



Lignin-Based Electrospun Carbon Nanofibers

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The article summarizes the scientific progress that has occurred in the past several years in regard to the preparation of carbon nanofibers from lignin as a low-cost environmentally-friendly raw material using electrospinning. It presents an overview of using lignin, electrospinning, and carbonization to convert lignin to carbon nanofibers. Lignin is a renewable source for carbon material, and it is very abundant in nature. It is mostly produced as a byproduct from the paper industry and biomass fractionation. Despite its extensive availability and beneficial properties, only a few studies have reported on its use in electronic applications. Lignin-based carbon nanofibers have a high surface area, high porosity, and good electrical conductivity; thus, it is proposed that they are suitable for future energy storage applications.

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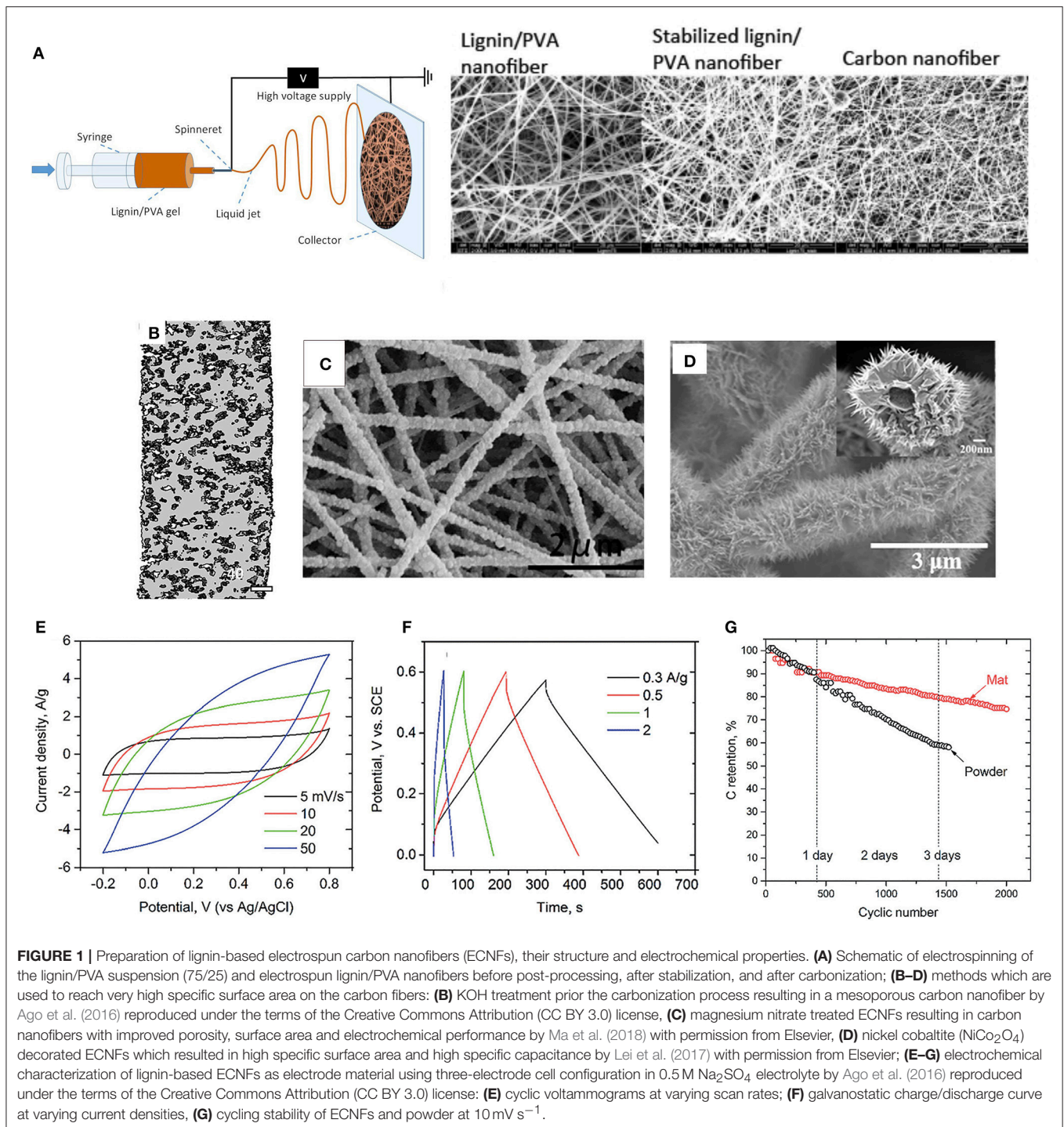
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INTRODUCTION

