Investigation of Opuntia ficus-indica Mucilage Nanofiber Membrane Filtration for Water Systems

Rasudha Muppaneni

University of South Florida, rmuppaneni@mail.usf.edu

Follow this and additional works at: https://scholarcommons.usf.edu/etd

Part of the Electrical and Computer Engineering Commons

Scholar Commons Citation

This Thesis is brought to you for free and open access by the Graduate School at Scholar Commons. It has been accepted for inclusion in Graduate Theses and Dissertations by an authorized administrator of Scholar Commons. For more information, please contact scholarcommons@usf.edu.
Investigation of Opuntia ficus-indica Mucilage Nanofiber Membrane

Filtration for Water Systems

by

Rasudha Muppaneni

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Electrical Engineering
Department of Electrical Engineering
College of Engineering
University of South Florida

Major Professor: Sylvia Thomas, Ph.D.
Norma Alcantar, Ph.D.
Andrew Hoff, Ph.D.

Date of Approval:
March 11, 2015

Keywords: Electrospinning, Arsen, Cactus Mucilage, Polystyrene, Life Cycle Analysis, Poly Vinyl Alcohol

Copyright © 2015, Rasudha Muppaneni
DEDICATION

I would like to dedicate this thesis, and all that comes after, to my family and friends. Thank you for all the love and support you have showered on me. I am grateful to each one of you for guiding me and helping me to reach higher goals and make my dreams come true.
ACKNOWLEDGMENTS

I would first like to thank my major professor, Dr. Sylvia Thomas, for her words of encouragement, and without whom I would not have come this far. I will forever be grateful to my AMBIR group members for always lending a helping hand whenever I needed. I would like to thank all my professors at USF and my undergraduate school. Thanks to Dr. Alcantar and her research group, especially Fei Guo and Daniella Stebbins, for helping me with the equipment procedures. I would also like to thank NSF (Award # 1241582) for funding this project.
# TABLE OF CONTENTS

LIST OF TABLES .................................................................................................................. iii

LIST OF FIGURES .................................................................................................................. iv

ABSTRACT ............................................................................................................................... vii

CHAPTER 1: INTRODUCTION ................................................................................................. 1
  1.1 Thesis Structure ............................................................................................................. 1
  1.2 Background and Motivation ......................................................................................... 1
  1.3 Significance of the Project ........................................................................................... 2
  1.4 Research Objectives ..................................................................................................... 3

CHAPTER 2: MATERIAL OF CHOICE .................................................................................... 4
  2.1 Cactus Mucilage ......................................................................................................... 4
    2.1.1 Composition of Cactus Mucilage ....................................................................... 5
    2.1.2 Preparation of Cactus Mucilage Solution ........................................................ 5
    2.1.3 Calculations for Preparing the Solution ............................................................ 7
  2.2 Poly Vinyl Alcohol (PVA) ........................................................................................... 8
    2.2.1 Composition of Poly Vinyl Alcohol ................................................................. 8
    2.2.2 Preparation of Poly Vinyl Alcohol and Mucilage Solution ................................ 9
    2.2.3 Calculations for the Preparation of the Solution ............................................. 11
  2.3 Polystyrene (PS) ......................................................................................................... 12
    2.3.1 Preparation of Polystyrene Solution .................................................................. 13
    2.3.2 Calculations for the Preparation of the Solution ............................................. 14
  2.4 Polystyrene with Toluene ........................................................................................... 15

CHAPTER 3: PROCESS OF CHOICE ..................................................................................... 17
  3.1 Introduction ................................................................................................................. 17
  3.2 Methodology .............................................................................................................. 18
  3.3 Results ......................................................................................................................... 19
    3.3.1 PVA and Mucilage Solution ............................................................................. 19
    3.3.2 Polystyrene and Mucilage Solution .................................................................. 19

CHAPTER 4: SCANNING ELECTRON MICROSCOPY ................................................................. 21
  4.1 SEM Analysis of Polystyrene: Mucilage Nanofibers ................................................. 21
  4.2 SEM Analysis of PVA: Mucilage Nanofibers ............................................................ 23

CHAPTER 5: VISCOSITY ..................................................................................................... 25
  5.1 Introduction ................................................................................................................. 25
LIST OF TABLES

Table 1 Contact Angle Measurements.................................................................32
LIST OF FIGURES

Figure 1 Composition of Proteins, Monosaccharides and Polysaccharides of Mucilage are (a) Arabinose, (b) Galacturonic Acid, (c) Galactose, (d) Rhamnose and (e) Xylose .................................................................6

Figure 2 Mucilage Solution .................................................................................7

Figure 3 11% Weight/Weight Low Molecular Weight PVA Solution ......................9

Figure 4 Procedure for Preparing PVA and Mucilage Solutions ..........................10

Figure 5 11% Weight/Weight High Molecular Weight PVA Solution ..................11

Figure 6 PVA and Mucilage Solution ..................................................................12

Figure 7 Procedure for Preparing Polystyrene and Mucilage Solution ...............13

Figure 8 Polystyrene Solution .............................................................................14

Figure 9 Polystyrene and D-Limonene Mixed with Mucilage on the Heater with a Stirrer ...............................................................15

Figure 10 Electrospinning Setup .......................................................................20

Figure 11 SEM Characterization of Polystyrene: Mucilage 30:70 .......................21

Figure 12 SEM Characterization of Polystyrene: Mucilage 50:50 .....................22

Figure 13 SEM Characterization of Polystyrene: Mucilage 70:30 .....................23

Figure 14 SEM Characterization of 11% Low Molecular Weight PVA: Mucilage 70:30 Nanofibers .................................................................24

Figure 15 Fungilab Smart L Series Rotational Viscometer ..................................26

Figure 16 Viscosity Measurements for 30:70 Polystyrene: Mucilage ..................27

Figure 17 Viscosity Measurements for 50:50 Polystyrene: Mucilage ..................27

Figure 18 Viscosity Measurements for 70:30 Polystyrene: Mucilage ..................27
Figure 19 Graph Plotted Between Concentrations versus Average Viscosity ........................................28
Figure 20 Spin-Coated Solution of PVA: Mucilage ........................................................................31
Figure 21 Spin-Coated Solution of Polystyrene: D-Limonene ..........................................................32
Figure 22 Filters Coated with Mucilage Nanofibers .........................................................................36
Figure 23 Glass Columns Filled with Sand and Nanofibers .................................................................37
Figure 24 Filtration System for Coated Filters ....................................................................................37
Figure 25 Filtration System for Columns .............................................................................................38
Figure 26 Filter Coated with Only Mucilage ......................................................................................38
Figure 27 Filter Coated with Mucilage under Heat Conditions ............................................................39
Figure 28 PVA: Mucilage 70:30 under Ultra Violet Conditions .........................................................39
Figure 29 70:30 PVA: Mucilage Nanofibers on Filter Paper ...............................................................40
Figure 30 Concentration of Arsenic in Solutions before (45 ppb) and after Filtration Treatment for Direct Filtration using Filter 1, 2, and 4 and Columnar Filtration ..............................................41
Figure 31 Comparison of the Amount of Arsenic Absorbed in the PVA: Mucilage Samples .............42
Figure 32 70:30 Polystyrene: Mucilage Nanofibers ............................................................................43
Figure 33 50:50 Polystyrene: Mucilage Nanofibers ............................................................................43
Figure 34 Comparison of the Amount of Arsenic Absorbed in (a) Control Sand (b) 70:30 PS: Mucilage Nanofibers and (C) 50:50 PS: Mucilage Nanofibers ..................................................44
Figure 35 Fourier Transform Infrared Radiation Spectroscope .........................................................46
Figure 36 Infrared Radiation Spectra of Polystyrene: Mucilage 50:50 ..............................................46
Figure 37 Infrared Radiation Spectra of Polystyrene: Mucilage 30:70 ..............................................47
Figure 38 Infrared Radiation Spectra Mucilage ..................................................................................47
Figure 39 Infrared Radiation Spectra of Low Molecular Weight PVA ................................................48
Figure 40 Infrared Radiation Spectra of PVA: Mucilage 50:50 .................................................................48
Figure 41 Infrared Radiation Spectra of Polystyrene .................................................................................49
Figure 42 Infrared Radiation Spectra of Polystyrene: Mucilage 70:30 ..........................................................49
Figure 43 Infrared Radiation Spectra of PVA: Mucilage 70:30 .................................................................50
Figure 44 Chart to Determine the Functional Groups with their Respective Wavelengths ...............................51
Figure 45 Life Cycle Analysis of Characterization in IMPACT 2002+ .........................................................54
Figure 46 Life Cycle Analysis of Damage Assessment in IMPACT 2002+ .....................................................54
Figure 47 Life Cycle Analysis of the Characterization using BEES V4.02 ......................................................55
Figure 48 Comparison of Components from Characterization using BEES V4.02 and IMPACT 2002+ Methods ..............................................................................................................................................55
ABSTRACT

