



Faculty of Engineering

**Bamboo-derived Cellulose/PVDF Hollow-fibre Membrane via Polymer
Binder Coating for Treatment of Peat, River and Rain Water**

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Bamboo-derived Cellulose/PVDF Hollow-fibre Membrane via Polymer
Binder Coating for Treatment of Peat, River and Rain Water

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A thesis submitted

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DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Malaysia Sarawak. Except where due acknowledgements have been made, the work is that of the author alone. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.



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ABSTRACT

This thesis covers the research regarding the characterization of coated and uncoated pristine Polyvinylidene fluoride (PVDF) hollow fiber membranes with the developed bamboo derived cellulose/polymer coating using Polyvinylidene fluoride (PVDF) and Polyvinyl alcohol (PVA) polymers as separate binders and (with/without) Graphene oxide (GO) as additive. The research was conducted in three experimental stages according to coating solution formulation: cellulose/PVDF coating, cellulose/PVA coating and graphene oxide incorporated (cellulose/PVDF and cellulose/PVA) coatings. The membranes were characterized via Fourier transform infrared analysis (FTIR), Scanning electron microscopy (SEM), X-Ray diffraction analysis (XRD), Pure water flux performance, Porosity and Mean pore size analysis. The optimal performing membrane was further tested using real water sources such as rainwater, river water, and peat water for water flux performance and iron removal was evaluated via atomic absorption spectroscopy (AAS). The focus on iron removal as the targeted heavy metal contaminant is due to its common availability in natural water quality issues, especially in peaty regions of rural Borneo. The FTIR, SEM and XRD results confirmed successful surface modification after coating. Among the coating systems, cellulose/PVA showed to be the best performing formulation. The optimum sample is PV01 at lowest cellulose weightage (0.25g), achieved the highest pure water flux of $860.74 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ in comparison to the control at $157.89 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$. After filtering the real water sources, the AAS showed no detectable iron in rainwater, complete iron removal from river, and up to 46% iron removal for peat water. The addition of GO reduced performance at mid-high cellulose weightage, likely due to coating density and pore restriction.

Keywords: Cellulose, Hollow Fiber Membrane PVDF, PVA, Surface Coating

**MEMBRAN GENTIAN BERONGGA SELULOSA/PVDF TERBITAN BULUH
MELALUI SALUTAN PENGIKAT POLIMER UNTUK RAWATAN AIR GAMBUT,
AIR SUNGAI DAN AIR HUJAN**

ABSTRAK

Tesis ini merangkumi kajian mengenai pencirian membran gentian berongga Polyvinylidene fluoride (PVDF) asli yang bersalut dan tidak bersalut, dengan salutan selulosa/polimer terbitan buluh yang dibangunkan menggunakan polimer Polyvinylidene fluoride (PVDF) dan Polyvinyl alcohol (PVA) sebagai pengikat berasingan serta Graphene oxide (GO) sebagai bahan tambahan, sama ada dengan atau tanpa GO. Kajian ini dijalankan dalam tiga peringkat eksperimen berdasarkan formulasi larutan salutan, iaitu salutan selulosa/PVDF, salutan selulosa/PVA, dan salutan yang mengandungi graphene oxide bagi sistem selulosa/PVDF dan selulosa/PVA. Membran telah dicirikan melalui analisis Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray diffraction analysis (XRD), prestasi fluks air tulen, porositi, dan analisis saiz liang purata. Membran yang menunjukkan prestasi optimum diuji selanjutnya menggunakan sumber air sebenar seperti air hujan, air sungai, dan air gambut bagi prestasi fluks air, manakala penyingkiran besi dinilai melalui atomic absorption spectroscopy (AAS). Fokus terhadap penyingkiran besi sebagai bahan cemar logam berat sasaran adalah disebabkan oleh kewujudannya yang lazim dalam isu kualiti air semula jadi, terutamanya di kawasan bergambut luar bandar Borneo. Keputusan FTIR, SEM, dan XRD mengesahkan pengubahsuaian permukaan yang berjaya selepas proses salutan. Antara sistem salutan yang dikaji, selulosa/PVA menunjukkan formulasi dengan prestasi terbaik. Sampel optimum ialah PV01 pada kandungan selulosa terendah, iaitu 0.25 g, yang mencapai fluks air tulen tertinggi sebanyak $860.74 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$ berbanding sampel kawalan sebanyak 157.89 L

$m^{-2} h^{-1} bar^{-1}$. Selepas penapisan sumber air sebenar, analisis AAS menunjukkan tiada besi yang dapat dikesan dalam air hujan, penyingkiran besi sepenuhnya daripada air sungai, dan sehingga 46% penyingkiran besi bagi air gambut. Penambahan GO mengurangkan prestasi pada kandungan selulosa sederhana hingga tinggi, berkemungkinan disebabkan oleh ketumpatan salutan dan sekatan liang.

Kata kunci: Selulosa, Membran Gentian Berongga PVDF, PVA, Salutan Permukaan

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LIST OF ABBREVIATIONS

A0	Control PVDF Hollow Fiber Membrane
AAS	Atomic Absorption Spectrometry
DI	Deionised Water
FTIR	Fourier Transform Infrared Spectroscopy
P01	Cellulose/PVDF at 0.25g
P02	Cellulose/PVDF at 0.5g
P03	Cellulose/PVDF at 0.625g
P04	Cellulose/PVDF at 0.725g
P05	Cellulose/PVDF at 1.0g
PV01	Cellulose/PVA at 0.25g
PV02	Cellulose/PVA at 0.5g
PV03	Cellulose/PVA at 0.625g
PV04	Cellulose/PVA at 0.725g
PV05	Cellulose/PVA at 1.0g
PG01	Graphene oxide Cellulose/PVDF at 0.25g
PG02	Graphene oxide Cellulose/PVDF at 0.5g
PG03	Graphene oxide Cellulose/PVDF at 0.625g
PG04	Graphene oxide Cellulose/PVDF at 0.725g
PG05	Graphene oxide Cellulose/PVDF at 1.0g
PVG01	Graphene oxide Cellulose/PVA at 0.25g
PVG02	Graphene oxide Cellulose/PVA at 0.5g
PVG03	Graphene oxide Cellulose/PVA at 0.625g

PVG04	Graphene oxide Cellulose/PVA at 0.725g
PVG05	Graphene oxide Cellulose/PVA at 1.0g
PVA	Polyvinyl alcohol
PVDF	Polyvinylidene fluoride
SEM	Scanning Electron Microscopy
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Study Background

In Borneo, there are a large communities living in rural villages surrounded by peatlands, rivers and experiences rainy seasons. Water treatment for these areas is crucial for the development of the community and accommodating towards basic human needs. Researching water treatment alternatives utilizing PVDF membranes may be the solution towards a simple way to directly treat and attain clean water sources. PVDF membranes are suitable for this purpose because of their excellent thermal and chemical stability, resistance to solvents and oxidants, and good membrane-forming ability (Gopakumar et al., 2017a).

Peat is organic soil formed as a result of incomplete disintegration and humification of marsh plants under high humidity conditions. As peat water is derived from peat soil, a major fraction of the natural organic matter present in peat waters is composed of humic substances, which are complex macromolecular products of the chemical and biological degradation of plant and animal residues, including lignin, carbohydrates, and proteins. These substances consist of complex macromolecular structures containing aromatic rings and aliphatic chains, contributing to partial hydrophobicity (Cruz-Silva et al., 2019). Such characteristics influence their interaction with membrane surfaces and can adversely affect filtration performance. The presence of humic substances, along with multivalent ions such as calcium, further complicates water treatment due to their role in forming stable complexes in aqueous systems (He et al., 2017). These challenges highlight the importance of improving membrane surface properties, particularly hydrophilicity, to enhance water transport performance in organic-rich water matrices.

Membrane technologies are widely recognized for their efficiency, modularity, and scalability for water treatment applications (R. Baker 2012; Geise et al., 2010). An example of a membrane technology is the use of Polyvinylidene fluoride (PVDF) hollow fiber membranes, which is extensively used in water treatment applications due to their excellent chemical resistance, thermal stability, and mechanical strength (R. W. Baker 2012; Liu et al., 2011; Kang & Cao, 2014). PVDF polymer membranes generally has intrinsic hydrophobicity, which remains a key limitation, affecting water permeability and interaction with various real water sources such as peat, river and rain water. PVDF hollow fiber membranes was justifiably selected over flat sheet membranes due to their superior performance and structural advantages in water treatment applications. Studies have shown PVDF hollow fiber membranes to provide higher water flux due to their self-supporting geometry, porosity and density, hence allowing more efficient water permeation within a given module volume. The overall productivity is enhanced, due to the significantly large surface area-to-volume ratio, which enables greater effective filtration area in a compact system (R. W. Baker 2012; Gopakumar et al., 2019). On the other hand, flat sheet membranes generally involve the development of more complex module designs and has lower packing efficiency with less surface area-to-volume ratio. Therefore, PVDF hollow fiber membranes are more suitable for achieving high water flux performance in a single given module (R. Baker 2012; Gopakumar et al., 2019). The strategy of using surface modification techniques on PVDF membranes has been widely explored, which studies shown that it could improve water treatment performance. Several surface modification techniques include blending, grafting, and coating has been investigated by researchers, with coating methods offering advantages in terms of simplicity and post-fabrication adaptability (Rana & Matsuura, 2010; Zhang et al., 2016). Cellulose and its derivatives are significantly researched and applied in

many different applications due to their abundance in supply, biodegradability, and inherent hydrophilic properties (Abouzeid et al., 2018; Mohammed Nishil et al., 2018). In particular, bamboo-derived cellulose presents a sustainable and locally available resource with high potential for membrane modification purposes (Liew et al., 2015). Some studies showcases that the incorporation of cellulose and their derivatives into PVDF membranes could improve hydrophilicity and permeability (Bai et al., 2012; Wang et al., 2021; Wu et al., 2024). However, majority of existing research focuses on mixed-matrix membranes with cellulose being incorporated in the main matrix during the membrane fabrication process (Koriem et al., 2023; Salem et al., 2019; Yu et al., 2009). These approaches could enhance membrane properties, however, they often involve complex fabrication processes and limited flexibility for post-treatment modification. In contrast, surface coating strategies on stock/pristine hollow fiber membranes remain comparatively underexplored, especially in the context of utilizing cellulose-based materials.

In this research, PVDF and PVA powders are deployed separately as binder materials in the bamboo cellulose-based coating solution. PVDF provides strong adhesion and chemical stability, ensuring durability of the coating layer, while PVA also provides strong adhesion, it also enhances hydrophilicity and film-forming ability due to its hydroxyl-rich structure (Li et al., 2010; Li et al., 2019). Graphene oxide (GO) has been widely explored as a functional additive in membrane modification due to its high surface area and its abundance in oxygen functional groups. The incorporation of GO as additive into the cellulose/polymer coating solution further enhances the hydrophilic properties, hence, improving water flux performance due to enhanced surface wettability and efficient interactions between surface water molecules (Perreault et al., 2015; Sun et al., 2022). The synergistic combination of GO, PVDF, PVA powders and bamboo cellulose as a coating solution is therefore expected

to improve coating stability, increase surface wettability, and enhance overall water flux performance of the pristine PVDF hollow fiber membrane system. As numerous studies demonstrated improved membrane performances, many are limited to evaluations using model solutions or single contaminants, without considering the samples variables from real water sources such as peat, river, and rainwater (Xu et al., 2022; Sahu et al., 2023). In addition, comprehensive physicochemical characterisations are often not emphasized with the correlation of surface modifications with water flux performance. This creates a gap in understanding the relationship between coating structure, membrane properties, and practical filtration performance.

To address these limitations, this study focuses on the development of a cellulose/polymer coating on pristine PVDF hollow fiber membranes and its effect on membrane properties and performance. The research emphasizes fundamental characterization, including surface morphology, infrared spectral analysis, crystallinity, porosity, and pore size, alongside water flux performance. In addition, practical validation is incorporated through iron removal analysis using Atomic Absorption Spectroscopy (AAS). This is to provide a simple method of evaluation for membrane performance in treating real water samples in particular, peat and river waters as iron content are in abundance (Fakhfekh et al., 2017). The significance of this research stems from developing and characterizing the physiochemical properties of polymer/cellulose surface coating on PVDF hollow fiber membrane for the purpose of water treatment. The scope of this study is limited to coating development, short-term water flux performance, and physicochemical characterization of cellulose/polymer-coated PVDF hollow fiber membranes. Long-term fouling behavior, mechanical properties under extended operation, and large-scale implementation are beyond the scope of this research. By clearly defining these boundaries,

this research aims to provide a focused evaluation of cellulose/polymer-coated PVDF hollow fiber membranes, contributing to the advancement of surface modification strategies for water treatment applications.

1.2 Problem Statement

The main engineering challenge comes from the hydrophobic nature and low surface energy of the PVDF polymer for membrane technologies which lowers water permeability and water flux. This limitation decreases filtration capabilities and increases energy demands, as it takes longer to yield the desired filtered water, making PVDF polymers for membrane technologies less effective for practical real water treatment applications (He et al., 2017). Past studies demonstrated improvements in PVDF membrane performance with the incorporation of cellulose and/or its derivatives into the membrane matrix, enhancing hydrophilicity, permeability, and membrane microstructure (Song Zhang et al., 2016; X. Yu et al., 2024; Lv, Zhang, Zhang, Zhao, et al., 2018a; Wu et al., 2024). However, most approaches rely on blending and mixed-matrix techniques, where hydrophilic materials are distributed within the bulk of the matrix mixtures rather than concentrated on the surface. This limits the potential of the surface properties for enhanced water permeability and water flux performance (L.-Y. Yu et al., 2009; Salem et al., 2019; O. A. Koriem et al., 2023; H. Bai et al., 2012).

In comparison to PVDF flat sheet membranes, PVDF polymer as hollow fiber membranes offer higher packing density, greater surface area-to-volume ratio, and with increased water permeability, making them suitable for compact systems (Baker, 2012; Asiri et al., 2024). Their scalability and flexibility in module design further supports implementation for rural applications (Liu et al., 2011; Acarer Arat et al., 2025). Despite these developments, there are limited studies exploring the use of sustainable biomass

resources such as bamboo-derived cellulose as a coating material. Pairing the bamboo cellulose in combination with polymers (e.g., PVDF and PVA) acting as binders, could enhance coating stability and water flux performance (Li et al., 2010; Li et al., 2019; Razmgar, Saljoughi, and Mousavi 2018). Therefore, a research gap exists in developing an effective surface modification strategy that directly enhances membrane surface hydrophilicity and water flux without altering the bulk structure of PVDF hollow fiber membranes. This research proposes the use of bamboo cellulose as the primary hydrophilic coating material due to its abundance and favourable surface chemistry (Liew et al., 2015; Abouzeid et al., 2018). PVDF and PVA are used as polymer binders to improve coating adhesion and film formation (Li et al., 2010; Li et al., 2019), while graphene oxide is incorporated to enhance coating uniformity and surface characteristics due to its high hydrophilicity and functional groups (An et al., 2017; Sun et al., 2022).

1.3 Research Gap

The utilization of cellulose-based materials with PVDF hollow fiber membranes has predominantly been achieved through blending and crosslinking strategies to form mixed-matrix or composite membranes (Lv, Zhang, Zhang, Zhao, et al., 2018a; Wu et al., 2024; Koriem et al., 2023). While these approaches enhance membrane hydrophilicity and permeability, they primarily modify the bulk structure rather than directly targeting the membrane surface, where key water transport interactions occur (Liu et al., 2011; Geise et al., 2010). Consequently, the development of surface coating strategies for PVDF hollow fiber membranes remains insufficiently investigated. A key challenge in this area is the stable deposition of hydrophilic cellulose onto the inherently hydrophobic PVDF surface, as its low surface energy limits interfacial interactions and coating stability (Kang & Cao, 2014; Liu et al., 2011).

Previous studies have demonstrated that surface modification using intermediate functional layers can improve compatibility and adhesion (Zhang et al., 2016; Wang et al., 2021). In addition, PVA and PVDF polymers have shown good compatibility within membrane systems and the ability to enhance hydrophilicity, permeability, and water flux performance (Li et al., 2010; Li et al., 2019; Razmgar et al., 2018). These characteristics suggest their potential to function as polymeric binders in cellulose-based surface coatings. However, their roles in such coating systems have not been systematically compared. In particular, the influence of different binder systems on surface chemistry, morphology, structural characteristics (e.g., crystallinity and porosity), and water flux performance remains insufficiently investigated.

In addition, conventional coating techniques such as spraying and simple dip-coat-dry processes often result in non-uniform layers and limited control over coating morphology (Zhang et al., 2016). Dip-coating in combination with phase inversion technique offers the potential to form a more uniform and stable secondary membrane layer through controlled solvent–nonsolvent exchange, improving the integration of coating layer and membrane substrate (Awanis Hashim et al., 2012; Ma et al., 2017; Baker, 2012). In addition, graphene oxide (GO), known for its high hydrophilicity and functional groups, has been widely incorporated into membrane systems to enhance surface characteristics (An et al., 2017; Sun et al., 2022; Perreault et al., 2015). However, its role within cellulose/polymer coating systems remains insufficiently investigated.

1.4 Hypothesis

- i. The tested water flux, morphological, infrared spectral, and structural characteristics (e.g. crystallinity and porosity) shown increased water treatment performance for

coated PVDF hollow fiber membrane samples in comparison to the PVDF hollow fiber membrane control sample.

- ii. The bamboo cellulose loading in the coating formulation will influence the amount of coating deposition on the surface of the membrane. The cellulose may improve surface hydrophilicity, however higher cellulose loading may increase surface coverage, which could partially restrict pore accessibility, and reduce water transport, Hence, the optimal pure water flux is expected to occur at lower cellulose weight ratios.
- iii. The added graphene oxide may show signs of increase porosity due to their nanoparticles; this may lead to improve pure water flux.

1.5 Objectives

- i. To develop bamboo cellulose/polymer coating using PVA and PVDF polymer as separate binders, with and without graphene oxide additives, for depositing onto the surface of pristine PVDF hollow fiber membranes via dip-coating coupled with phase inversion.
- ii. To characterize the effects of the coating formulations regarding surface chemistry, morphology, structural characteristics, porosity, and pure water flux performance of PVDF hollow fiber membranes.
- iii. To determine the optimal coated PVDF hollow fiber membrane based on pure water flux performance, evaluating the practical filtration performance using rainwater, river water, and peat water samples, including iron removal analysis using Atomic Absorption Spectroscopy (AAS).

