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## A Brief Review on Electrospun Lignin Nanofibres

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**Abstract.** Application of bio-based materials in various fields is currently gaining momentum; and lignin occupies a prominent place among such materials. Integrating lignin with synthetic plastics is an innovative approach in the development of sustainable polymers. However, blending lignin with other polymers is not an easy process because of its brittleness and low dispersibility. Grafting of lignin with poly(methyl methacrylate) via atom transfer radical polymerisation enhances the miscibility of lignin with other polymers. A number of publications describe the applications of electrospun lignin nanofibres in various fields. Often discarded as an unwanted component this material has a vast potential for various applications. With the advent of electrospinning technique, lignin-based nanofibres used as an alternative to conventional lignin due to its exceptional property and thus find use in various biomedical as well as electronic applications. Antimicrobial properties of these nanofibres could be exploited effectively in the current pandemic situation. Numerous reviews have been lately published on lignin and lignin-based materials. Most of them focus on the applications of lignin, that is why the idea of the present review is to concentrate entirely on lignin-based nanofibres prepared by the electrospinning process and consolidate the applications of these electrospun lignin nanofibres.

**Keywords:** electrospinning, lignin nanofibres, carbon nanofibres, kraft lignin, organosolv lignin, alkali lignin.

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## **Краткий обзор по применению нановолокон лигнина, полученных электроспиннингом**

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**Аннотация.** В настоящее время стремительно возрастает использование в различных областях материалов на биологической основе, лигнин занимает важное место среди таких материалов. Создание соединений лигнина с синтетическими пластиками можно рассматривать как инновационный подход в разработке экологически безопасных полимеров. Однако соединение лигнина с другими полимерами – это не простой процесс из-за его хрупкости и неспособности к хорошей дисперсии. Сополимер лигнина с полиметилметакрилатом, полученный посредством радикальной полимеризации с переносом атома, может обладать лучшей смешиваемостью. В ряде недавно опубликованных работ описаны перспективы применения в различных областях нановолокон лигнина, полученных электроспиннингом. Этот многообещающий материал часто выбрасывают как нежелательный компонент, хотя он имеет огромный потенциал для использования в различных сферах. С появлением технологии электроспиннинга получаемые с его помощью нановолокна на основе лигнина благодаря исключительным свойствам стали находить применение в различных областях биомедицины и электроники. Нановолокна, обладающие антимикробными свойствами, могут быть эффективно использованы в нынешней ситуации пандемии. В настоящее время опубликовано множество обзоров, посвященных применению лигнина и материалов на его основе. Идея настоящего обзора состоит в том, чтобы полностью сконцентрироваться на нановолокнах на основе лигнина, полученных в процессе электроспиннинга, и охарактеризовать области их применения.

**Ключевые слова:** электроспиннинг, лигниновые нановолокна, углеродные нановолокна, крафт-лигнин, органосольвентный лигнин, щелочной лигнин.

## Introduction

Lignins are aromatic polymers that occur mainly in secondarily thickened plant cell walls. Lignin is the second most abundant organic polymer on earth after cellulose. It accounts for approximately 30 % of the organic carbon existing in the biosphere and is the main biorenewable source of aromatic structures. It is the major constituent of the plant body responsible for inhibition of diffusion of enzymes into wood and creates strong cell walls in plants. Lignin is a complex amorphous highly branched polymer. It consists of three phenylpropane monomers, coniferyl, sinapyl, and p-coumaryl alcohols which are termed as lignols. These monolignols generate guaiacyl, syringyl and p-hydroxyphenyl residues respectively in the lignin polymer. The composition and ratio of lignols in the structure of lignin is largely influenced by both the nature of a plant and the environment in which the plant dwells. Industrial lignins are mainly classified as kraft lignin, lignosulfonates, organosolv lignin and steam-exploded lignin based on the preparation methods (Calvo-Flores, Dobado, 2010). Following the go green trend, researchers are keenly interested in replacing synthetic materials with bio-based materials, such as cellulose, lignin, tannin etc. (Domínguez-Robles et al., 2020). Due to their high porosity and large surface area, nanofibres have various applications such as air filters (Chang, Chang, 2016), protective clothes (Baji et al., 2020), drug delivering agents (Li et al., 2021), scaffolds (Wang et al., 2018a), supercapacitors (Zhu et al., 2020), catalists (García-Mateos et al., 2018), solar cell electrodes (Zhao et al., 2018) etc. Electrospun lignin nanofibres deserve special attention because of their biocompatibility, biodegradability and magnificent selectivity (Gupta et al., 2015). The nature of electrospun fibres crucially depends on the attributes of the spinning solution, namely its viscosity, electrical conductivity and surface