This work investigates the fabrication, characterization and testing of Opuntia ficus-indica mucilage nanofibers to be utilized in water filtration systems. These mucilage nanofibers are formed using different polymers through a process called electrospinning. The polymers used to promote the formation of nanofibers are poly vinyl alcohol (PVA) and polystyrene (PS). The mucilage is a jelly like substance extracted from the pads of the cactus plant. It is a mixture of proteins, complex polysaccharides and monosaccharides. It is an inexpensive, non-toxic, biodegradable and biocompatible material which is present in abundance. The mucilage extracted from the pads is mixed with acetic acid to form the mucilage solution. The mucilage solution is then mixed by volume with co-spinning polymers, PVA and PS. PVA is a synthetic polymer that is water-soluble, and this work considers two types of PVA differentiated based upon molecular weight, such as low molecular weight PVA and high molecular weight PVA. Polystyrene is a synthetic polymer extracted from a monomer styrene, and it is inexpensive, biodegradable, and abundant. The polystyrene, in its solid form, is further decomposed using a solvent called D-Limonene. D-Limonene is a biodegradable, non-toxic solvent formed from the citrus extract of orange peelings. The PVA and PS solutions are mixed in several different volume ratios with the mucilage solutions. These solutions were electrospun and consistent nanofibers were obtained using the low molecular weight PVA solutions and the polystyrene solutions. The fibers and polymeric solutions were characterized by scanning electron microscopy (SEM), contact angle measurements, viscosity, and FTIR. Resulting mucilage nanofiber membranes were characterized by atomic fluorescence spectrometry (AFS) filtration.
testing. In addition, a life cycle analysis using the SimaPro software was performed to understand the environmental impact of solutions used to fabricate the mucilage nanofiber membranes. Characterization results confirm the formation of PVA:mucilage and PS:mucilage nanofibers. Filtration testing of the nanofiber membranes indicates better performance with membranes formed by PS: mucilage solutions as compared to PVA: Mucilage solutions. Overall, this work has shown that natural materials, such as cactus mucilage, can be synthesized with polymeric solutions to form environmentally friendly water filters.
CHAPTER 1: INTRODUCTION

1.1 Thesis Structure

The first chapter discusses the background, motivation and significance of this project. Chapter 2 elaborates on the materials selected to fabricate nanofiber membranes. Chapter 3 explains the electrospinning process and process parameters. Chapters 4-9 discuss the characterization of the polymeric mucilage solutions and nanofiber membranes. More specifically, chapter 4 looks at fiber diameter and morphology via Scanning Electron Microscopy; chapter 5 explains the viscosity of the solutions; chapter 6 investigate the hydrophobicity via contact angle measurements; chapter 7 elaborates on nanofiber filtering captured from atomic fluorescence spectrometry testing; chapter 8 shows a spectral analysis from FTIR data to help identify components of the polymeric solutions; and chapter 9 presents the Life Cycle Analysis of PVA, PS, and polymeric mucilage materials and the impacts on the environment. Chapter 10 summarizes the results of this project, presents concluding remarks and future works.

1.2 Background and Motivation

The long-term objective of this work is to develop an inexpensive, sustainable water filtration system that is economical, such that people in rural areas can afford to obtain safe consumable water. The natural material driving this technology is a substance called mucilage, which extracted from cactus pads that are available in abundance in all parts of the world. The mucilage has an interesting property of absorbing the harmful chemicals present inside the water like bacteria, E.coli, arsenic. To help maintain global sustainability, researchers need to
investigate ways to clean our water resources [1]. These ways must be inexpensive, natural and non-toxic. We have only 1% of the available freshwater useful for drinking [1]. One out of every 12 people does not have clean water to drink [1]. There are billions of people who do not practice adequate sanitation. Water contains different contaminants such as inorganic compounds like metals (arsenic, lead, sediments), microorganisms (waste, viruses), and synthetic organic compounds (pesticides, herbicides), which need to be removed from the water.

Analysts say that the nanofiber market will emerge rapidly in the next decade as vital components for filtration systems. These nanofibers belong to the nanotechnology family and have unique properties, which can lead to versatile application opportunities in many areas. Nanofibers are used in a vast variety of applications such as filter media, protective coatings, cosmetics, bio devices, sensors, and tissue engineering. Nanofiber membranes are currently being investigated as effective devices for the treatment of contaminated water by the toxic ions and the microorganisms. A cost effective process used to fabricate these nanofibers is called electrospinning.

1.3 Significance of the Project

We chose Oputia ficus-indica also called prickly pear cactus plant, as it is readily available and in all parts of the world. It is studied that this prickly pear can store water in itself and has water purification abilities. It absorbs the harmful chemicals present inside the water and purifies it. Other than water filtration, these cactus mucilage nanofibers can be used in various applications like tissue scaffolding, cell culturing, air filtration, gas filtration, tissue engineering, drug delivery, textiles, enzyme carrier, sensors and many other uses. This research is mainly to investigate if the nanofibers purify the contaminated water and compare them to the industrial filters. This will be helpful for making a water filtration system that can be affordable,
biodegradable and sustainable. This could be used by millions of people across the globe. The nanofibers are obtained through the electrospinning technique. Electrospinning is a simple, reliable and inexpensive method for producing the nanofibers.

1.4 Research Objectives

The research objectives of my thesis are:

• To fabricate and characterize electrospun nanofibers of poly vinyl alcohol (PVA) and polystyrene(PS) solutions formulated using an environmentally friendly solvent D-limonene

• To investigate the filtration capability of fabricated PVA-Mucilage and Polystyrene-Mucilage membranes.

• To estimate the impact of produced nanofibers on the environment.
CHAPTER 2: MATERIAL OF CHOICE

2.1 Cactus Mucilage

The flesh of the prickly pear cactus is called mucilage. This mucilage is a gummy substance that helps us to retain water in the hottest weather conditions. This mucilage is extracted by boiling the cactus pads. When it is added to the dirty water, the larger dirt particles settle out of the water due to high molecular weight of the mucilage. This high molecular weight of the mucilage is because it swells in water. Dr. Alcantar’s research group has performed experiments and has data concluding that mucilage absorbs harmful chemicals inside the water such as bacteria and arsenic [2]. Even the trace of arsenic inside the water can cause serious health problems. This project aims to provide a mechanism to assist in the removal of arsenic from water and make it more acceptable for drinking. The mucilage is of two types:

- Gelling extract
- Non-gelling extract

The non-gelling (NE) mucilage extract was obtained from Opuntia ficus-indica pads by a method which is proposed by F. Goycoolea [3]. The structure of the cactus mucilage changes on exposure to contaminants. Depending upon the pH values, ion content, contaminant concentration, mucilage extraction process, the temperatures at which it is processed, the mucilage is considered to be sensitive and hence the extraction process is very important to follow [6]. The pads are washed with DI water and diced. They are soaked in a NaOH 1% solution and then heated up to boiling point. Once the pads are cooked, they are blended or liquefied. The mucilage mixture at this point has a pH level of 4 and is neutralized to pH level of
7 by NaCl of 1M. The neutralized mixture is then separated from the solids by centrifugation. The solid remaining is used for gelling mucilage extract while the liquid is used for the non-gelling mucilage extract used in this project. The liquid is then filtered for any remaining solids and precipitated with an equal volume of acetone. The precipitate is then dried at room temperature. The resulting precipitate is what is used as cactus mucilage for the following solution mixtures.