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This literature review highlights the development of bamboo cellulose/polymer coated PVDF hollow fiber membranes for water treatment applications. Firstly, the literature review discusses on membrane technology and membrane separation processes before focusing on the types of membranes used in water treatment applications. The subsequent sections include literature reviews on hollow fiber membranes, PVDF membrane properties, PVDF hydrophobicity, surface modification strategies, bamboo cellulose and coating strategies, polymer binders, additives such as graphene oxide, membrane performance evaluation and methods for characterization. The literature review also highlights relevance of real water (e.g. peat, river and rain water) flux evaluation and iron removal analysis after filtration.

Furthermore, it is also structured to address the research gap in regards to the development of a cellulose/polymer coating systems for surface modification via coating onto pristine PVDF hollow fiber membrane. While past studies demonstrated the potential of cellulose and cellulose derivatives, PVA, PVDF and graphene oxide in improving membrane hydrophilicity and water permeability, majority of these studies focuses on blended or newly fabricated composite membranes, in comparison to post fabrication surface modification coating of pristine PVDF hollow fiber membranes specifically. In addition, there are limited studies conducted an investigation regarding polymer binders influence as coating, surface morphology, pore related properties, water flux output and filtration performance.

Hence, this literature review considers the relevance of utilizing real water sources (e.g. peat water, river water and rainwater) and comparing it with deionized water as a pure water flux baseline performance. The addition of heavy metal removal (e.g. iron) analysis after filtration further contextualizes the filtration performance for real water application of the coated membranes beyond just pure water flux performance. This inclusion of filtration performance positions this thesis research within the research gap between material coating development, fundamental membrane characterisation and practical water treatment evaluation.

2.2 Membrane Technology for Water Treatment

Membrane technology has an essential role for water treatment applications, due to the membranes ability to separate contaminants from feed concentrates (e.g. raw water sources) based on the particle size, electrical charge, water diffusivity and solubility. In comparison to older conventional water treatment processes, membrane systems could be developed and categorized depending on membrane pore size, material properties, module configuration, and operating pressure (R. W. Baker 2004). Polymeric membranes are widely utilized due to its characteristics when using the Loeb-Sourirajan technique (phase inversion process) of membrane development. The phase inversion could be described as changing a one-phase casting solution into two separate phases. During the phase inversion process, a liquid polymer solution is precipitated (e.g. in a water bath) into two phases; a solid, polymer rich phase that forms the main bulk of the membrane and a liquid, a polymer poor phase which forms the membrane pores (R. W. Baker 2004). The phase inversion process enables progress in membrane technology regarding interfacial polymerization, multilayer composite casting and coating for high performance membranes (R. W. Baker 2004).

In general, membrane filtration systems could be classified into two main operating modes, which are; dead end filtration and cross flow filtration. In dead end filtration system, feed concentrate stream moves perpendicular to the membrane surface. This operating mode allows suspended particles and rejected solutes to gradually accumulate and deposited on the surface of the membrane. This accumulation leads to the formation of a cake layer on the surface, which decreases permeate water flux over periods of time operating (R. W. Baker 2004). In contrast, cross flow filtration system, feed concentrate stream flows tangentially along the membrane surface, which allows circulation of feed concentrate and help limit the accumulation of suspended particles and rejected solutes, hence reducing fouling. Furthermore, cross flow filtration systems produce two set of streams, which is the clean particle free permeate and a mixture of retentate and feed concentrate (due to circulating system). The equipment required for cross flow filtration is more complex, but the membrane lifetime is expected to last longer than dead end filtration system (R. W. Baker 2004). In contrast, phase inversion membranes could be fabricated into various module configuration, depending on the intended water treatment application. Module configurations include plate and frame (e.g. using flat sheet membranes), hollow fiber, spiral wound, tubular, vibrating, and rotating membrane modules (Xu et al. 2022; R. W. Baker 2004). Among these module configurations, hollow fiber membranes are particularly relevant to this research due to high membrane surface area per module volume hence higher permeate output and using a cross flow filtration system would lower retentate accumulation, which all are suitable for a compact water treatment system for rural regions in Borneo.

2.2.1 Membrane Separation Processes

The separation mechanism could be categorized via pore flow or solution diffusion separation mechanisms. In pore flow membranes, water and solutes move through

membrane the pores, and the separation rely on the pore size and solute particle size. The pore flow separation mechanism is commonly associated with microfiltration (MF) and ultrafiltration (UF) membranes. On the other hand, solution diffusion membranes are generally dense or non porous, where solute first dissolve into the membrane and then diffuse through the membrane under a pressure driven force. The solution diffusion mechanism is mainly associated with nanofiltration (NF) and reverse osmosis (RO) membranes (Xu et al. 2022, R. W. Baker 2004; R. Baker 2012).

A membrane separation performance is highly influenced by the membrane pore size, pore structure, porosity, hydrophilic behaviour, surface chemistry and the operating pressure. Hence, a membrane separation performance is usually evaluated via parameters such as water flux, rejection coefficient, porosity, mean pore size, and flux recovery ratio (R. W. Baker 2004; R. Baker 2012). Mainly, water flux, porosity and mean pore size are sufficient for a fundamental evaluation of a membrane water treatment performance. Relative to this current research, these parameters are significant for phase inversion membranes, where the membrane morphology is strongly influenced by the concentration of the polymer based casting solution, casting solution modifiers (e.g. binders and additives) and the precipitation medium (e.g. deionized water bath) (R. W. Baker 2004; R. Baker 2012). Some of these parameters are utilized by past studies to evaluate PVDF and cellulose based membranes correlate membrane structure to the filtration performance, which includes water flux, porosity, and mean pore size (Liu et al., 2011; Hossein Razzaghi et al., 2014; Malucelli et al., 2021; Wang et al., 2021; Wu et al., 2024).

This current research also utilizes the fundamental evaluation of the membrane filtration performance related to the coated and uncoated PVDF hollow fiber membranes. These evaluation of membrane separation performance showcases properties which directly

influence how water passes through the membrane and how the coated surface interacts with different water sources (e.g. deionized water, peat water, river water, and rainwater).

2.2.2 Types of Membranes for water treatment

Based on a studies on polymer based membranes for drinking water purification, the membranes used for water purification could be classified similarly in this chapter section 2.2 and 2.2.1 according to the membrane separation mechanism (pore flow and solution diffusion), operating mode (dead end and cross flow), and module configuration type (plate and frame, hollow fiber, spiral wound, tubular, vibrating, and rotating) (Xu et al. 2022, R. W. Baker 2004; R. Baker 2012). In contrast, there are mainly 4 types of membranes for water purification, categorized according to the pore size:

- i. Microfiltration (MF)
- ii. Ultrafiltration (UF)
- iii. Nanofiltration (NF)
- iv. Reverse osmosis (RO)

Classification via pore size commonly used due to its direct effect on the filtration of specific contaminants. (Xu et al. 2022). The MF membranes have pores with a size range of 0.1 to 5 μm , commonly used in purifying drinking water. These membranes can effectively filter particles, asbestos, and bacteria with a diameter of 0.1 to 10 μm at low pressure (less than 2 psi) or via gravity filtration (H. Ma, Chu, and Hsiao 2012). MF membranes typically function only through the pore-flow mechanism. With pore sizes ranging from 0.01 to 0.1 μm , UF membranes can effectively filter out dissolved bio-macromolecules like proteins, viruses, and pyrogens. As a result, UF membranes have a broad range of uses in both industrial and community wastewater treatment, with typical operating pressure of 10-30 psi. The structure of UF membranes typically consists of an anisotropic Loeb and Sourirajan

structure and operates based on pore-flow. NF membranes, on the other hand, are utilized to filter substances with sizes ranging from 0.001 to 0.01 μm , such as multivalent ions and surfactants. These membranes operate through a combination of pore-flow and solution-diffusion and typically require a pressure of 100 psi (Lee, Elam, and Darling 2016; Geise et al. 2010; Nyuk Khui et al. 2025). RO membranes are known to be non-porous in comparison to other types of membranes such as MF, UF, and NF, with pore sizes less than 1 nm. They work based on the mechanism of solution-diffusion, where solutes pass through the membrane by dissolving in the membrane structure and moving through it due to applied pressure. RO membranes are typically used for desalinating brackish groundwater and seawater (Lee, Elam, and Darling 2016; Xu et al. 2022; R. Baker 2012; Nyuk Khui et al. 2025).

2.3 Hollow Fiber Membranes for Water Treatment

Hollow fiber membranes have made a big impact on the advancement of membrane technology across different industries. This is due to the hollow fiber membranes unique mechanical properties, where the pore development of the membrane and shape creates a self supporting structure and the ease of scaling via modular units for a wide range of applications (Kamaludin et al. 2022). The most prominent feature of hollow fiber membranes is their large surface area per module volume, if compared to flat sheet membranes, the bundles of hollow fiber membranes has an effective filtration output, increasing water flux performance (Kamaludin et al. 2022; Asiri et al. 2024). Hollow fiber membranes could be used in a variety of filtration processes with industry applications includes; membrane bioreactors, reverse osmosis pre-treatment, industrial water or wastewater treatments (Kamaludin et al. 2022; Akhondi et al. 2017). This configuration enables hollow fiber membranes to have a larger effective filtration surface area within a small module volume,

hence making them an effective filtration unit for water treatment applications (R. W. Baker, 2012; Akhondi et al., 2017). This is especially useful for rural regions in Borneo where space could be limited and logistics are a challenge. The common water treatment processes for hollow fiber membranes include microfiltration and ultrafiltration processes, where the pore flow separation mechanism dictate the removal of suspended solids, organic matter, microorganisms and other particulates solute/contaminants (Kang & Cao, 2014; R. W. Baker, 2012). This is effectively useful for treating raw water sources from the rural regions of Borneo (e.g. peat, river and rainwater). However, although hollow fiber membranes could remove suspended solids, organic matter, microorganisms from raw water sources, their performance are still limited by fouling, pore blockage, hydrophobic surface properties, and mechanical stress during operation (Akhondi et al., 2017; Rana & Matsuura, 2010). Inherently hydrophobic polymers such as PVDF as hollow fiber membrane are particularly affected by these limitations. Hence, surface modification techniques and coating strategies are often incorporated during development to enhance the PVDF hollow fiber membrane hydrophilicity, flux stability and filtration performance (Kang & Cao, 2014; R. W. Baker, 2012).

2.4 Polyvinylidene fluoride (PVDF) as Hollow Fiber Membrane

Polyvinylidene fluoride (PVDF) is a semicrystalline polymer, which exhibits good mechanical strength, thermal stability, chemical resistance and good aging resistance, which benefits water treatment operation longevity (Kang and Cao, 2014; Gopakumar et al. 2017b). The membrane forming capabilities of PVDF polymer ranges from the development flat sheets, hollow fibers or tubular membranes. In contrast, PVDF for hollow fiber membrane development is widely produced via Loeb-Sourirajan non-solvent induced phase separation process with a hollow fiber spinneret system (Kang and Cao, 2014; R. W. Baker, 2004).

PVDF hollow fiber membranes are widely utilized for water treatment application as it provides a higher packing density and higher membrane surface area within a compact system (R. W. Baker 2012). The shape and size of the hollow fiber allow large numbers to be packed into a single system, hence increasing the filtration output over a smaller operating area (R. W. Baker 2012). The PVDF hollow fiber membranes water treatment performance is highly influenced by membrane morphology, pore structure, surface chemistry, and hydrophilicity. However, pristine PVDF characteristically is hydrophobic in nature, hence could restrict the water transport effectiveness and increase fouling tendency. Therefore, surface modification techniques and coating formulations are common place in membrane technology research, with the goal to improve the hydrophilic properties and water flux performance of PVDF based membrane systems (Liu et al., 2011; Kang & Cao, 2014).

2.4.1 Comparison Between PVDF to other Polymers

Polymeric membranes are widely used in water treatment because they are relatively easy to fabricate, cost-effective, and adaptable into different configurations such as flat sheet, tubular, and hollow fiber modules. Examples of commonly utilized polymers for membrane development include Polyvinylidene fluoride (PVDF), polysulfone (PS), polyethersulfone (PES), cellulose acetate (CA) and polyamide (PA). Generally, all polymers have their specific advantageous properties and limitations, depending on the desired application, operating environment, membrane configuration and the desired separation process (Geise et al., 2010; Lee et al., 2016).

PVDF as a polymeric membrane, has some advantages in comparison to other polymeric membranes. In regards to chemical resistance and thermal stability, PVDF is considerably more durable under challenging operating conditions in comparison to cellulose based membranes (e.g. cellulose acetate and nanocellulose) which are much more

sensitive towards chemical degradation and pH variations. On the other hand, cellulose based membranes are more hydrophilic in comparison to PVDF based membranes, hence compensates for the lack of durability with better filtration capabilities due to its hydrophilic properties (Liu et al., 2011; Kang and Cao, 2014). The importance of good mechanical properties for hollow fiber membranes is essential, as the membrane fibers must be able to withstand various pressure fluctuations, flowrates, handling during maintenance and assembly, as well as varied exposure to different contaminant particle size (membrane contact damage). Hence, the rationale behind PVDF selection as the ideal polymer for hollow fiber membrane for this thesis research (Rana and Matsuura 2010; Liu et al., 2011; Kang and Cao, 2014).

Recent studies focus on blending PVDF as the main membrane matrix with hydrophilic materials such as cellulose, cellulose nanocrystals, cellulose nanofibers, polyvinyl alcohol (PVA), and graphene oxide (GO) to enhance structural properties, durability, improved surface wettability and water flux performance (Bai et al., 2012; Gopakumar et al., 2017a; Wang et al., 2021; Acarer-Arat et al., 2024). The approach by recent studies is principally relevant to this research, where bamboo derived cellulose is utilized as part of a coating system following similar phase inversion technique for deposition, modifying the surface properties of pristine PVDF hollow fiber membranes.

The comparison of the hydrophilic or hydrophobic properties PVDF, PES and PS based membranes for water treatment applications, PVDF is considered to be more hydrophobic than PES and PS membranes. The hydrophobic properties correlates to the structural formation of the membrane and structural stability, however it produces a challenge for water treatment application, due to hydrophobic surfaces interact weakly with water molecules (Liu et al., 2011). The utilization of PES and PS polymers as membranes

are attributed to their good film forming ability and thermal stability, however often requires additional hydrophilic materials and surface modification as well, in order to improve fouling resistance and water permeability (Geise et al., 2010; Lee, Elam, and Darling, 2016).

Overall, PVDF offers a strong combination of mechanical strength, chemical stability, thermal resistance, and membrane-forming ability. Nevertheless, its hydrophobic nature limits its direct performance in water filtration. An increase in hydrophilicity is believed to enhance water filtration performance for membranes. The formation of a pure water layer on a highly hydrophilic surface could prevent the adhesion and accumulation of hydrophobic contaminants and improve water filtration performance (Rana and Matsuura 2010; Kang and Cao 2014). As a result, majority of the current research conducted to make PVDF membranes more hydrophilic through various methods, including improving the preparation process and modifying existing membranes. However, the highly hydrophobic nature and low surface energy of PVDF membranes result in poor anti-fouling performance and low permeability, making them challenging for practical use (He et al. 2017; Nyuk Khui et al. 2025). One alternative to overcome these drawbacks without compromising much on the base properties of the PVDF membrane is to incorporate hydrophilic materials such as cellulose, nanocellulose and/or other cellulose derivatives via coating (H. Bai et al. 2012; Lv, Zhang, Zhang, Zhao, et al. 2018b; Song Zhang et al. 2016; Wang et al. 2021; X. Yu et al. 2024).

2.5 Cellulose for PVDF Membrane Surface Modification

Cellulose is known to be a sustainable organic polymer which is in abundance in nature and largely used for membrane development and modifications. This is due to the cellulose properties such as hydrophilicity, low toxicity, water stability, and good mechanical properties (Peng et al. 2022). These properties presents cellulose as suitable

hydrophilic material for water treatment applications, where hydrophilicity, surface wettability, water flux and water transport mechanism are factors affecting filtration performance (Peng et al. 2022).

According to past studies, the advancements of cellulose and its derivatives have further improved the potential utilization of cellulose in water treatment applications, as cellulose could improve membrane surface area, incorporate more hydrophilic hydroxyl groups and enhanced surface functionality of the membrane (Mohammed, Grishkewich, and Tam 2018; Yang et.al, 2021). In addition, studies also demonstrated that the incorporation of cellulose derivatives such as nanocellulose, cellulose nanocrystals and cellulose nanofibers as a matrix blend, improves membrane hydrophilicity, surface functionality and water filtration performance (Mohammed, Grishkewich, and Tam 2018; Yang et.al, 2021). In regards to ultrafiltration membranes, the incorporation of cellulose as a coating could improve the interaction between water molecules and the membrane surface due to the presence of the nanocellulose hydroxyl groups, hence supporting surface wettability and water permeability. In past studies, nanocellulose modified polymeric membranes could reduce occurrence of biofouling and improve their utilization for wastewater treatments (Yang et al. 2021).

There are several methods which have been reported for the incorporation of nanocellulose into polymeric membranes, which are; the preparation of hybrid nanocomposite membrane with the blending of cellulose nanocrystals and/or cellulose nanofibers with polymer solutions. The development of hybrid nanofibrous polymeric membranes via impregnation of cellulose nanocrystals and/or cellulose nanofibers into electrospun scaffolding, and the fabrication of thin film blended nanocomposite polymeric

membranes through vacuum filtration or solution casting onto polymer substrates (Abouzeid et al. 2018).

2.5.1 Comparison between Cellulose Matrix Blend and Coating

In regards to PVDF hollow fiber membranes, the utilization of cellulose based surface modification techniques is particularly relevant, this due to the pristine PVDF hollow fiber membrane inherently hydrophobic nature, which limits surface wettability and potential water flux performance. The incorporation of cellulose as a coating layer could act as a surface modification technique by introducing hydroxyl groups on the surface of the PVDF hollow fiber membrane, hence improving the PVDF hollow fiber membrane affinity toward water molecules. This surface modification technique via coating application has great potential in improving surface wettability while also influencing surface roughness, porosity and pore related properties which overall influences the PVDF hollow fiber membrane water flux performance. This matches the role of cellulose molecular coating in improving the hydrophilic surface functions of PVDF membranes (Wang et al., 2021).

