

Suchitha Devadas

Dr. Erick S Vasquez, Dr. Garry Crosson, Dr. Keny Crosson

## Objective

- The specific objective of this work is to produce nanofiber mats comprised of lignin (low sulfonate content) with polyacrylonitrile (PAN) polymer using an electrospinning process.
- The proposed research will aid in the fabrication of an efficient lignin nanofiber as an emergent green approach in toxin removals from water.
- Produced PAN/lignin nanofibers will also have potential use in many other chemical separation adsorption and other water treatment technologies, which overcome many traditional physical and chemical treatments.

## Introduction

- Emerging contaminants in water, potentially causing detrimental effects on human health and the ecosystem, have become a global issue. To fulfill a raise in freshwater demands treating wastewater and contaminated water to improve its quality to a satisfactory level without causing no harm to the human population is challenging.
- To solve this problem, traditional methods are implemented such as filtration, irradiation, thermal regeneration, and others but have several drawbacks. Henceforth, implementing the efficient process which is cost-effective and easier to operate with lesser or no hazardous intermediates formation is necessary.
- This proposed work aims at fabricating nanofibers using lignin and PAN with an electrospinning technique (**Figure 1**). Lignin is a bio-safe organic polymer, found in cell walls for plants, available as a second abundance macromolecule after cellulose. It is also a by-product from the paper industry and textile industries.
- Electrospinning is an easier and cost-effective approach to the manufacturing of micron-sized and nano-sized fibers. Nanofibers produced have a high surface area, high porosity and have an applications as a filter, medical materials, membranes, sensors, coating material, and many others. Electrospinning setup consists of a syringe pump, a sample collector drum, and a D.C voltage supply.

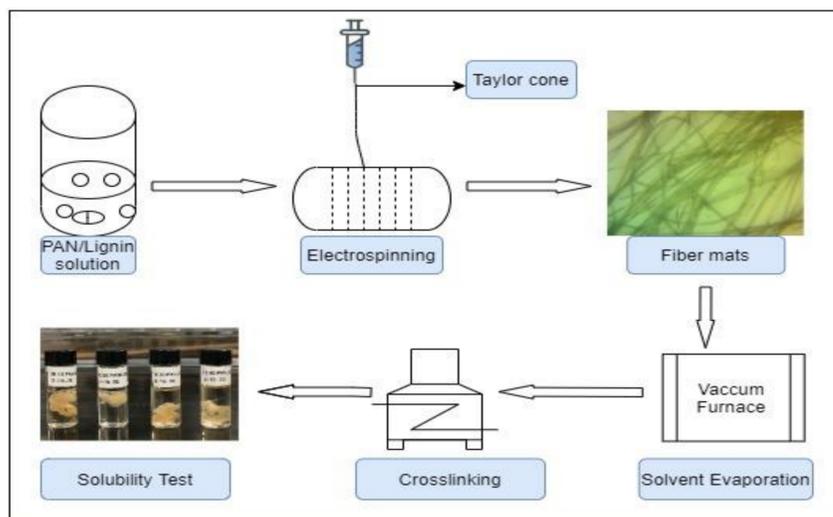


Fig 1: Diagrammatic representation of nanofiber production using electrospinning

- The obtained nanofibers have many potential future applications such as removing pharmaceutical contaminants, dyes, and heavy metals.

## Methodology

- Polymeric solution was prepared by dissolving PAN (Mw=100,000 g/mol) and lignin in N,N-Dimethylformamide (DMF). The weight percentage of the solution components were 10wt% PAN and lignin (weight content ratios are shown in **Table I**), and 90wt% DMF. The mixtures were vigorously magnetic stirred at 50°C for 12hrs until PAN and lignin were mixed fully.
- Electrospinning was performed by loading solution into 10ml of syringe with a feeding rate of 0.003ml/min and 17kV voltage was applied to the spinning solution. The distance between the collector and the syringe pump was 20cm. The samples were collected on rotatory cylindrical drum.
- Optical microscope and scanning electron microscope was utilized to study the morphology of the fibers. Before electrospinning, the fluid rheology was studied using Anton Paar 302 Rheometer to study the fluid viscosity.
- Solvent evaporation of the remaining DMF solvent was removed from fabricated nanofibers in vacuum furnace at 50°C for 4hrs.
- Fiber characterization was studied using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The crosslinking of PAN/lignin fiber was determined using these results.
- Solubility test for both before and after crosslinking of nanofiber mats approximately 3mg were added to a 6ml vial containing purified water. It was shaken well and left for 24hrs to check the solubility of lignin and PAN is insoluble in water by its nature.