2.1.1 Composition of Cactus Mucilage

Mucilage consisting of the proteins, monosaccharides and the polysaccharides is also a clear and colorless compound. The also contain chains of different sugars. Their chemical structures are shown in figure [1]. It is also made of a linear chain of rhamnose, galactose and galacturonic acid with arabinose and xylose combined as side chains. It also contains different sugars that have a capability to interact with the other metals, biological substances and cations. Mucilage is a neutral, the temperature, and irrigation [3]. crop but is also dependent on complex carbohydrate composed of 55 sugar residues including arabinose (67.3%), galactose (6.3%), rhamnose (5.4%), and xylose (20.4%), and a galacturonic acid [3,4]. It contains other organic compounds that give it the ability to interact with metal like K, Ca, Mg, Fe and others [3].

2.1.2 Preparation of Cactus Mucilage Solution

Cactus mucilage is prepared by mixing acetic acid and deionized (DI) water in 50% weight-to-weight ratio. The weights are taken with the help of a weighing balance. Initially a beaker is taken on the weighing balance and is tarred to zero. The weight of water is 1 gram. The weight of acetic acid is 1.3 grams.
Figure 1 Composition of Proteins, Monosaccharaides, and Polysaccharides of Mucilage are (a) Arabinose, (b) Galacturonic Acid, (c) Galactose, (d) Rhamnose, and (e) Xylose [1].

Water is first measured on the weighing balance and acetic acid is poured into the water in 50% w/w ratio. We choose acetic acid to make the solution as it is a weak acid and it is harmless. It can be diluted easily and is biocompatible when mixed with mucilage. The role of acetic acid in the preparation of mucilage solution is to breakdown the carbohydrates such as chitosan and cellulose.

Now take a weighing paper on the weighing balance and tare it to zero. 4% weight-to-weight ratio of mucilage powder is added to the solution of acetic acid and water slowly such that no lumps are formed. The mixture of mucilage, acetic acid and water is heated and stirred with the help of a magnetic stirrer at 600 rpm and 60°C. It is allowed to stir for approximately 10-12 hours. It is covered with the help of a thin parafilm such that the solution won’t evaporate. There would still be small particles left after the stirring. We need to grind the whole solution with the help of a tissue grinder so that a homogeneous solution is formed.
2.1.3 Calculations for Preparing the Solution

The calculations show the amount of mucilage, acetic acid and distilled water used in the preparation of the solution.

\[
\text{Mucilage} = \frac{4}{100} (\text{Mucilage} + \text{acetic acid} + \text{DI water})
\]

\[
\text{Mucilage} = 0.04 (\text{Mucilage} + \text{acetic acid} + \text{DI water})
\]

Acetic acid: DI water = 1:1

\[
\text{Acetic acid + water} = \text{Solution}
\]

\[
\text{Mucilage} = 0.04 (\text{Mucilage} + \text{solution})
\]

\[
\text{Mucilage} - 0.04 (\text{Mucilage}) = 0.04 \text{ Solution}
\]

0.96 Mucilage = 0.04 Solution

\[
\text{Mucilage} = 0.04/0.96 (\text{Solution})
\]

Therefore,

\[
\text{Mucilage} = 0.4166 (\text{Solution})
\]

We know that,

1 gram = 1 ml
Consider 15ml of acetic acid and 15ml of DI water. The amount of mucilage required is

\[
\text{Mucilage} = 0.04(\text{Mucilage} + 15 + 15) \\
\text{Mucilage} = 0.04(\text{Mucilage} + 30) \\
\text{Mucilage} - 0.04(\text{Mucilage}) = 1.2 \\
0.96(\text{Mucilage}) = 1.2 \\
\text{Mucilage} = 1.2/0.96
\]

Hence,

\[
\text{Mucilage} = 1.25 \text{ grams}
\]

Therefore we need 1.25 grams of mucilage for 30ml of the solution. As the amount of the solution changes, the amount of mucilage changes respectively.

2.2 Poly Vinyl Alcohol (PVA)

Poly Vinyl Alcohol is chosen for this study, as it is an odorless, nontoxic, biodegradable, biocompatible and water-soluble polymer. It is used as a co-spinning polymer in the process of electrospinning. It is used as a co-spinning polymer as it helps in dissolution. From the previous studies it is known that this polymer is highly flexible and has high tensile strength. It is resistant to solvents and oil. While electrospinning PVA as a co-spinning polymer we must monitor the concentrations of carbohydrates to PVA. PVA was successfully spun with other natural polymers and carbohydrates and hence is considered a good choice for electrospinning.

2.2.1 Composition of Poly Vinyl Alcohol

Poly Vinyl Alcohol is of two types with different molecular weights, namely: the Low Molecular Weight (LMW) PVA and the High Molecular Weight PVA. The molecular weight of the LMW PVA is 28.4 M. It is mixed in different compositions like 7%, 9% and 11%. The molecular weight of HMW PVA is 80 M. it is mixed in different compositions like 9% and 11%.
2.2.2 Preparation of Poly Vinyl Alcohol and Mucilage Solution

Poly vinyl Alcohol is prepared by mixing the powered PVA with deionized water. The weighing paper is taken on the weighing balance and is tarred to zero. The LMW PVA shown in figure 3 has a molecular weight of 27,000 is weight to 7%, 9% and 11% volume-to-volume ratio of the solution. The powdered PVA is added slowly into the DI water such that there are no clusters are formed. The mixture is heated and stirred and heated at 900 rpm and 90°C for about 2 hours until a homogeneous solution is formed. It is covered with the help of a thin parafilm such that the solution won’t evaporate. The same process repeats for the High Molecular Weight PVA shown in figure 5 has molecular weight ranging from 85000-1, 24,000 as explained in figure 4.

![Image](image.jpg)

Figure 3 11% Weight/Weight Low Molecular Weight PVA Solution
Figure 4 Procedure for Preparing PVA and Mucilage Solutions
2.2.3 Calculations for the Preparation of the Solution

The calculations below give us the amount of PVA and water we use to prepare the solutions.

\[
PVA = \frac{R}{100} (PVA + \text{Water})
\]

\[
R = \text{Ratio} = 7, 9, 11
\]

\[
100 \times PVA = R \times (PVA) + R \times (\text{Water})
\]

\[
(100 - R) \times PVA = R \times (\text{Water})
\]

Therefore,

\[
PVA = \left(\frac{R}{(100 - R)}\right) \times [\text{Water}]
\]

Consider,

\[
R = 7\% \text{ and water} = 10\text{ml}
\]

\[
PVA = (7/93) \times 10
\]

\[
PVA = 0.752 \text{ grams}
\]
Hence for 7% volume-to-volume ratio of 10ml of the solution we need 0.752 grams of PVA. As the amount of solution changes, the amount of PVA changes respectively. The homogeneous mixture of the PVA and Mucilage solution shown in figure 6 is used for the electrospinning. The sample shows the 70:30 PVA: Mucilage solution.

