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# Product Innovations from Electrospun Nanofibres

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**Abstract**

The article reviews some significant research trends in the development of innovative products from electrospun nanofibres. In one area of investigation, high surface area Poly (Lactic Acid) (PLA)/Tea Polyphenols (TPs) porous Composite Nanofiber Membranes (CNFMs) were prepared successfully by electro spinning and applied to adsorption of silver ions. In another area of research electrospun PVA/$SiO_2$ separator membranes were presented and their electrochemical performance was evaluated for use in Li-ion batteries. Polyvinyl Alcohol (PVA) was used to prepare nanofiber based membranes due to advantages such as low cost, water solubility, and biodegradability. In yet another work, a mixture of Formic Acid (FA), Acetic Acid (AA), and acetone was used, for the first time, as a ternary solvent system to dissolve Poly (E-Caprolactone) (PCL). In addition, as a biomaterial reinforcement, various amounts of Cellulose Microfibrils (CMF) (1.5, 3, and 5wt. %), extracted from rice husk, were added to PCL solution, and subsequently the prepared suspensions were individually electrospun.

**Keywords:** Biocomposite; Electro spinning; Membrane; Adsorption; Polyvinyl alcohol; Battery separator

## Introduction

The environmental pollution is a crucial problem of the era, which arises from industrial activities, for the sake of industrialization chemical, cosmetics, agriculture, mining, textiles, and leather introduced a large number of chemical substances into environment [1-5]. The quality of the environment has also deteriorated due to industrialization, which releases many pollutants into the atmosphere [6,7]. Considering the high toxicity of these materials, the removal of heavy metals has attracted a significant amount of attention.

Owing to a number of merits like high energy density, long cycle life, low maintenance, long shelf life, and low self-discharge Li-ion batteries hold prospects for electric vehicles and portable electronics. However, improving battery safety is crucial to increase application of lithium ion batteries in new energy storage devices [8-11].

Biopolymers have attracted attention since synthetic polymers are creating environmental problems. Many research investigations have been focused in replacing long-term degradable polymers by such eco-friendly polymers in many end uses like packaging, transportation, medicine etc [12-14]. Mostly, biopolymers are biocompatible and biodegradable and decompose without any adverse effects on the environment. Tissue engineering (Li et al., 2014), drug delivery (Ribeiro et al., 2014), and disease diagnosis methods, as subgroups of medicine, are the areas in which biopolymers have attracted a tremendous attention [15,16]. Poly (E-Caprolactone) (PCL), Poly (Lactic Acid) (PLA), Poly (Lacticco-Glycolic Acid) (PLGA) and poly (vinyl alcohol), to name just a few, are examples of biopolymers [17-23]. Among them the former is interest of the current study due to good solubility, low cost, excellent processability and acceptable viscoelastic properties as well as FDA approval holding [24,25].

## Porous Composite Nano Fibre Membranes

Chemical precipitation, membrane filtration, electrochemical methods, ion exchange, adsorption, and so on, are some methods considered in heavy metal removal from water [26,27]. The adsorption is considered to be the most significant of the different methods to remove heavy metal from water, owing to its economy, versatility, energy-efficiency, and requirement of no additional reagents [28,29]. Electrospun nanofibers have been widely used as nanofibrous adsorbents for removal of heavy metals from aqueous solutions, due to their large specific surface area, high porosity, and small interconnected pores [30-35]. A wide variety of low-cost adsorbents such as algae, chitosan, alginate, fungi, Tea Polyphenols (TPs), and lignin were studied to evaluate their potential as viable alternatives to the mostly used expensive adsorbents [36-38]. Among these adsorbents, TPs are mainly consisted of Epicatechin (EC), Epicatechin gallate (ECG), Epigallocatechin (EGC) and Eepigallocatechin gallate (EGCG), in which the EGCG makes up about 50-60 % of the total TPs and possess various biological activities [39]. The multiple pyrogallol and catechol structures of these compounds make TPs highly water-soluble and excellent antioxidant properties [40]. Up to now, TPs have been used as both reducing and stabilizing agents for adsorbing and restoring silver, gold, palladium, and iron nanoparticles, due to their strong surface adhesion ability [41-46]. In this paper, high-specific surface area Poly (Lactic Acid) (PLA)/TPs porous Composite Nanofiber Membranes (CNFMs) were prepared by electro spinning and used in the adsorption of silver ions. The surface morphology of the electrospun PLA/TPs CNFMs before and after adsorption of silver ions were investigated by Scanning Electron Microscopy (SEM). The results showed the average diameters of the nanofibers increased with the increase in TPs. And the porosity of porous CNFMs was higher than that of nonporous CNFMs. And with the increase in TPs, the porosity of nonporous CNFMs decreased. In addition, the adsorption capacities for silver ions of PLA/TPs CNFMs with varying quantities of TPs were confirmed by atomic absorption spectrophotometer. And the amounts of silver element in the CNFMs after adsorption of silver ions were determined using Energy Dispersive Spectrometer Test (EDS). Studies have been carried out on the influences of porous