Table I: Weight % ratios of PAN to lignin in solution with total polymer content percent of 10wt%

PAN	Lignin
100	0
90	10
80	20
50	50
20	80

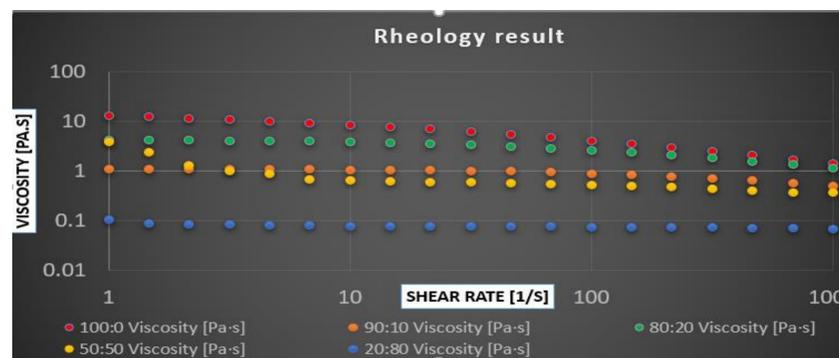


Fig 2: Viscosities of 10wt% total polymer concentration at different PAN/lignin ratios before electrospinning.

## Future Research

- Crosslinking of lignin nanofibers using the thermal analysis study from TGA and DSC to improve the morphology of so far produced nanofibers.
- FT-IR and TEM analysis to study the practical arrangement in fibers to study its physical properties.
- Investigating the solubility ratio of lignin from nanofiber mats using UV-Vis and to study capability of nanofibers to separate contaminants from water under laboratory conditions.

## Experimental Results

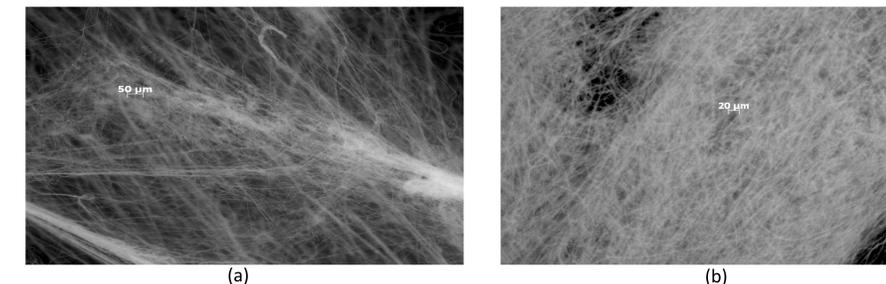


Fig 3: Optical microscope images of obtained PAN/lignin nanofibers (a) 90:10 and (b) 50:50 at magnification scale 20 in a bright field.

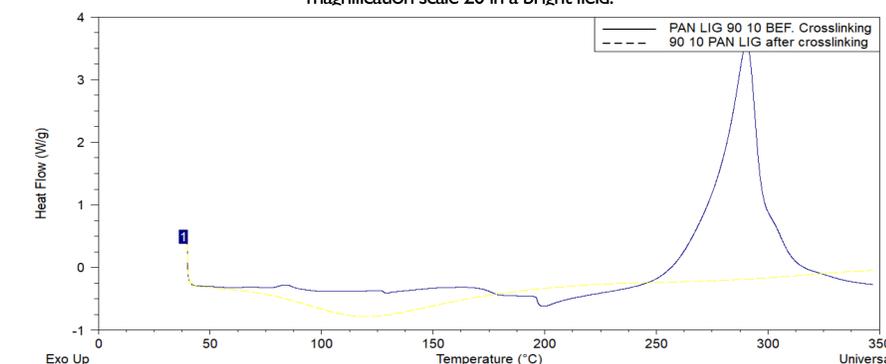


Fig 4: Thermal analysis of produced nanofibers DSC results of 90:10 PAN/lignin before and after crosslinking at 300°C.



Fig 5: Solubility test of produced nanofibers before and after crosslinking in purified water for 24hrs

## Conclusion

- Produced nanofibers over has larger pore size and surface area for separation than traditional technique and also lignin is a bio-safe and cost-effective polymer.
- The viscosity of PAN in DMF was high, but viscosity gradually decreased with addition lignin to PAN and DMF (from Fig 2).
- Based on DSC results, heat treatment of nanofibers was done at 300°C in a tube furnace with a rate of 5°C/min, which stabilized the PAN nanofibers but the lignin nanofibers needs the heat treatment at temperature around 800°C for 1hr to make fiber mats insoluble in water.
- Heat treatment at 300°C the nanofiber color changed from cream to black.
- Nanofibers between 100:0 and 50:50 was insoluble in purified water but, ratios from 60:40 to 20:80 showed solubility of lignin in water (shown in Fig 5).

## References

- Beck, Rika J., et al. "Electrospun lignin carbon nanofiber membranes with large pores for highly efficient adsorptive water treatment applications." *Journal of water process engineering* 16 (2017): 240-248.
- Al-Ajrash, Saja M. Nabat, et al. "Experimental and Numerical Investigation of the Silicon Particle Distribution in Electrospun Nanofibers." *Langmuir* 34.24 (2018): 7147-7152.