![Figure 6 PVA and Mucilage Solution](image)

**2.3 Polystyrene (PS)**

Polystyrene is used as another co-spinning polymer as it is biodegradable and biocompatible. It is available in abundance in nature. It is naturally transparent, clear, hard rather brittle. It is inexpensive. It is a long chain of hydrocarbons with alternate carbons attached to phenyl groups. To break the hydrocarbon chain we need another co spinning polymer called the D-Limonene. D-Limonene is a citrus extract from an orange substrate. It is used in our daily lives. Polystyrene is hydrophobic in nature which means it doesn’t dissolve in water. On the weighing balance, the weighing paper is placed and is tarred to zero. Polystyrene is solid in
nature and the D-Limonene is liquid. So we need to consider the weight of the D-Limonene for the weight-to-weight ratio of polystyrene and D-Limonene. 20% weight-to-weight of polystyrene and D-Limonene are mixed. The polystyrene dissolves completely into D-Limonene.

2.3.1 Preparation of Polystyrene Solution

Figure 7 Procedure for Preparing Polystyrene and Mucilage Solution
This mixture is heated and stirred at 900 rpm and 90°C for 3 hours until the solution is homogeneously mixed as described in figure 7. It is covered with a thin parafilm so that the solution won’t evaporate as shown in figure 8.

![Figure 8 Polystyrene Solution](image)

### 2.3.2 Calculations for the Preparation of the Solution

The calculations below give us the amount of polystyrene and D-Limonene required in the preparation of the solution.

\[
\text{Polystyrene} = \frac{20}{100}(\text{Polystyrene}+\text{D-Limonene})
\]

\[
\begin{align*}
\text{PS} &= 0.2(\text{PS}+\text{D-Limonene}) \\
(1-0.2)\text{PS} &= 0.2 \ (\text{D-Limonene}) \\
0.8 \text{ PS} &= 0.2 \ (\text{D-Limonene}) \\
\text{PS} &= 0.2/0.8(\text{D-Limonene}) \\
\text{PS} &= 0.25(\text{D-Limonene})
\end{align*}
\]

But we are considering the weight percentages, so the weight of D-limonene must be considered.

\[
\text{Weight of D-limonene} = 0.841 \text{ g/ml}
\]
Consider we are taking 20ml of D-Limonene

\[ \text{PS} = 0.25 \times 0.841 \, \text{g/ml} \times D \]
\[ \text{PS} = 0.210 \times D \]

Hence for 20ml of D-limonene solution we need 4.205 grams of polystyrene. As the amount of solution increases, the amount of polystyrene increases.

Figure 9 Polystyrene and D-Limonene Mixed with Mucilage on the Heater with a Stirrer

2.4 Polystyrene with Toluene

A mixture of polystyrene and toluene is made. Toluene was used in the form of FORM 66. FORM 66 was a replacement for toluene provided by the GREEN Company. Toluene was replaced instead of D-Limonene just to check if we were obtaining standalone membranes. It was mixed in a ratio of 70:30 polystyrene: mucilage. Fibers were obtained but not the standalone membrane. For 50:50 and 30:70 volume-to-volume mixtures of polystyrene: mucilage no fibers
were obtained. And toluene is not bio degradable and is harmful to the environment, hence we have performed the further test only on polystyrene and D-Limonene.
CHAPTER 3: PROCESS OF CHOICE

3.1 Introduction

Nanofiber membranes are currently being investigated as effective devices for the treatment of water contaminated by toxic metal ions, organic and inorganic solutes, and microorganisms. A cost effective process used to fabricate these nanofiber membranes is electrospinning. Electrospinning is one of the broadly used reliable and cost effective techniques that are used to produce polymeric nanofiber membranes with fiber diameter ranging from several micrometers down to several hundred nanometers for a wide range of applications [3]. These electrospun nanofiber membranes show the potential to filter toxic pollutants from the water [4]. The unique features of nanofiber membranes, like high surface area to volume ratio and highly controllable nano pore size, enhance the filtration capability. Nanofiber membranes are being used for filtration, but most of them are fabricated with non-organic materials that are not environmentally friendly. This study investigates the use of the abundantly available natural material, Opuntia ficus-indica also called as cactus mucilage, as a tool for nanofiber membrane filtration. Mucilage is a non-toxic, biodegradable, non-toxic and a biocompatible material that can be extracted from the cactus plant. Ofi or prickly pear is a very resourceful plant with various medicinal benefits, and has been used for treating arteriosclerosis, diabetes, and gastritis and hyperglycemia by rural Mexican people [4, 5]. Ofi has also been studied for its anti-oxidant properties and its ability to remove toxic contaminants from water. The literature reviews indicate that the cactus mucilage has the ability to be used as the sustainable water filtration method.
3.2 Methodology

The fabrication technique used to produce the PVA: mucilage and PS: A mucilage nanofiber membrane was the electrospinning process. The electrospinning setup is shown in figure. The setup uses high voltage (20 kV ~ 22 kV) to create an electric field between the tip of a needle (18 ~ 20 gauge) and a grounded collector plate. There are various electrospinning parameters that affect the fiber formation. They are voltage applied, infusion rate set in the syringe pump, concentration, homogeneity and molecular weight of the polymer. A voltage supply is used to create a strong electric field between the needle tip and the collector plate. The syringe pump is programmed to dispense a controlled volume of the solution. The infusion rate is set from 1-5 µl/min. As the solution is infused, because of the high electric field, the solution forms a cone at the needle tip and it is pulled to the collector plate. As the solution leaves the needle, the solvent begins to evaporate leaving the polymer behind as a thin fiber strand.

A Taylor cone of the polymeric solution is formed and due to surface tension threads of fibers that are produced. It has the characteristics of both electro spraying and electrospinning of the fibers. The process can be performed at room temperatures to produce the threads from the prepared solutions. Hence this process is suitable for the making of fibers using complex and large compounds. The 11% weight to weight of high molecular weight PVA solution was electrospun without combining the solution with the mucilage. The solution was neither electrospinning nor electro spraying. The polymer was stuck at the tip of the needle and became air tight inside the syringe. The solution was not infused even if the infusion rate was increased. Hence PVA and Mucilage solution was not sustainable for water filtration even though we obtained fibers using the low molecular weight PVA. This can be used for other applications.
3.3 Results

3.3.1 PVA and Mucilage Solution

Initially the low molecular weight PVA was electrospun without the mucilage. We could obtain the LMW PVA fibers. Then the different ratios of 30:70, 50:50 and 70:30 solutions of 11% weight to weight low molecular weight PVA:Mucilage were taken into the syringe simultaneously. They were put onto the Harvard apparatus where the solution is infused from the syringe onto the collector plate. The fiber mesh was obtained on the collector plate due to the electric field created between them.

The 30:70 PVA: Mucilage solution was only electro spraying and we could not obtain any fibers. The 50:50 and 70:30 of PVA: Mucilage solutions formed fibers. But the drawback with these fiber meshes was that they disappeared when a water droplet was dropped on them. Thus this wasn’t useful for water filtration since we needed fibers that could withstand water, as they had to be used for several cycles of filtration. Figure 10 shows the laboratory setup of the electrospinning system.

