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Electrospun Cellulose Composite Nanofibers

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Abstract

This chapter deals with the structure, properties, and applications of electrospun-based cellulose composites. Extraction methods of cellulosic nanofibers from different sources are discussed in detail. Cellulose has the special advantage of high specific strength and sustainability, which make them ideal candidates for reinforcement in various polymeric matrices. Cellulose nanofibers find application in various fields, including construction, the automobile industry, and soil conservation. Cellulose, an eminent representative of nanomaterial obtained from various natural fibers, can be dissolved in various solvent systems, which are described in detail in this study. Thermoplastic-based electrospun cellulose nanocomposites and their applications are highlighted. This chapter describes current and future applications of electrospun cellulosic nanofibers in various fields.

1   Introduction

Science and technology continue to move toward renewable raw materials, such as plant fibers and biomass materials, and more environmentally friendly as well as sustainable resources and processes [3, 6, 7, 99]. Micron-size, nano-scale fibers exhibit several unique properties, such as higher surface area and surface functionalization [100, 154], and possess excellent mechanical, optical, and electrical properties [243], making them desirable for various applications.

There are many methods for fabricating polymeric nanofiber, including phase separation, drawing, self-assembly, template, and electrospinning [120]. The majority of these techniques suffer from disadvantages, for example, using limited polymer, noncontrollable orientation and diameter of fiber, and noncontinuous process [123]. Compared with other nanofiber production processes, electrospinning has gained considerable attention because of its control of fiber dimension, structures, alignment, and porosity [23]. Electrospinning is a straightforward technique for fabricating nanofibers using polymer solutions or melt in submicron diameters [84, 155, 231]. Briefly, in these processes nanofibers are generated by applying an electric field between opponent electrodes, which causes a whipping motion of solution, creating a stretched, charged polymer jet, evaporation of solvent, and solidifying of the jet to form fiber [20, 183]. The morphology and diameter of the nanofibers manufactured by electrospinning depend on several parameters: voltage, needle-to-target distance, solution concentration, surface tension, viscosity, environment conditions, and so on [90].

Electrospun nanofibers have broad potential for different applications such as sensors, filtrations materials, tissue engineering, and composites [52, 127]; this is because of their high porosity, large specific surface area, and tiny pore size [212]. In recent decades, research has focused on electrospinning of nanofibers [208] and composite nanofibers [13, 50, 137, 255] from natural resources in order to

utilize them in different fields, especially in composite. The key advantages of renewable fillers such as chitin, cellulose, and starch are their sustainability, accessibility, cheap price, small energy consumption, and excellent mechanical properties. Cellulose is an abundant, biodegradable, and biocompatible polymer [142] that recently came into researchers' consideration for electrospinning. Despite the above-mentioned properties, electrospinning of cellulose is restricted owing to its nonsolubility in common solvents and thermal degradation before melting [62, 214]. In addition, cellulose nanowhisker (CNW), obtained through an acid hydrolysis process of cellulose, is recognized as a new nano material to reinforce other materials [172]. Cellulose does dissolve in some solvents, such as lithium chloride-dimethylacetamide (LiCl/DMAc), N-methylmorpholine-N-oxide (NMMO), and ionic liquids (ILs) as a "green" solvent [1]. Obviously, each of these solvents has its own disadvantages. For example, NMMO requires elevated temperature and washing to eliminate residual NMMO; it also produces fibers with low uniformity [80].

The aim of this chapter is to investigate developments in the field of electrospinning of cellulose and cellulose composite nanofibers. Electrospun cellulose nanofibers have potential for use in a wide range of applications.

2   Electrospinning

2.1   Process

There are many methods for producing polymer nanofibers, including self-assembly, phase separation, and electrospinning [47, 156]. Electrostatic fiber spinning, or electrospinning, is a novel and interesting process for producing nano- to micro-fibers from solution or melt by electrostatic forces [144, 206]. Recently, it has gained increasing attention because of its simplicity and versatility in fabricating nanofibers [104, 220]. Formhal patented this method in 1934 [260]. Basically, there are two main differences between conventional fiber spinning and electrospinning. In traditional spinning, mechanical forces cause the production of microfibers, whereas electrospinning can produce fibers in nano size by electrical forces [221]. Electrospinning confers unique properties to nanofibers, such as large surface area, lower structural defect, and enhanced mechanical properties [88, 113]. Generally, the electrospinning apparatus comprises three main parts: a target to collect nanofibers, a high-voltage electric source that provides electric force for drawing the jet, and a syringe pump to carry the polymer solution [110]. Figure 12.1 displays a schematic of an electrospinning setup.

In a typical process, an electrostatic field between needle and collector is provided by a high-voltage electric source. Because of surface tension, the polymer drop remains at the tip of syringe [199]. High voltage induces charges to the solution [210]. In addition, ions act as an element to carry charges [188]. The polymer solution under the effect of electrostatic fields forms a conical-shaped droplet known as a Taylor cone at the tip of the capillary. At a critical value of applied voltage, electrostatic force overcomes the surface tension of the droplet and
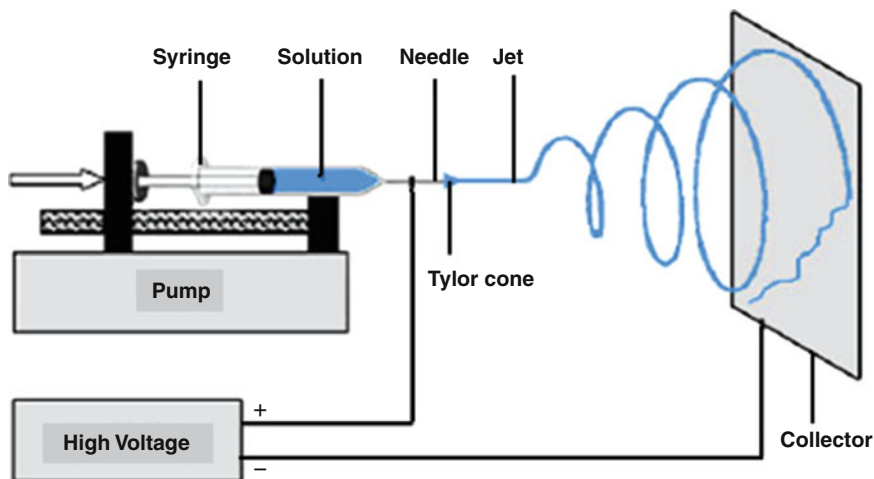
Fig. 12.1 Schematic of an electrospinning setup [259]

a fine, charged jet erupts from the cone tip [32, 118]. The polymer jet is only stable close to the tip of the capillary, after which it undergoes bending instability [182]. The electric field controls the path of the charged polymer jet. The jet is enlarged through spiraling loops. As the diameter of the loops grows, the jet becomes smaller in diameter and longer in length [43]. Finally, the solvent evaporates and solid nanofibers are deposited at the target [215]. Different structures of electrospun nanofibers, such as hollow, aligned, core-shell, and multilayer, can be fabricated by some modifications in the electrospinning setup [218].

Various forces impact the appearance of electrospun nanofibers. Viscoelastic forces are affected by solution concentration and viscosity as well as polymer molecular weight. Surface tension of electrospun nanofibers depends on surface tension of the solvent, average molecular weight of the polymer, and solution concentration. Gravitational forces are determined by the density of solution. Electrostatic forces change with electrostatic field and the conductivity of solution [235]. Columbic forces push apart neighboring charged species in the jet and cause drawing of the jet, while drag forces act between surrounding air and the jet. It is important to note that viscoelastic forces prohibit the jet from elongation, surface tension prevents from drawing of surface of the polymer jet, and electrostatic force is responsible for transferring the charged jet from the nozzle to target [158].

2.2   Parameters

One of the most important issues with regard to electrospinning is the morphology of resultant nanofibers, which depends on many factors. On the whole, these factors can be classified into three categories: processing parameters (voltage, flow rate, distance from nozzle to collector), solution characteristics (viscosity, polymer molecular

weight, concentration, surface tension, electrical conductivity), and ambient parameters (atmospheric pressure, temperature, and humidity) [60, 222, 226]. Morphology and characterization of electrospun nanofibers can be controlled by these conditions.