The cellulose coating on pristine PVDF hollow fiber membrane only functions as a surface modification technique, while in comparison to blended matrix modification techniques, cellulose material is incorporated directly into the polymer casting solution before membrane formation. According to past studies, this allows the cellulose to influence the internal membrane structure, porosity and pore formation, mechanical properties and hydrophilicity throughout the membrane matrix (Abouzeid et al., 2018; Mohammed et al., 2018). A cellulose blend matrix approach could improve overall membrane performance, however, the distribution of cellulose within the polymer matrix may be affected by possible aggregates and issues may arise regarding compatibility with the main polymer matrix when

undergoing phase inversion process for membrane development (Abouzeid et al., 2018; Mohammed et al., 2018).

In contrast, cellulose as surface modification coating applied onto the surface of an already formed membrane, such as a PVDF hollow fiber membrane, the modification is mainly concentrated at the surface level membrane to water interface. This allows a more water molecules to be in direct exposure of the cellulose hydroxyl groups, hence, could improve membrane surface wettability and water transport mechanism for improved pure water flux performance (Wang et al., 2021). On the other hand, the effectiveness of cellulose coating depends strongly on coating developed, solvent system for coating layer formation uniformity, cellulose distribution, membrane surface adherence as well coating stability. As irregular cellulose regeneration or aggregation may alter overall membrane surface porosity and pore related properties (Medronho & Lindman, 2014; Li et al., 2012). Hence, while cellulose blended matrix membranes may be able to modify the bulk properties, cellulose coating on PVDF hollow fiber membranes offers a targeted surface modification strategy which particularly useful for improving the hydrophilicity of readily available pristine PVDF hollow fiber membrane in the market, without significantly altering much of the original hollow fiber main characteristics.

2.5.2 Bamboo Cellulose Coating on PVDF Hollow Fiber Membrane

In this thesis research, bamboo derived cellulose was synthesized and used as the main coating material for the formulation of coating solution, due to bamboo being a sustainable and fast growing lignocellulosic biomass source for cellulose production apart from purely solid wood trees. Bamboo contains cellulose could be extracted through various chemical and mechanical treatments, making it a suitable raw material source for developing a bio based membrane surface modifying coating material (Liew et al., 2015). The utilization

of bamboo derived cellulose supports a more sustainable approach towards surface modification of pristine PVDF hollow fiber membrane by utilizing natural biomass sources local to the rural region of Borneo, while improving membrane surface hydrophilicity and membrane water interactions (Bai et al., 2012; Wang et al., 2021; Wu et al., 2024). Therefore, bamboo cellulose as coating material is expected to contribute towards a hydroxyl rich surface functionality which could enhance water interaction and improve pure water flux performance of pristine PVDF hollow fiber membrane (Abouzeid et al., 2018; Mohammed Nishil, Grishkewich, and Tam, 2018).

In order to utilize bamboo cellulose as coating, a coating solution procedure must be incorporated in this research. Hence, the dissolution of bamboo cellulose for coating application is an important step as cellulose has strong intra and intermolecular hydrogen bondings, making it difficult to dissolve in most commonly used solvents. In this research, the cellulose coating solution is developed using dimethylacetamide/lithium chloride (DMAc/LiCl) cellulose dissolution system, which follows selected parameters as reported by Sadeghifar et al. (2019) regarding cellulose weightage and solvent system (Sadeghifar et al. 2019). In addition, according to Sadeghifar et al. (2019) research, cellulose films was successfully developed via Loeb Sourirajan phase inversion technique in a water bath, where the cellulose films demonstrate good hydrophilic, mechanical, thermal, and ultraviolet resistance properties (Sadeghifar et al. 2019). Sadeghifar et al. (2019) research findings support this thesis research regarding the suitability of utilizing DMAc/LiCl as cellulose dissolution system for forming a stable cellulose based layer, adapted for coating applications on the surface of pristine PVDF hollow fiber membrane (Sadeghifar et al. 2019).

In regards to the arrangement and stability of cellulose on the membrane surface as coating depends on the exposure of hydroxyl groups at the membrane water interface during

filtration operations. If cellulose within the coating solution is poorly distributed, the resulting coating may have irregular and poor porosity, non-uniform pore size and reduces pore accessibility. In addition, with such surface structural irregularities, it could influence water transport mechanism and may limit the improvement in water flux performance. Hence, the dissolution and regeneration behavior of cellulose plays an important role in determining the precipitation (surface coating layer forming) during phase inversion, final surface morphology and stability of the coating layer. (Medronho and Lindman 2014; Wang et al. 2021; R. Li, Zhang, and Xu 2012).

Furthermore, it is possible that the developed cellulose solution may aggregate or has poor coating stability during phase inversion which limits functionality as a surface modification material for the pristine PVDF hollow fiber membrane. An unstable or uneven cellulose coating may not adhere, partially cause pore blockage and reduces its effective water transport channels. Even though cellulose coating has been shown to improve hydrophilic surface properties of PVDF membranes, overall, the coating structure, distribution and stability must be carefully formulated to successfully modify the membrane performance (Wang et al., 2021). Hence, while cellulose may be a potential sustainable hydrophilic water treatment membrane modifier, optimization of the bamboo cellulose coating formulation is necessary to achieve improved wettability, coating stability, and pure water flux performance.

2.6 Polymer Binders in Cellulose Coating System

To overcome the limitation of cellulose coating system via DMac/LiCl solvent system, in this thesis research, the use of polymers as a binder in the cellulose solution is suggested. The concept of utilizing similar polymers as binder material may provide good adhesion between the cellulose and the neat PVDF hollow fiber membrane surface. Hence,

may produce good coating stability for the cellulose due binder chemical compatibilities to the neat PVDF hollow fiber membrane. Polyvinylidene fluoride (PVDF) and Polyvinyl alcohol (PVA) was suggested as binder material and Graphene oxide (GO) additive for this project.

The use of PVDF as a binder for membrane coating application provides numerous synergistic effects when combined with a hydrophilic material such as cellulose. Surface coating using cellulose alone often led to poor adhesion and lacks mechanical integrity onto the hydrophobic PVDF hollow fiber membrane surface. For the formulation of Cellulose/PVDF coating solution, the PVDF binder plays a stabilizing role which suppresses excessive swelling or dissolution of cellulose in aqueous environments (Adaval et al. 2022). This is important parameter for preserving overall coating stability and water treatment performance of the coated PVDF hollow fiber membrane. Even though the PVDF binder is hydrophobic nature, the robust film forming ability of PVDF binder could integrated during the dissolution of cellulose coating solution, hence, assisting adherence of cellulose onto the pristine PVDF hollow fiber membrane surface. Overall, the cellulose/PVDF coating strategy may provide a balanced solution by combining the hydrophilic properties of cellulose with the structural and adhesive strength of PVDF binder (Varanasi, Low, and Batchelor 2015; Joshi et al. 2023; Wang et al. 2021; Malucelli et al. 2021; X. Bai et al. 2025; G N et al. 2020).

Similarly, the use of PVA as binder for membrane coating application provides numerous synergistic effects when combined with another hydrophilic material such as cellulose. The concept for using PVA the same as PVDF as a binder material, where it also provides good film forming capabilities allow the coating to form a stable and continuous layer. The differences lie with PVA strong hydrogen bonding with cellulose and the

integration of both materials on the PVDF hollow fiber membrane surface may significantly improves hydrophilicity compared to uncoated PVDF membranes (M. Li et al. 2019; Choi and Nam 2025; N. Li et al. 2010; Çifci and Kaya 2010).

The utilization of both Cellulose/PVDF and Cellulose/PVA formulated coatings may improve the hydrophilic properties and pure water flux performance of pristine PVDF hollow fiber membrane. However, it should be noted that both coatings may differ in surface coating structure development during phase inversion (e.g. pores and porosity), adhesion, stability. As for cellulose/PVA coatings, PVA acts as a hydrophilic binder which forms hydrogen bonds with cellulose, which may improve water permeability and coating flexibility during applications. However, PVA is water soluble and may require further crosslinking procedures for long term durability for the coated membranes. In contrast, cellulose/PVDF coatings uses a hydrophobic but compatible PVDF binder to anchor cellulose onto the PVDF hollow fiber membrane surface. As result, it may improve mechanical stability and resistance to delamination under active filtration. The characterization of Cellulose/PVDF and Cellulose/PVA coated PVDF hollow fiber membrane is investigated in this project for further understanding of their capabilities.

2.7 Graphene Oxide as Additive in Cellulose/Polymer Coating System

In this thesis research, Graphene oxide (GO) is added as an additive during the developed of Cellulose/PVDF and Cellulose/PVA coating solution. The characteristics of the added graphene oxide in separate coating systems are investigated for improved hydrophilic properties, pore related properties and water flux performances of coated pristine PVDF hollow fiber membrane. Graphene oxide has a considerably high surface area and has large amounts of oxygen containing functional groups ($-\text{OH}$, $-\text{COOH}$, $-\text{C=O}$) (Ayyaru,

Linh, and Ahn 2019). The incorporation of graphene oxide in both Cellulose/PVDF and Cellulose/PVA coating solution may improve cellulose distribution and adhesive properties for anchoring the formulated coating onto the surface of pristine PVDF hollow fiber membrane. Past studies claim graphene oxide utilized at low loadings at around 0.1 to 0.5 wt% enhances the membrane hydrophilic properties, structural integrity and pure water flux performances without compromising the membrane mechanical stability. The graphene oxide as additive may be multifunctional role, which makes it a valuable additive in blended coatings aimed at improving pure water flux performance and durability of PVDF hollow fiber membranes (Y. L. Liu et al. 2020; Ayyaru, Linh, and Ahn 2019; An et al. 2017; Majumder and Gangopadhyay 2022; Perreault, Fonseca de Faria, and Elimelech 2015; Sahu et al. 2023).

According to a study by Ayyaru, Linh, and Ahn (2019), it reports regarding the incorporation of graphene oxide with zinc oxide (ZnO) as a blend in PVDF ultrafiltration membranes could enhance antifouling properties and membrane hydrophilicity, which indicate the role of graphene oxide as a functional hydrophilic additive (Ayyaru, Linh, and Ahn 2019). Similarly, according to Lv et al. (2018), their study reports regarding graphene oxide/cellulose nanocrystal functionalised PVDF membrane has exhibited improved surface properties and antifouling behaviour. This suggest that graphene oxide could interact well with cellulose based materials and in PVDF membrane systems. Overall, in this thesis research graphene oxide is considered as a functional additive which may enhance the cellulose/polymer coating system, however its characteristic effect must be evaluated in compilation with SEM morphological analysis, porosity, mean pore size, FTIR, XRD and water flux performances.

2.8 Pure Water Flux Performance

Pure water flux performance is an important variable used to analyze the permeability performance of the PVDF hollow fiber membrane samples. In particular to water treatment or filtration applications, it serves as a benchmark characterization for the PVDF hollow fiber membrane samples. The pure water flux performance refers to the volumetric flow rate of distilled or deionized water permeating through the PVDF hollow fiber membrane under a working pressure, regulated by the properties of the PVDF hollow fiber membrane such as area and porosity over time (Awanis Hashim et al. 2012; Asiri et al. 2024; Akhondi et al. 2017). The pure water flux is calculated according to the equation below (Awanis Hashim et al. 2012):

$$\text{Pure water flux, } JW = \frac{V}{ATP} \quad \text{Equation 2.1}$$

JW: Pure water flux (L/m²·h·bar)

V: Pure water permeated volume (L)

A: Effective area of membrane (m²)

T: Filtration time (h)

P: Functioning pressure (bar)

The pure water flux performance gives an understanding about the PVDF hollow fiber membrane porosity, pore size and hydrophilicity. A high pure water flux generally indicates good water permeability, which is desired for water treatment applications with high output. On the other hand, a high pure water flux may also indicate high porosity percentage with large pore size, in return enables good water permeability. In contrast, subsequent porosity analysis supplements the pure water flux results, as it could identify

changes in porosity percentage and pore sizes and the correlation to water flux performance (Trisnawati, Suryanti, and Pramono 2024; Hossein Razzaghi et al. 2014; Acarer Arat et al. 2025).

The influence of modification techniques regarding water flux performance are not always positive and/or linear. Even though the hydrophilic coating manages to improve wettability, the excess coating deposition may influence membrane pore blockage, hence also influencing hydraulic resistance. According to Li et al. (2010), increased PVA content in PVDF/PVA blend hollow fiber membrane may increase pure water flux performance, but it also reduces the membrane rejection performance (Li et al. 2010). Li et al. (2010) highlights a common flux selectivity trade off observed when developing water treatment membrane. Some for example of pure water flux performance of various modified PVDF membrane could be observed in Table 2.1. Overall, regarding pure water flux performance for this research, the surface coating modification or the blending of different hydrophilic additives like graphene oxide, PVA and cellulose may lead to improving the pure water flux by pore development for water transport channels and improving water wettability.

Table 2.1: Examples of Pure Water Flux Performance of Various Modified PVDF Based Membranes

Type of Membrane	Highest Pure Water Flux Performance	Study Highlights	References
Wet Spinning surface modification of PVDF hollow fiber membrane	406.200 L m ⁻² h ⁻¹ at 1 bar	Modified membranes demonstrate enhanced pure water flux performance, approximately 400-fold higher in comparison to the control PVDF hollow fiber membrane.	Awanis Hashim et al. (2012)

Table 2.1 continued

PVDF blended nanocrystalline cellulose ultrafiltration membrane	230.800 L m ⁻² h ⁻¹ ,	The modified composite blend membrane demonstrates an increased in pure water flux performance up to 47.5% in comparison to the pristine PVDF membrane.	Bai et al. (2012)
Composite PVDF membrane fabricated via cellulose molecular coating	9591.000 L m ⁻² h ⁻¹ ,	Develop cellulose molecular coating demonstrated significant enhancement of water flux performance in comparison to the control, of approximately 14 times the value.	Wang et al. (2021)
Direct coating of cellulose hydrogel on PVDF membrane	2675.200 L m ⁻² h ⁻¹	Study highlight influence of direct coating could improve separation performance; however, their application is based on oil/water separation.	X. Yu et al. (2024)
PVDF membrane with Nanocellulose	228 L m ⁻² h ⁻¹	Shows nanocellulose can improve PVDF membrane flux and rejection performance	Wu et al. (2024)
PVDF/PVA blend hollow fiber membrane	>140 L m ⁻² h ⁻¹	The pure water flux increased with increased PVA content, study also highlights flux selectivity trade off with increased PVA content.	Li et al. (2010)

Based on the studies in table 2.1, modification via incorporation of hydrophilic properties from hydrophilic materials generally enhances PVDF membrane pure water flux

by increasing surface wettability and improving water transport channels, reducing hydraulic resistances (Wang et al., 2021; Awanis Hashim et al. 2012; Li et al., 2010). However, pure water flux improvement depends strongly on the type of hydrophilic material, technique of depositing the hydrophilic material and formulation. Cellulose may enhance hydrophilic properties as well as improving pore structure, while PVA could increase water affinity via available hydrophilic functional groups (Bai et al., 2012; Lv, Zhang, Zhang, Zhao, et al., 2018b; Wang et al., 2021; Wu et al., 2024; Li et al., 2010; Li et al., 2019). The addition of graphene oxide may also contribute to hydrophilic properties due available oxygen containing functional groups, however due to the physical properties of graphene oxide itself, there is possibility of excessive loading may cause aggregation or pore blockage, even at lower weightages (An et al., 2017; Ayyaru, Linh, and Ahn, 2019; Majumder and Gangopadhyay, 2022; Sun et al., 2022). Hence, membrane modification do involve trade offs between improved water flux, coating stability, pore related properties and water transport channels. This highlights this thesis research, at it evaluates pure water flux performance in relation to porosity, mean pore size, morphology and iron removal performance for real water sources (e.g. peat, river and rain water) rather than relying on simply pure water flux alone as performance parameter.

2.9 Porosity and Mean Pore Size

The analysis of porosity and mean pore size are important factors in supplementing the pure water flux analysis of the PVDF hollow fiber membrane samples. The porosity and mean pore size govern the transport and separation performance of PVDF hollow fiber membrane samples. The properties influence the PVDF hollow fiber membrane permeability, selectivity, and fouling resistance, which are important conditions in water treatment applications. Increased porosity with decreased mean pore size may lead to

decreased pure water flux, while increased porosity with increased mean pore size may lead to an increased pure water flux (Trisnawati, Suryanti, and Pramono 2024). The varied porosity and mean pore size will be able to showcase the surface coating capabilities of this project for the PVDF hollow fiber membrane. The membrane samples porosity and mean pore size could be determined via the gravimetric method based on equations below (Rahman et al. 2024; Yuliwati et al. 2011) :

$$Porosity, \varepsilon = \frac{\left(\frac{W_{wm} - W_{dm}}{\rho_{water}} \right)}{\left(\frac{W_{wm} - W_{dm}}{\rho_{water}} \right) + \left(\frac{W_{dm}}{\rho_{PVDF}} \right)} \quad \text{Equation 2.2}$$

Porosity Percentage, $\varepsilon\%$

$$= \frac{\left(\frac{W_{wm} - W_{dm}}{\rho_{water}} \right)}{\left(\frac{W_{wm} - W_{dm}}{\rho_{water}} \right) + \left(\frac{W_{dm}}{\rho_{PVDF}} \right)} \times 100 \quad \text{Equation 2.3}$$

W_{wm} : Weight of wet membrane (g)

W_{dm} : Weight of dry membrane (g)

ρ_{water} : Density of water (0.998 g/cm³)

ρ_{PVDF} : Density of PVDF polymer (1.740 g/cm³)

The mean pore size calculated following the Guerout-Elford-Ferry equation:

$$Mean\ Pore\ Radius\ (m) = \sqrt{\frac{(2.9 - 1.75\varepsilon) \times 8\eta LQ}{\varepsilon A(\Delta P)}} \quad \text{Equation 2.4}$$

ε : membrane porosity

η : water viscosity (0.00089 Pa·s)

L: membrane thickness (m)

Q: volume of water permeation (m³/s)

A: effective area of membrane (m²)

ΔP: pressure applied (Pa)

$$\text{Mean Pore Size (m)} = 2 \times (\text{Mean Pore Radius}) \quad \text{Equation 2.5}$$

The surface modification via coating could influence membrane porosity and mean pore size by either developing more selective porous layer on the surface or narrowing pore openings of the pristine PVDF hollow fiber membrane. The added hydrophilic coating materials such as bamboo cellulose may improve hydrophilicity at the surface contact area by increasing surface wettability and creating more water transport channels. On the other hand, excessive coating deposition may partially block membrane pores and reducing the mean pore size. An example is a study by Bai et al. (2012), which reports the developed PVDF/nanocrystalline cellulose membrane demonstrated improved pure water flux with increased porosity and modified pore structure (Bai et al. 2012). This result indicates the relationship between cellulose incorporation, pore characteristics and water permeability. In another by Li et al. (2010), their developed PVA modified PVDF hollow fiber membranes, reported as the PVA weightage increases, pure water flux also increases. However, this results also has a trade off, which is the reduction in rejection performance. This highlights relationship between hydrophilic materials added influencing filtration selectivity and between pore accessibility (Li et al. 2010). In regards to the addition of graphene oxide, the hydrophilic material may also affect pore structure and water flux due the available oxygen containing functional groups improving the membrane hydrophilicity. However, poor dispersion or excessive loading could cause aggregation and pore blockage during

applications (Ayyaru, Linh, and Ahn, 2019; Sun et al., 2022). Therefore, in cellulose/PVA/PVDF/GO coating systems for pristine PVDF hollow fiber membrane, the porosity and mean pore size should be interpreted together with pure water flux and morphology, in order to determine the coating improves water transport mechanism or restricting pore accessibility. Table 2.2 showcases some examples of porosity and comparative relationship with performance observation for modified PVDF membranes.