3.3.2 Polystyrene and Mucilage Solution

The polystyrene solution was electrospun using the electrospinning setup. The nanofibers were obtained. Different ratios of 30:70, 50:50 and 70:30 ratios of polystyrene: mucilage were electrospun. Fibers were obtained at all the three ratios. The 30:70 polystyrene: mucilage solution had fibers but it was also electrospaying simultaneously. We could obtain fibers using 50:50 and 70:30 polystyrene: mucilage. These fiber meshes did not dissolve in water and hence they were further tested for the filtration process.
Figure 10 Electrospinning Setup
CHAPTER 4: SCANNING ELECTRON MICROSCOPY

4.1 SEM Analysis of Polystyrene: Mucilage Nanofibers

Polystyrene is used as another co-spinning polymer. D-limonene, a citrus extract from an orange peeling, is used to breakdown the polystyrene into a solution form. A solution of 20%w/w of polystyrene and D-limonene is made. This is mixed with the mucilage in different ratios of 30:70, 50:50, and 70:30. The mixture is heated and stirred at 60°C and 600 rpm until they are mixed well. These samples are then electro spun and fibers are formed. The results show that the 30:70 polystyrene: mucilage solution forms smaller fibers as compared to the 50:50 and 70:30 polystyrene: mucilage solutions. We could conclude that 50:50 and 70:30 polystyrene: mucilage formed larger fibers compared to 30:70 polystyrene: mucilage from SEM characterization.

Figure 11 SEM Characterization of Polystyrene: Mucilage 30:70
Figure 11 shows bead formation with the fibers. The viscosity for this 30:70 solution is lower and the material is trending toward being hydrophilic in nature, meaning, it has the potential to dissolve in water. This is due to the nature of the mucilage, which is 70% of the solution, to be hydrophilic. For water filtration testing, the resulting solution and materials need to be hydrophobic in nature, such that it doesn’t dissolve in water. The resulting fibers for the 30:70 v/v solution have an average diameter of about 334 nm.

![SEM Characterization of Polystyrene: Mucilage 50:50](image)

Figure 12 SEM Characterization of Polystyrene: Mucilage 50:50

Figure 12 shows less beading in the fibers formed using the 50:50 v/v solution as, compared to the 30:70 solution. It has a higher viscosity of 82 cp vs. 45 cp for 30:70. Due to these results, the nanofibers formed from the 50:50 v/v solution have the potential to be used for water filtration. The resulting fibers for the 50:50 v/v solution have an average diameter of 611 nm.
Figure 13 SEM Characterization of Polystyrene: Mucilage 70:30

Figure 13 shows no beading in the fibers formed using the 70:30 v/v solution. Results in Chapter 5 and 6 show results of the 70:30 solution having higher viscosity and being hydrophobic in nature. The resulting fibers for the 70:30 v/v solution have an average diameter of about 206 nm.

4.2 SEM Analysis of PVA: Mucilage Nanofibers

Mucilage extraction varies from plant to plant therefore extractions from different pads of the Ojí cactus were used. There was no significant difference between the NE of one pad to another in the formation of nanofibers in this study. Future study can determine the effectiveness of the mucilage fibers and its water filtration uses. The PVA solutions at 7% gave us no fibers. It had only beads. This leads to the hypothesis that not enough polymers were present to form fibers. At 9% solution fibers were produced with the volume ratio of 70:30. There are enough PVA polymers in the solution to help the mucilage fiber into forming. The 30:70 and 50:50 volume ratios do not have sufficient polymer chains to produce fibers with no beads. It is hypothesized that at a higher PVA concentration 10% and above fibers will form that are thicker and less usable for filtration for a higher solubility in water.
Figure 14 SEM Characterization of 11% Low Molecular Weight PVA: Mucilage 70:30 Nanofibers [1]
CHAPTER 5: VISCOSITY

5.1 Introduction

Process parameters that may affect the formation of fibers are the infusion rate, applied voltage, temperature, and distance to the collector etc. Although there are many operational and material parameters those effect fiber formations, viscosity has more significant effect. The viscosity of a fluid is a measure of its resistance to flow [8]. Shear refers to the force required to move a layer of fluid with respect to another layers. Friction is directly proportional to the force required to move the liquid. More the friction more is the force required to move the liquid.

\[
\text{Viscosity} = \eta = \frac{F'}{S} = \text{shear stress} \times \text{shear rate}
\]

Two equal areas of parallel planes of fluid are moving with different velocities, \(V_1\) and \(V_2\), in the same direction and are separated by a distance \(dx\). The shear rate, or velocity gradient, is calculated as \(dv/dx\). F’ symbolizes the shear stress, which is defined as the force applied per unit area required to produce a shearing action (F/A).

5.2 Methodology

A Fungilab Smart L series rotational viscometer is used to measure the viscosity of these concentrations. Selected spindles are used as measurement tools through a calibrated torsion spring. Depending on the volume of the solution, a particular spindle is immersed in the test
fluid and rotates at a set speed (rpm). The spring will deflect as the fluid drags against the spindle and the viscometer calculates the viscosity.

Figure 15 FungiLab Smart L Series Rotational Viscometer

Cactus mucilage from the Opuntia ficus-indica is mixed with a polymer solution called polystyrene and a co-spinning polymer called D-limonene in different volume ratios of 30:70, 50:50, 70:30. This polymeric solution is electrospun into nanofibers, and the electrospun nanofibers from the volumetric ratios of the polymer solutions form a fiber membrane. In the case of low concentrations of the polymer solution, fibers may not be formed as the solution electrospays. These concentrations have a direct impact on the viscosity of the solution. These same solutions are electrospun to form cactus mucilage nanofibers and scanning electron microscopy (SEM) images are taken to characterize fiber formation and diameter.
5.3 Results

Figure 16 Viscosity Measurements for 30:70 Polystyrene: Mucilage

Figure 17 Viscosity Measurements for 50:50 Polystyrene: Mucilage
Figure 18 Viscosity Measurements for 70:30 Polystyrene: Mucilage

Figure 19 Graph Plotted Between Concentrations versus Average Viscosity
By evaluating the SEM images, it is shown that beading occurs for the lower polymer concentration of 30% and a fiber diameter of 334 nm (Figure 16). As the polymer concentration increases, viscosity increases, and there are fewer beads as in Figure 17 for the 50:50 solution. Fiber diameter for the 50:50 solution show ranges from 488 nm to approximately 2.5 µm. Figure 18 shows almost no beading in the fiber formation with a fiber diameter of 206 nm for a 70:30 solution.

Viscosity measurements were evaluated for polystyrene as a co-spinning polymer in the solvent D-Limonene and mucilage in the following weight ratios: 30:70, 50:50, 70:30. SEM images were taken to characterize impact on the fiber formation. Figure 16, figure 17 and figure 18 shows an average viscosity of 45 cp for 30:70, 82 cp for 50:50, and 122 cp for 70:30 solutions. From the above results a relation has been obtained between the formations of beads to the viscosity. As the viscosity is becoming larger the formation of beads is low and vice versa.
CHAPTER 6: CONTACT ANGLE MEASUREMENTS

6.1 Introduction

The contact angle is the angle formed by a tangent drawn at the surface of interaction between a solid and a liquid to the surface of the solid. The contact angle explains us about the wettability phenomena. This wettability is given by the Young equation. The contact angle is measured for a solid, liquid and a vapor. The contact angle for these surfaces is different at different temperatures. They have a unique contact angle at equilibrium temperatures. In practice, the contact angle ranges between the maximum contact angle to the minimum contact angle. The maximum contact angle is called advancing contact angle and the minimum contact angle is called the receding contact angle. The contact angle at the equilibrium temperatures is in between these values. The contact angle shows the strength of the liquid, solid and the vapor interactions.

It allows us to determine if the nature of the materials is hydrophobic or hydrophilic. The more the contact angle, the more is its hydrophobicity. The lesser the contact angle, the more is its hydrophilicity.