Polymer chain entanglement and, consequently, viscosity of the solution are determined by molecular weight. Furthermore, molecular weight is a crucial factor in choosing the lowest polymer concentration for electrospinning [217]. In general, when the molecular weight increases, the viscosity will be raised, and, therefore, higher chain entanglement leads to formation of a continuous polymer jet rather than droplets or beads [95]. Beads are generated in some conditions during the electrospinning process. Solution viscosity, surface tension, and charge density are the most important factors influencing the formation of beads [129, 140]. Spinnability of a polymer solution is determined by solution concentration. Concentration also has an effect on the surface tension and the viscosity of a polymer solution. Solutions that are too dilute form droplets, as a result of surface tension, prior to reaching the collector. On the other hand, polymer solution that is too concentrated cannot be electrospun because of high viscosity. Therefore, optimum concentration is required for producing nanofibers [207].

Solution viscosity plays an important role in determining the morphology and diameter of fibers. Generally, a solution with higher viscosity yields fibers with larger diameter. It has been documented that when viscosity is very low, no continuous fiber will form; with very high viscosity, the jet will eject with great difficulty. Maximum viscosities for spinning from 1 to 215 P have reported by researchers [28]. On the whole, if all other conditions remain constant, surface tension will determine the upper and lower limitations of electrospinning. Lower surface tension, which and is preferred for electrospinning, leads to lower electrostatic fields and helps to obtain fibers without beads [79].

The electrical conductivity of a solution is an important factor in electrospinning [115]. It reveals the charge density and repulsion in the polymer solution and, therefore, the amount of elongation a jet. Greater conductivity can cause the jet to carry more charges and smaller-diameters fibers will form [79, 173]. There is not a general pattern to follow for choosing specific values of parameters such as viscosity, conductivity, concentration, and surface tension because they change from one system (polymer-solvent) to another [75].

Choice of solvent is critical from two points of views: quick solvent evaporation and phase separation, which occurs before the fiber is deposited on the collector. Thus, the vapor pressure of solvent influences the rate of evaporation and the time required for drying of nanofibers [210].

Applied voltage or field strength is one of the most crucial parameters in the electrospinning process and has been studied widely. Increasing voltage causes a higher electric field. The effect of raising voltage on the diameter of resultant fibers is a controversial topic in electrospinning [42]. Several researchers have reported that increasing applied voltage leads to formation of thinner fibers because of increasing electrostatic forces [101, 160, 232]. Surprisingly, some authors showed that thicker fibers are produced by increasing voltage [46]. Other researchers have stated that there is no significant correlation between

Table 12.1 Effect of various electrospinning factors on the morphology of fibers [28]

Parameters	Effect on fiber morphology
Solution parameters	
Viscosity	Low causes bead generation; high increases fiber diameter, beads disappear
Concentration	Increase in fiber diameter with increase of concentration
Molecular weight	Reduction in the number of beads and droplets with increase of molecular weight
Conductivity	Decrease in fiber diameter with increase in conductivity
Surface tension	No conclusive link with fiber morphology, high surface tension results in instability of jets
Processing parameters	
Voltage	Decrease in fiber diameter with increase in voltage
Distance from needle to collector	Generation of beads with too small and too large distance, minimum distance required for uniform fibers
Flow rate	Decrease in fiber diameter with decrease in flow rate, generation of beads with too high flow rate
Ambient parameters	
Humidity	High humidity results in circular pores on the fiber
Temperature	Increase in temperature results in decrease in fiber diameter

fiber diameter and applied voltage [9, 14, 44]. This inconsistency can be explained as follows: in a conventional apparatus, the solution erupts downwards And, therefore, by increasing voltage, a greater electrostatic force is induced on the jet and the flow rate will boost. When gravitational force is accompanied by electrostatic force, this phenomenon will be reinforced. Sometimes, electrostatic and gravity forces can detach a drop prior to formation of an initiation cone [2]. In addition, increasing the applied voltage increases beaded fiber formation [260].

Distance from the nozzle to the target is one of the most studied parameters in the research. In the region where whipping instability occurs, the travel time and solvent evaporation rate can be changed by altering the distance from the tip to the collector. In short distances, the solvent cannot evaporate fully [108, 154]. Solution flow rate must be studied for the characterization of nanofibers. Typically, thinner fibers can be obtained by a lower solution feed rate. Flow rates that are too high may also cause formation of beads because of insufficient solvent evaporation before reaching the target [173].

A number of investigations have evaluated the effect of solution and processing parameters on electrospun nanofiber morphology. The influences of spinning environment conditions (like temperature, humidity, and pressure) need further study, however. In terms of the effect of temperature, it is accepted that by increasing solution temperature, nanofibers with smaller diameters can be produced because of the reduction in viscosity [229]. Humidity is another parameter that influences the evaporation rate of solvent, and has been observed that higher humidity leads to larger fiber diameter [82]. Table 12.1 describes the overall effect of electrospinning parameters on the morphology of nanofibers [28].

As stated in previous sections, electrospinning is a simple method for producing fibers in nano scale, and therefore many researchers have investigated

Table 12.2 Development of electrospinning

Year	Progress	References
1882	Balance of fluid conducting masses that are charged by electric potential	Lord Rayleigh [184]
1914	Electrical discharge from liquid points	Zeleny [250]
1934	Process and apparatus for fabricating artificial filaments	Formhal [65]
1955	Dispersibility of some liquids in aerosols using high voltage	Drozin [55]
1971	Acrylic microfibers produced by electrospinning	Baumgarten [26]
1981	Study electrospinning using polymer melts	Larrondo and Manley [125]
1996	Study electrospinning of various polymers	Reneker and Chun [187]
1997	Electrospinning of calf thymus Na-DNA with concentration from 0.3 % to 1.5 %	Fang and Reneker [61]
1998	Electrospinning of some polymer/solvent for producing ultrafine nanofibers	Jaeger et al. [96]
1999	Formation of beaded nanofibers in electrospinning of polyethylene oxide (PEO) solutions	Fong et al. ([64]
2000	A mathematical model for analysis of bending instability	Reneker et al. [189]
2001	Utilizing stability theory to forecast when electrospinning takes place	Hohman et al. [86]
2002	Electrospun membrane from cellulose acetate (CA) with three different solvents	Liu and Hsieh [139]
2003	Producing core-shell nanofibers by electrospinning	Sun et al. [213]
2004	Theoretical investigation of allometric scaling laws in various stages of electrospinning	He et al. [83]
2005	Effect of connection between concentration and viscosity on morphology of electrospun poly methyl methacrylate (PMMA)	Gupta et al. [76]
2006	Altering of energy in the alignment of molecules by computer simulation of EVOH (ethylene/vinyl alcohol copolymer) with different solvents	Lu et al. [143]
2007	Electrospinning of conductive microfluid approach to produce core/shell and hollow composite nanofibers by electrospinning	Srivastava et al. [209]
2008	Electrospinning of aligned nanofibers using nonconductive ferrite magnet target	Yang et al. [241]
2009	Co-electrospinning of thermoset polymer	Reddy et al. [185]
2010	Electrospinning of cyclodextrin as a non-polymeric system	Celebioglu and Uyar [37]
2011	Electrospun nylon-6 nanofibers using AFM probe as a tip	Gururajan et al. [77]
2012	Eliminating whipping motion and produce electrospun nanofiber from PEO and polystyrene (PS) with a fast-rotating collector	Kiselev and Rosell-Llompart [116]

about it. Table 12.2 shows the chronological events that take place in the development of electrospinning. It is undeniable that, during recent decades, electrospinning has gained more attention in different fields.

Electrospinning fabricates nanofibers with small diameter and, consequently, large surface area, high porosity, great pore interconnection [102], and higher mechanical

properties [119]. Based on these unique properties, electrospun nanofibers can be utilized in such fields as biomedical [18, 98, 135, 225, 252], filtration [24, 29, 180], sensors [53, 242], catalyst [45], energy and environment [219], and protective cloth [74, 128].

3   Cellulose

Cellulose forms the basic material of all plant fibers. It is generally accepted that cellulose is a linear condensation polymer consisting of D-anhydroglucopyranose units joined together by β-1, 4-glycosidic linkages. Cellulose is thus a 1, 4–βDglucan [164]. The molecular structure of cellulose, which is responsible for its supermolecular structure, determines many of its chemical and physical properties. In the fully extended molecule, the adjacent chain units are oriented by their mean planes at the angle of 180° to each other. Thus, the repeating unit in cellulose is the anhydro cellobiose unit, and the number of repeating units per molecule is half the Degree of Polymerization (DP). This may be as high as 14,000 in native cellulose. The mechanical properties of natural fibers depend on the cellulose type. Each type of cellulose has its own cell geometry, and the geometrical conditions determine the mechanical properties. Solid cellulose forms a microcrystalline structure with regions of high order (i.e., crystalline regions) and regions of low order (i.e., amorphous regions). Cellulose is also formed of slender rod-like crystalline microfibrils. The crystal nature (monoclinic sphenodic) of naturally occurring cellulose is known as cellulose I. Cellulose is resistant to strong alkali (17.5 wt%) but is easily hydrolyzed by acid to water-soluble sugars. Cellulose is relatively resistant to oxidizing agents.