tension. If viscosity values are higher or lower than the required value, bead formation will occur instead of fibre formation (Kumar et al., 2019). Using a suitable spinning solution with required properties, electrospun nanofibres are produced, then stabilised thermally and carbonised.

MiJung Cho et al. (2020) used flax lignin to fabricate high performance electrospun carbon nanofibre mats. Their work opens a new avenue for utilisation of agricultural residue rich in flax fibre as a source for production of electrospun carbon nanofibres. Jose Francisco Vivo-Vilches et al. (2019) used three different types of lignin to make carbon nanofibres and analysed its ability as an electrode material. All fabricated fibres were subjected to electrochemical reactions and the samples prepared from kraft and phosphorus lignin exhibited superior properties.

Integrating lignin with synthetic plastics can be considered a promising approach in the development of sustainable polymers. However, blending lignin with other polymers is not an easy process because of its brittleness and low dispersion in many composites. Grafting lignin with poly(methyl methacrylate) via atom transfer radical polymerisation improves the miscibility of lignin with other polymers (Kai et al., 2015). Ying Zhao et al. (2018) used lignin as the precursor to develop flexible carbon nanofibre mats which are used as a counter electrode for solar cells. The outstanding features of the electrode are credited to the exceptionally high surface area and low charge transfer resistance of carbon nanofibre mats made of lignin.

Numerous reviews on lignin and lignin-based materials have been lately published. Most of them focus on lignin applications. The present review aims to concentrate entirely on lignin-based nanofibres prepared by the electrospinning process and consolidate the information on the applications of electrospun lignin nanofibres.

### Parameters involved in electrospinning of lignin

As mentioned above, creation of the appropriate spinning solution is very important for production of nanoscale fibres using the electrospinning technique. In 2007, Manuel Lallave et al. (2007) published the first research on the application of this technology to virgin lignin devoid of any binders. In their work, a lignin: ethanol solution of 1:1 w/w ratio with a viscosity of 350 to 400 cPs was used to make lignin fibres by following the coaxial electrospinning technique. The diameter of the fabricated fibre ranged between 400 nm and 2  $\mu\text{m}$ . The scanning electron microscopy (SEM) image of the fabricated nanofibres is shown in Figure 1. In addition, coaxial electrospinning method in a triaxial configuration was used by these authors to produce lignin hollow nanofibres (nanotubes). The flow rates for ethanol sheath: lignin solution: glycerine varied from 0.05/0.5/0.01 to 0.1/1/0.25 mL h<sup>-1</sup>, respectively. The tip-to-collector distance was maintained at 20–25 cm, and an electrical voltage of 12 kV was applied.

The common practice for producing lignin nanofibres with enhanced properties is adding binder polymers along with lignin. It was observed that water soluble polymers, such as polyethylene oxide (PEO) (Wang et al., 2020) and polyvinyl

alcohol (PVA) (Camiré et al., 2020), were ideal as binders in the process of electrospinning of lignin. Lignin-polyacrylonitrile (PAN) solutions in the N, N-dimethylformamide (DMF) solvent improve the electrospinning of lignin (Seo et al., 2011). Seo et al. (2011) observed that upon increasing the lignin content the viscosity of the solution changed, which led to the formation of beads and thicker fibres. R. Ruiz-Rosas et al. (2010) fabricated lignin-derived carbon fibres using platinum (Pt)-doped lignin: ethanol solution for electrospinning. The tip-collector distance was set as 20–25 cm, and the applied electrical voltage was 12 kV. Maintaining such conditions prevented the fibres from reaching places other than the collector, thus facilitating the collection of electrospun fibres. The obtained lignin fibres were thermostabilised in an oxidising environment. Then, the fibres were heated to 200 °C maintaining a heating rate of 0.05 °C/min for about 36 hours. The obtained fibres exhibited high resistance to oxidation because of the lack of surface defects and fairly regular structure. Oxidation resistance of lignin-derived carbon fibres was slightly reduced due to the presence of platinum.