6.2 Methodology

Contact angle is measured using the spin-coated solution over a glass slide. A droplet of the water is put onto the glass slide using the Hamilton Microliter Syringe. The image of this slide was recorded with the help of a microscope, which is zoomed to fit to the screen. The KSV Contact Angle Measurement Optical Contact Angle and the Pendant Drop Surface Tension
software, version 4.04 traces the edge of the droplet by drawing a tangent to the curve. The exterior angle between the sample solution on the surface of the glass slide and the water droplet is measured and is called the contact angle. Figure 20 shows the water droplet on the spin coated surface of the PVA: Mucilage solution. Figure 21 shows the water droplet on the spin coated surface of the Polystyrene: Mucilage solution. If the angle between the sample solution on the surface of the glass slide and the water droplet is greater than or equal to $90^\circ$, then the solution is hydrophobic in nature. If the angle between the sample solution on the surface of the glass slide and the water droplet is less than $90^\circ$, then the solution is hydrophilic in nature.

Figure 20 Spin-Coated Solution of PVA: Mucilage
6.3 Results

Table 1 Contact Angle Measurements

<table>
<thead>
<tr>
<th>S.NO</th>
<th>SOLUTION</th>
<th>CONTACT ANGLE IN DEGREES</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>MUCILAGE SOLUTION</td>
<td>21.10</td>
</tr>
<tr>
<td>2</td>
<td>LOW MOLECULAR WEIGHT POLY VINYL ALCOHOL SOLUTION 11% w/w</td>
<td>54.64</td>
</tr>
<tr>
<td>3</td>
<td>PVA:MUCILAGE 50:50</td>
<td>35.92</td>
</tr>
</tbody>
</table>
The contact angle measurement helps us to determine the hydrophobicity and the hydrophilicity of the solution. It gives us an idea of the surface tension. It also shows us the wetting phenomena. Wetting phenomena is well defined as the water on the sample of the solution. The contact angle of all the solutions we obtained was less than 90°. But the polystyrene: mucilage solutions had contact angle greater than PVA: mucilage. Hence polystyrene: mucilage has more contact angle compared to PVA: mucilage. Polystyrene: Mucilage is more hydrophobic compared to PVA: mucilage solutions.
CHAPTER 7: ATOMIC FLUORESCENCE SPECTROMETRY

7.1 Introduction

PS Analytical can be used for arsenic, selenium, antimony and other hydride forming elements. The fluorescence technique has a good sensitivity and linearity that provides low detection limits. The hydrides and excess hydrogen swept out the generation vessels through an argon stream into a chemically generated hydrogen diffusion flame. Samples levels can be quantified by reference to calibration prepared from a series of standard solutions.

7.2 Methodology

Firstly turn on the nitrogen and argon gases and check the pressure on the cylinder and lines. We need at least 400 psi on the cylinder. We need nitrogen of 80 psi and argon of 60 psi. We need to place the tubing on the respective reagents. The grey tubing goes into the reagent, the two green tubing blank in and blank out in the reagent blank. The sample tubing is placed in distilled water and samples. Connect the pump tubing and cassette head on the pump. We need to ensure that they are correctly attached. Turn on the computer and the instrument and let the instrument be warmed up for at least 30 minutes. Open the millennium software. Click on the analysis tab and turn on the instrument. Load the default calibrations. Go to the method tab and choose the method. Change the gain and range of the As concentration. Check if the gases and pumps are on. Using the lighter ignite the flame. After the flame is on, the lighter shows the flame status is present. Now the manual control page can be closed. The sequence must be organized in five steps:
• Three times reagent blank
• New calibration
• Drift monitor
• Five samples
• Drift monitor

Click the start button to start the above sequences. Click the export data and save the file as .xls. Put the tubing on distilled water with pumps and clean the instrument. Clean the Gas/Liquid Separator. Click the idle, off and exit.

7.2.1 Preparation of Sample Solutions

There are four solutions to be prepared:

• Solution 1: Reductant:

Add 2 grams of NaOH in 300mL distilled water and mix well. Add 7 grams of NaBH4 and complete the volume to 1000mL with distilled water. Filter the solution with 0.22µM membrane industrial filters.

• Solution 2: Potassium Iodide: (KI solution)

Dissolve 5 grams of ascorbic acid and 25 grams of KI in 50mL with distilled water.

• Solution 3: Reagent Blank: (30% HCl + 2% KI)

Add 300mL of HCl in 200mL of distilled water and all other sample constituents. Add 20mL KI solution and complete the volume to 1000mL with distilled water.

• Solution 4: final concentration
The final concentration of the standard solutions will depend on the expected concentration range of our samples. Starting by stock solutions you must to do diluted solutions and add 30% HCl, 2% KI solution and all other sample constituents.

7.2.2 Nanofiber Filtration Procedure

The test were performed using GVWP 0.22 membranes filters from Millipore coated with the mucilage nanofibers or columns (Pasteur glass pipets) filled with 0.5 g of pre-washed sand from Fisher Scientific and 0.01 g of the mucilage nanofibers.

Figure 22 Filters Coated with Mucilage Nanofibers
500 mL of 50 µg/L of arsenic solution was prepared from arsenic (V) oxide (Acros Organics). Aliquots of 25 mL of the arsenic solution were filtered through the filters or columns as in figure 23 and figure 24.
The coated filters were tested in 2 cycles (filtering 25 mL of 50 µg/L of arsenic solution in each cycle). Triplicates of the stock solution and samples from the columns were collected and analyzed.

7.3 Results

7.3.1 Removal of Arsenic from PVA: Mucilage Nanofibers

Five different samples are prepared for testing the AFS filtering tests.

• Sample 1: only a filter coated with mucilage

• Sample 2: only mucilage on a filter paper heated at 50°C for 1 hour
Figure 27 Filter Coated with Mucilage under Heat Conditions

- Sample 3: 70:30 PVA:Mucilage layer in U.V light for 24 hours

Figure 28 PVA: Mucilage 70:30 under Ultra Violet Conditions

- Sample 4 and 5: 70:30 PVA: Muc electro spun on filter paper and aluminum foil respectively.
The total arsenic content on the samples were measured before and after treatment using Atomic Fluorescence Spectrometry (AFS) from PS Analytical, model 10.055 MILLENIUM EXCALIBUR).

It is found that all the samples were dissolving when treated through water. Initially a sample reference was taken with the control sand. All the samples were run for 2 cycles. We could not get proper results. Sample 2 was coated with mucilage that could remove only 2.5% more arsenic than the reference sample. This sample was not significant in the second cycle as it absorbed only 0.1% arsenic. The sample 3 had the best performance compared to all the other samples. It could absorb 20% of arsenic but there was no second cycle treatment as we could not reuse the filter. The first sample filtration was very slow compared to all the other samples. We could not perform the filtration test on sample 4 as there was only a thin layer on the foil. It was not enough to use it in the machine. The sample 5 could remove the arsenic for two cycles of treatment. It removed 11.1% of arsenic in the first sample and 8.8% in the second sample.
Figure 30 Concentration of Arsenic in Solutions before (45 ppb) and after Filtration Treatment for Direct Filtration using Filter 1, 2, and 4 and Columnar Filtration.