Plant fibers are constituted of cellulose fibers consisting of helically wound cellulose microfibrils bound together by an amorphous lignin matrix. Lignin keeps the water in fibers, acts as protection against biological attack, and is a stiffener to give the stem its resistance against gravity forces and wind. Hemicellulose, found in natural fibers, is believed to be a compatibilizer between cellulose and lignin [81]. The cell wall in a fiber is not a homogenous membrane (Fig. 12.2) [191]. Each fiber has a complex, layered structure consisting of a thin primary wall that is the first layer deposited during cell growth encircling a secondary wall. The secondary wall is made up of three layers and the thick middle layer determines the mechanical properties of the fiber. The middle layer consists of a series of helically wound cellular microfibrils formed from long-chain cellulose molecules. The angle between the fiber axis and the microfibrils is called the microfibrillar angle. The characteristic value of microfibrillar angle varies from one fiber to another. These microfibrils have typically a diameter of about 10–30 nm and made up of 30–100 cellulose molecules in extended chain conformation that provide mechanical strength to the fiber.

The properties of cellulose fibers are affected by many factors, including variety, climate, harvest, maturity, retting degree, decortications, disintegration (mechanical, steam explosion treatment), fiber modification, textile, and technical processes (spinning and carding) [224]. In order to understand the properties of natural fiber-reinforced composite materials, it is necessary to know the mechanical, physical, and
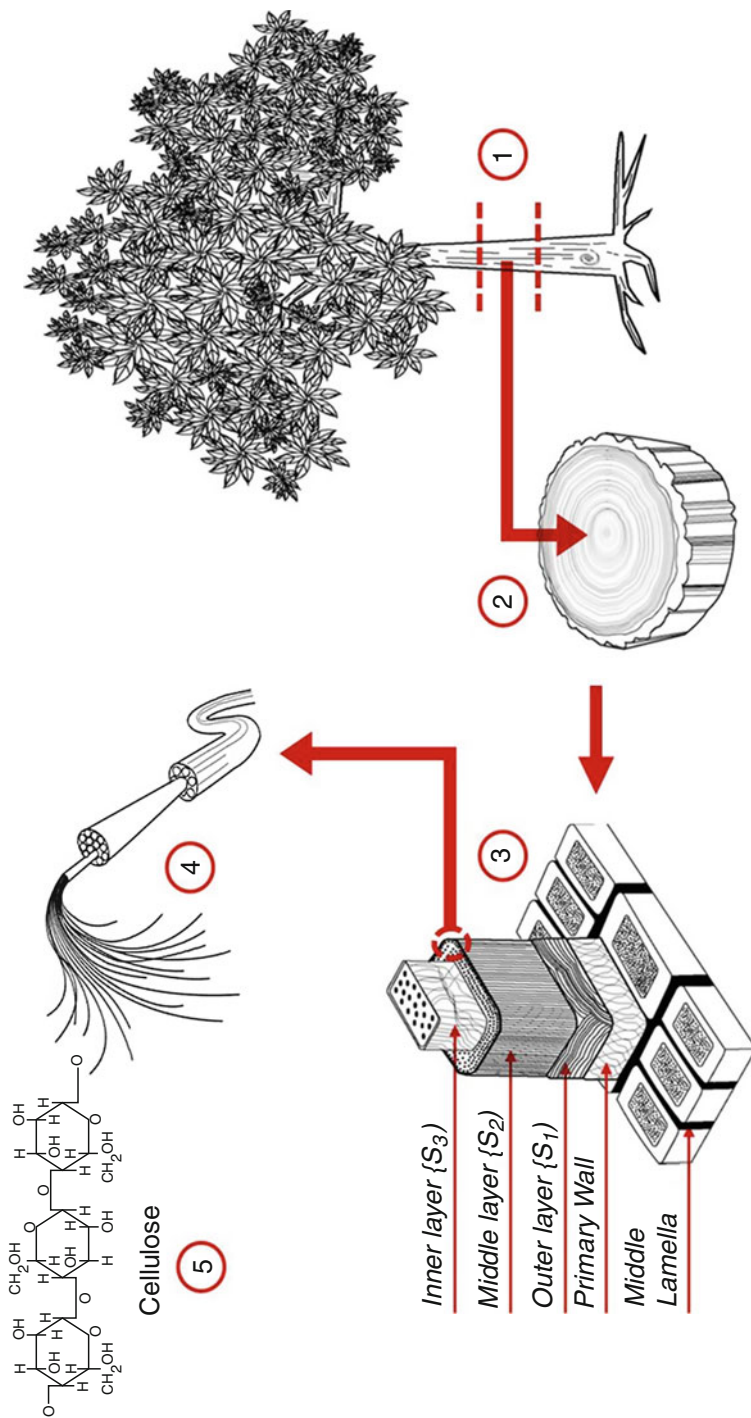
Fig. 12.2 Microscale to fiber bundles to cellulosic nanofibrils from plant fibers

chemical properties of natural fibers [4, 5]. Flax fibers are relatively strong fibers as compared to other natural fibers. The tensile strength of elementary fibers is in the range of 1,500 MPa and for technical fibers a value of approximately 800 MPa was observed at 3-mm clamp length [33]. Baley [25] and Lamy and Baley [124] investigated the modulus of flax fibers. The modulus of elementary fibers is dependent on the diameter of fiber and it ranges from 39 GPa for fibers having diameter approximately 35 μm to 78 GPa for fibers having 5-μm diameter. This variation is related to the variation in relative lumen size between fibers having different diameter. An average Young's modulus of 54 GPa was observed after numerous tensile tests on single flax fibers and the results are within the range of moduli measured on technical fibers. The mechanical, chemical, and physical properties of plant fibers are strongly harvest dependent, influenced by climate, location, weather conditions, and soil characteristics. These properties are also affected during the processing of fiber such as retting, scotching, bleaching, and spinning [97, 230].

4   Electrospun Cellulose Nanofibers

4.1   Cellulose Solvent Systems

Renewable biopolymers, particularly cellulose, have been attracted more attention in nanotechnology [70]. Owing to the fact that cellulose cannot be melted, it must be functionalized or prepared into solution form for electrospinning [68]. The hydrogen bond in cellulose is an obstacle for dissolving it in common solvents. However, some solvents, including NMMO/H_2O, LiCl/DMAc, and ionic liquids (ILs), have been utilized to direct dissolution of cellulose for electrospinning process (Lee et al. 2009). Some investigations related to direct dissolution of cellulose for electrospinning process are briefly described here.

4.1.1   *N*-methylmorpholine-*N*-oxide (NMMO)/H_2O

Monohydrated NMMO is probably the most used non-derivatizing solvent for cellulose in industry, and it is used in the manufacturing of man-made fibers [103]. NMMO belongs to the group of one-component tertiary amine oxide solvents for which the first reports on dissolution of cellulose appeared as early as 1939 (Fig. 12.3). It may be misleading to consider tertiary amine oxide solvents as true one-component solvent systems as they usually must be dissolved either in water or organic solvent (dimethyl formamide (DMF) or dimethyl sulfoxide (DMSO)) due to their solid and/or explosive nature at room temperature [31]. On the other hand, NMMO loses its capability to dissolve cellulose upon hydration by two or more water molecules [132].

The mechanism of cellulose dissolution in this family of solvents is accompanied by the strong intermolecular interaction between cellulose and a strong N → O dipole. The interaction may be interpreted as the formation of a hydrogen bond complex with a superimposed ionic interaction, as shown in Fig. 12.4 [63].