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structure on adsorption properties of PLA/TPs CNFMs. The results showed that as TPs increased in these CNFMs, the adsorption properties were enhanced. And the porous structure of nanofibers could promote the adsorption of silver ions.

PLA/TPs CNFMs with high specific surface area have been designed by electro spinning and applied to the adsorption of silver ions. The surface morphology and structure, such as nanofiber diameter, porous structure, and porosity, of the electrospun PLA/TPs CNFMs before and after adsorption of silver ions were investigated by SEM and capillary flow porometry. The results showed with the increase in TPs the average diameters of the CNFMs increased [47]. The porosity of porous CNFMs with 0.032g TPs was higher than that of nonporous CNFMs with 0.032g TPs, and the porosity of nonporous CNFMs decreased with the increase in TPs contents. In addition, the adsorption capacities for silver ions of PLA/TPs CNFMs with varying quantities of TPs were confirmed by atomic absorption spectrophotometer. The effects of porous structure on adsorption properties of PLA/TPs CNFMs were investigated. EDS has been used to determine the amounts of silver element in the porous and nonporous CNFMs with 0.032g TPs after adsorption of silver. The results showed that with the amount of TPs increased the adsorption properties were enhanced due to strong surface adhesion ability of TPs. And the porous structure of nanofibers could promote the adsorption of silver ions due to larger specific surface area and higher porosity of porous CNFMs.

## Nanofiber Separator Membranes for Lithium Ion Batteries

The physical contact of electrodes is impeded by separators thereby avoiding short circuiting and simultaneously permitting mobility of Li ions between electrodes during the phases of charging and decharging. The separators demand certain requisites that include specific thickness, suitable pore size, good chemical/thermal stability, and high Liþ ion permeability. Microporous polyolefin based membranes have been used as separators. However, their poor thermal stability and low porosity limit their electrochemical performance. At elevated temperatures, separators with low thermal stability could cause some safety issues. Designing separator with highly porous structure and high thermal stability are crucial for high performance lithium ion batteries [48-51]. Electrospun nanofiber membranes have been presented and superior electrochemical performance was obtained by using highly porous separators. But a number of investigations reported petroleum based polymers including poly acrylonitrile which is highly expensive and non-biodegradable and hazardous solvents have to be used during processing of these polymers [52]. In this study, Polyvinyl Alcohol (PVA), water soluble, biodegradable, and environmentally friendly polymer, was used to prepare highly porous nanofiber based separator membranes and solgel techniques was utilized for the first time to fabricate high performance PVA separators for Li-ion batteries. Incorporation of inorganic particles in separator membranes has been presented as an effective way to improve thermal stability and electrochemical performance coated $SiO_2$ on both sides of polyethylene separator and ionic conductivity was increased up to 8.1104S/cm and better cycling performance was reported [53-55]. $SiO_2$/poly (vinylidene fluoride-hexafluoro propylene)-coated Poly (Ethylene Terephthalate) (PET) nonwoven was presented with improved ionic conductivity and cycling performance compared to bare PET separator [56]. In another study, PVdF/PMMA/$SiO_2$ separator was studied and the capacity was increased up to 158mAh/g as $SiO_2$ was introduced [57]. $SiO_2$ has been coated on both sides of separator [58]. Likewise, researchers have reported on $SiO_2$ nanoparticles/poly (vinylidene fluoride-hexafluoropropylene) layers-coated polyethylene separators [59]. However, high amount of $SiO_2$ loading could cause agglomerations and pulverization, leading to slow kinetics. In addition, introducing $SiO_2$ nanoparticles in polymer solution could cause agglomerations and bead formation resulting in poor cycling performance. In this study, sol gel technique was applied to prepare $SiO_2$ containing PVA fibrous separator without nanoparticle agglomerations and bead formation, resulting in enhanced electrochemical performance.