Electrospun carbon nanofibers (ECNFs) are part of a technological revolution due to their operational simplicity and extraordinary properties, such as being lightweight, having a high surface area, and exhibiting good thermal and electrical conductivity (Lallave et al., 2007). ECNFs have a wide range of applications in areas such as energy conversion and storage (Peng et al., 2016), sensing and diagnostics (Huang et al., 2010), aerospace (Wu et al., 2013), and micro/nano electromechanical systems (Arshad et al., 2011). However, the widespread use of ECNFs is limited due to the high cost of the precursor material and the low yield. Currently, polyacrylonitrile (PAN), produced from depleting petroleum resources, is the prevalent precursor material used to produce ECNFs (Inagaki et al., 2012). The solvents required to convert electrospun nanofibers from the PAN precursor polymer, i.e., dimethylformamide (DMF), are toxic. Therefore, it is essential to develop an alternative, greener precursor material based on renewable plant-based materials.

Lignin, a natural polymer present in lignocellulosic plants, is one of the best substitute materials for producing carbon nanofibers (Baker and Rials, 2013). According to a recent research report, the lignin market is growing at a compound annual growth rate (CAGR) of 2.2%, and it is estimated to generate revenues of 913.1 million USD by 2025 (Grand View Research Inc., 2017). Using the electrospinning method, dissolved lignin suspensions can be processed into submicron and nanoscale fibers. After Lallave et al. (2007) reported the use of electrospinning for fabricating lignin-based carbon fibers, the number of studies related to converting lignin into carbon nanofibers has steadily grown. The recent progress in lignin-based carbon nanofibers has substantially increased the intention of using them for technical applications, such as energy storage (dye-sensitized solar cells [DSSC], batteries, capacitors, and supercapacitors) (Fang et al., 2017). An ISI Web of Science literature search showed that, currently, only 61 research papers have been published on lignin, electrospinning, and carbon fibers. Researchers in Republic of China, the United States, and Canada have been most active in this field.



slow heating and a long duration are recommended during the stabilization procedure. As an outcome of the stabilization procedure, the size of the fibrous mat shrinks and the color slightly darkens. For example, Lallave et al. (2007) stabilized the fiber mat in air at $0.25^\circ\text{C min}^{-1}$ from room temperature to 200°C for 24 h. Lignin was oxidized and the fiber weight was reduced (81 wt%) due to the release of water. No fusion of the fibers was

obtained after stabilization, and the fiber size (diameter) in the resulting product was similar to the size of the original mat. You et al. (2015) shortened the stabilization time from 38 to 3 h using hexamethylenetetramine. According to Yen and Chang (2016), stabilization is a crucial step because it prevents fiber fusion and rupture in the subsequent carbonization step. In contrast, Cho et al. (2018) reported that it is possible to obtain uniform

lignin-based carbon nanofibers without the thermal stabilization step by adding small amounts of nanocrystalline cellulose.

Carbonization is the final step in the conversion of lignin-based ECNFs. Here, the carbon atoms are cross-linked at temperature ranging from 600 to 1,500°C in an inert atmosphere. The non-carbon elements evaporate as a mixture of different gases in the applied high temperature range. The remaining mat is nearly 100% pure ECNFs. As an outcome of the carbonization procedure, the size of the fibrous mat shrinks more and the color becomes black. Moreover, a decrease in the fiber diameter has been observed as the carbonization temperature increases (Ruiz-Rosas et al., 2010). A higher carbonization temperature has been found to result in a higher degree of structural ordering in carbon (Rodríguez-Mirasol et al., 1996), and lignin-based carbon fibers with higher electrical conductivity were obtained by increasing the carbonization temperature from 600 to 900°C (Aslanzadeh et al., 2017).