Table 2.2: Examples of Porosity and Performance Observations for Modified PVDF membranes

Type of Membrane	Porosity	Related performance observation	Reference
PVDF/nano crystalline cellulose blended flat sheet UF membrane	65%	Pure water flux at $230.8 \text{ L m}^{-2} \text{ h}^{-1}$, increased 47.5% higher than pristine PVDF membrane.	Bai et al. (2012)
PVDF hollow fiber membrane with PEG 400 additive with varied spinning conditions	>61.61% (PEG 400 loading), up to 43.39 (varied experimental air gap)	According to PEG 400 loading, the membrane increased porosity and pure flux. The experimental air gap influenced porosity and pore structure development.	Zakria et al. (2021)

Table 2.2 continued

PVDF/PVP flat sheet UF membrane modified with GO-PVA-NaAlg hydrogel	70.8–80.5%	Pure water flux increased up to $103.2 \text{ L m}^{-2} \text{ h}^{-1}$; however, excessive hydrogel caused pore blockage.	Moghadam and Hemmati (2023)
Hydrophilically modified PVDF/PVA blend flat sheet membrane	Up to 80% depending on polymer content formulation	According to the study, the higher the polymer concentration, it reduces porosity and flux, with PVA ratio affecting developed pore structure.	Zhang et al. (2016)
Porous PVDF-HFP hollow fiber membrane modified by stretching ratio	>80%	According to the study, the stretching ratio altered pore size while maintaining high porosity of the modified membrane.	Fazli et al. (2024)

2.10 Morphological Properties (Scanning Electron Microscopy SEM)

To study the morphological properties of coated PVDF hollow fiber membrane includes the use of morphological testing procedures. The test is carried out to analyze the surface structure of the membrane at a microstructure level to observe surface roughness, presence of voids, crack, fracture, etc (Choi and Nam 2025; Malucelli et al. 2021). The equipment used to analyze the surface microstructure of composite materials can be applied to membranes, which includes an optical microscope, scanning electron microscope, and transmission electron microscope, as according to ASTM E2015-04 (2021) as the testing

protocol. Utilizing scanning electron microscopy (SEM) for analyzing coated PVDF hollow fiber membrane surface microstructures, the key features to look out for in the imagery include the deformation, voids, cracks, fractures, visible pores and surface roughness.

The surface morphology is particularly important for coated PVDF hollow fiber membranes due different formulation of coating solution used in this research, hence may produce different surface features which directly influence water flux performance. The addition of bamboo cellulose, PVA and graphene oxide in this research may introduce more hydrophilic properties on surface layer, adding more porous structures and water transport channels, while not altering the main body of the pristine PVDF hollow fiber membrane (Bai et al., 2012; Wang et al., 2021; Wu et al., 2024). The PVA binder formulated coatings may form a smoother and more continuous hydrophilic film due to its film forming ability, however, similar to many cases on excessive coating deposition, it may cause pore blockage and reduces effective pore accessibility (Li et al., 2010; Choi and Nam, 2025). In contrast, for PVDF binder formulated coatings may also provide better compatibility with the pristine PVDF membrane surface. However, as the PVDF polymer binder is hydrophobic nature, it may limit surface wettability, if the coating formulation is not balanced with the hydrophilic bamboo cellulose coating formulation. Overall, the SEM analysis should be able to observe surface morphology such as cracks, voids, coating uniformity for coating deposition as well as pore visibility, surface roughness and possible agglomeration sites. These morphological characteristics are closely related to filtration or water flux performance, well distributed pores may support higher water flux results, pore blockage, uneven and thick coating or particle aggregation may increase hydraulic resistance during water treatment operations (Rana and Matsuura, 2010; He et al., 2017; Cruz-Silva et al., 2019).

2.11 Infrared Spectral Properties

The Fourier transforms infrared (FTIR) spectroscopy is an infrared spectral analysis via FTIR spectrometer. It is generally utilized to identify available functional groups of the sample tested. The test sample is exposed to infrared radiation and the frequency graph could be adjusted to identify either transmittance or absorbance. The infrared radiation causes the functional groups to vibrate, which were then recorded, analyzed and grouped accordingly. The functional groups could vibrate when exposed to infrared radiation, at certain frequencies the test sample could absorb the infrared radiation. Depending on the bonds and functional groups present in the test sample, the frequency graph recorded will show levels and trend of peaks for analysis. It is known that the vibration frequency causes peaks to appear, in which leads to the illustration of the molecule's 'fingerprint' (Aup-Ngoen and Noipitak 2020; Muvhiiwa et al. 2019; Mohamed et al. 2017).

The 3 spectrum types for FTIR analysis are far infrared, mid-infrared and near-infrared. The standard tested wavelength range for a sample following ASTM E168-16 (2016) and ASTM E1252-98 (2021) standards for qualitative and quantitative analysis are between 4000 cm^{-1} and 400 cm^{-1} , with mid-infrared range divided into 4 regions. The presence of the functional groups could be identified according to trends, significant spikes or wide peaks appearing in the region. The 4 recognizable regions are from 4000 cm^{-1} to 2500 cm^{-1} are single bond functional groups e.g., (O-H, C-H and N-H), from 2500 cm^{-1} to 2000 cm^{-1} are triple bond functional groups, from 2500 cm^{-1} to 2000 cm^{-1} are double bond functional groups and ranging from 1500 cm^{-1} to 600 cm^{-1} is recognize to be the tested sample 'fingerprint' region (Aup-Ngoen and Noipitak 2020; Muvhiiwa et al. 2019; Mohamed et al. 2017). In perspective of the coated PVDF hollow fiber membranes samples, the presence of functional groups would describe changes in chemical structures as result of

the different coating solutions e.g., (Cellulose/PVDF, Cellulose/PVA, graphene oxide) applied onto the surface of neat PVDF hollow fiber membrane.

In surface coated or matrix blend modified PVDF membrane studies, the utilization of FTIR is commonly used to identify available functional groups and to confirm the added surface coating have been successfully incorporated and improve hydrophilic properties of the PVDF membrane. In this research, bamboo cellulose based coatings PVDF membranes, the availability and/or increased intensity of broad O–H stretching bands around 3200–3600 cm^{-1} and C–O/C–O–C related bands in the fingerprint region are an indication of the presence of cellulose based materials and improved hydrophilic properties (Bai et al., 2012; Wang et al., 2021; Wu et al., 2024). For PVA binder formulated bamboo cellulose based coating systems, the availability and/or increased intensity of O–H and C–O related functionally group supports the presence of the PVA hydrophilic polymer components which supports improved water affinity and water flux performance (Li et al., 2010; Li et al., 2019; Choi and Nam, 2025). In contrast, with the addition of graphene oxide as additives in the formulated bamboo cellulose/polymer coating systems, FTIR could identify the presence of oxygen containing groups such as hydroxyl, carbonyl and carboxyl groups, which are associated with improved surface hydrophilicity and possible interaction with cellulose or the polymeric binders (e.g. PVDF and PVA) (Ayyaru, Linh, and Ahn, 2019; Sun et al., 2022; Majumder and Gangopadhyay, 2022). The FTIR analysis results of coated and uncoated PVDF hollow fiber membranes should be interpreted together with SEM, porosity and water flux performance data, as the presence of hydrophilic functional groups does not automatically confirm proper coating deposition and overall membrane water treatment performance. Thus, in this study, the FTIR analysis provides a chemical evidence of cellulose/polymer/graphene oxide surface coating formation, while the practical

significances are evaluated in relation to the membranes sample morphology, pore structure and water flux performances.

2.12 X-ray Diffraction Analysis (XRD)

X-ray Diffraction Analysis (XRD) is used to verify the range of crystallinity of the samples and to observe the availability of either a crystalline or amorphous phase. The samples were analyzed at room temperature using X-ray diffractometer with $\text{CuK}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The XRD analysis for samples is recorded within a scattering angle (2θ) ranging from 5 to 90° at a scanning rate of 2min^{-1} . This procedure is in accordance with the ASTM F3419-22 (2022) standard (ASTM F3419-22 2022). The sample preparation involves drying the membrane and placing it flat to ensure consistent reflection. The XRD analysis is crucial for confirming successful coating or additive incorporation, where reduced crystallinity or peak shifts can indicate blending, dispersion, or phase transformation within for the coated and uncoated PVDF hollow fiber membranes investigated in this project. The control neat PVDF hollow fiber membrane may exhibits characteristic (2θ) peaks at 18.5° , 20.0° , and 26.5° . This is typically associated with the α -phase, which is semicrystalline and non-polar. These peaks may appear sharp and strong, which reflects on the PVDF hollow fiber membrane high crystallinity (Acarer-Arat et al. 2024; Yuan et al. 2020; B. Ma et al. 2017; Arif et al. 2019).

In regards to modified PVDF hollow fiber membrane systems, changes in XRD peak intensities, broadening and/or shifting could provide support of structural changes related to coating deposition or added polymeric binders and additives. In this research bamboo cellulose may introduce additional crystalline domains due to the ordered of cellulose the structure, at the same time influencing the crystallinity (e.g. amorphous phase) and pore formation of the coated PVDF hollow fiber membrane (Bai et al., 2012; Acarer-Arat et al.,

2024; Wu et al., 2024). For the polymeric binders such as PVDF and PVA, XRD analysis detect alterations of the crystalline arrangement of the formulated cellulose/PVA layer, this is due to the hydrogen bonding interactions between PVA and cellulose. This influence availability of peaks around 15–16° and 22–23° for cellulose and PVA may show a broad peak around 19–20° (Bai et al., 2012; Li, Zhang, and Xu, 2012; Liew et al., 2015; Rahman et al., 2024; Li et al., 2019; Miri et al., 2015). On the other hand, for the PVDF binder in cellulose/PVDF coating formulations, may contribute towards coating compatibility with the pristine PVDF membrane surface, with available peak similar to pristine PVDF hollow fiber membrane at 18.5°, 20.0° and 26.5° (Acarer-Arat et al., 2024; Yuan et al., 2020; Ma et al., 2017; Arif et al., 2019). The addition of graphene oxide may also affect crystallinity by interacting with polymer chains and influencing dispersion within the bamboo cellulose/polymer binder coating system. The available peaks may appear at 10–13° range. (Ayyaru, Linh, and Ahn, 2019; Sun et al., 2022; Adaval et al., 2022; El Miri et al., 2015; Sun et al., 2022). The changes in crystallinity are important as it may influence the membrane structural stability, pore related properties and water transport behaviour. Hence, the XRD results should be analysed in compilation with SEM morphology, FTIR functional group analysis, porosity, mean pore size and water flux performance in order determine the coating improves membrane overall performance and surface structure without restricting water permeability.

2.13 Atomic Absorption Spectroscopy (AAS) Analysis.

The Atomic Absorption Spectrometry (AAS) is an analytical technique used to quantitatively determine the concentration of metal elements in liquid samples. AAS measure the absorption of light by free ground state atoms in the gas phase. Each individual metal absorbs light at a specific wavelength, and the amount of light absorbed is directly

proportional to the concentration of that element in the liquid sample (Çifci and Kaya 2010; Fakhfekh et al. 2017). AAS analysis is done by first atomizing and converting the liquid sample into free atoms. This could be done via Flame AAS or an electrothermal graphite furnace (GFAAS). A light beam specific to the target heavy metal element (e.g., iron, lead, zinc), passes through the vaporized liquid sample. The atoms absorb part of this light, and the spectrometer detects and measures the intensity loss to calculate the metal concentration. AAS is known for its high sensitivity, capable of detecting heavy metals typically concentrations in parts-per-million (ppm). In this project, AAS analysis is conducted for only the optimal coated PVDF hollow fiber membrane sample iteration, for the best attempt to test the coated PVDF hollow fiber membrane treatment for rain, river and peat water. Since AAS is heavy metal specific, iron (Fe) is selected for investigation.

The AAS analysis is suitable for the quantification of targeted heavy metal removal (e.g. iron), however the application towards filtered and unfiltered natural water sources (e.g. peat, river and rainwater) requires careful deliberation. For peat water, dissolved organic matter, humic substances and suspended contaminants could contribute towards matrix interference during heavy metal detection. Hence, proper calibration and sample preparation (e.g. no cross contamination) are important to improve analytical reliability. If comparing inductively coupled plasma mass spectrometry (ICP-MS) and AAS analysis, it generally offers lower detection limits and multi element (e.g. heavy metals) detection capabilities. For inductively coupled plasma optical emission spectroscopy (ICP-OES) in comparison to AAS analysis, it is also considered to be suitable for simultaneous multi heavy metal analysis. In contrast, these techniques are generally more expensive which requires more complex instrumentation and sample preparations. Utilizing UV-Vis spectrophotometry may be simpler and lower cost for heavy metal detection, however it usually requires colour

forming reagents and may precipitate or altered in coloured natural water sources, especially for peat and river water. Hence, AAS is more appropriate for this thesis research, firstly due to the current availability of lab instrumentation, and the analysis could focus specifically on iron concentration before and after filtration rather than broad multi element screening. The main rationale for iron concentration detection is due to its abundance in peat and river water sources, as rural regions in Borneo are surrounded by peaty soils rich in iron content leaching into those natural water sources. The iron removal obtained from AAS analysis could be linked to the coated membrane filtration performance, where changes in surface chemistry, pore structure, coating layer formation and hydrophilic functional groups from coating deposition may influence iron retention through filtration, surface interaction or adsorption related mechanisms (Çifci and Kaya, 2010; Fakhfekh et al., 2017; Arthanareeswaran et al., 2007).

CHAPTER 3

METHODOLOGY

3.1 Methodology Flowchart Representing by Phases

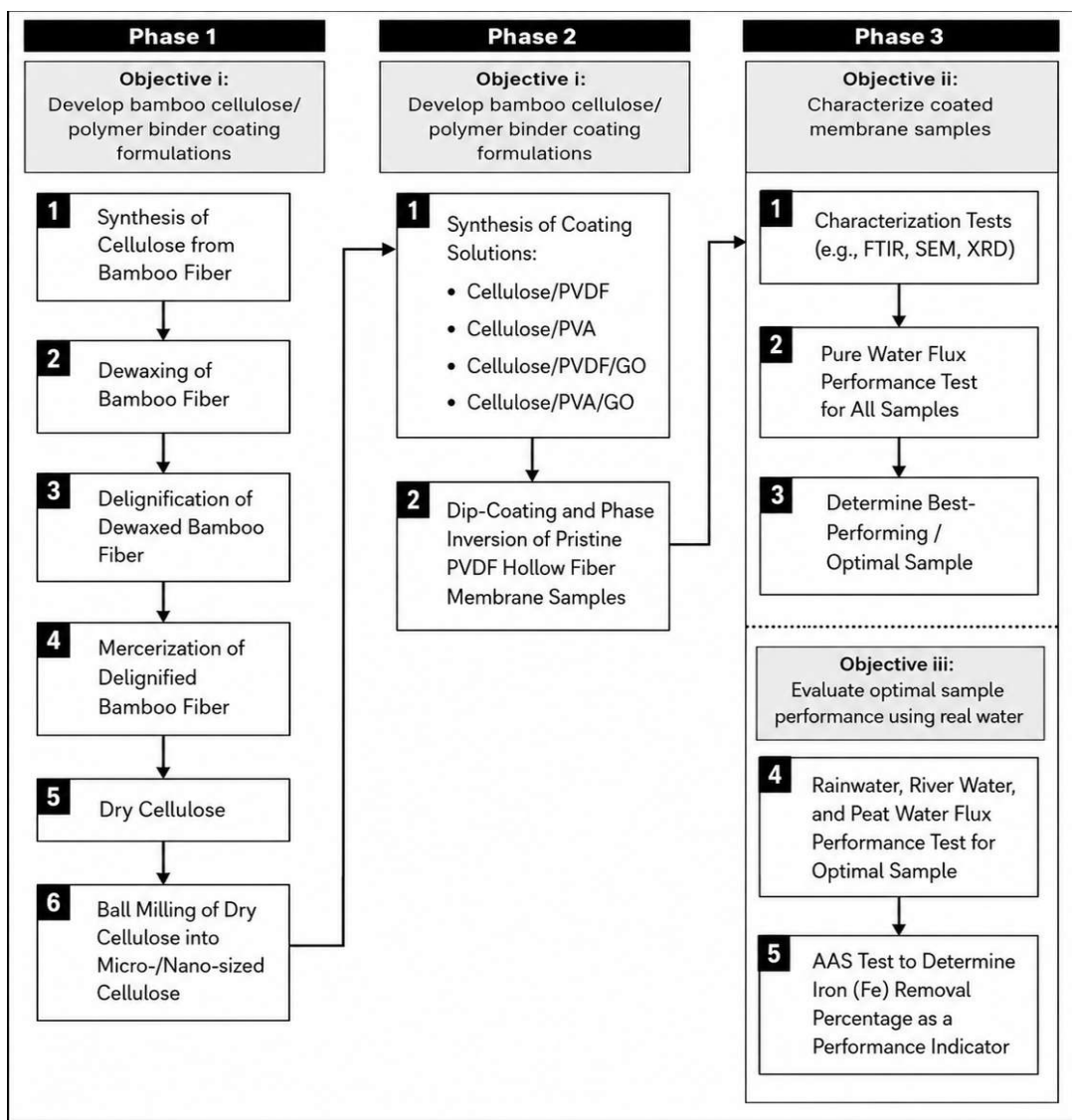


Figure 3.1: Methodology Flowchart Representing by Phases

The methodology flowchart represents the entire research project into 3 phases. Phase 1 involves synthesizing a micro-nano size cellulose from raw green bamboo via dewaxing, delignification, mercerization, drying and ball milling processes. Phase 2 involves

synthesizing the coating solution using the synthesized cellulose, powder PVDF, PVA and Graphene oxide (GO) additive, and coating the neat PVDF hollow fiber membrane. Phase 3 involves characterization test such as FTIR, SEM, XRD, AAS, Porosity and water flux performance for all water samples (e.g. peat, river, rain, deionized water). These characterization test showcases the properties and performance for coated and uncoated PVDF hollow fiber membrane.