Although there are a great potential to use the mucilage nanofibers to remove arsenic from water, work on the composition and solubility of the nanofibers will be required in order to achieve better results. At this point, the coated filters have removed a maximum of 13.8 % (20 % minus 6.2 % obtained on the control using only the filter apparatus) of the arsenic from the solution.
7.3.2 Removal of Arsenic from Polystyrene: Mucilage Nanofibers

Three cycles of 70:30 PS: mucilage samples are taken as in figure 32. The first cycle output concentration is 30.1326 ml. The second cycle output concentration is 25.34421 ml. The third cycle output concentration is 23.69612 ml. It is observed that this sample could run all three cycles. An average of 9.26% arsenic is removed by the 70:30 PS: mucilage nanofibers. Three cycles of 50:50 PS: mucilage samples are taken as in figure 33. The first cycle output concentration is 21.46663 ml. The second cycle output concentration is 26.07304 ml. The third cycle output concentration is 22.23318 ml. An average of 18.93% of arsenic is removed by the 50:50 PS: mucilage nanofibers. It is observed that the 50:50 sample absorbed more arsenic than the 70:30 sample.
Figure 32 70:30 Polystyrene: Mucilage Nanofibers

Figure 33 50:50 Polystyrene: Mucilage Nanofibers
In an effort to evaluate the functionality of PVA:Mucilage and PS: Mucilage nanofibers, atomic fluorescence spectrometry (AFS) from PS Analytical was used to evaluate electrospun nanofiber membranes made from volume ratios of 30:70, 50:50 and 70:30. The mucilage nanofiber membranes were used as filtration devices for 50 ppb arsenic solutions. PVA: mucilage nanofiber membranes were found to dissolve upon repeated cycling of water solutions. This is contributed to the hydrophilic nature of the PVA and mucilage. On the other hand, results PS: mucilage show that on performing the AFS test on 70:30 PS: Mucilage a nanofiber membrane, 9.72% of arsenic is removed from the water, and the 50:50 PS: Mucilage nanofiber membrane can remove 18.93% arsenic from figure 34.

In conclusion, it should be noted that this natural, biodegradable, cheap mucilage nanofiber filter using a 50:50 v/v solution is comparable to a traditional sand columnar filtration result of 18.33%. Further investigations will be performed using PS: mucilage nanofiber membranes to complete a comprehensive study.
CHAPTER 8: FOURIER TRANSFORM INFRARED SPECTROMETRY

8.1 Introduction

FTIR stands for Fourier Transform Infrared Spectrometry. It is defined as the technique that deals with the infrared regions. The FTIR technique is used to determine the C-H and C=O bonds. They use the sampling technique that examines the samples directly in the solid or liquid without any further reflection. This technique is called the attenuated total reflection. It uses the property of the total internal reflection. It shows the presence of the functional groups in the solution.

8.2 Procedure

The FTIR setup consists of a beam splitter, fixed mirror and another mirror that moves back and forth. The beam splitter transmits half of the radiations and it reflects the other half of the radiation. When the infrared radiations pass through the beam splitter, they separate into two beams. One beam passes through the fixed mirror and the other beam is reflected back through the moving mirror. The FTIR has high spectral accuracy with high signal-to-noise ratio. It has high sampling rate and can sample the wavelengths without any prior preparations.

8.3 Results

The results below show us the presence of functional groups inside the solution. Figure 35 shows us the instrument used for Fourier Transform Infrared Radiation Spectroscope.
Figure 35 Fourier Transform Infrared Radiation Spectroscope

Figure 36 Infrared Radiation Spectra of Polystyrene: Mucilage 50:50
Figure 37 Infrared Radiation Spectra of Polystyrene: Mucilage 30:70

Figure 38 Infrared Radiation Spectra of Mucilage
Figure 39 Infrared Radiation Spectra of Low Molecular Weight PVA

Figure 40 Infrared Radiation Spectra of PVA: Mucilage 50:50
Figure 41 Infrared Radiation Spectra of Polystyrene

Figure 42 Infrared Radiation Spectra of Polystyrene: Mucilage 70:30
Figure 43 Infrared Radiation Spectra of PVA: Mucilage 70:30

The graphs are plotted both in transmittance and observance. It is our choice to choose against which parameter we are plotting. Transmittance is generally the traditional method for performing the FTIR. Observance is the present method to perform FTIR. We choose transmittance as it is the chemist's best choice and easy to evaluate the results. Every graph depicts the presence of their respective functional groups in them. The wavenumbers corresponding to the peaks show the bonds present in the solution. Based on the wave numbers and the chart present in figure 44 we can determine the functional groups present at the peaks. The wavenumbers are obtained by a process of total internal reflection where its critical angle is said to be greater than 90°. When the light is passed through the crystal, it reflects some of its light and refracts some part of it. So,
### Table of Characteristic IR Absorptions

<table>
<thead>
<tr>
<th>Frequency, cm(^{-1})</th>
<th>Bond</th>
<th>Functional Group</th>
</tr>
</thead>
<tbody>
<tr>
<td>3640–3610 (s, sh)</td>
<td>O–H stretch, free hydroxyl</td>
<td>alcohols, phenols</td>
</tr>
<tr>
<td>3500–3200 (s, h)</td>
<td>O–H stretch, H–bonded</td>
<td>alcohols, phenols</td>
</tr>
<tr>
<td>3400–3250 (m)</td>
<td>N–H stretch</td>
<td>1’, 2’ amines, amides</td>
</tr>
<tr>
<td>3300–2500 (m)</td>
<td>O–H stretch</td>
<td>carboxylic acids</td>
</tr>
<tr>
<td>3330–3270 (n, s)</td>
<td>=C≡C–H: C–H stretch</td>
<td>alkenes (terminal)</td>
</tr>
<tr>
<td>3100–3000 (s)</td>
<td>C–H stretch</td>
<td>aromatics</td>
</tr>
<tr>
<td>3100–3000 (m)</td>
<td>=C–H stretch</td>
<td>alkenes</td>
</tr>
<tr>
<td>3000–2850 (m)</td>
<td>C–H stretch</td>
<td>alkenes</td>
</tr>
<tr>
<td>2830–2695 (m)</td>
<td>H–C≡O: C–H stretch</td>
<td>aldehydes</td>
</tr>
<tr>
<td>2260–2210 (v)</td>
<td>C≡N stretch</td>
<td>nitriles</td>
</tr>
<tr>
<td>2260–2100 (w)</td>
<td>=C≡C– stretch</td>
<td>alkenes</td>
</tr>
<tr>
<td>1760–1665 (s)</td>
<td>C≡O stretch</td>
<td>carbonyls (general)</td>
</tr>
<tr>
<td>1760–1690 (s)</td>
<td>C≡O stretch</td>
<td>carboxylic acids</td>
</tr>
<tr>
<td>1750–1735 (s)</td>
<td>C≡O stretch</td>
<td>esters, saturated aliphatic</td>
</tr>
<tr>
<td>1740–1720 (s)</td>
<td>C≡O stretch</td>
<td>aldehydes, saturated aliphatic</td>
</tr>
<tr>
<td>1730–1715 (s)</td>
<td>C≡O stretch</td>
<td>α, β–unsaturated esters</td>
</tr>
<tr>
<td>1715 (s)</td>
<td>C≡O stretch</td>
<td>ketones, saturated aliphatic</td>
</tr>
<tr>
<td>1710–1665 (s)</td>
<td>C≡O stretch</td>
<td>α, β–unsaturated aldehydes, ketones</td>
</tr>
<tr>
<td>1680–1640 (m)</td>
<td>=C≡C– stretch</td>
<td>alkenes</td>
</tr>
<tr>
<td>1650–1580 (m)</td>
<td>N–H bend</td>
<td>1˚ amines</td>
</tr>
<tr>
<td>1600–1585 (m)</td>
<td>C–C stretch (in–ring)</td>
<td>aromatics</td>
</tr>
<tr>
<td>1550–1475 (s)</td>
<td>N–O asymmetric stretch</td>
<td>nitro compounds</td>
</tr>
<tr>
<td>1500–1400 (m)</td>
<td>C–C stretch (in–ring)</td>
<td>aromatics</td>
</tr>
<tr>
<td>1470–1450 (m)</td>
<td>C–H bend</td>
<td>alkanes</td>
</tr>
<tr>
<td>1370–1350 (m)</td>
<td>C–H rock</td>
<td>alkanes</td>
</tr>
<tr>
<td>1360–1290 (m)</td>
<td>N–O symmetric stretch</td>
<td>nitro compounds</td>
</tr>
<tr>
<td>1335–1250 (s)</td>
<td>C–N stretch</td>
<td>aromatic amines</td>
</tr>
<tr>
<td>1320–1000 (s)</td>
<td>C–O stretch</td>
<td>alcohols, carboxylic acids, esters, ethers</td>
</tr>
<tr>
<td>1300–1150 (m)</td>
<td>C–H wag (–CH(_2)X)</td>
<td>alkyl halides</td>
</tr>
<tr>
<td>1250–1020 (m)</td>
<td>C–N stretch</td>
<td>aliphatic amines</td>
</tr>
<tr>
<td>1000–650 (s)</td>
<td>=C–H bend</td>
<td>alkenes</td>
</tr>
<tr>
<td>950–910 (m)</td>
<td>O–H bend</td>
<td>carboxylic acids</td>
</tr>
<tr>
<td>910–665 (s, b)</td>
<td>N–H wag</td>
<td>1’, 2’ amines</td>
</tr>
<tr>
<td>900–675 (s)</td>
<td>C–H “oop”</td>
<td>aromatics</td>
</tr>
<tr>
<td>850–550 (m)</td>
<td>C–Cl stretch</td>
<td>alkyl halides</td>
</tr>
<tr>
<td>725–720 (m)</td>
<td>C–H rock</td>
<td>alkanes</td>
</tr>
<tr>
<td>700–610 (b, s)</td>
<td>=C≡C–H: C–H bend</td>
<td>alkenes</td>
</tr>
<tr>
<td>690–515 (m)</td>
<td>C–Br stretch</td>
<td>alkyl halides</td>
</tr>
</tbody>
</table>

