxyapatite + CNT [220, 221], curcumin [146], silicon carbide [222]
Electrospinning of polymer particles	Polystyrene [223–225], PS/PBA/P(S-*co*-BA)[129], P(S-*co*-BA), P(S-*co*-BA-*co*-AMA), P(S-*co*-BA-*co*-DAAM) [130], poly (styrenemethyl methacrylate-acrylic acid) (poly(St-MMA-AA)) [226, 227], PMMA [228], polyurethane [229, 230], PLGA [231], P(HA-*b*-EO) [232], PVDF [233], PVA [234], P(MAA-*co*-DVB)/P (MAA-*co*-TRIM) [163], P(NIPAM-*co*-AA-*co*-MBAAm) [235], poly (*N*-isopropylacrylamide-*co*-tert-butyl acrylate) [167], P(DMA-*co*-EDMA-*co*-4VP)/ P(DMA-*co*-EDMA-*co*-AMSA) [142]
Electrospinning of natural systems	M13 viruses [236], adipose-derived stem cells [237], *Lysinibacillus* sp. [238]
Electrospinning of emulsions	
Water-in-oil emulsion	PEG/PLA [239, 240], PDLLA [169,241,242], PLGD [243], cellulose nanocrystals/PLA [244]
Oil-in-water emulsion	PEO [245], PVA [246, 247], PVP [248, 249]
Polymer–polymer emulsion	PAN [156, 250]

Some solutions to this problem may come from taking advantage of the blend electrospinning technique discussed in the next section. Some natural polymers that may be difficult to electrospin in aqueous solutions such as chitosan and silk fibroin may be mixed with electrospinnable polymers like PVA, PEO, or PAA to increase their processability while including properties of the natural polymer. Through a careful polymer selection, nanofibers can have additional properties including antibacterial or antifouling properties, for example, chitosan nanofibers intrinsically exhibit antibacterial activity but require blending to make more easily spun [257].

2.2.2 Blend electrospinning

Blend electrospun fibers are produced from polymer solutions with or without other additives. Factors that influence the fiber formation are related to the properties of the polymer solution and electrospinning environment. In coaxial electrospinning, two solutions are electrospun simultaneously through different capillary channels into a nozzle. This generates composite nanofibers with a core–shell structure [258]. The term blend electrospinning can refer to a number of scenarios. Typically, blends are a mixture of two or more components, which are mixed together in order to gain properties from both materials; PEO/PVA for instance is a common blended fiber [259]. The term "emulsion electrospinning" has also been used to describe a type of blend electrospinning [118].

The main contribution blend electrospinning adds to the green electrospinning process is in the use of natural additives to reduce the synthetic materials. There are natural polymers that have relied heavily on addition of bioactive reagents, including essential oils, polyphenols, herbal extracts, honey, and other natural components [260, 261]. Cinnamaldehyde, for example, is a volatile essential oil that has been shown to eradicate pathogens nonspecifically and has been added to electrospun fibers [262].

"Green/Safe/Clean Electrospinning," which can be considered as an approach to toxicology, safety, and environmental issues, has been the focus of many research publications in recent years. Although the optimization of process parameters with benign solvents and biopolymers is more difficult, promising results have been obtained for the generation of green nanofibers particularly in biomedical and healthcare applications.

Abbreviations

AMSA	Acrylamidoethylsulfonic acid
BSA	Bovine Serum Albumin
CNT	Carbon nanotube
CMC	Carboxymethyl cellulose

CD	Cyclodextrin
DMAc	Dimethyl acetamide
DMSO	Dimethyl sulfoxide
DVB	Divinylbenzene
ECH	Epichlorohydrin
EGCG	Epigallocatechin-3-gallate
EDMA	Ethylene dimethacrylate
GPE	Gel polymer electrolyte
T_g	Glass transition temperature
HFIP	Hexafluoroisopropanol
HAP	Hydroxyapatite
HPβCD	Hydroxypropyl-β-cyclodextrin
HPγCD	Hydroxypropyl-γ-cyclodextrin
IC	Inclusion complex
ILs	Ionic liquids
T_m	Melting temperature
MAA	Methacrylic acid
MβCD	Methyl-β-cyclodextrin
NIPAM	*N*-isopropylacrylamide
DMF	*N*,*N*-dimethyl formamide
DMA	*N*,*N*-dimethylacrylamide
MBAAm	*N*,*N*-methylenebis(acrylamide)
ODA-MMT	Octadecyl amine-montromorillonite
O/W	Oil-in-water
PDLLA	Poly(DL-lactic acid)
PLA	Poly(lactic acid)
PLGA	Poly(lactic-*co*-glycolic acid)
PHA	Poly(hexamethylene adipate)
PBA	Poly(n-butyl acrylate)
P(S-*co*-BA-*co*-AMA)	Poly(styrene-*co*-*n*-butyl acrylate-*co*-allymethacrylate)
P(S-*co*-BA-*co*-DAAM)	Poly(styrene-*co*-*n*-butyl acrylate-*co*-diacetone acrylamide)
P(S-*co*-BA)	Poly(styrene-*co*-*n*-butyl acrylate)
Poly(St-MMA-AA)	Poly(styrenemethyl methacrylate-acrylic acid)
PCL-*b*-MPEG	Poly[(epsilon-caprolactone)-*block*-(methoxypolyethylene glycol)]
PHA-*b*-MPEG	Poly[(hexamethylene adipate)-*block*-(methoxypolyethylene glycol)]
PAA	Polyacrylic acid
PAN	Polyacrylonitrile
PEG	Polyethylene glycol
PEO	Polyethylene oxide
PEI	Polyethylenimine
PMMA	Polymethyl methacrylate
PU	Polyurethane
PVA	Polyvinyl alcohol
PVDF	Polyvinylidene fluoride
PVP	Polyvinylpyrrolidone
AgNPs	Silver nanoparticles
SCL	Sodium carbonate lignin
TTIP	Titanium(IV) isopropoxide
TFA	Trifluoroacetic acid

TFE	Trifluoroethanol
TRIM	Trimethylolpropane trimethacrylate
VOCs	Volatile Organic Compounds
W/O	Water-in-oil
WPU	Waterborne polyurethane
VP	4-vinylpyridine
EDC/NHS	1-ethyl-3-(3-dimethylaminopropyl)carbodiimide / *N*-hydroxysuccinimide
$[C_4mim]Cl$	1-butyl-3-methylimidazolium chloride
$[C_4mim]Br$	1-butyl-3-methylimidazolium bromide
$[C_4mim][OAc]$	1-butyl-3-methylimidazolium acetate
$[C_2mim][BA]$	1-ethyl-3-methylimidazolium benzoate
$[C_2mim][OAc])$	1-ethyl-3-methylimidazolium acetate
$[C_2mim][OLac]$	1-ethyl-3-methylimidazolium lactate

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