3.1.1 Materials

Bamboo Fiber was processed following procedures by Liew et al., 2015 from mature Bamboo culms from Bau, Sarawak (Liew et al., 2015). All the following chemicals used are analytical grade: Hydrogen peroxide, (CAS No.: 7722-84-1, molecular weight: 34.01g/mol, purity: 3%), (Sigma Aldrich, MY), Acetic acid, (CAS No.: 64-19-7, molecular weight: 60.05g/mol, purity: $\geq 99.7\%$), (Sigma Aldrich, MY), Titanium (IV) oxide, (CAS No.: 13463-87-7, molecular weight: 79.87g/mol, purity $\geq 99.5\%$), (Sigma Aldrich, MY), Sodium hydroxide, (CAS No.: 1310-72-2, molecular weight: 40g/mol, purity: $\geq 98\%$), (Sigma Aldrich, MY), Toluene, (CAS No.: 108-88-3, molecular weight: 92.14g/mol, purity: 99.8%), (Sigma Aldrich, MY), Ethanol, (CAS No.: 64-17-5, molecular weight: 46.07g/mol, purity: $\geq 99.5\%$), (Sigma Aldrich, MY), Poly(vinylidene fluoride) PVDF powder, (CAS No.: 24937-79-9, molecular weight: 530,000g/mol), (Sigma Aldrich, MY), Polyvinyl alcohol (PVA) powder (99% purity), (Sigma Aldrich, MY), Graphene oxide paste (GO) Graphene Oxide Paste (Sigma Aldrich, MY), Lithium Chloride (LiCl), (CAS No.: 7447-41-8), (Sigma Aldrich, MY), N, N-Dimethylacetamide (DMac), (CAS No.: 127-19-5), (Sigma Aldrich, MY), Iron Standard for AAS, 1 g/L Fe in nitric acid (nominal concentration) (Sigma Aldrich, MY), PVDF hollow fiber membrane from (Sigma Aldrich, MY) supplied by University Malaysia Sarawak (UNIMAS).

3.1.2 Synthesis of Bamboo Cellulose

The synthesis of bamboo cellulose follows the procedures according to Liew et al. via chemical treatment with mechanical process (Liew et al., 2015). The dewaxing, delignification, and mercerization procedures are the main procedure for removing noncellulosic components via stages from bamboo fiber, while preserving the cellulose integrity. The dewaxing process using a toluene–ethanol mixture removes surface waxes, oils, and extractives, which is essential for later stages of the synthesis of cellulose as those compounds effect chemical penetration. The delignification process using hydrogen peroxide and acetic acid at 130 °C targets the removal of lignin and hemicellulose from the prior dewaxing stage, hence yielding better cellulose purity. Without the delignification stage, it may reduce the cellulose purity and influence between the cellulose/polymer coating solution formulation. Mercerization using 6 wt% NaOH at 80 °C further removes remaining hemicellulose and impurities while promotes swelling of the cellulose fibers, hence increases the surface roughness and exposing the hydroxyl groups. These changes are expected to improve cellulose dispersion, cellulose/polymer coating formulation interaction, and coating formation onto the surface of the PVDF hollow fiber membrane. Furthermore, the grinding and ball milling of dried cellulose after the mercerization process, helps reduce the cellulose particle size into micro/nano sized particles. This increases the available surface area and improving the potential interaction between cellulose, polymer binders, and graphene oxide in the coating solution. In contrast, these treatments were adapted in order to synthesize cellulose from bamboo fiber with suitable purity, morphology and particle size for stable coating formation and improved membrane water flux performance (Liew et al., 2015).

All chemical preparation steps were conducted using appropriate laboratory safety measures such as conducting the experiments under a fume hood, using a laboratory coat,

chemical resistant gloves and safety goggles. This is due to the use of volatile organic solvents, strong alkaline solution, oxidizing thiagents, and exposure to high temperature lab equipments. After stages of synthesizing cellulose from bamboo fibers, all samples were rinsed thoroughly with deionized water until neutral pH was achieved in order to minimize residual chemical contamination prior to further processing.

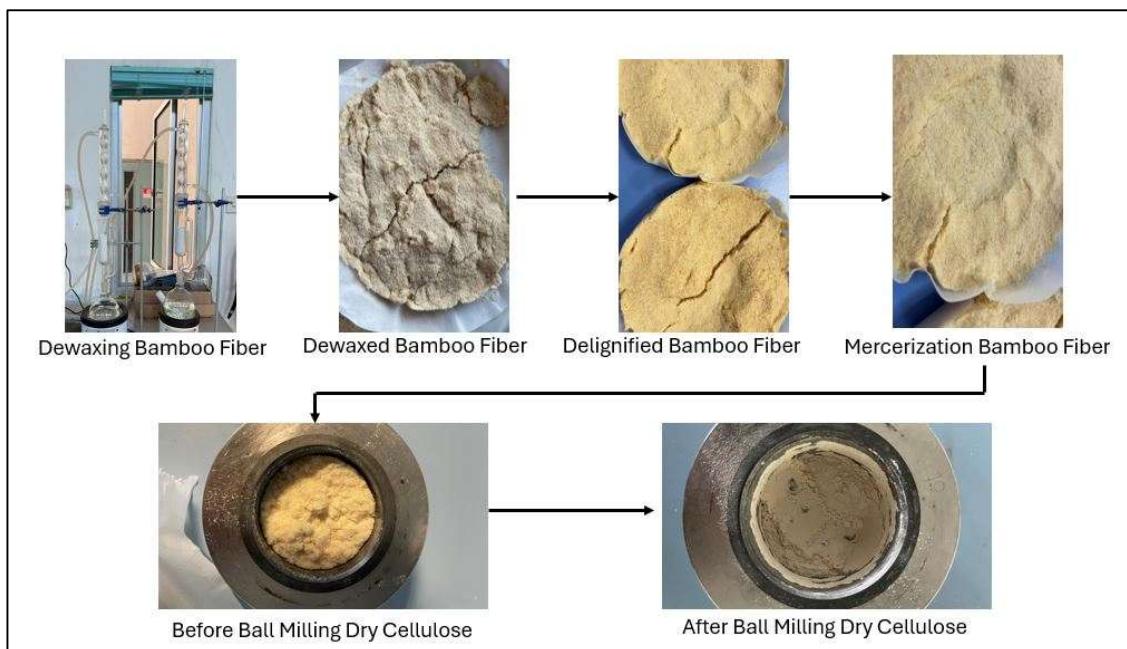


Figure 3.2: Illustration of Synthesis of Bamboo Cellulose from Bamboo Fiber Flowchart

3.1.3 Dewaxing of Bamboo Fiber

A 400 mL toluene and 200 mL ethanol are mixed to produce toluene-ethanol ratio of 2:1 ratio. To undergo dewaxing a Soxhlet extractor and Liebig condenser was used. Approximately 10 g of Bamboo Fiber was scooped into a membrane tube and then placed into the extraction thimble. The toluene-ethanol solution was heated until it is visibly boiling above 110 °C. After 10–12 cycles of extraction via Soxhlet extractor, the resulting fiber is oven dried at 35°C for 24 hours. The dried sample was labelled as Dewaxed Bamboo Fiber (DBF).

3.1.4 Delignification of Bamboo Fiber

Delignification solution uses 82.3 g of 35 wt% hydrogen peroxide and 106.2 g of 99.8 wt% acetic acid with small amount of titanium (IV) oxide as catalyst. This solution is for the delignification of 30 g of DBF and is heated up to 130°C. After 2 hours, the solution is cooled to room temperature. The treated fiber is filtered and rinsed with deionized (DI) water until the pH 7, followed by drying at 35°C for 24 hours. The dried sample was placed into a bottle and kept in a dark and cool place for alkaline treatment. The sample is identified as Delignified Bamboo Fiber (DLBF).

3.1.5 Mercerization

DLBF is immersed into a 6 wt% of sodium hydroxide solution. The mixture is consistently stirred with a magnetic stirrer and heated to 80°C for 2 hours. After 2 hours the heating is stopped and cooled to room temperature. The stirring can be stop after 8 hours or the day after. The mixture was rinsed continuously with DI water until the product reached pH 7. The treated product was then filtered using Buchner flask, rinsed with DI water until the pH level reached 7, and oven dried at 30°C The cellulose is then ground using a grinder followed by ball milling for 1 hour until a fine powder is formed.

3.1.6 1st Experiment: Cellulose/PVDF Coating Solution

Cellulose/PVDF dissolution for coating solution formulation follows some parameters according to Sadeghifar et al. 2019 using Lithium chloride (LiCl) and N, N-Dimethylacetamide (DMac). The DMac/LiCl solvent system is suitable to assist cellulose dissolution and/or dispersion during coating solution preparation (Sadeghifar et al. 2019). The procedure proceeds as follows; Firstly, (0.25g) PVDF powder is dissolved in beaker 42g DMac, stirred with a magnetic stir bar, making a PVDF/DMac solution. Second, the desired

cellulose iteration (0.25g, 0.5g, 0.625g, 0.725g, 1.0g) and 3g LiCl is added into the PVDF/DMAc solution. The mixture was heated up to 130°C and stirred with a magnetic stir bar. The temperature is held for 20-30mins until there is visible homogenous consistency of dissolution has occurred. The mixture was allowed to cool down slowly and continuously stirred for 12 hours at room temperature. The end product is the Cellulose/PVDF coating solution. According Sadeghifar et al. 2019 the LiCl/DMAc solvent is suitable for cellulose dissolution and film formation via Loeb-Sourirajan phase inversion technique. In this research, LiCl/DMAc solvent system was utilized for attaining a cellulose dissolution from the synthesized bamboo cellulose. While the addition of PVDF binder in the solvent system improves coating compatibility with the pristine PVDF hollow fiber membrane surface due to polymer similarity, allowing better integration of the cellulose containing coating layer during the subsequent coating and phase inversion process (Sadeghifar et al. 2019; Liu et al., 2011; Awanis Hashim et al., 2012). The following table illustrates the cellulose/PVDF sample name and weight ratio for developing the coating solution.

Table 3.1: Cellulose/PVDF sample name and weight ratio for developing the coating solution.

Sample Name	Cellulose Weight	PVDF Weight	DMAc Weight	LiCl Weight
P01	0.250g	0.25g	42g	3g
P02	0.500g	0.25g	42g	3g
P03	0.625g	0.25g	42g	3g
P04	0.725g	0.25g	42g	3g
P05	1.000g	0.25g	42g	3g

3.1.7 2nd Experiment: Cellulose/PVA Coating Solution

The methodology for the 2nd experiment Cellulose/PVA coating solution follows the same procedure as the 1st experiment for Cellulose/PVDF, the differences is the replacement of polymer binder, with the PVDF powder (0.25g) being replaced with PVA powder (0.25g). The following table illustrates the cellulose/PVA sample name and weight ratio for developing the coating solution.

Table 3.2: Cellulose/PVA sample name and weight ratio for developing the coating solution.

Sample Name	Cellulose Weight	PVA Weight	DMac Weight	LiCl Weight
PV01	0.250g	0.25g	42g	3g
PV02	0.500g	0.25g	42g	3g
PV03	0.625g	0.25g	42g	3g
PV04	0.725g	0.25g	42g	3g
PV05	1.000g	0.25g	42g	3g

PVA was selected as a polymer binder due to its hydrophilic nature and compatibility with PVDF based membrane systems. Some studies demonstrated that the incorporation of PVA into PVDF membranes could improve hydrophilicity, permeability, and water flux performance (Li et al., 2010; Li et al., 2019; Razmgar et al., 2018). Hence, in this research, PVA is expected to act as a hydrophilic binder that assists cellulose dispersion and coating formation, while promoting hydrophilic properties onto the surface of the pristine PVDF hollow fiber membrane. PVA as binder could attain dissolution using DMac/LiCl solvent system, at the same time enables the nonsolvent phase inversion to occur when submerged

in the deionized water bath. Precipitation during phase inversion does not occur with the dissolution of PVA in the same substrate as the water bath. In addition, the cellulose does not dissolve in water, hence the need for a DMac/LiCl solvent system as a stable medium for dissolution and coating formation when the developed coating solution precipitates during the phase inversion stage (Sadeghifar et al. 2019; Li et al., 2010; Li et al., 2019; Razmgar et al., 2018). As both experiment 1st and 2nd utilize the DMac/LiCl solvent system, it allows the comparison with PVDF as a binder, where PVA is expected to contribute more strongly to hydrophilicity, while PVDF may contribute more strongly to the pristine PVDF hollow fiber compatibility.

3.1.8 3rd Experiment: Graphene oxide Cellulose/PVDF and Graphene oxide Cellulose/PVA Coating Solution

The methodology for the 3rd experiment Graphene oxide (GO) Cellulose/PVDF and Graphene oxide (GO) Cellulose/PVA coating solution is the same as 1st experiment Cellulose/PVDF and 2nd experiment Cellulose/PVA coating solution, with the addition of Graphene oxide paste (0.105g) placed at the same time as the mixing the PVDF or PVA powder as binder in the DMac solution, prior to adding the cellulose iterations and LiCl. The following table illustrate the graphene oxide cellulose/PVA sample name and weight ratio for developing the coating solution.

Table 3.3: Graphene oxide Cellulose/PVDF sample name and weight ratio for developing the coating solution.

Sample Name	Cellulose Weight	PVDF Weight	Graphene Oxide Weight	DMac Weight	LiCl Weight
PG01	0.250g	0.25g	0.105g	42g	3g
PG02	0.500g	0.25g	0.105g	42g	3g

Table 3.3 continued

PG03	0.625g	0.25g	0.105g	42g	3g
PG04	0.725g	0.25g	0.105g	42g	3g
PG05	1.000g	0.25g	0.105g	42g	3g

Graphene oxide (GO) was incorporated in the cellulose/polymer DMac/LiCl solvent system as an additive due to its hydrophilic oxygen containing functional groups, which could contribute to improved surface characteristics and hydrophilicity of polymeric membranes (An et al., 2017; Perreault et al., 2015). In the coating solution, GO may also interact with cellulose and PVA through hydrogen bonding, while improving the distribution of hydrophilic components within the coating layer (El Miri et al., 2015).

Some studies demonstrated GO modified PVDF membranes shown that the dispersion of GO in the system could influence membrane morphology and water transport behaviour (Sun et al., 2022). Hence, in this research, GO was added to evaluate its effect on the surface chemistry, morphology, porosity, crystallinity and water flux performance of the coated PVDF hollow fiber membranes. The utilization of DMac/LiCl solvent system is to maintain a common processing medium for all coating formulations. By incorporating GO within the same DMac/LiCl solvent system, it allows GO, cellulose, and the polymer binder (PVDF and PVA) to be mixed within a single coating medium (Awanis Hashim et al., 2012; Baker, 2012).

Table 3.4: Graphene oxide Cellulose/PVA sample name and weight ratio for developing the coating solution.

Sample Name	Cellulose Weight	PVA Weight	Graphene Oxide Weight	DMac Weight	LiCl Weight
PVG01	0.250g	0.25g	0.105g	42g	3g
PVG02	0.500g	0.25g	0.105g	42g	3g
PVG03	0.625g	0.25g	0.105g	42g	3g
PVG04	0.725g	0.25g	0.105g	42g	3g
PVG05	1.000g	0.25g	0.105g	42g	3g

3.1.9 Coating of PVDF Hollow Fiber Membrane Preparation

Prior to coating the PVDF hollow fiber membrane, the coating solutions were first examined visually for solution uniformity. The absence of visual cues such as sediments, phase separation, floating particles, or large agglomerates was taken as an indicator of proper dissolution of the coating solution before proceeding with dip-coating process. Visual inspection alone may not confirm complete molecular dissolution. However the effectiveness of the coating deposition was subsequently verified indirectly through membrane characterization, which includes; infrared spectral analysis for functional groups, scanning electron microscopy for coating morphology, crystallinity analysis, porosity and mean pore size evaluation.

Prior to the coating process, the pristine PVDF hollow fiber membrane of a length of 20cm and 1mm diameter was kept clean and dry at room temperature in a sealed container. The coating of the pristine PVDF hollow fiber membrane follows the principle of dip coating time/process according to Wenten et al., 2020, where the pristine PVDF hollow fiber

membrane was dip for 2s and withdrawn from the solution for 10s (Wenten et al., 2020). The membrane was dip in the coating solution with 40ml volume. Subsequent to the dip coating, a glass rod is used to remove dripping or excess coating solution to yield a thin layer coating. The coating membrane was immersed in a deionized water bath to allow the coating to precipitate on the surface of the membrane and the remaining solvent (DMAc) migration to water. The coated membrane was then taken out from the water bath and dried at room temperature for 24 hours. Once dried, the coated hollow fiber membrane can be used for application.

It is noted that the experimental withdrawal speed and coating thickness were not directly measured in this study. However, the immersion time, withdrawal duration, coating solution volume, membrane length and drying time were kept constant for all samples to ensure comparative reproducibility. Potential defects such as cracks, uneven coating, or delamination were assessed visually and through SEM morphology analysis. No chemical crosslinking or curing was applied; therefore, the coating stability was attributed to phase inversion-assisted deposition and polymer binder interaction. This limitation is acknowledged, particularly for PVA-containing coatings, where water sensitivity may affect long-term coating stability.