The earliest studies of electrospinning cellulose from NMMO water solutions [87] reported formation of fibers between 3 and 10 μm in diameter and noted

Fig. 12.3 Structures of one-component tertiary amine oxide solvents for dissolution of cellulose

NMMO

N-Methylpiperdine-*N*-oxide

Et₃N → O

Triethylamine-*N*-oxide

N,N-Dimethylcyclohexylamine-*N*-oxide
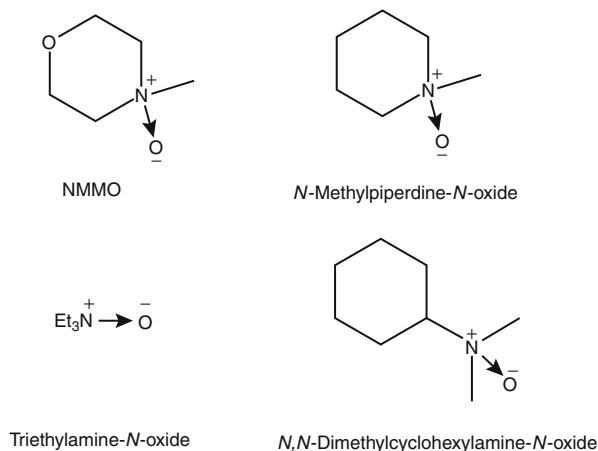
Fig. 12.4 A hydrogen bond complex between cellulose and NMMO during the dissolution

Cell —— O —— H
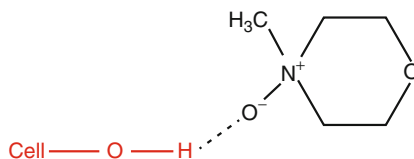
instability of fibers collected directly on a grounded surface. Fibers collected directly on a grounded surface would retain NMMO solvent and, over time, lose fiber shape and revert to film morphology. To overcome this difficulty, fibers were collected in a water bath and coagulated as excess solvent diffused into the bath. Finer fibers, with diameters ranging from 250 to 750 nm, were prepared by making two significant changes to the spinning apparatus [114]. First, the temperature of the spinning unit was carefully controlled between 70 °C and 110 °C, and increasing temperature was correlated with a measurable decrease in the spinning dope viscosity and a corresponding decrease in fiber diameter. Secondly, a rotating disk formed the grounded collector for the fibers.

Another approach to coagulating electrospun cellulose fibers was to use a flowing water bath [121]. Coagulation was found to be limited when fibers were electrospun directly onto a stationary water surface. Fibers would build up on the fiber surface and eventually spread into a film. The addition of a surfactant to the stationary bath allowed fibers to sink but did not prevent film formation. Incorporation of a moving bath prevented electrospun fibers from agglomerating and allowed fibers with diameters between 200 and 500 nm to be collected. Only Khil et al. [109] reported a successful collection of cellulose fibers electrospun from nNMMO/H₂O solution without incorporating a coagulation step. Fibers were successfully collected on a rotating, translating mandrel and washed with distilled water after the electrospinning process was complete.

4.1.2 Lithium Chloride (LiCl)/Dimethylacetamide (DMAc)

As stated previously, LiCl/DMAc is a common solvent system which can be applied for direct electrospinning of cellulose. However, this system has its own benefits and drawbacks. Various sources of cellulose can be dissolved in LiCl/DMAc with different range of concentrations without any side reactions, however, preparation of solution for electrospinning is challenging [202]. Frey et al. reported electrospinning of cellulose in LiCl/DMAc as a solvent system and produced a non-woven mat with diameter 250–750 nm [112]. They pretreated cellulose with water, then solvent was exchanged at 55 °C with DMAc before preparing the electrospinning solution. It was observed that with the combination of heated collector to remove the DMAc and water coagulant to eliminate LiCl, stable and dry cellulose fiber could be fabricated. No obvious degradation and no changes in thermal stability of cellulose were seen because of electrospinning. The authors claimed that these cellulose fibers have potential for filtration. In 2006, Kim et al. used the same process to produce cellulose nanofiber by the solvent system and obtained the same results [114]. They compared the solvent with NMMO/H$_2$O solvent and showed the amorphous structure for LiCl/DMAc in comparison with various amounts of crystallinity in the NMMO/H$_2$O system.

Two years later, a novel method was used to improve orientation of cellulose fibers to improve piezoelectricity of an electro-active paper actuator (EAPap) [247]. The procedure was composed of four steps: solution preparation (cellulose and LiCl/DMAc) and electrospinning, vacuum drying at ambient temperature, filling pores of electrospun cellulose with DMAc solution, and wet-drawing up to 10 % strain. Results of X-ray diffraction (XRD) and scanning electron microscopy (SEM) analysis exhibited greater crystalinity and multi-layer structure of electrospun cellulose compared with a spin-cast film of cellulose [247]. In line with previous work, Lee et al. fabricated electrospun cellulose membrane in LiCl/DMAc solvent at 2 wt% concentration (Lee et al. 2009). Hot air (around 80 °C) was utilized on a rotating collector to remove moisture and solvent. In addition, they used mechanical drawing to increase orientation and crystallinity of the membrane and therefore could use the membrane for an EAPap application.

4.1.3 Ionic Liquids (ILs)

The disadvantages of traditional processes for dissolution of cellulose, such as xanthate and cuprammonium processes, include environmental problems, high cost, use of uncommon solvents with great ionic strength [258], necessity of multiple steps pre-treatment, and prolonged stirring [69]. ILs are organic salts with low melting point, below 100 °C [126, 149]. They are used extensively to dissolve cellulose [186, 253]. Room-temperature ionic liquids (RTILs) consist of an inorganic or organic anion and an organic cation [162]. In 1934, Graenacher discovered that cellulose can be dissolved in molten salt. Some decades later, Roger and coworkers investigated the dissolution of cellulose in ILs, which could be melted in lower temperatures [228].

Several common ILs have been examined to dissolve cellulose and biopolymers: 1-butyl-3-methylimidazolium chloride (BMIMCl), 1-ethyl-3-methylimidazolium chloride (EMIMCl), 1-butyl-3-methylimidazolium acetate (BMIMAc), and 1-ethyl-3-methylimidazolium acetate (EMIMAc) [111, 117]. Liebert and Heinze reported that only ILs with imidazolium, pyridinium, and ammonium cations can dissolve cellulose [136]. In another words, they imply that cellulose only interacts with organic salt including asymmetric cations. Some researchers believe that particularly the anion parts of ILs can disrupt the hydrogen bonding of cellulose and dissolve it [216].

ILs exhibits notable and attractive characteristics, such as very low vapor pressure, great thermal stability, and capability to use at moderate or ambient temperatures. Because of nontoxicity and recyclability they are known as a "green solvents." In addition, by blending different anions and cations, some properties of ILs such as solubility, polarity, and viscosity can be changed [157, 237, 249]. For first time, in 2006, branched and smooth surface electrospun cellulose fibers of micron to nanometer size were prepared using BMIMCl [227]. Briefly, the solution was prepared by heating of IL to 70 °C, adding cellulose, and then irradiation with microwave for 4–5 min. They observed that there is not any N or S in elemental analysis (C, H, N, S) of resultant fibers, which means that no RTIL exists in the fibers using a bath containing ethanol coagulant. A thromboelastography (TEG) test, which measures clotting of human blood, was similar to control sample. Two years later, Xu et al. introduced DMSO as a co-solvent, which reduced surface tension and viscosity, increased conductivity, and improved spinability of a solution including native cellulose with 1-allyl-3-methylimidazolium chloride (AMIMCl) [240]. They also reported that fiber formation can be enhanced by using a rotating collector covered by copper wires and increasing environmental humidity. Furthermore, FT-IR and XRD analysis showed a mostly amorphous structure for electrospun cellulose fibers. Electrospinning of cellulose with AMIMCl was not possible.

After modifying an electrospinning apparatus by adding a chamber with constant temperature to the syringe and also using a water bath for coagulation of fibers, Quan et al. fabricated cellulose fibers with BMIMCl [181]. They claimed that addition of DMSO to the solvent leads to smooth fibers with smaller diameter in comparison to only using BMIMCl. By increasing concentration from 1.5 % to 5 %, the morphology of fibers changed from blocks to thin fibers (at 4 %) and then large fibers (at 5 %). Based on XRD test, crystallinity of electrospun fibers was higher than regenerated films, however, thermogravimetric analysis (TGA) revealed lower thermal stability for electrospun fibers (Fig. 12.5).