GREEN CARBON FIBERS FOR ENERGY STORAGE APPLICATIONS

Lignin-based carbonized carbon fibers have been reported to have a high specific surface area, high porosity, high electrical conductivity, good mechanical properties, high oxidation resistance, and superior thermal stability. These unique properties of lignin-based ECNFs make them ideal resources for low-cost energy storage applications, especially as green electrode materials for supercapacitors. Supercapacitors are considered to be very promising devices for energy storage due to their high power density, long cycling stability/durability, short charging times, and environmental friendliness (Ma et al., 2018). **Table 1** summarizes recent studies on the use of lignin-based ECNFs in energy storage applications showing the material combination used for the electrospinning, the carbonization temperatures, the surface activating agents that were used, the surface area measured by the Brunauer-Emmett-Teller (BET) method, the electrolyte used, and the specific capacitance for energy storage applications.

Lai et al. (2014b) reported on the properties of ECNFs from a lignin-PVA nanofiber mat followed by stabilization and carbonization to be used in supercapacitors. The carbonized mat was found to be highly effective as binder-free electrodes. The gravimetric capacitance of 64 Fg⁻¹ was achieved at a current density of 400 mA g⁻¹ and 50 Fg⁻¹ at 2,000 mA g⁻¹. The membrane showed a capacitance retention of 10% after 6,000 cycles of charge/discharge. Ago et al. (2016) reported on the conversion of lignin-PVA fiber mats into a mesoporous carbon fiber mat for supercapacitor electrode. The fiber mat was stabilized at 250°C; it was then impregnated with potassium hydroxide (KOH), and then carbonized at 900°C. The resulting fiber mat exhibited a high surface area, up to 2005 m²g⁻¹, and a large pore volume, 0.7 cm³g⁻¹ with 70% mesoporosity, which greatly improved the material's electrochemical performance (**Figures 1B,E,F**). The specific capacitance of 205 Fg⁻¹ was measured, and 83% of the capacity could be maintained after 1,500 cycles and **Figure 1G**).

Ma et al. (2016) applied manganese (IV) dioxide (MnO₂) nanowhiskers for the decoration of carbonized ECNFs from the lignin-PVA mat, and then tested this material as an electrode material for supercapacitors. The small fiber diameters of 200 nm and the high surface area of 583 m²g⁻¹ exhibited superior supercapacitor performance with a high gravimetric capacitance of 83.3 Fg⁻¹, as well as an energy density of 84.3 WhKg⁻¹ and a power density of 5.72 kWkg⁻¹, making the material suitable for application in a high-performance supercapacitor. The improved supercapacitor performances were explained by the MnO₂ surface coating, which provided high specific/gravimetric capacitance and a large reaction surface area, resulting in fast reaction kinetics. Same authors, Ma et al. (2018) used magnesium nitrate hexahydrate (Mg(NO₃)₂·6H₂O) as an additive in lignin-polyvinylpyrrolidone (PVP)-based carbon nanofibers resulting in a carbon nanofiber mat with a specific surface area of 1,140 m²g⁻¹, increased mesoporosity (2–4 nm pore size) (shown in **Figure 1C**), and specific capacitance of 248 Fg⁻¹ at a current density of 0.2 Ag⁻¹. They also reported a good cycling stability of 97% capacitance retention after 1,000 cycles.

Deposition of silver (Ag) nanoparticles to lignin-PVA derived ECNFs was reported as a cost-effective and highly efficient electrocatalyst in alkaline fuel cells; the suitable activating agent can provide additional functionality as well as higher crystallinity due to the particle size growth (Lai et al., 2014a). That study observed the electrochemical activity of Ag/ECNFs electrocatalyst systems toward oxygen reduction reaction; they found that the mass activity of 15 wt% Ag/ECNFs system was 119 mA g⁻¹. You et al. (2015) reported the fabrication of lignin-based activated carbon fibers (ACFs), without using an additional polymer, followed by stabilization, carbonization, and steam activation for electric double layer capacitor application. The nitrous oxide (N₂) adsorption and desorption measurement indicated that the ACFs had a high specific surface area of 2,185 m²g⁻¹ and large pores (0.5–1.3 nm range). These favored the good electrical capacitance of 133.3 Fg⁻¹ at a current density of 1 Ag⁻¹. Wang et al. (2017) used iron oxide (Fe₂O₃ and Fe₃O₄) particles to decorate acetic acid lignin-PEO based carbon nanofibers, and then used the carbon nanofibers as an electrode material for a pseudocapacitor. The iron oxide-lignin based composite ECNF showed excellent electrochemical performance due to the reversible multi-electron redox faradaic reactions. The specific capacitance of 72.1 Fg⁻¹ at a constant current density of 500 mA g⁻¹ was achieved. Hu et al. (2014) used a KOH solution to activate lignin-PEO-based carbon fibers as the electrode material for a supercapacitor. The prepared ACFs showed a high microporosity (0.75 nm pore size) and high specific capacitance (344 Fg⁻¹) at a low electrode mass of 1.8 mg and a scan rate of 10 mVs⁻¹. ACFs also showed a low equivalent series resistance (ESR) of 1.71 Ω and outstanding cycling stability. Recently, Lei et al. (2017) decorated nickel cobaltite (NiCo₂O₄) on carbon nanofibers of lignin-PAN blend solution using the hydrothermal method, and then utilized the material as a flexible electrode for an asymmetric supercapacitor (**Figure 1D**). They obtained the following electrochemical properties: a specific capacitance of 1,757 Fg⁻¹ at 2 mA cm⁻² and an excellent cycling stability with