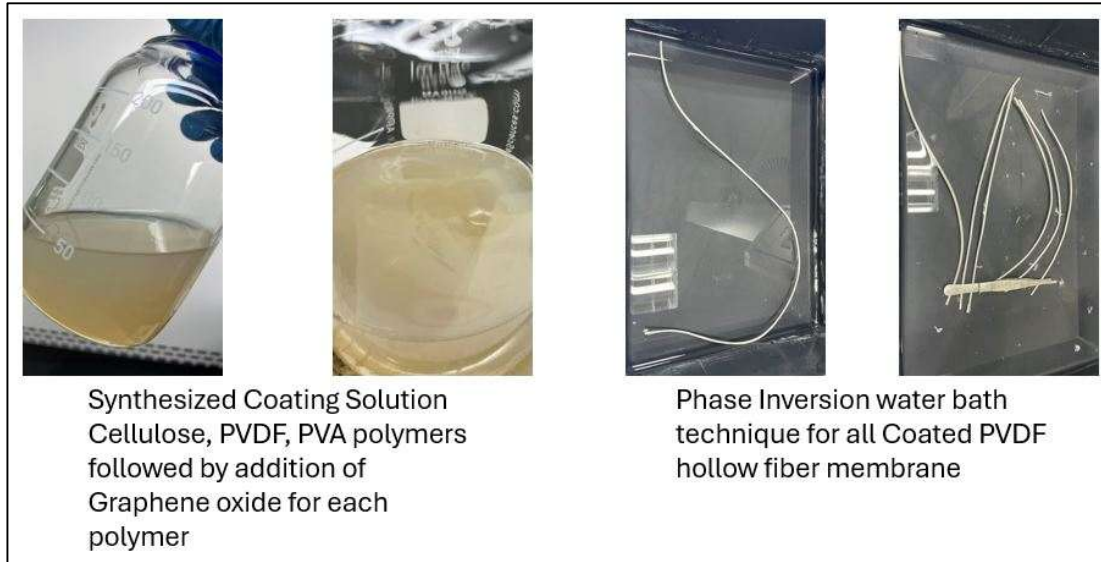


Figure 3.3: Illustration of coating solution and coated PVDF membrane with synthesized coating solution

3.2 Water Flux Test as Membrane Performance Test

Prior to the water flux performance test, a single 10 cm hollow fiber membrane sample was cut and prepared for water flux testing rig. The water flux performance test was firstly conducted for 15 to 20 minutes at a functioning pressure 1 bar to achieve a stable water flux prior to the actual readings to be recorded. The water flux test was conducted at an operating pressure of 1 bar as it was selected to represent a low-pressure filtration condition suitable for evaluating membrane permeability for typical to household functioning pressure (rural area water supply) while minimizing energy demand for practical water treatment applications (Baker, 2012; Liu et al., 2011). Furthermore, this allows the membrane sample to properly wet, swell and drives the water to start permeating the through the membrane sample. The water flux readings are recorded for the total of 1 hour at the functioning pressure of 1 bar. The water flux is calculated according to the Equation 2.1 (Awani Hashim et al. 2012). For reliability, the water flux test was repeated 3 times for each

membrane samples and the average water flux value was used for comparison. As standard deviation was not included, the subsequent results were discussed based on comparative trends in average water flux performance. This limitation is acknowledged, as reporting standard deviation would provide a more complete indication of measurement variability. The water flux test was conducted for deionized water, peat water, river water and rain water samples, for all coated membrane samples and the pristine PVDF hollow fiber membrane (control) sample.

3.3 Characterization Tests of PVDF Hollow Fiber Membrane Samples

3.3.1 Fourier Transform Infrared (FTIR) Analysis of PVDF Hollow Fiber Membrane Samples

Fourier transform infrared (FTIR) spectroscopy was conducted according to the ASTM E168-16 (2016) and ASTM E1252-98 (2021) standards for qualitative and quantitative analysis. Prior to analysis, the PVDF hollow fiber membrane samples were dried at room temperature to remove residual moisture. A small 2mm size membrane sample was cut and placed directly onto the attenuated total reflectance (ATR) crystal surface. The sample was pressed gently against the ATR crystal to ensure good contact during spectral analysis. The spectrum scanning was conducted in the wavenumber range of 4000 to 400 cm^{-1} for each sample (ASTM E168-16 2016; ASTM E1252-98 2021).

FTIR analysis was selected as a characterization test for this research to identify the characteristic functional groups available of coated and uncoated pristine PVDF hollow fiber membrane. FTIR could also identify if deposition of cellulose, PVDF and PVA as binder material, as well as graphene oxide as additives were successful (Liu et al., 2011; Kang & Cao, 2014). The verification of characteristic C–F and C–H absorption bands, signify the presence of PVDF polymer, while the incorporation of cellulose could be supported by the

increased intensity of O–H and C–O related bands (Bai et al., 2012; Wang et al., 2021). Hence, an FTIR analysis is important for verifying surface chemical modification after coating and for supporting the clarification hydrophilic behaviors, water flux performance, and membrane structural characteristics (Wang et al., 2021; Wu et al., 2024).

3.3.2 Scanning Electron Microscopy (SEM) Analysis of PVDF Hollow Fiber Membrane Samples

Scanning electron microscopy (SEM) for the samples was conducted according to ASTM E2015-04 (2021), in regards to electron microscopy testing procedure. Prior to SEM analysis, the membrane samples were dried completely at room temperature to remove residual moisture. Small 2mm sections of each membrane samples were cut and mounted onto SEM sample stubs using conductive carbon tape. The samples were then sputter coated with a thin conductive gold layer, in order to minimize surface charging during scanning. During scanning, magnifications of 500x and 1000x were utilized to observe the surface of the samples a total of 6 samples was be tested using SEM (ASTM E2015-04 2021). The SEM analysis was mainly used for qualitative comparison of surface morphology analysis, including coating coverage, surface roughness appearance, visible pores, cracks, agglomeration and possible coating defects. Quantitative pore size or roughness analysis was not conducted using SEM; therefore, pore related properties were evaluated separately through porosity and mean pore size measurements in subsequent testing.

3.3.3 X-ray diffraction Analysis (XRD) Crystallinity Analysis of PVDF Hollow Fiber Membrane Samples

X-ray Diffraction Analysis (XRD) is used to verify the range of crystallinity of the samples and to observe the availability of either a crystalline or amorphous phase. The membrane samples were analyzed at room temperature using a Rigaku Smart Lab X-ray diffractometer with $\text{CuK}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The XRD analysis for the control and

coated samples were recorded within a scattering angle (2θ) ranging from 5 to 90° at a scanning rate of 2min⁻¹. This procedure was carried out in accordance with the ASTM F3419-22 (2022) standard (ASTM F3419-22 2022). Crystallinity analysis was utilized to determine the bamboo cellulose/polymer binder coating and graphene oxide additives altering the structural arrangement on the surface of the PVDF hollow fiber membrane. The phases of crystallinity as analyzed by XRD could influence pore structure and water transport behaviour, hence XRD analysis supports the evaluation of coating effects on membrane flux performance (Geise et al., 2010; Liu et al., 2011; Ma et al., 2017).

3.3.4 Porosity and Mean Pore Size Analysis of PVDF Hollow Fiber Membrane Samples

The membrane samples porosity was determined using the gravimetric method based on Equation 2.2 and 2.3 (Rahman et al. 2024; Yuliwati et al. 2011). The membrane samples with the same effective length of 10cm were dried before measurement and their dry weights were recorded. Subsequently, the samples were then immersed in deionized water for at least 20 minutes to allow swelling and pore filling to occur. After immersion, excess surface water was removed carefully using tissue paper and the wet membrane samples were immediately measured. The mean pore size of both control and coated sample was calculated following the Guerout-Elford-Ferry equation. The mean pore radius was obtained from Equation 2.5 and the mean pore diameter was determined by multiplying the value of the mean pore radius by two (Rahman et al. 2024; Yuliwati et al. 2011). Overall, the measurement was repeated 3 times for each membrane samples and the average value was used for comparison. As standard deviation was not included, the subsequent results were discussed based on comparative trends in average porosity and mean pore size analysis. This limitation is acknowledged, as reporting standard deviation would provide a more complete indication of

measurement variability. It is noted that, the coating thickness was not directly measured, the porosity values were considered as the porosity of the entire membrane sample. Hence, the porosity and mean pore size was interpreted alongside other analysis such as SEM images and water flux performance to supplement the research regarding the influence of the coating layer on pore accessibility and water transport.

3.3.5 Atomic Absorption Spectrometry (AAS) Analysis for Iron removal after filtration as Membrane Performance Test

The Shimadzu Flame Atomic Absorption Spectrometry 'AA-7000' spectrometer is used to identify the concentration of heavy metal element present in the rainwater, river water and peat water after filtering using the optimal performing sample. This analysis measured the wavelength of electromagnetic radiation emitted by a light source using the prepared standard solution. Prior to analysis, all real water samples (peat, river and rain water) were allowed to settle to remove suspended particles and stored clean sample bottles to prevent contamination. Acid digestion was not conducted; this analysis was limited to the detectable dissolved or readily measurable iron fraction rather than total iron content. Therefore, the AAS results were interpreted as an indication of iron concentration change after membrane filtration. A calibration curve was prepared using standard Iron (Fe) solutions at concentrations of (0.0, 0.1, 0.5, 1.0, and 2.0 ppm) from Sigma. The blank solution was used to zero the instrument before analysis.

The Fe concentration of each water sample was determined by comparing its absorbance value with the calibration curve. The reference electrode was silver/silver chloride and the carrier gas is high-purity nitrogen gas (Çifci and Kaya 2010; Arthanareeswaran et al. 2007; Fakhfekh et al. 2017). Each test was repeated 3 times and the average concentration was used for comparison. As standard deviation was not included, the

subsequent results were discussed based on comparative trends in average iron concentration in (ppm). This limitation is acknowledged, as reporting standard deviation would provide a more complete indication of measurement variability. The percentage of iron removal was calculated based on the Fe concentration before and after filtration. As the AAS analysis was conducted only for the optimal coated membrane sample, the results were used as a practical validation of the selected membrane performance in real water filtration rather than a full comparative analysis among all membrane formulations. This limitation was acknowledged as testing all coated samples would provide broader comparative insight into the relationship between coating formulation and iron removal performance. However, the AAS analysis was limited to the optimal coated membrane selected based on pure water flux performance. As the optimal sample has the potential to provide the most effective filtration output among the tested formulations/iterations.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Characterization of Cellulose/PVDF Coated PVDF Hollow Fiber Membrane – 1st Experiment

4.1.1 Overview for 1st Experiment

This section covers the characterization for 1st experiment for Cellulose/PVDF coated and uncoated PVDF hollow fiber membrane. The objective of this section was to evaluate the effects on the physicochemical properties and water flux performance from coating cellulose/PVDF via phase inversion on the surface of pristine PVDF hollow fiber membrane. The characterization for 1st experiment include; Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray diffraction (XRD), water flux performance, and porosity analysis. The characterization conducted evaluates the effect of the coating formulation regarding chemical structure, surface morphology, crystallinity, hydrophilicity and porosity variation after surface coating.

4.1.2 FTIR Analysis for Cellulose/PVDF Coated PVDF Hollow Fiber Membrane

According to the FTIR graph for Cellulose/PVDF coated PVDF hollow fiber membrane, it could be observed that there is a visible difference in peak intensity between coated samples and the control. Peaks for the control indicates a characteristically pristine sample of PVDF hollow fiber membrane. The control sample shows an identifiable stretching at C-H stretching region with bands located at 3371.56 and 2931.80 cm^{-1} , this is indicative of $-\text{CH}_2$ groups for PVDF polymer structure (Liu et al., 2011; Kang & Cao, 2014). Another identifiable stretching region at the C-F stretching region with band located at 1174.65 and 1107.14 cm^{-1} , the intense stretching and peaks at the C-F stretching region is

one of the key areas to identify the existence of PVDF of the PVDF hollow fiber membrane control sample tested (Awanis Hashim et al., 2012; Liu et al., 2011).

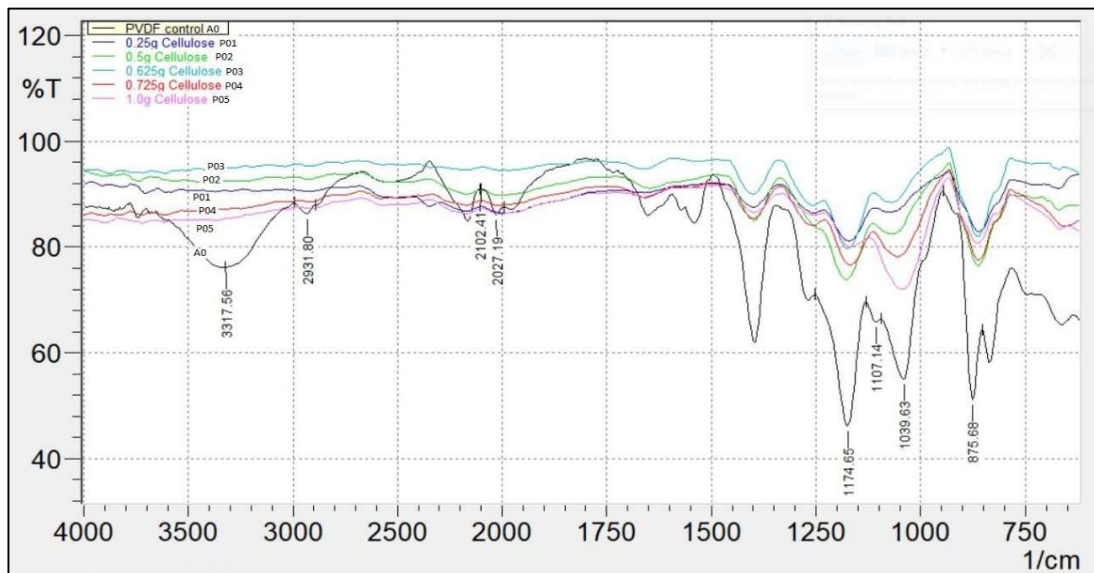


Figure 4.1: FTIR for Cellulose/PVDF coated PVDF hollow fiber membrane and Control sample

In regards to the effect of Cellulose/PVDF coating on PVDF hollow fiber membrane, the main identifiers could be observed at the O-H stretching region between the 3700 to 3000 cm^{-1} , due to the broad and gradual bands this is indicative of hydroxyl groups showcasing the addition of cellulose on the sample (Bai et al., 2012; Wang et al., 2021). It could be observed, at higher cellulose content, some samples showed increased peak intensity. In addition, while higher cellulose loading appeared to partially mask the characteristic PVDF peaks, it is signifying that the cellulose rich coating became more dominant on the membrane surface (Wang et al., 2021; Wu et al., 2024). In the C-O and C-F stretching region, the Cellulose/PVDF coated samples demonstrates broad peaks which are indicative of the availability of PVDF polymer structure or content between the 1170 to 840 cm^{-1} region, and cellulose at the 1124 to 1087 cm^{-1} region (Liu et al., 2011; Kang & Cao, 2014; Bai et al., 2012).

A study on PVDF composite membranes blended with nano-crystalline cellulose also feature similar regions in their FTIR results for cellulose availability at the 1124 to 1087 cm^{-1} region and as well as the 3700–3000 cm^{-1} O–H stretching region (Mohammadpour et al., 2024). The FTIR finding are consistent with studies related to cellulose/PVDF based membranes, where cellulose incorporation was confirmed by the identification, increased availability of hydroxyl related absorption bands and alteration in the C–O, C–F stretching region (Bai et al., 2012). The broad O–H absorption observed in the coated samples indicates the presence of cellulose on the PVDF membrane surface, while the changes in peak intensity within the C–O and C–F stretching region suggest modification of the pristine PVDF hollow fiber membrane surface. Similarly, the surface chemical changes have been reported for cellulose molecular coating and nanocellulose-modified PVDF membranes, which were associated with improved hydrophilicity, permeability and water flux performance (Wang et al., 2021; Wu et al., 2024). In comparison with other studies regarding cellulose/PVDF membrane, this present research differs by applying cellulose/PVDF coating onto a stock preformed pristine PVDF hollow fiber membrane rather than incorporating cellulose as a blended matrix directly during membrane development. An example is a study by Bai et al. (2012), which reported nanocrystalline cellulose-blended PVDF composite membranes, while Lv Zhang, Zhang, Zhao et al. (2018 a, b) reported modified PVDF ultrafiltration membranes using cellulose nanocrystals to improve water treatment performance and properties (Bai et al., 2012; Lv, Zhang, Zhang, Zhao, et al., 2018a; Lv, Zhang, Zhang, Zhao, et al., 2018b). Overall, the FTIR results shows that there is evidence of cellulose availability in the samples tested, successfully demonstrating Cellulose/PVDF coating on pristine PVDF hollow fiber membrane.

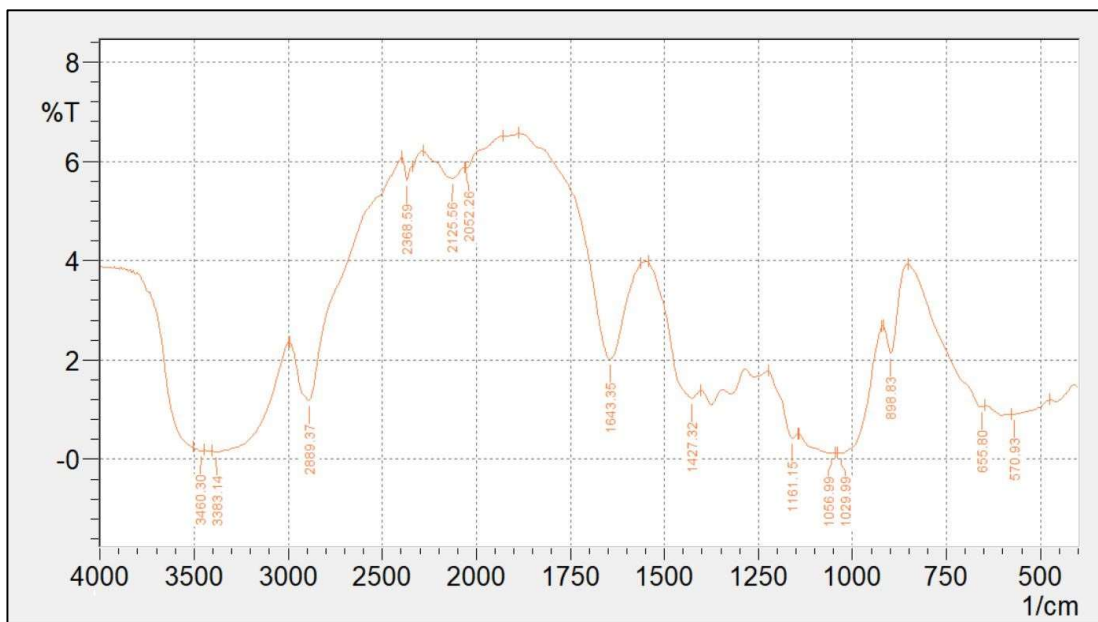


Figure 4.2: FTIR of the Extracted Cellulose for Coating Solution

The FTIR results shown in Figure 4.2 shows the extracted cellulose with similar graph patterns and functional groups of a successfully extracted cellulose. According to Liew *et al.* (2015) the functional group at 1514 cm^{-1} is attributed to the C=C stretching vibration in the aromatic ring of lignin. The extracted cellulose in Figure 4.2 did not show this stretching hence indicates that lignin was removed during the extraction process Liew *et al.* (2015). The broad and intense peaks observed at 3460 and 3383 cm^{-1} resemble the O–H stretching vibrations indicating the presence of hydroxyl groups. The peak at 2889 cm^{-1} is attributed to C–H stretching, while the band at 1427 cm^{-1} is associated with –CH_2 bending vibrations. The intense peak at 1643 cm^{-1} corresponds to the H–O–H bending vibration of adsorbed water in carbohydrates, and the peak at 1029 cm^{-1} is related to C1–H deformation vibrations characteristic of cellulose. This results for the extracted cellulose from bamboo fiber matches the results according to Liew *et al.* (2015). Other studies also match these results in regards to available functional groups for a successfully extracted cellulose (Abderrahim et al., 2015; O.Brian et al., 2022; Weng et al., 2015).