Formation of uniform fibres by addition of poly(ethylene oxide) (PEO) was studied by Ian Dallmeyer et al. (2014). The process

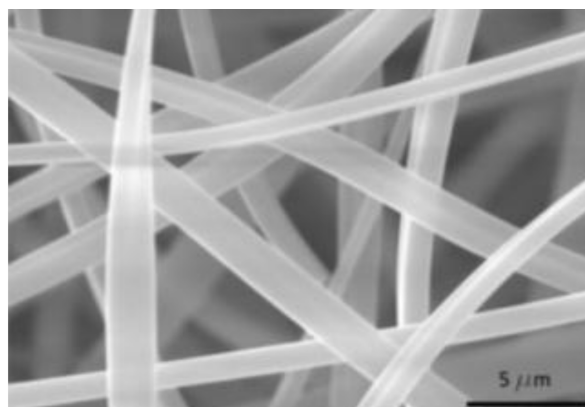


Fig. 1. SEM image of fabricated Alcell lignin electrospun nanofibres (Lallave et al., 2007)

occurred at the tip-collector distance of 14 to 20 cm, the potential difference between 9 and 14 kV, and the continuous flow rate of 0.03 mL/min. The fabricated electrospun fibres were thermostabilised under oxidative condition: they were heated at a constant heating rate to a temperature of 250 °C and held isothermally for 1 hour. The properties of the fabricated sample suggested that lignin polymers have potential applications as precursors for flexible carbon electrodes. Figure 2 illustrates the process of electrospinning of lignin and shows the SEM images of the sample prepared by this method.

### Applications of electrospun lignin nanofibres

Applications of lignin fibres can be significantly improved by converting them to nanofibre structures. Electrospinning is a charge-assisted technique that allows to do it. Lignin nanofibre production is challenging because of the complex lignin's structure, but electrospun lignin nanofibres can be prepared using proper solvent combinations and incorporating secondary supporting polymers. These nanofibres

find applications in various fields of material chemistry (Deeraj et al., 2021) (Table).

Camire et al. (2020) prepared electrospun fibres of alkali lignin with the addition of PVA polymer and investigated the adsorption of fluoxetine (pharmaceutical contaminant) in an aqueous solution. They used different combinations of lignin and PVA and tested their performance. The researchers observed that nanofibres with a diameter of 156 nm adsorbed 70 % of fluoxetine in the tested solution. This accounts for the removal of 32 ppm of contaminants in water.

Salami et al. (2017) obtained polycaprolactone (PCL)/lignin nanofibres by changing the weight percentage of lignin and used them to produce scaffolds for biomedical applications. They observed that the mechanical properties of lignin-loaded samples were better than those of pure PCL samples. The introduction of lignin to PCL scaffolds enhanced their hydrophobicity and porosity. The authors determined that 10 % lignin-loaded samples had better physical, morphological and mechanical properties and were ideal for cell tests. They concluded that these

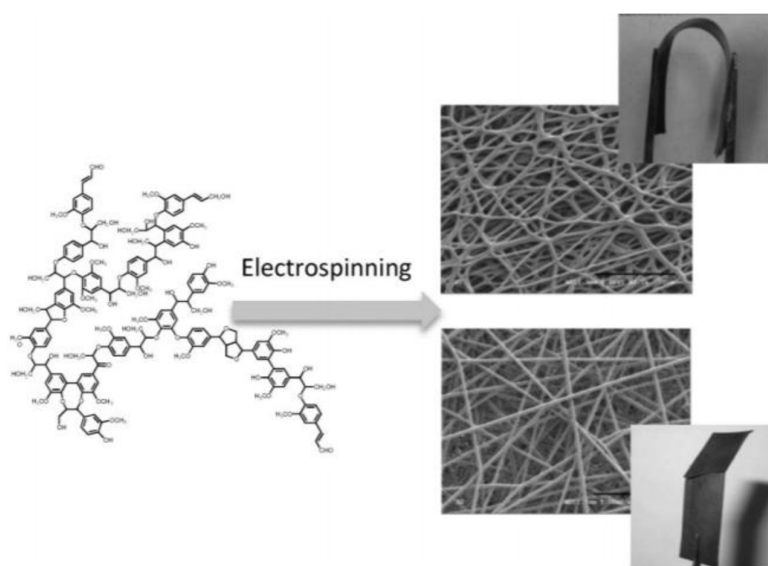


Fig. 2. Images of electrospun lignin derived carbon fibres and their SEM images (Dallmeyer et al., 2014)