m=medium, w=weak, s=strong, n=narrow, b=broad, sh=sharp

---

**Figure 44 Chart to Determine the Functional Groups with their Respective Wavelengths**
CHAPTER 9: LIFE CYCLE ANALYSIS

9.1 Introduction

Life cycle assessment (LCA) is an environmental accounting tool to analyze the impact of a process, product or system over its entire life cycle. This study helps to apprehend the environmental impact of processing and producing Opuntia ficus-indica (Ofi)-cactus mucilage nanofiber membranes, as a tool for filtration systems. The cactus mucilage is mixed with different polymer solutions namely: Poly vinyl alcohol (PVA), Polystyrene and D-Limonene (PLYD). The LCA compares PVA: mucilage (30:70), Polystyrene-D-Limonene: mucilage (70:30) solutions. The different stages considered for comparing the above solutions are their preparation methods, power consumption in the processing, energy absorbed in heating and stirring the mixtures. One gram of mucilage solution is taken as the basic functional unit to make a direct comparison for all the solutions. All the above solutions are compared using Simapro7® software. The BEES V4.02/characterization method is used. The results show a comparison of global warming, ozone depletion, acidification, water intakes, eco-toxicity, eutrophication, smog, depletion of natural resources and various other factors. These results indicate that PLYD: mucilage is more sustainable than PVA: mucilage.

9.2 Procedure

The life cycle analysis requires more data of the product right from its scratch to know its effect on the environment. The functional unit is considered as 1 gram of mucilage nanofibers.
The whole inventory is executed from open literature. The raw materials and processes are available in the database. Nine samples were considered, namely:

- Only Polystyrene
- Polystyrene: Mucilage 30:70
- Polystyrene: Mucilage 50:50
- Polystyrene: Mucilage 70:30
- PVA:Mucilage 30:70
- PVA:Mucilage 50:50
- PVA:Mucilage 70:30
- Only Mucilage
- Polystyrene:Toluene (Random mixture)

The LCA characterization is done using the IMPACT 2002+ and BEES V4.0. The damage assessment is done using the IMPACT 2002+. The results hence show that D-Limonene is a better solvent than compared to toluene. Toluene is a toxic substance and is harmful to the environment.

On comparing the carcinogens, ozone layer depletions, ionizing radiations from the results obtained through characterizations through IMPACT and BEES method we can conclude that polystyrene and mucilage with D-Limonene has less environmental impacts compared to all the other solutions. The results show that the D-limonene is more reliable as it causes less damage to the environment compared to toluene. The D-Limonene has comparatively less impact on the environment.
9.3 Results

Figure 45 Life Cycle Analysis of Characterization in IMPACT 2002+

Figure 46 Life Cycle Analysis of the Damage Assessments in IMPACT 2002+
Figure 47 Life Cycle Analysis of the Characterization using BEES V4.02

Figure 48 Comparison of Components from Characterization using BEES V4.02 and IMPACT 2002+ Methods
CHAPTER 10: SUMMARY AND CONCLUSION

In an effort to evaluate the functionality of PVA:Mucilage and PS: Mucilage nanofibers, atomic fluorescence spectrometry (AFS) from PS Analytical was used to evaluate electrospun nanofiber membranes made from volume ratios of 30:70, 50:50 and 70:30. The mucilage nanofiber membranes were used as filtration devices for 50 ppb arsenic solutions. PVA: Mucilage nanofiber membranes were found to dissolve upon repeated cycling of water solutions. This is contributed to the hydrophilic nature of the PVA and mucilage.

On the other hand, results of studies on PS: mucilage show that on performing the AFS test on 70:30 PS: Mucilage a nanofiber membrane, 9.26% of arsenic is removed from the water, and the 50:50 PS: Mucilage nanofiber membrane can remove 18.93% arsenic. It should be noted that this natural, biodegradable, cheap mucilage nanofiber filter using a 50:50 v/v solution is comparable to a traditional sand columnar filtration result of 18.33%. This work and the future work will help us in gaining an understanding of natural polymer meshes, and the use of Ofī as a membrane filter. The further contribution to the work would be testing the produced nano fiber meshes that can be used for water filtration systems. More studies are needed on the characteristics of the mucilage nanofibers. Testing is needed to see if the mucilage in nanofiber form can continue to interact with the particulates in the water and the effectiveness of a cactus mucilage filtration system in comparison to other similar filtration systems. There is hope that with this and other studies an accessible and sustainable water filtration and purification system can be developed.
The Fourier transform infrared spectrometry helps us in identifying the functional groups in the solution based on their wavelengths. It helps us to know the presence of the bonds in the solution. The mucilage can be identified in the polystyrene: mucilage solution. The functional groups present in the mucilage solution can be identified. They are a combination of linear chains of various types of sugars [1]. The O-H bonds present in the mucilage solution help us to absorb arsenic from the water [2]. We could verify the peaks of the PVA: Mucilage solution from the prior results obtained. The Scanning electron microscopy and the viscosity measurement results give us a conclusion that as the viscosity increases the bead formation on electrospinning the solution decreases. It also concludes that as the concentration of the solution increases the bead formation decreases. On analyzing environmental impacts of the solutions, we come to a conclusion that polystyrene: D-limonene: Mucilage has less effect on the environment than compared to the others.

In an effort to move this technology forward, the following needs to be studied:

• To investigate the robustness of the mucilage membranes for industrial application by studying the increase in the membrane thickness.

• To improve the efficiency of the membrane filtration by increasing the number of filtration cycles and the life span of the membrane.

• To investigate the removal of additional contaminants in water.

From the results of viscosity, scanning electron microscopy and fourier transform infrared spectrometry I have met my first research objective which is to fabricate and characterize the electrospun nanofibers of poly vinyl alcohol and polystyrene solutions formulated using the environmentally friendly solvent called the D-Limonene. From the results of the contact angle measurements and atomic fluorescence spectrometry I have met my second
research objective which is to investigate the filtration capability of fabricated PVA-Mucilage and Polystyrene-Mucilage membranes. From the results of the life cycle analysis I have met my third research objective which is to estimate the impact of the produced nanofibers on the environment.
REFERENCES