4.1.3 SEM Analysis for Cellulose/PVDF coated PVDF Hollow Fiber Membrane

According to Table 4.1, it could be observed that A0 has a surface structure which is generally smooth and compact with minimal visible pores. This result matches few studies in regard to neat PVDF, where water flux or permeability of the membrane is lower as compared to modified PVDF membrane (Ma et al. 2017; H. Bai et al. 2012).

Table 4.1: SEM of Cellulose/PVDF coated PVDF hollow fiber membrane and Control sample

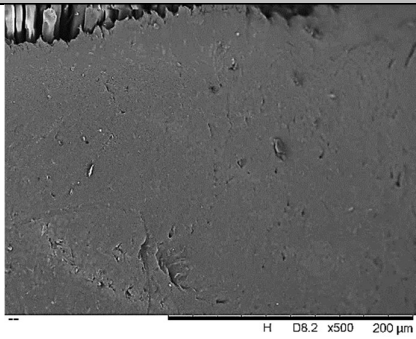
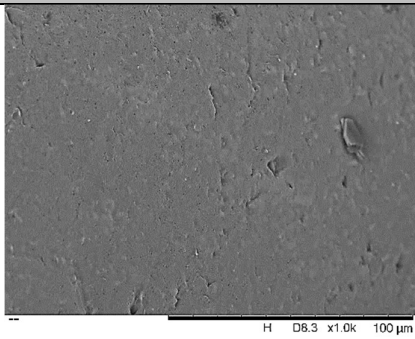
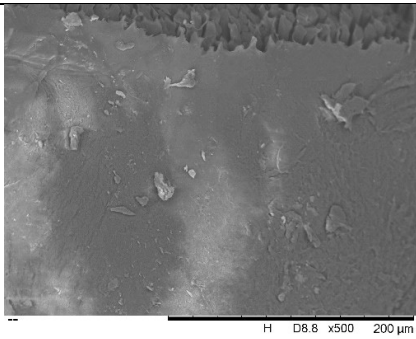
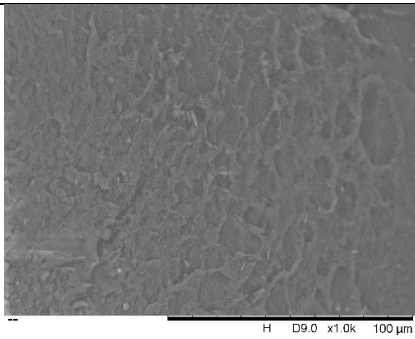
Sample	500x Magnification	1000x Magnification
A0 Pristine PVDF hollow fiber membrane		
P01 Cellulose at 0.250g		

Table 4.1 continued

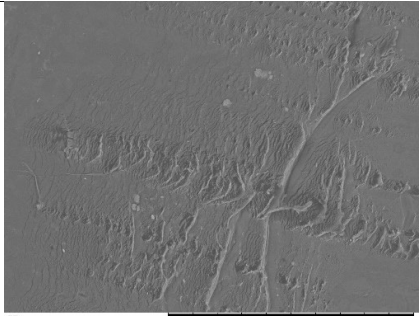
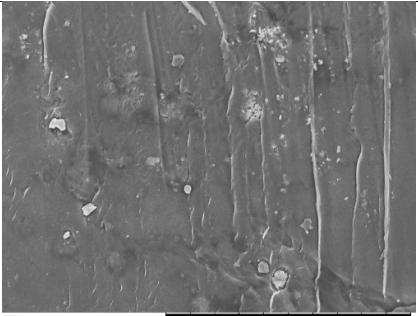
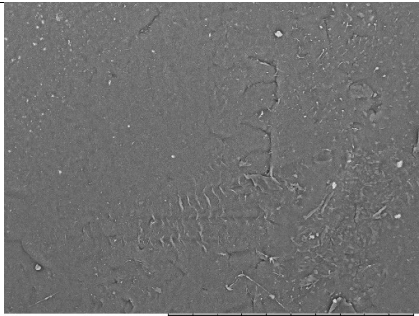
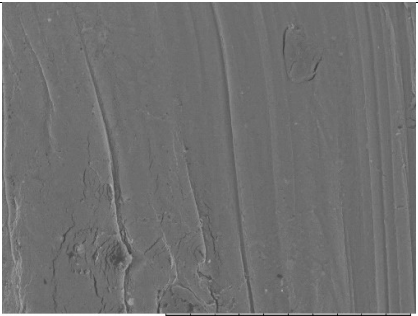
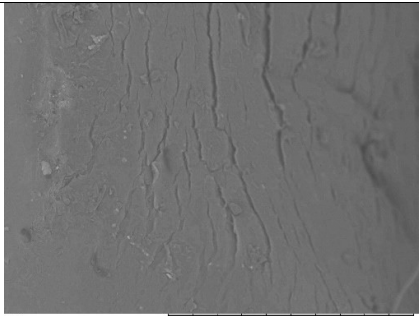
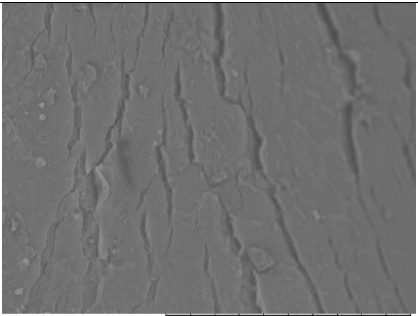
P02 Cellulose at 0.500g	 SEM image showing a textured surface of cellulose at 0.500g. The scale bar indicates 200 μm. Metadata: H D8.6 x500 200 μm.	 SEM image showing a textured surface of cellulose at 0.500g. The scale bar indicates 100 μm. Metadata: H D9.0 x1.0k 100 μm.
P03 Cellulose at 0.625g	 SEM image showing a textured surface of cellulose at 0.625g. The scale bar indicates 200 μm. Metadata: HL D8.1 x500 200 μm.	 SEM image showing a textured surface of cellulose at 0.625g. The scale bar indicates 100 μm. Metadata: H D8.7 x1.0k 100 μm.
P04 Cellulose at 0.725g	 SEM image showing a textured surface of cellulose at 0.725g. The scale bar indicates 200 μm. Metadata: H D8.9 x500 200 μm.	 SEM image showing a textured surface of cellulose at 0.725g. The scale bar indicates 100 μm. Metadata: H D9.0 x1.0k 100 μm.

Table 4.1 continued

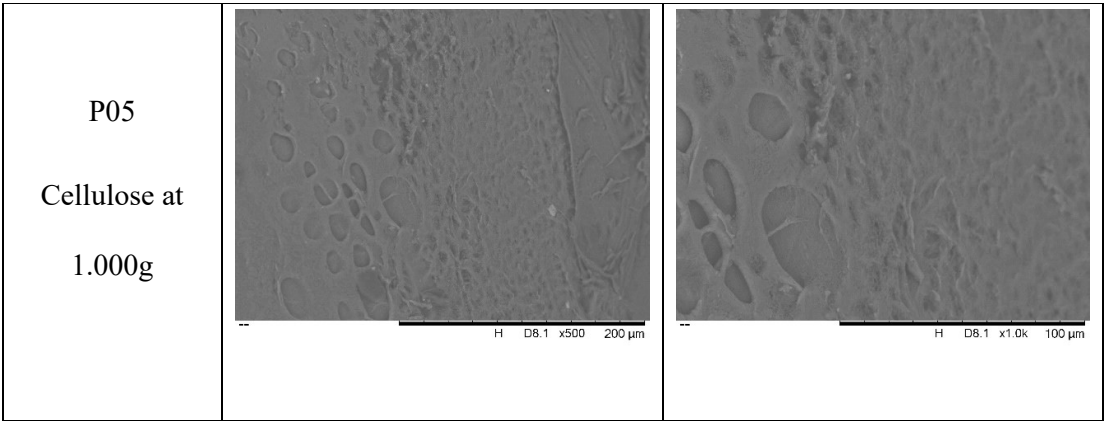


Table 4.1 showcases the SEM at 500x and 1000x magnification for pristine PVDF hollow fiber membrane A0 (control) and coated Cellulose/PVDF samples P01, P02, P03, P04, and P05. For the scanning electron imagery for P01 and P05, the surface structure may share some similarity with rough and non-uniformity with visible pores and voids, however in respect to the pure water flux performance, P01 has the higher pure water flux in comparison to P05. Hence, P05 surface structure may be the result of a thicker coating due being the higher cellulose content of 1.0g (Ma et al. 2017). It is also possible that the 0.25g PVDF weightage in the coating solution may not be able to be a suitable binder when cellulose weightage is above 100% of PVDF binder weightage. P02 surface structure shows more rougher surface structure with visible coating lines, it could be indicative that the rougher surface may bring better pure water flux performance, as P02 has the best pure water flux performance among all coated samples (Ma et al. 2017; O. Koriem et al. 2022). P03 and P04 shows signs of cracking with P04 being the sample with most cracking surface structure, no visible pores or voids are observed as compared to the rest of the samples. P03 and P04 has similar coating lines at P02 which could be indicative that cellulose above 0.25g may yield cleaner coating layer. In relation to pure water flux performance, P03 and P04 are the

first to show signs of reduced pure water flux, followed by P05 being the lowest among the coated samples (Ma et al. 2017; O. Koriem et al. 2022).

4.1.4 XRD Analysis for Cellulose/PVDF coated PVDF Hollow Fiber Membrane Sample

The XRD analysis for cellulose/PVDF coated PVDF hollow fiber membrane (P01-P05) and pristine PVDF hollow fiber membrane (A0) was conducted as a qualitative evaluation supported by semi-quantitative comparison of diffraction peak intensity. The crystalline and amorphous phase characteristics of pristine PVDF hollow fiber membrane (control) and cellulose/PVDF coated PVDF hollow fiber membranes were evaluated based on the observed 2θ ($^{\circ}$) peak positions, relative peak intensity and peak broadness. The identification of crystalline or amorphous phases was verified by comparing research findings regarding observed XRD diffraction patterns PVDF membranes and cellulose/PVDF based membranes.

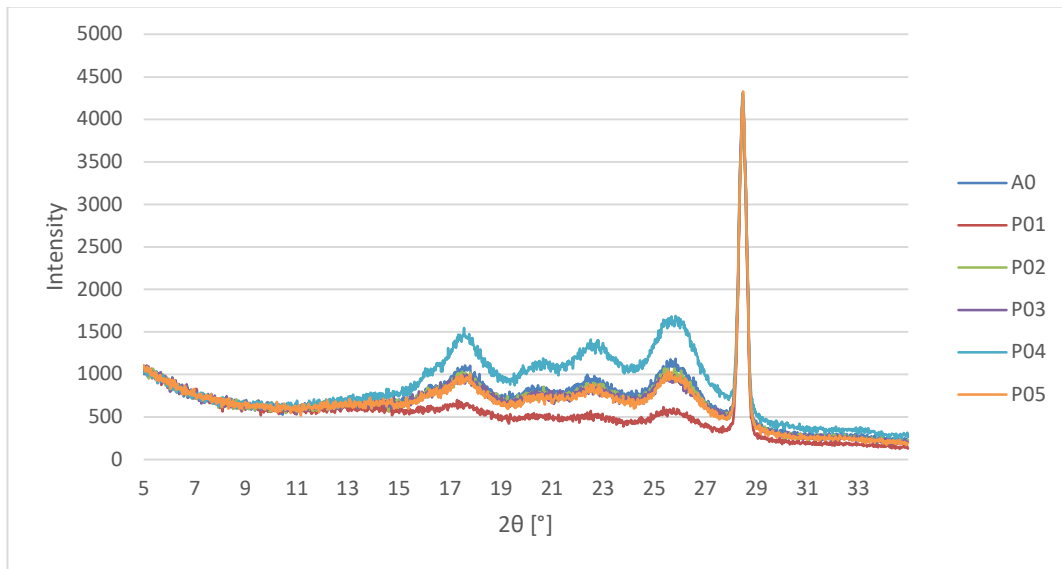


Figure 4.3: The combine XRD graph for Cellulose/PVDF coated PVDF hollow fiber membrane and Control sample

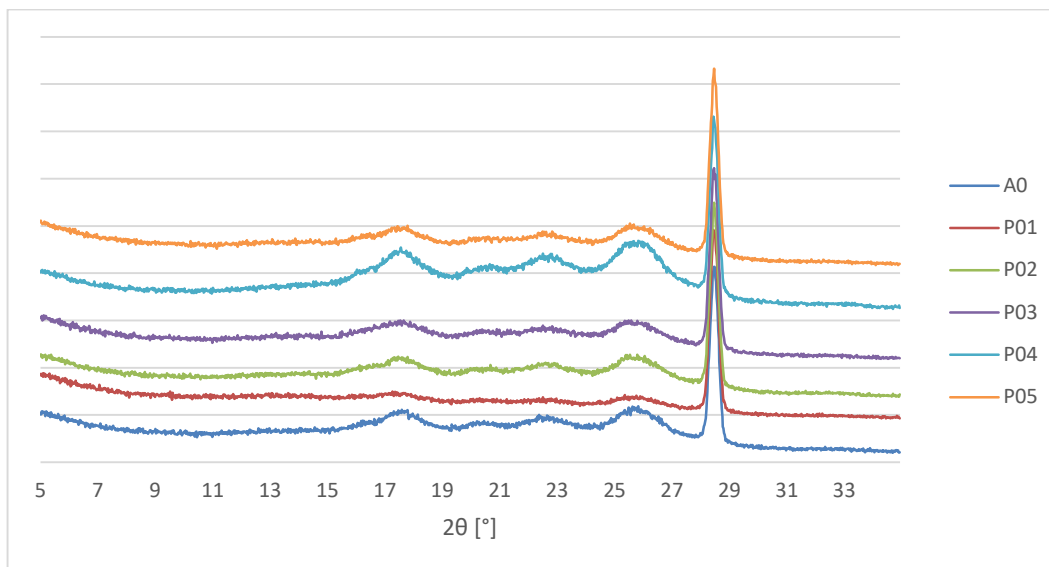


Figure 4.4: The stacking XRD graph for Cellulose/PVDF coated PVDF hollow fiber membrane and Control sample

It is observed that in Figure 4.3 combine and Figure 4.4 stacking XRD graph for Cellulose/PVDF coated PVDF hollow fiber membrane and control sample, shows differences in intensity, illustrating the deposition and availability of cellulose on the coated PVDF hollow fiber membrane. In regards to membrane analysis using XRD, the aim is to identify the range of phases between amorphous and crystallinity of membrane. This will support evidences of improved pure water flux performance, as amorphous phase for water filter membranes is related to hydrophilic properties, which improves water flux performance. In contrast, semi-crystalline or crystalline phases relates to hydrophobic properties, hence may exhibit lower pure water flux performance (Acarer-Arat et al., 2024; Yuan et al., 2020).

In Figure 4.3 for the combine XRD graph, it could be observed that the control neat PVDF hollow fiber membrane sample shows peaks indicative of a semi-crystalline neat PVDF membrane material similar in comparison to other studies analysing neat PVDF as a control (Acarer-Arat et al., 2024; Ma et al., 2017; Yuan et al., 2020). The key peaks “ 2θ (°)”

of PVDF control sample are within the 18°, 20°, 26° and 28° mark. Sharper and intense peaks are observed, which are signs of the crystalline phase (Acarer-Arat et al., 2024; H. Bai et al., 2012; Ma et al., 2017; Yuan et al., 2020). By observing both stacking and combine XRD graphs (Figure 4.3 and Figure 4.4), the addition of cellulose showcases peaks at similar range as the pristine PVDF hollow fiber membrane (control). All the coated samples are lower intensity in comparison to the control. The peaks are noticeably broader with moderate peaks, which some studies share similar findings regarding the surface modification influencing the membrane to exhibit amorphous phases (Acarer-Arat et al., 2024; Ma et al., 2017; Yuan et al., 2020). This result respects the pure water flux results where the coated samples surface modification influenced the membrane to exhibit amorphous phases (hydrophilic properties), hence supports this research experimental result outcome of improved pure water flux performance. A more amorphous and cellulose rich coating layer may promote water transport by increasing surface hydrophilicity and reducing the dominance of the hydrophobic properties from crystalline phase of the pristine PVDF hollow fiber membrane and the PVDF binder. This interpretation is consistent with studies showing that modification of PVDF membranes can alter crystallinity, improve hydrophilicity, and enhance water flux or antifouling performance (Yuan et al., 2020; Acarer-Arat et al., 2024; Wu et al., 2024). In addition, the XRD results supplements the FTIR and SEM results (1st experiment) by showcasing the cellulose/PVDF coating modified the structural characteristics of the pristine PVDF hollow fiber membrane.