The effect of water bath compared with water/ethanol bath to remove EMIMAc solvent from electrospun cellulose fibers was the topic of another investigation [159]. Ribbon fibers that were produced by water bath had lower IL extraction rate and greater surface area compared with cylindrical fibers from a mixture of water/ethanol as coagulant. These ribbon fibers showed a bovine serum albumin (BSA) affinity adsorption capacity. In the same year, Freire et al. added 1-decyl-3-methylimidazolium chloride (C_{10}MIMCl) to the same ionic liquid and obtained
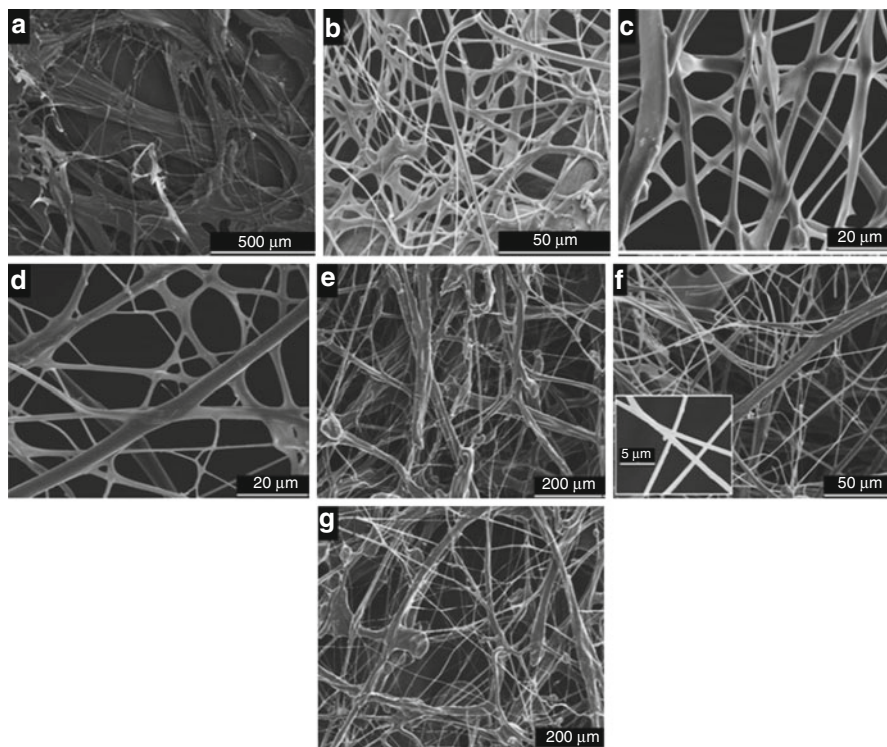
Fig. 12.5 SEM images of electrospun cellulose/BMIMCl with different cellulose concentrations:(**a**) 1.5 wt%, (**b**) 2.4 wt%, (**c**) 3 wt%, (**d**) 4 wt%, (**e**) 5 wt%, (**f**) 4 wt% with adding DMSO, (**g**) 5 wt% with adding DMSO [181]

fibers with more homogeneity and smaller diameters [66]. Fourier transform infrared spectroscopy (FT-IR) and XRD tests revealed lower crystallinity using a binary solvent system and also found that thermal stability of cellulose fibers by EMIMAc and EMIMAc/C_{10}MIMCl solvents was higher than regenerated cellulose casting film. Cellulose electrospun fibers and casting film were mostly amorphous with few crystalline parts.

The effect of three different co-solvents, DMAc, DMF, and DMSO, on the electrospinning of cellulose by EMIMAc was evaluated [78]. The viscosity of system based on DMSO was higher than two other systems, but by decreasing mole of IL solvent, the surface tension decreased. The binary solvent systems containing DMF and DMAc showed the highest and lowest conductivity, respectively. It was observed that the best cellulose fiber was formed by DMSO as a co-solvent. Ahn and co-workers increased spinnability of hemp fibres in EMIMAc by treatment of fibers with sodium hydroxide (NaOH) and sodium chlorite ($NaClO_2$) to reduce lignin content [11]. They obtained thinner fibers with uniform size distribution by decreasing lignin content. When lignin concentration was more than 6 %, only drops were formed. The author, in the same year, demonstrated that,

Table 12.3 Effect of different ionic liquids, coagulant, and collector on the diameter of electrospun cellulose nanofiber

Type of ionic liquid	Collector and coagulant	Nanofiber diameter	References
BMIMCl	Ethanol bath	Micro- to nanometer	Viswanathan et al. [227]
AMIMCl	Aluminum foil, aluminum foil in ethanol bath, rotating drum	100–800 nm	Xu et al. [240]
BMIMCl	Water bath, at constant temperature chamber (100 °C)	500–800 nm	Quan et al. [181]
EMIMAc	Coagulation bath filled with water or water/ethanol	100 nm	Miyauchi et al. [159]
EMIMAc and C_{10}MIMCl	Water coagulation bath	470 ± 110 nm for EMIMAc, 120 ± 55 nm for EMIMAc/C_{10}mimCl solvent	Freire et al. [66]
EMIMAc	Rotating collector submerged in water	–	Hardelin et al. [78]
EMIMAc	Rotating wired cylinder, ethanol as coagulant	–	Ahn et al. [11]
EMIMAc	Rotating wired cylinder, Ethanol as Coagulant	–	Ahn et al. [12]

in spite of the type of co-solvent (DMF or DMAc), higher concentration of co-solvent leads to thinner, uniform, high-crystalline fibers with more thermal stability in electrospinning solution of cellulose with EMIMAc [12]. Furthermore, it was observed that the influence of DMF on the diameter and crystallinity of fibers is more meaningful than DMAc (Table 12.3).

4.1.4 Trifluoroacetic Acid (TFA)

Ohkawa reported the first instance of electrospinning of two types of cellulose (cotton and wood pulp) with trifluoroacetic acid (TFA) or TFA/methylene dichloride (MC) as a solvent at room temperature [168]. The results showed that both solvents had similar effects. Also, FT-IR exhibited no residual solvent in fabrics, and XRD showed an amorphous structure of samples. In addition, electrospinning of pre-spun cellulose loaded with drugs indicated a large surface area of fabrics for releasing drugs and no phase separation between matrix and drugs. In line with Ohkawa's work, Montano-Leyva et al. used TFA, TFA/water, and TFA/acetic acid to prepare electrospun nanofiber from wheat straw [161]. They found that two binary solvent systems produce drops due to failure in solvent evaporation. Nonwoven fibers with tubular aggregates were fabricated by optimum condition of electrospinning (concentration 4 %, distance between needle to collector 7 cm, flow rate 1.5 mL/h, voltage 15 kV) with TFA. It was observed that the glass transition temperature of nanofibers was greater than cellulose, whereas degradation temperature was lower than cellulose. FT-IR showed formation of pure nanofibers and XRD exhibited reduction in crystallinity of nanofibers.

4.1.5 Other Direct Solvents

Ionic liquid solvents for cellulose, including BMIMCl, are touted as environmentally friendly solvents specifically because they are nonvolatile. Because evaporation of the solvent is not a possibility in electrospinning cellulose from ionic liquids, a coagulation bath is necessary. Cellulose fibers with diameters ranging from 500 nm to 10 μm have been formed by electrospinning cellulose from BMIMCl solution into an ethanol bath [227]. Cellulose solvents based on ethylene diamine/ potassium thiocyanate (ED/KSCN) are similar to LiCl/DMAc in constituents of sufficiently volatile solvent and a salt. Diameters of fibers depended significantly on cellulose DP. Fibers spun from high molecular weight (DP. 1000) cotton cellulose had very fine segments with intermittent beads. Similar structures reported in LiCl/ DMAc fibers [67] indicate that the nonvolatile salt component of the solvent may be incorporated in the beads. At low concentrations of lower molecular weight cellulose (DP ¼ 140), only droplets were formed. When a sufficient concentration of the low molecular weight cellulose was incorporated in the solvent, large fibers with a twisting structure were collected.

5 Electrospinning of Cellulose Composite Nanofibers

5.1 Polysaccharides

It is generally agreed that nanotechnology and new products such as nanocomposites from cellulose have the potential of to change the forestry industry and achieve new markets [58]. Cellulose and its particles have properties such as low cost and density, high strength and stiffness, renewability, availability, and biodegradability that make them attractive to use as a reinforcement for other materials [73, 148]. One type of cellulose particle is cellulose nanowhisker (CNW). By dissolving the amorphous region of cellulose via acid hydrolysis, rod-like CNW or cellulose nanocrystal (CNC) [85] with the length of few hundred nanometers and diameter of 5–20 nm can be obtained [27, 57].

5.1.1 Cellulose

Magalhaes et al. reported on a co-electrospinning process to generate nanofibers composed of cellulose in NMMO/H$_2$O in the shell and a CNC suspension in the core, using hot air (∼120 °C) blown on the syringe and a stationary collector immersed in cold water (10 °C) [145]. It was found that with reduction of volume ratio of shell solution to core suspension, voltage and flow rate tend to fabricate individualized fibers. Lower viscosity of NMMO/H$_2$O led to easier electrospinning of CNC suspensions compared with a NMMO/DMSO solvent system. Interestingly, crystallinity and physical characterization of composite nanofibers were lower than that of electrospun neat cellulose. It was observed that absence of CNC causes greater crystallinity indices of FT-IR. The incorporation of this particle as a reinforcing

phase into a matrix and generation of nanocomposites is a result of this obvious improvement in mechanical characterization at very low concentration [35]. Finally, from differential thermogravimetric (DTG) thermograms and FT-IR test's Magalhaes et al. concluded that the concentration of CNC and the formulation of cellulose solution are two critical parameters during co-electrospinning of cellulose/CNC nanocomposites [145].