Table. Electrospun lignin nanofibres and their applications (PVA – Polyvinyl alcohol, PAN – Polyacrylonitrile, PEO – Poly(ethylene oxide))

Electrospinning material	Solvent	Applications	Reference
PVA-alkali lignin	Aqueous solution of NaOH	Adsorption of pharmaceutical contaminants in wastewater	Camiré et al., 2020
PVA-lignin	Dimethyl sulfoxide (DMSO)	Microelectrodes	Roman et al., 2019
PAN-lignin	N, N-dimethylformamide (DMF)	Supercapacitor electrodes	Zhu et al., 2020
PVA-lignin	Water	Absorbent for water purification	Roman et al., 2019
Silver nanoparticles loaded PVA-lignin	Methanol: Water (60: 40)	Antimicrobial agents	Aadil et al., 2018
PAN-enzyme hydrolysis lignin (EHL)	N, N-dimethylformamide (DMF)	Binder-free supercapacitor electrodes	Wang et al., 2018b
PCL-lignin	1,1,1,3,3,3-hexafluoro-2-propanol (HFP)	Healthcare	Kai et al., 2017
PAN-lignin	N, N-dimethylformamide (DMF)	Supercapacitors	Jayawickramage et al., 2019
PVA-alkali lignin	Water	Counter electrodes of dye-sensitized solar cells	Ma et al., 2016
Kraft lignin-cellulose acetate	Acetone: N, N-dimethylcyclohexylamine (2:1 volume ratio)	Anode for high-performance sodium-ion batteries	Jia et al., 2018
PAN-lignin	N, N-dimethylformamide (DMF)	Electrodes for sodium ion batteries	Jin et al., 2014
PEO-lignin / N-doped (urea)	N, N-dimethylformamide (DMF)	Anode materials for lithium ion batteries	Wang et al., 2013
PEO-kraft lignin	N, N-dimethylformamide (DMF)	Piezoresistive sensors	Wang et al., 2020
Kraft lignin	N, N-dimethylformamide (DMF)	Supercapacitor	Schlee et al., 2019
H <sub>3</sub> PO <sub>4</sub> -lignin	Ethanol	Catalysis	García-Mateos et al., 2018

PCL/lignin composites have excellent potential for medical applications. Aadil et al. (2018) used electrospinning to prepare silver nanoparticle loaded PVA-acacia lignin fibre mats. Examining morphological characteristics of the resultant fibres showed that their average diameter was at nanoscale. The presence of silver nanoparticles was confirmed by XRD. The antimicrobial activity of these fibres against *Bacillus circulans* (MTCC7906) and *Escherichia coli* (MTCC739) was investigated. In both cases, significant antimicrobial activity was observed. The authors concluded that these silver nanoparticle-loaded

fibres are potential materials for wound dressing, membrane filtration, and antimicrobial fabrics. Joo-Hyun Hong et al. (2019) prepared lignin/PCL electrospun mats for antifungal treatment of wood. Pathogenic fungi can deteriorate wood and are responsible for timber loss, whereas prevention of fungal attacks is costly. In their work, lignin/PCL mats were investigated for their anti-fungal properties against the sapstain fungi, *Grosmannia koreana*, and *Ophiostoma floccosum*. The authors report that mat-covered pin sapwood showed better resistance to fungal infection than unprotected sapwood. They also

observed that these mats performed as good mechanical protection against moisture. That is why these environmentally friendly mats can enhance wood trade globally. C.-Y. Chang and F.-C. Chang (2016) focused on studying the performance of lignin fibres as filter media. They produced electrospun mats from lignin and lignin/PEO combinations and investigated the filtration efficiency of these mats. They determined that composite filters created of lignin/PEO mats and surgical mask filter layers had filtration efficiency comparable to that of N95 respirators that filter 95 % of airborne particles. This allowed them to conclude that these lignin/PEO mats have a potential for air filtration applications.