Electro spinning of water soluble, biodegradable PVA polymer has been used to produce PVA based separator membranes. Electrochemical performance was further improved by introducing $SiO_2$ via sol gel technique. Highly-porous nanofibrous structure was observed and the physical properties including porosity, electrolyte uptake were improved by increasing $SiO_2$ content. The cells containing $SiO_2$/PVA separator membranes showed good cycling and C-rate performance owing to enhanced ionic conductivity and interfacial resistance [60]. It is, therefore, demonstrated that $SiO_2$/PVA separator membranes are promising environmentally friendly separator candidate for high-performance Li-ion batteries.

## Solent based Electrospun Biocomposites

Moreover, its lower rate of degradation than PGA or PLA, satisfies the needs for fabrication of implants, drug delivery devices, scaffolds, and sutures that are supposed to have longer degradability [61]. But, the hydrophobic nature of PCL has been the focus of scientists to overcome this setback by blending with hydrophilic polymers and also incorporation of fillers [62]. Meanwhile, nanofillers from natural resources are charming candidates [63]. In the recent years, cellulose, particularly in the form of nano-scale filler, has pushed a massive part of the scientific activities toward itself [64]. The root of such interest has mostly originated from the growing environmental concerns. In addition, cellulose resources are the most abundant, cheap, and renewable. Biodegradability, biocompatibility, large surface area, high aspect ratio (from 3 to 20 nm in diameter and up to few micrometers in length), high modulus and strength, and lower density compare to inorganic fillers are other distinct characteristics of cellulosic nanofillers [65,66]. Generally, there are various shapes of such particles which depend on the original source (plant) and extraction method of cellulose. Cellulose nanoparticles are classified as nano crystalline cellulose (5-70 nm in diameter and 100-250 nm in length) and micro fibrilated cellulose (5-60 nm in diameter and few micrometers in length) [67]. Rice production, as the main food in many countries, results in rice husk waste which accounts for 20% of raw rice [68]. It was the great motivation for this study to choose rice husk as the rich source for cellulose extraction. Incorporating fillers into polymer matrices leads to creation of polymeric composites. For decades, many attempts have been carried out to develop micro- and nano-porous composite structures, due to their beneficial properties and this ended to great achievements in various applications including filtration, sensor, medicine (e.g. tissue engineering), etc. [69]. The electro spinning is a popular and flexible method for fabrication of structures with submicron porosity in comparison with other porous manufacturing techniques such as gas foaming and phase separation [70-77]. The simplicity, low cost of equipment, controllable morphology, and scalable one-step approach are some rationales to choose this technique for preparing high porous electrospun PCL/CMF biocomposites [78]. Some researchers have attempted to exploit the benefits of both electro spinning and nanoscale cellulose in biopolymers. The reinforcement influence of cellulose Nanocrystals (CNC) into electrospun PLA scaffolds for bone tissue engineering has been studied [79]. In another effort, nanofibrous mats of polyethylene oxide/CNC with heterogeneous and homogeneous microstructures were fabricated and characterized [80]. Recently, three component bio nano composites of PVA/nHAp reinforced by Cellulose Nanofibers (CNF) was developed by for bone tissue engineering [81,82]. Skin tissue engineering application of Polylactide-Polyglycolide (PLGA) nanofiber membranes incorporated by CNC was also investigated [83]. In situ generation of CNC in PCL was carried out via post electro spinning as they firstly prepared electro spunfibers of Cellulose Acetate (CA) and PCL and subsequently CA was deacetylated to CNC by alkaline saponification [84,85]. Further, research workers took efforts to introduce CNC, extracted from ramie, into PCL (dissolved in dichloromethane) and then the electrospun mats were analyzed from morphological, thermal, and mechanical aspects. In the current work, the authors, for the first time, proceeded with the electro spinning of PCL incorporated by CMF and addressed the issues associated with the electro spinning process when CMFs were included [86]. Furthermore, the work innovates a ternary solvent system used as PCL solvent, for the fabrication of electrospun PCL/CMF fibers with uniform morphology. Finally, the developed nanofibrous biocomposites were characterized morphologically and structurally in different ratios of CMF.