They also reported on a slightly modified process utilizing a rotating wire collector to fabricate oriented cellulose/CNC composite fibers and to prevent agglomeration [146]. They found that in co-electrospinning of this mixture, suitable dispersion of CNC in cellulose can be obtained by DMSO suspension in comparison with water suspension. It is worth mentioning that reinforced cellulose nanocomposite fibers had lower crystallinity, but greater alignment and adhesion of amorphous cellulose fibers resulted in higher tensile stress and modulus of elasticity of cellulose film reinforced with CNC.

5.1.2 Chitosan

Chitosan is a polysaccharide that is prepared from de-acetylation of chitin [153]. Chitin is extracted from crustaceans (shrimp and crabs) or is produced via a fungal fermentation [239]. Chitosan has promising properties such as good biocompatibility [167], wound healing characteristics, biodegradability, antimicrobial activity [71, 201], chemical-resistance, nontoxicity, and good film-forming ability [208], and has potential to be used in various industries. The rigidity and brittle properties of chitosan, along with its weak solubility, are the main reasons for restricted utilization of this polymer [200]. Because chitosan is powerfully interacting polymer, it can be blended with other materials [36, 59] like polylactic acid (PLA) [171], collagen (Chen et al. 2007), polyvinyl alcohol (PVA) [194], PEO [51], and polycaprolactone (PCL) [203]. Blending of cellulose and chitosan, similar to other mixing systems, yields the desirable advantages of both materials [56]. Because generating electrospun chitosan nanofiber with EMIMAc is difficult (because of its restricted solubility and cationic nature). Park et al. prepared electrospun hybrid nanofibers using EMIMAc, 0.25 wt% chitosan, and 0.5 wt% cellulose [170]. Energy-dispersive X-ray spectroscopy (EDS) nitrogen mapping showed homogeneous dispersion of chitosan on the surface. The micron-size composite nanofibers created a large surface area and can retain a good amount of water as a wound dressing.

5.2   Lignocellulosic Component

5.2.1 Lignin

Lignin is the most abundant phenolic polymer in the plant cell wall, which principally produced by the Kraft pulping method [138]. The two main types of lignin are guaiacyl and guaiacyl-syringyl [106]. Two approaches are applied to incorporate

lignin in polymer materials. The first one incorporates a large quantity of lignin to synthetic polymers, which leads to reduction of the mechanical characterization of the materials. The second approach is the addition of a lower concentration of lignin [178]. In general, lignin is a material with great recovery potential, low cost, low degree of pollution, and availability from different renewable resources [176]. In addition, it reinforces the middle layer of wood-plants and cell walls [8], conducts water, and defends plants against pathogens [196].

Ago and coworkers studied the effect of various compositions of CNC on the electrospinning of lignin-PVA matrix [10]. Lignin-PVA bead-free fibers were generated by weight ratio of 75 to 25. Furthermore, addition of lignin caused an increase in conductivity and viscosity and a decrease in surface tension of this bi-component solution. Three separated domains were obtained by a ternary diagram including beaded fiber, fibers without beads, and macro-size phase separation. Electrospinning of two component lignin-based composite nanofibers using PEO [49] and polyacrylonitrile (PAN) [204] have been reported. In the multi-component case (lignin/PVA/CNC), Ago's investigation revealed that, with addition of CNC, the viscosity and surface tension of solution increased due to the interaction between CNC and lignin, specifically at 75:25 of lignin to PVA [10]. They concluded that this interaction, basically from hydrogen bonding, led to better dispersion of CNC in the lignin-PVA matrix and improved thermal stability of the composite fibers.

5.3   Protein Polymer

5.3.1 *Bombyx mori* Silk Fibroin

Silk fiber is composed of fibroin cores surrounded by sericin [174]. The fibroin consists of heavy-chain and light-chain macro molecules linked together through a disulfide bond [93]. These fibers are rigid and strong, and they are generated from aqueous solution using silkworms at ambient temperature [17]. Some desirable characterizations of silk are low in vivo degradation rate, biocompatibility, easy chemical modification, and luster [190]. In the case of silk nanofibers, high surface area and surface energy, great strength, conductivity. and thermal properties make them good candidates for textile, biomedical. and electrical fields [21].

Huang et al. employed CNW to reinforce electrospun silk fibroin (SF) nanofiber [89]. They observed that the diameter of CNW-SF composite nanofibers is 77–160 nm because of increasing conductivity of solution, whereas in the case of unreinforced nanofibers it is 250 nm. The CNW oriented along the axis of fiber, was well dispersed in the matrix, and uniform and smooth composite nanofibers without bead were generated (Fig. 12.6).

In addition to blending CNW with silk, there has been extensive interest in expansion of nanofibers based on silk with other materials such as carbon nanotubes (CNT) [22, 105], chitosan [34], and hydroxyapatite [236]. Huang and co-workers displayed that the Young's modulus and tensile strength of CNW/SF nanofibers grew with an increase in the CNW concentration due to the formation of hydrogen

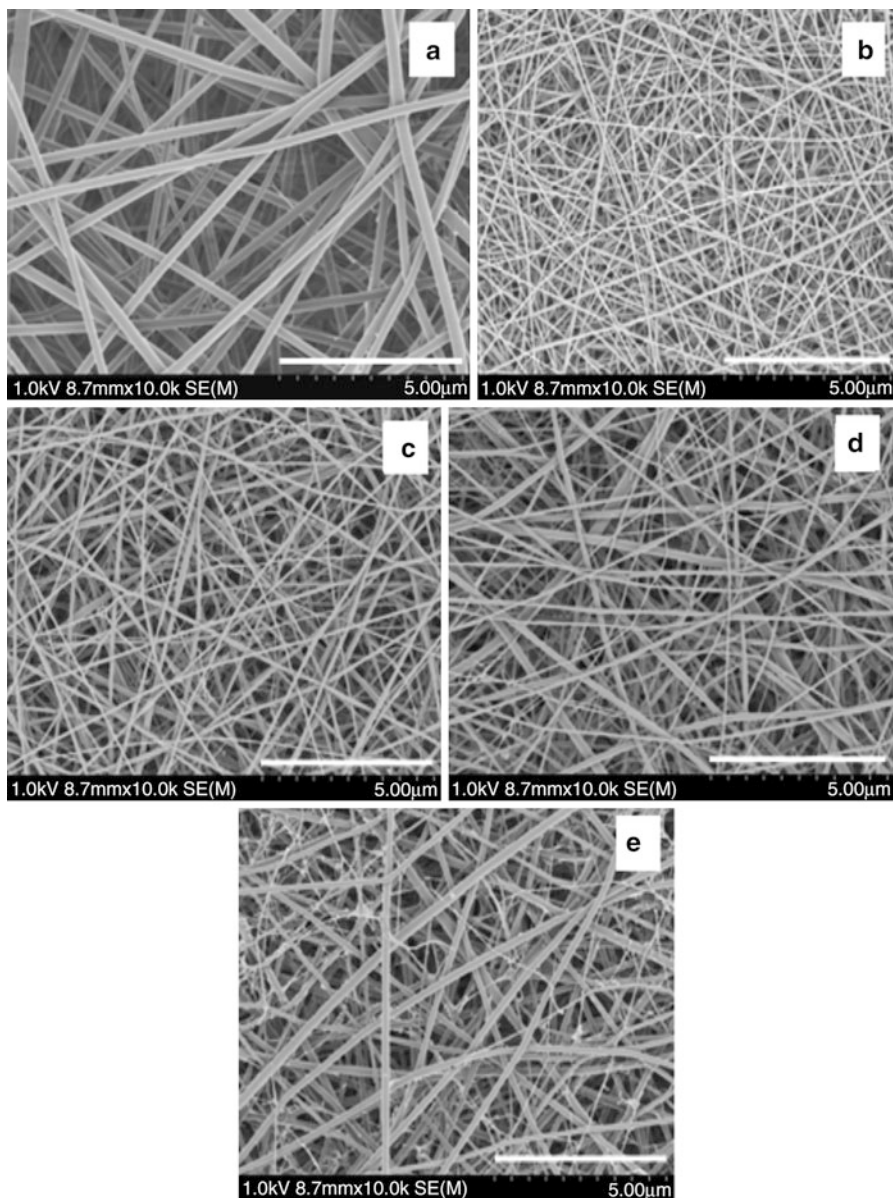**Fig. 12.6** FE-SEM micrographs of electrospun SF nanofiber with different CNW values: (**a**) 0, (**b**) 1, (**c**) 2, (**d**) 3, and (**e**) 4 w/w% [89]

bonds between hydroxyl groups of CNW and hydroxyl, amide, carboxyl, and amine groups of SF and good dispersion of CNW in SF [89]. They reported that the reduction in the strain at break is due to the needle structure of CNW and the semi-rigid structure of cellulose.