#### **Applications of lignin-derived electrospun carbon nanofibres**

Lignin is a low-cost biosource for preparation of carbon nanofibres (Svinterikos et al., 2020). Carbon materials derived from lignin have multi-functional applications, especially in material electronics (Zhu et al., 2020). Roman et al. (2019) prepared lignin nanofibres from lignin, PVA, and dimethylsulfoxide (DMSO) combinations employing the electrospinning technique. The obtained lignin fibre mats were carbonised, and electrochemical measurements were carried out. They also prepared lignin-based twisted carbon nanofibres by twisting lignin nanofibres precursor mats. The twisting level influenced the electrochemical capacitance (from 330 mF·g<sup>-1</sup> to a few mF·g<sup>-1</sup>) and conductivity (from 11 to 22 S·cm<sup>-1</sup>) of the twisted carbon fibres. They concluded that these twisted carbon nanofibres could be promising candidates as microelectrodes.

Beck et al. (2017) prepared porous lignin carbon nanofibre membranes for applications in adsorptive water treatment. They determined that the prepared nanofibre membranes had the surface area of 583 m<sup>2</sup>/g, pore diameter of 3.5 nm and pore volume of 0.29 cm<sup>3</sup>/g which enhanced

their adsorptive performance. They estimated that energy consumption in water treatment could be reduced by 87 % by using these nanofibre membranes. Zhang et al. (2020) functionalised lignin-derived carbon fibres with air plasma. Prepared by this technique oxygen/nitrogen co-doped lignin fibre electrodes exhibited good specific capacitance (344.6 F/g). In addition, the water contact angle was observed to decrease by 64 %. Thus, these materials display excellent electrochemical performance. Dai et al. (2019) prepared electrospun lignin/polyacrylonitrile derived carbon fibres with nitrogen-sulphur and graphene by electrospinning, carbonisation, and subsequent activation. They found the exceptional supercapacitor performance of the resultant material. They also observed that doped carbon fibres increased the energy density (4.12 to 9.28 Wh kg<sup>-1</sup>) and barely reduced power density of the obtained supercapacitor. Kai et al. (2015) obtained lignin– poly(methyl methacrylate) (PMMA) copolymers using the atom transfer radical polymerisation technique, then blended it with poly( $\epsilon$ -caprolactone) and prepared nanofibres employing the electrospinning technique. Mechanical performance of lignin–PMMA copolymer loaded samples were observed to have improved mechanical properties. The cell culture studies on electrospun nanofibre mats showed that they were biocompatible and helped in the proliferation of cells. The authors concluded that these materials have a potential in biomedical applications. Zhang et al. (2019) investigated the Safranin T (dye) adsorption of alkali lignin/PVA composite membranes. The results indicate that there are strong intermolecular hydrogen bonds between lignin and PVA. The results also show that the capacity of adsorption is improved with an increase in pH and temperature of the dye. Moreover, the absorption exhibited a close proximity to Langmuir isotherm. The kinetics was in accordance with pseudo second order type

models. They also observed excellent desorption behavior and concluded that these results indicate that the prepared composite fibre membrane can be used for dye removal.

### Conclusion and prospects

The review clearly shows a number of ways for effective utilisation of a biowaste material, such as lignin, after its transformation into nanofibres using electrospinning. Through careful choice of the solvent and processing parameters lignin can be converted into nanoscale fibres. Electrospun lignin can also be used as a precursor of carbon nanofibres. Carbonised as well as non-carbonised

electrospun lignin nanofibres are effectively used for various industrial, medical, textile and biomedical applications. The combination of lignin with suitable polymeric substrates often makes the process easier. However, only a few polymers, such as PVA, PAN, PEO etc. are mostly used. There is a vast potential for replacing these polymers with novel biomedical polymers. Fabrication of hybrid fibres using lignin along with other nanofillers is another area that is less explored. The development of these novel lignin derived nanofibres using electrospinning as a tool could definitely increase the applications of lignin nanofibres.

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