A novel ternary solvent system including formic acid, acetic acid, and acetone was employed for preparing PCL solution and the suspensions were incorporated with 1.5, 3, and 5wt. % of CMF content. Using this new PCL solvent system resulted in an optimum PCL electro spinning solution compared with double FA/AA system causing higher quality and thinner PCL fibers. Addition of CMF content increased fiber diameter and broadened fiber diameter distribution which attributed to the viscosity increase and CMFs agglomeration [87]. Apart from SEM morphological characterization, WAXS and DSC measurements were carried out. It was proved that the crystallinity of PCL was enhanced by CMF addition, which was maximized at 1.5wt. % CMF, and then it was reduced at further CMF incorporation. Furthermore, the hydrophilicity and degradation rate of fibrous bio nano composites were explored. It was cleared that CMF addition reduced hydrophobicity of PCL and also fastened its degradation in PBS solution. Regarding the mechanical properties, PCL nanofibers containing 1.5wt. % CMF recorded the highest tensile modulus and strength which were reduced upon higher loading of CMF, while the maximum elongation at break was obtained at 3wt. % of CMF.

## Conclusion

Various techniques such as scanning electron microscopy, energy dispersive spectrometer test, universal testing machine, and so on have been used to study the morphology and properties of the electrospun PLA/TPs CNFMs before and after adsorption of silver. Also, atomic absorption spectrophotometer has been used to determine the adsorption capacities of PLA/ TPs CNFMs for silver ions. The adsorption properties of silver ions by the electrospun PLA/TPs CNFMs has been observed to be good as revealed by the adsorption tests, and the adsorption of silver ions could be enhanced by porous structure. The $SiO_2$ has been incorporated into PVA nanofibers by Sol gel method. The PVA separator membranes have been observed to have better electrochemical properties with highly porous structure. The porosity, liquid electrolyte uptake, and ionic conductivity have been more enhanced by incorporation of $SiO_2$, which on the other hand reduced interfacial resistance. Furthermore, when PVA/$SiO_2$ separator membranes were assembled into lithium/lithium iron phosphate cells, higher cycling and C-rate performance was observed compared to those using commercial microporous polyolefin membrane. Adding acetone to FA/AA solvent system led to fabrication of uniform electrospun nanofibers with the average diameter of 178±38nm. The mean electrospun fibre diameter was increased by introduction of CMF, mainly owing to the increase in the solution viscosity. Further, wider diameter distribution in the presence of CMF has been confirmed by Scanning Electron Microscopy (SEM). In order to investigate the super molecular structure and thermal behavior of fibrous bionanocomposites the electrospun fibers were also analyzed by means of Wide Angle X-Ray Scattering (WAXS) and Differential Scanning Calorimetry (DSC). Because of introduction of CMF the characterizations of both positively affect the crysatllinity of PCL. The maximum crystallinity has been observed by the DSC measurements by introduction of 1.5wt. % CMF. The influence of CMF addition on the hydrophilicity of PCL has also been studied by contact angle measurement, where a reduced trend in contact angle has been noticed after loading CMF. Also, in vitro degradability of the bionanocomposite nonwoven has been investigated in PBS solution. Under the presence of CMF the degradation rate has been improved. Further, tensile mechanical assessment has been conducted and CMF inclusion had a reinforcing impact on electrospun PCL. The maximum modulus and Ultimate Tensile Strength (UTS) have been attained at 1.5wt. % CMF addition to PCL.

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