5.4 Synthetic Fiber

5.4.1 Meta-aramid

Aramids are classified as strong artificial and heat-resistant fibers [30]. Because of their high rigidity, backbone conjugation, and large amount of hydrogen bonding, meta-aramid (Ar) fibers can be difficult to dissolve in common solvents [244, 256]. Yao et al. prepared Ar-cellulose electrospun composite nanofiber using LiCl/DMAc solvent and compared it with electrospun composite nanofiber of Ar-CA [245]. The homogenous solution of Ar-cellulose and Ar-CA was formed due to new interactions between amide and hydroxyl groups of Ar and cellulose or CA. Also, by increasing the concentration and weight ratio of composite solution, the number of beads was reduced. It was found that higher viscosity and surface tension of Ar/cellulose made it less favorable for electrospinning than Ar-CA, and uniform nanofibers were formed by a solution containing 2:1 Ar/CA and 6:1 Ar/cellulose. Compared with the polymers, composite nanofibers exhibited less stable thermal properties because hydrogen bonds were broken and crystallinity was reduced after dissolving. The Ar/CA and Ar/cellulose showed three and two weight loss processes, respectively. By incorporation of cellulose or CA, new interactions were formed and led to increasing modulus and improvement in mechanical characteristics of composite nanofibers.

5.5 Thermoplastic Polymers

5.5.1 Poly(ethylene-co-vinyl alcohol) (EVOH)

Hybrid CNW and EVOH fibers generated by means of electrospinning have a more uniform morphology than pure polymer electrospun fibers. The treatment of purified bacterial cellulose with sulfuric acid is an effective way to extract cellulose nanowhiskers, consisting of highly crystalline cellulose I structures of nanofibrils aggregates. However, the thermal stability of the repeatedly washed cellulosic material was seen to decrease after the sulfuric acid treatment. A morphology of fewer beaded fibers, with the smallest diameter, was obtained with centrifuged CNWs and an increase in the concentration up to 8 wt%.

Taking into account the relative increase in the glass-transition temperature (Tg) as well as the degree of incorporation of CNW into the electrospun fibers calculated from the FT-IR spectra, it was confirmed that a more effective incorporation of nanowhiskers was achieved when adding them in the form of a centrifuged precipitate without applying sonication [152]. Sonication was found to more efficiently increase the Tg of the composite per filler content, probably due to stronger induced interfacial interaction of the CNWs with the matrix. Additionally, sonication improved the incorporation level of the nanofiller when added as a freeze-dried product, although it produced the contrary effect on centrifuged nanowhiskers. These results highlight the adequacy of the method developed for the incorporation and proper dispersion of CNW into EVOH matrixes, which yielded novel hybrid electrospun fibers.

5.5.2 Poly Methyl Methacrylate (PMMA)

Uniform fibers composed of PMMA reinforced with progressively increasing contents of CNC, up to 41 wt% CNC, have been successfully produced by electrospinning. The morphological, thermal, and nano-mechanical properties of the composite submicron fibers were investigated. The CNCs derived from wood pulp by sulfuric acid hydrolysis were well dispersed in solutions of PMMA and the processing solvent DMF prior to fiber formation. Well-formed fibers with controllable diameters were generated reproducibly at all CNC contents investigated. including 41 wt%. The orientation of the CNCs along the fiber axis was facilitated by the electrospinning process and was observed directly via microscopy examination. Shifts in thermal transitions of PMMA with increasing CNC content suggest hydrogen bonding interactions between CNC hydroxyl groups and carbonyl groups on the PMMA matrix. Nano-scale dynamic mechanical analysis (nanoDMA) was performed using nano indentation on single fibers perpendicular to the fiber axis. Many of the current challenges associated with single fiber nano indentation are addressed, such as fiber diameter range and minimum, depth to diameter ratio, and valid depth range under these experimental conditions. Fibers that contained 17 wt% CNCs showed a modest increase of 17 % in the storage modulus of PMMA, a high modulus polymer of interest for transparent composite applications [54].

The electrospinning process also facilitated alignment of CNCs along the fiber axis, making it feasible to use aligned electrospun fibers as an aid for CNC alignment in polymer composites. Thermal analysis revealed that the glass transition temperature of PMMA was increased with incorporation of CNCs, suggesting hydrogen bonding interactions between carbonyl in the ester groups of PMMA and hydroxyl groups on CNC surface. These studies present a method for incorporating evenly distributed CNCs into fibers, thin films, and composites in a manner that facilitates CNC alignment. The demonstrated alignment of CNCs along the fiber's long axis provides a new approach to orient CNCs with desired directions in polymer composites. For example, it may be feasible to achieve alignment of CNCs or whiskers in both thin-film and bulk polymer composites through application of pressure at an elevated temperature to layers of one-dimensional aligned nanocomposite fiber mats interspersed with the matrix polymer.

5.5.3 Polystyrene (PS)

PS is a thermoplastic polymer. It is a vinyl polymer that is one of the most widely used plastics; only polythene is more common. PS is an inexpensive and hard plastic with limited flexibility, and it is a colorless in pure conditions. The most serious deficiencies are its low impact strength and poor chemical resistance. It can be cast into moulds with fine detail. Solid polystyrene is used, for example, in plastic models, CD and DVD cases, packaging materials, insulation and foam drink cups, toys, and the housings of things like hairdryers, computers, and kitchen appliances. Table 12.4 lists the mechanical properties of PS.

The electrospinning process was performed at room temperature in a solution of cyclohexane, DMF, and tetrahydrofuran (THF) (70 %, 20 %, and 10 %, respectively), while the ratio of SEBS-g-MA to the solution was 10 %. An increase in

Table 12.4 Properties of PS

Properties	Polystyrene
Density	0.00800–2.14 g/cc
Hardness, shore D	36.0–83.0
Tensile strength, yield	10.3–539 MPa
Elongation at yield	0.600–75.0 %
Modulus of elasticity	0.00120–35.2 GPa

water content of the solution and a decrease of feeding rate resulted in deformation of nanofibers and thinning the fibers, respectively. The former was caused by aggregation of CNW and also slower evaporation of solvents in the presence of water contents [19]. In the parallel study, the same polymer as above was electrospun with cellulose CNW using two different methods. The single-nozzle method in which the polymer solution does not have any water content was preferable to the coaxial nozzle method with regard to thermal, mechanical, and morphological properties.

5.5.4 Polyethylene Oxide (PEO)

PEO is a synthetic polyether that is readily available in a range of molecular weights. Low molecular weight (Mw < 1,000) PEO is viscous and colorless liquid, while higher molecular weight PEO is a white solid with melting points proportional to molecular weight, to an upper limit of about 67 °C. It is a water-soluble thermoplastic resin, as well as being soluble in many organic solvents (e.g., methylene chloride, ethanol, toluene, acetone, and chloroform). PEO is used as a thickener in drug delivery, in tissue engineering scaffolds, and in other applications in many industries [257]. A series of PEO/CNC composite nanofibrous mats with different CNC loadings was successfully fabricated via an electrospinning process. A morphological investigation of the obtained nanofibrous mats demonstrated that the transition from homogeneousness to heterogeneousness in their microstructures was achieved by tailoring the concentration of electrospinning solutions from 5 to 7 wt%. PEO/CNC nanofibers became more uniform and finer with the increased CNC content because of the enhanced electric conductivity of electrospinning solutions (Fig. 12.7).