TABLE 1 | Electrospun carbon fibers tested in energy storage applications.

References	Material combination	Carbonization (°C)	Surface activation	BET (m ² g ⁻¹)	Electrolyte	Spec. cap. (Fg ⁻¹)
Lai et al., 2014b	Kraft lignin:PVA (70:30)	1,200	No	–		64
You et al., 2015	Hexamine:Acetic acid lignin (10:90)	1,000	900°C steam	2185	1 M TEMABF ₄ /PC	133
Ago et al., 2016	Kraft lignin:PVA (75:25)	900	KOH prior carbonization	2005	0.5 M Na ₂ SO ₄	205
Ma et al., 2016	Lignin:PVA	1,200	MnO ₂	583	1.0 M LiPF ₆	83.3
Wang et al., 2017	Acetic acid lignin:PEO (30:70)	900	Fe(acac) ₃	–	1 M Na ₂ SO ₃	72.1
Ma et al., 2018	Kraft lignin:PVP (1:2)	800	Mg(NO ₃) ₂ ·6H ₂ O	1140	6 M KOH	248
Lei et al., 2017	Kraft lignin:PAN (50:50)	1000	NiCO ₂ O ₄	–	2 M KOH	1757
Hu et al., 2014	Lignin:PEO (9:1)	850	KOH	–	6 M KOH	344
Kim et al., 2007	PAN:ZnCl ₂	800	No	550	6 M KOH	130
Niu et al., 2011	PAN:PVP	970	CO ₂	531	1 M H ₂ SO ₄	221
Lai and Lo, 2015	PAN:PMMA (50:50)	800	No	248	2 M KOH	195

~138% capacitance retention after 5,000 cycles at 7 mAcm⁻². Comparing these numbers with PAN-based carbon nanofibers in **Table 1** the lignin based carbon fibers show promising electrochemical performance.

Zhao et al. (2018) prepared a flexible carbon nanofiber mat from a lignin-PVA solution and used it as a binder-free counter electrode for DSSC, instead of supercapacitors. The mat was carbonized at 1,500°C, and it was reported to have a relatively high specific surface area of 941 m²g⁻¹; it also provided a maximum photoelectric conversion efficiency of up to 7.6% due to its high specific surface area and low charge transfer resistance, which is of the same magnitude as the conventional and expensive Pt material used for DSSC.

CONCLUSIONS

Developments in the manufacture and properties of lignin-based carbon nanofibers has significantly increased the intention of their use for technical applications, such as green electrode materials for energy storage applications. Green carbon nanofibers prepared using the electrospinning technique have been shown to have a high surface area, high porosity, high electrical conductivity, and superior graphite content. This

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brief literature review provides a step-by-step understanding of the lignin material, the preparation of the spinning solution, the electrospinning parameters, and the conversion process involved in the preparation of green carbon nanofibers. It also includes a concise summary of the recent electrochemical studies on lignin-based carbon nanofibers for energy storage applications. In the near future, a more detailed study about electrospinning of native lignin-based carbon nanofibers needed in order to implement for an exciting possibility as a “greener” electrode material.

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All authors listed have made a substantial, direct and intellectual contribution to the work, and approved it for publication.

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Conflict of Interest Statement: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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