With the decrease in the needle diameter, as-spun nanofibers showed improved size uniformity. The heterogeneous composite mats were composed of rigid-flexible bimodal nanofibers. It was also indicated that CNCs effectively improved the mechanical properties of both types of nanofibrous mats. This was ascribed to the efficient stress transfer from PEO to CNCs, originating from their strong interactions and the uniform dispersion and high alignment of CNCs in the electrospun fibers. When a smaller diameter needle was used in the spinning of homogeneous network mats, enhanced thermal and mechanical properties were obtained. Moreover, the mechanical properties of heterogeneous nanofibrous mats were better than those of their homogeneous counterparts for all compositions (0–20 wt% CNC contents). It was demonstrated for the first time
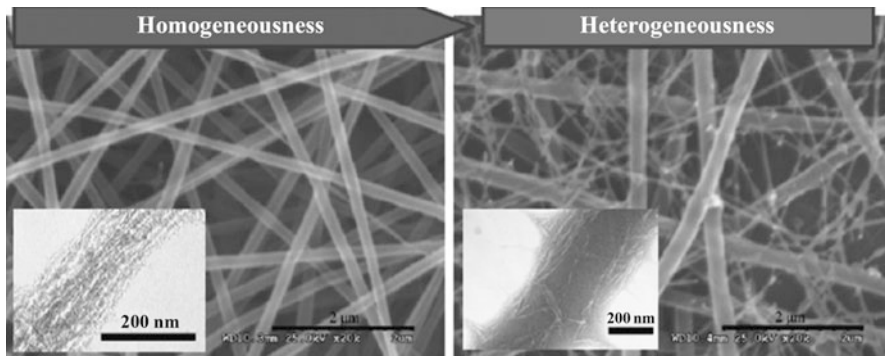
Fig. 12.7 SEM images of electrospun PEO

that the heterogeneous nanofibrous microstructure made of rigid-flexible bimodal nanofibers is especially beneficial to the mechanical properties of electrospun composite nanofibrous mats [75, 195, 223].

5.5.5 Polylactic Acid (PLA)

PLA/CNW composite nanofibers have been successfully produced by electrospinning through mixtures of cellulose whiskers with PLA solution. The effects of CNW on the microstructure and thermal behavior of electrospun PLA nanofibers have been investigated in this study [7]. The PLA/CNW composite nanofibers were successfully produced by electrospinning the mixtures of cellulose whiskers with PLA solution. The diameters of PLA and its composites were around 300 nm. Scanning electron micrographs showed that the CNWs do not protrude out of the outer surfaces of PLA nanofibers. The existence of CNWs in the electrospun PLA matrix nanofibers and the microstructural evolution were investigated using X-ray diffraction. FT-IR analysis showed various functional groups present in the PLA/CNW composites attributing to their chemical interactions [141]. The electrospun PLA and PLA/CNW composites revealed low crystallinity due to the rapid solvent evaporation and relatively slow crystallization kinetics characteristic of PLA. The electrospun nanofibers showed particularly different thermal behavior from that of the solution-cast films.

The nanofibers of pure PLA and PLA/CNW experience two consecutively overlapping crystallization processes. The CNWs act as heterogeneous sites for nucleation of PLA by decreasing the cold crystallization onset temperature. The incorporation of CNW into PLA nanofibers is expected to improve mechanical properties and bring new functionalities to the electrospun matrix nanofibers.

5.6   Carbon Nanotube (CNT)

Since the introduction of the CNT in 1991, it has the subject of many studies [92]. CNTs are fabricated by folding graphene sheets in a cylindrical form [38, 251]. Generally, there are two types of CNT: single-wall carbon nanotube (SWCNT) and

multi-wall carbon nanotube (MWCNT) [166, 246]. The nano diameter of CNT makes it more desirable than micrometer graphite fibers [198]. Geng et al. noticed that CNTs have 10,000 or higher aspect ratio, diameter of a few nanometers, 1–2 TPa elastic modulus, and higher electrical conductivity compared with copper wire [72]. In addition, they can be semi-conducting or metallic based on diameter and wrapping angle [150].

CNTs have potential applications in such fields such as electronics and sensors, specifically as a reinforcement in order to generate new, stronger composite materials [15, 147]. The surface nature of CNTs and interactions between them can cause agglomeration, so good dispersion of CNTs in the polymer matrix is necessary for better mechanical and thermal properties for nanocomposites [151]. In this perspective, various CNT-reinforced polymers have been fabricated by the electrospinning process with aliginate [94], PAN [179, 234], PVA [248], polybutylene terephthalate (PBT) [197], and PCL [192].

5.6.1 Multi-wall Carbon Nanotube (MWCNT)

Dry-jet-wet electrospinning was applied to fabricate smooth-surface white cellulose and black cellulose/MWCNT composite fibers with AMIMCl [254]. The storage modulus and tensile strength of composite nanofibers containing 4 wt% MWCNTs increased 2.5 times and 40 % at ambient temperature. In addition, the greatest conductivity was obtained in this composition. The results also displayed better mechanical properties with increasing temperature. MWCNT are composed of several sheets [48, 122] with graphene layer separation of 0.34 nm, 1 nm diameters, and large aspect ratio [177]. Zhang et al. reported that improved mechanical characteristics and thermal properties were related to good interaction of MWCNT and cellulose and, therefore, good distribution and alignment of MWCNT in the matrix [254].

Preparation of hybrid nanofibers with 200–500 nm diameter composed of 5 wt% bacterial cellulose (BC), AMIMCl, DMSO, and 0.02 wt% acid-treated MWCNTs by the electrospinning process was the main objective of an investigation conducted by Chen and his groups [41]. Acid treatment was done to create functional groups at the surface of MWCNT and to decrease its length. These fibers assembled on rotating parallel wires with a 1-cm gap and coagulated in ethanol. BC is a type of cellulose that can be produced using many microorganisms. Despite the properties of cellulose, BC shows its own particular properties such as great degree of polymerization [205], high purity, and high mechanical performance [163]. Chen et al. demonstrated that pure BC, electrospun BC, and electrospun BC/MWCNT have degradation temperatures around 203 °C, 237 °C and 260 °C, respectively [41]. It was noted that good dispersion, alignment, and addition of MWCNT leads to increasing modulus as well as the tensile strength of the hybrid fibers to around 290 % and 280 %. It was found that the conductivity of the BC/MWCNT composite nanofibers increased.

5.6.2 Single-wall Carbon Nanotube (SWCNT)

The SWCNT consists of a single sheet [165, 211] with 0.4–2 nm diameter, lengths up to 1.5 cm. and aspect ratio of over 10 million [16]. Factorial testing as well as design of experiments (DOE) were two means that Pankonian et al. used to

determine the optimum condition for electrospinning of cellulose and cellulose/ SWCNT in DMAc [169]. For electrospinning of cellulose, they applied hot air blowing (around 90 °C) to the needle, while for cellulose/SWCNT an unheated setup was utilized. Desirable voltage and flow rate for electrospinning of cellulose were 20 kV and 0.90–1.50 mL/h, in contrast to 14 kV and 2.5–4.5 mL/h for cellulose embedded in SWCNT.

6   Applications of Electrospun Cellulose Composite Nanofibers

Over the past few years, increasing attention has been paid to composite materials based on natural fibers as a renewable resources instead of artificial fibers because of their lower cost and environmentally friend nature [107]. Among these renewable resources, cellulose has gained more consideration because of its abundance and notable characteristics [238]. The excellent mechanical properties of cellulose, which are derived from its strong hydrogen bonds [175], lead to increasing interest to incorporate cellulose with other materials for fabrication of composite fibers. As mentioned in previous sections, electrospinning is a simple and versatile method [233] that can be employed to produce continuous, long, highly oriented fibers with a good drawing rate [261], from micron to nanometer [91]. Many applications have been determined for electrospun hybrid fibers—filtration, energy, biotechnology, and sensors, to name a few [193]. Various applications have defined for electrospun cellulose composite nanofibers, including antibacterial textiles for wounds [170], filtration and catalysis [89], precursors for production of cellulose-based carbon fibers [254], and electrical and mechanical applications [41].

7   Conclusion

Recent advances in the field of electrospun nanofibers demonstrate their enormous potential in various applications. In this respect, Cellulose Nanofibers (CNFs) have been examined because of their properties such as renewability, biodegradability, and low cost. The main obstacle that must be overcome for effective electrospinning of cellulose is the need for a superior solvent, inasmuch as cellulose cannot be dissolved in common solvents. However, the use of different solvent systems like NMMO/H_2O, LiCl/DMAc, and ionic liquids has shown that electrospinning of cellulose is possible. A cellulose solution for electrospinning is prepared by dissolving a suitable amount of cellulose samples in various solvent systems and stirring until a homogenous solution is obtained. In spite of significant improvements, a comprehensive study is needed to overcome the disadvantages of the current solvent systems, including environmental problems, the high temperature required for electrospinning, and lack of control.

Cellulose's strong hydrogen bonds and the high surface area to volume ratio in nano cellulose lead to high mechanical strength of this material. For this reason, electrospun cellulose hybrid nanofibers have attracted much attention.

Briefly, solutions for electrospinning of cellulose composite nanofibers are prepared by incorporating the desired weight percentage of cellulose with various matrixes and suitable solvents, and then stirring to obtain an entirely transparent solution. Although much research has been concentrated on this concept, it is still a new subject that needs further studies.

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