It should be noted that the XRD analysis in this research was limited to qualitative and semi-quantitative interpretation based on peak position, peak intensity and peak broadening. Peak deconvolution and peak area integration were not performed for this XRD analysis, hence full crystallinity percentage was not calculated. Therefore, the XRD results

should be interpreted as relative crystallinity trends rather than crystallinity values. However, from the observed reduction in peak intensity and increased peak broadness, provides valid supporting evidence that cellulose/PVDF coating, modified structural characteristic of the surface of the pristine PVDF hollow fiber membrane, which supports the pure water flux performance.

4.1.5 Water Flux Analysis for Cellulose/PVDF coated PVDF Hollow Fiber Membrane Sample

Table 4.2: Pure water flux performance data for control and coated samples

Sample (0.25g PVDF Powder)		Pure water flux (L/m ² ·h·bar)
A0	PVDF (control)	157.89 ± 1.57
P01	cellulose 0.25g	432.91 ± 3.07
P02	cellulose 0.5g	483.85 ± 3.44
P03	cellulose 0.625g	388.60 ± 2.18
P04	cellulose 0.725g	249.56 ± 2.19
P05	cellulose 1.0g	180.81 ± 1.88

Pure water flux experimental test was conducted using pure deionized water for all developed Cellulose/PVDF coated PVDF hollow fiber membrane comparing with the neat PVDF hollow fiber membrane control. The pure water flux test was conducted at 1 bar functioning pressure for the total time of 1 hour per sample. It could observe that the all the Cellulose/PVDF coated PVDF hollow fiber membrane samples have increased in pure water flux performance in comparison to the PVDF hollow fiber membrane control pure water flux of 157.89 ± 1.57 L/m²·h·bar. P02 being the highest pure water flux achieve at 483.85 ± 3.44 L/m²·h·bar followed by P01 at 432.91 ± 3.07 L/m²·h·bar and then drops in performance starting from P03, P04 and P05 respectively. It could be stated that the optimal weightage of

cellulose content is P02 at 0.5g, where it has the highest pure water flux achieved for the 1st experiment. The pure water flux performance for the best performing coated membrane sample P01 and P02 is comparable to other studies which uses also PVDF and Cellulose. Trisnawati, Suryanti, and Pramono (2024) reported that cellulose incorporation improved the pure water flux of PVDF/cellulose membranes from 17.08 to 31.07 L/m²h, representing nearly a two-fold increase compared with the control (Trisnawati, Suryanti, and Pramono 2024). This supports the present findings, where P01 and P02 showed approximately three-fold improvement compared with the neat PVDF control. Similarly, Bai et al. (2025) reported that cellulose acetate blending and cellulose nanocrystal doping in PVDF membranes improved water flux by approximately two to three times compared with the control membrane, which was attributed to increased porosity, larger pore size, and improved hydrophilic behaviour (X. Bai et al. 2025).

On the other hand, the water flux performance could be interpreted with several limitations. Mainly the test was conducted for only 1 hour, so the long term stability or durability of the cellulose/PVDF coated membrane under continuous filtration was not explored. Pressure fluctuation and extended filtration testing period would be ideal to further determine the improved flux could be maintained over time or whether compaction, pore blockage, coating detachment may occur. Hence, while the pure water flux performance results demonstrate the fundamental permeability improved for coated membranes, further long term filtration experiment would support the stability and performance of the cellulose/PVDF coated PVDF hollow fiber membranes.

4.1.6 Porosity and Mean Pore Size Analysis for Cellulose/PVDF coated PVDF Hollow Fiber Membrane Sample

Table 4.3: Characteristic of Porosity and Mean pore size of control and coated samples via Gravimetric method

Sample (0.25g PVDF Powder)		Porosity (%)	Mean Pore Size (μm)
A0	PVDF (control)	69.73 ± 0.91	2.0208 ± 0.0098
P01	cellulose 0.25g	72.67 ± 0.74	3.7622 ± 0.0140
P02	cellulose 0.5g	72.67 ± 0.60	3.7186 ± 0.0117
P03	cellulose 0.625g	76.13 ± 0.72	3.0452 ± 0.0127
P04	cellulose 0.725g	76.13 ± 0.82	2.7608 ± 0.0103
P05	cellulose 1.0g	79.58 ± 0.71	2.7587 ± 0.0127

Table 4.3 shows the porosity and mean pore size of pristine PVDF hollow fiber membrane and cellulose/PVDF coated PVDF hollow fiber membrane samples via gravimetric method. The results are evaluated using standard deviation calculation to reflect the variability between repeated measurements. Overall, all coated samples showed higher porosity and mean pore size compared with the pristine PVDF hollow fiber membrane A0 (control). According to table 4.3, the porosity increased from $69.73 \pm 0.91\%$ for the A0 (control) to $72.67 \pm 0.74\%$ and $72.67 \pm 0.60\%$ for P01 and P02, respectively. These two samples the best pure water flux performance among the coated membranes. The mean pore size also increased from $2.0208 \pm 0.0098 \mu\text{m}$ for A0 to $3.7622 \pm 0.0140 \mu\text{m}$ for P01 and $3.7186 \pm 0.0117 \mu\text{m}$ for P02. The increased in porosity and mean pore size suggests that the cellulose/PVDF coating successfully improved the membrane pore structure and provide more accessible channels for water permeation.

Furthermore, for P03 to P05, the porosity continued to increase, reaching $76.13 \pm 0.72\%$, $76.13 \pm 0.82\%$, and $79.58 \pm 0.71\%$, respectively. However, the mean pore size

decreased to $3.0452 \pm 0.0127 \text{ }\mu\text{m}$, $2.7608 \pm 0.0103 \text{ }\mu\text{m}$, and $2.7587 \pm 0.0127 \text{ }\mu\text{m}$, respectively. This indicates that at higher cellulose weightage did not necessarily improve water flux performance, despite the increase in porosity. It is the reduction in mean pore size which may limits water transport by narrowing the available permeation channels. In addition, excessive cellulose content may have caused swelling or partial pore blockage within the coating layer, reducing the effective pore accessibility for water permeation. According a study by Trisnawati, Suryanti, and Pramono (2024), a similar behaviour was reported, where the modified membrane experience swelling and alters the pore structure, hence influencing pure water flux performance (Trisnawati, Suryanti, and Pramono 2024).

From the observed trend of porosity and mean pore size, it is consistent with previous studies on Cellulose/PVDF based membrane system. According to Acarer Arat et al. (2025), the incorporation of cellulose nanocrystals in PVDF based membrane system increases porosity, however, changes in pore structure also influenced water flux behaviour. Hence, the current results suggests that the optimum cellulose weightage is the balance between porosity and pore size properties as well as water flux performance. In this study, P01 and P02 showed the most favourable porosity and mean pore size for water transport, while the higher cellulose weightage from P03 to P05 may exhibit increased porosity, but the reduction in mean pore size negatively effects water permeability (Acarer Arat et al. 2025).

4.1.7 1st Experiment Conclusion

In conclusion, the use of Cellulose/PVDF as a phase inversion coating as a surface modification technique on neat PVDF hollow fiber membrane is successful as observe in the SEM, FTIR and XRD analysis. In comparison to the control, the coated samples have improved pure water flux performance up to three folds the value for samples P01 and P02 which is the best performing membrane. The correlation between cellulose weightage,

porosity, mean pore size and pure water flux is apparent where the increased porosity with smaller mean pore size has an effect on permeability of the coated membrane samples. Hence gradually reducing pure water flux performance with increased cellulose weightage.

4.2 Characterization of Cellulose/PVA Coated PVDF Hollow Fiber Membrane – 2nd Experiment

4.2 Overview for 2nd Experiment

This section covers the characterization for 2nd experiment for Cellulose/PVA coated and pristine PVDF hollow fiber membrane. The objective of the 2nd experiment was to evaluate the effectiveness of cellulose/PVA as an alternative hydrophilic coating formulation for improving the surface properties and filtration performance of pristine PVDF hollow fiber membranes. The characterization for 2nd experiment includes FTIR, SEM, XRD, Water flux performance and porosity analysis.

Based on the hypothesis of this research, the 2nd experiment is regarding the incorporation of PVA as binder with cellulose would improve the hydrophilicity and water flux performance of the membrane. Due to the coating deposition, hydrophilic functional groups present in both cellulose and PVA was expected to enhance water permeation, modify the membrane surface morphology as well as influencing the pore related properties of the coated membrane. FTIR was used to identify chemical functional groups and confirm the presence of cellulose/PVA coating, SEM was used to observe surface morphology and coating distribution, XRD was used to evaluate crystalline or amorphous structural changes, while water flux and porosity analyses were used to evaluate the membrane permeability and pore structure.

As the 2nd experiment showed to be the optimal/best pure water flux performance compared with the 1st and the subsequent 3rd experiment, a further filtration tests was added

in this section using real water sources. Peat water, river water and rain water flux was conducted to evaluate the practical performance of Cellulose/PVA coated PVDF hollow fiber membrane. In addition, atomic absorption spectroscopy (AAS) analysis was included to determine the filtration capability of the optimal samples, particularly in relation to iron removal. Overall, the 2nd experiment was conducted to determine whether cellulose/PVA coating improved hydrophilicity, water permeability and water flux performance compared to other coating formulations.

4.2.1 FTIR Analysis for Cellulose/PVDF Coated PVDF Hollow Fiber Membrane

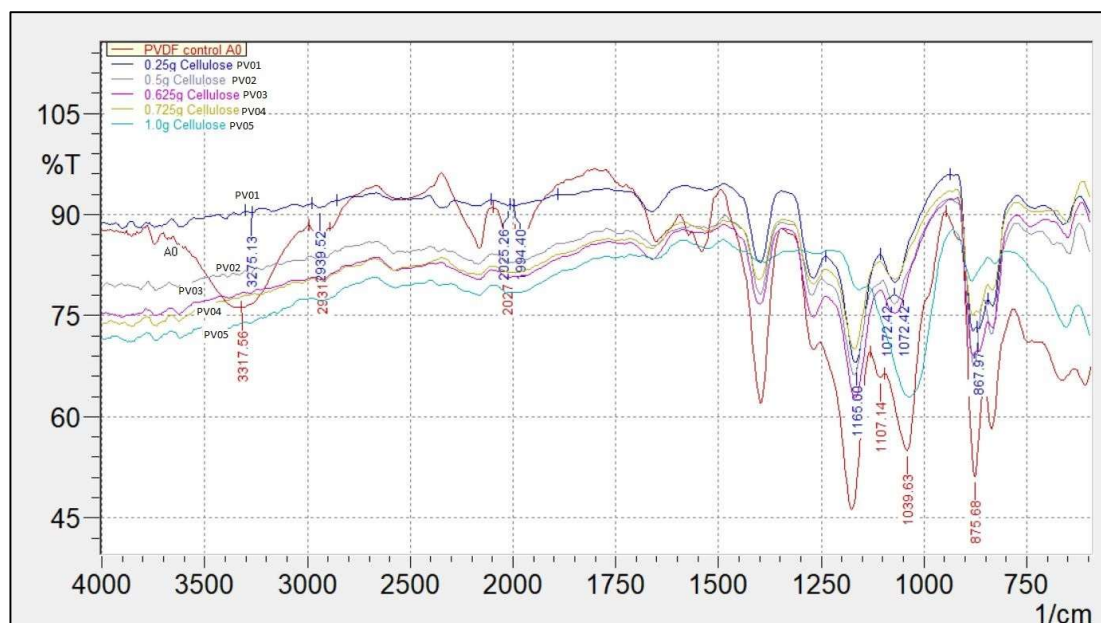


Figure 4.5: FTIR for Cellulose/PVA coated PVDF hollow fiber membrane (PV01, PV02, PV03, PV04, PV05) and Control sample (A0)

In the figure above, the FTIR graph for Cellulose/PVA coated PVDF hollow fiber membrane and control sample, there is a visible difference in peak intensity between coated samples and the control. The control sample A0 shows an identifiable stretching at C-H stretching region with bands located at the 3300 and 2900 cm^{-1} , this is indicative of $-\text{CH}_2$ groups for PVDF. The regions between 1100 and 1180 cm^{-1} has a deep stretching and peaks

at the C-F stretching region is one of the key areas to identify the existence of PVDF of the PVDF hollow fiber membrane control sample tested. The key functional groups in regard to the Cellulose/PVA coating on PVDF hollow fiber membrane are the O-H and C-O stretching regions. These regions will showcase the presence of Cellulose/PVA on the sample. The O-H stretching region 3500 to 3000 cm^{-1} shows a gradual increasing trend, indicative of the presence of cellulose and PVA (Razmgar, Saljoughi, and Mousavi 2018; Trisnawati, Suryanti, and Pramono 2024). This could be seen for all coated samples (PV01 to PV05), however PV01 has the highest intensity at this region, which corroborates with hydrophilic behaviours, improving the water flux performance (Razmgar, Saljoughi, and Mousavi 2018). The presence of cellulose could be observed at the regions 3300, 1600 and 1000 cm^{-1} , which is validated with the cellulose synthesis study (Liew et al. 2015) and a similar study regarding PVDF membrane modify with polymeric additives (PVA) (Mohamat et al. 2023).

4.2.2 SEM Analysis for Cellulose/PVDF Coated PVDF Hollow Fiber Membrane

According to Table 4.4 below, it showcases the SEM imagery at 500x and 1000x magnification for pristine PVDF hollow fiber membrane A0 (control) and Cellulose/PVA coated PVDF hollow fiber membrane samples PV01, PV02, PV03, PV04, and PV05. The control (A0) has a surface structure which is mostly smooth and even with minimal cracking and voids. This SEM imagery matches similarly to some studies with pristine PVDF hollow fiber membrane demonstrated the permeability and water flux performance of the membrane is lower in comparison to the modified PVDF hollow fiber membranes (Oppong et al. 2025; Kamaludin et al. 2022).

Table 4.4: SEM of Cellulose/PVA coated PVDF hollow fiber membrane and Control sample

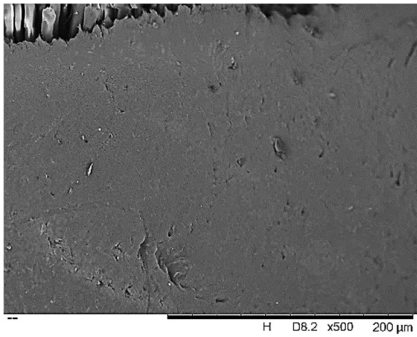
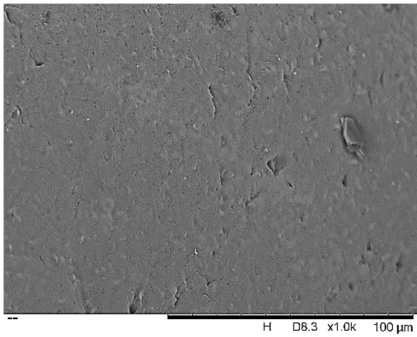
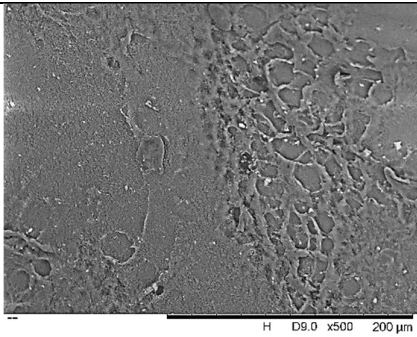
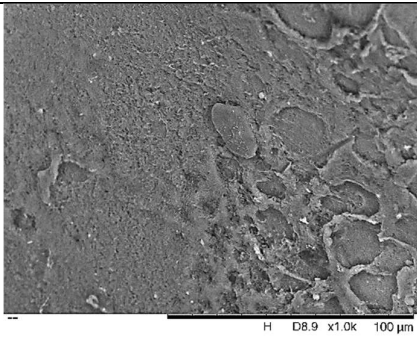
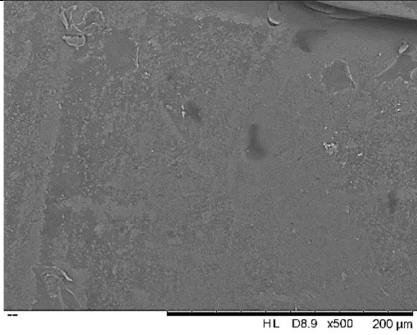
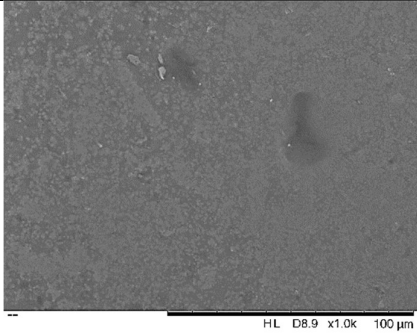
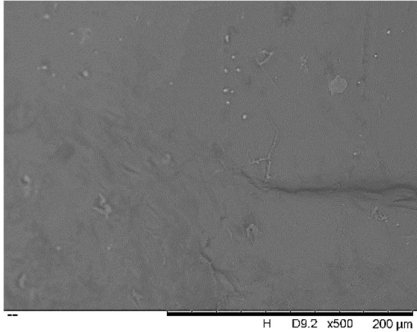
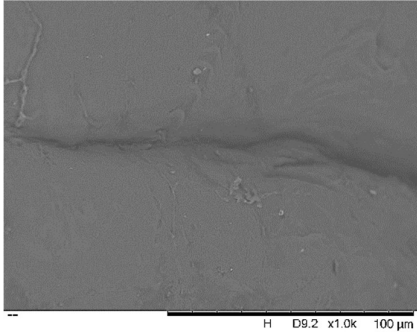
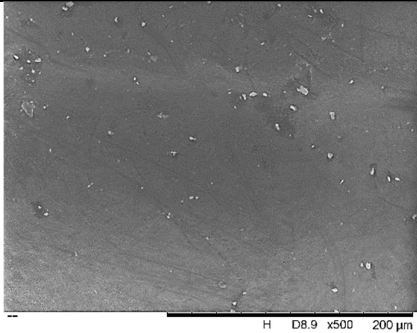
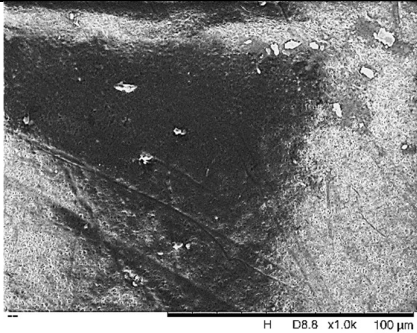
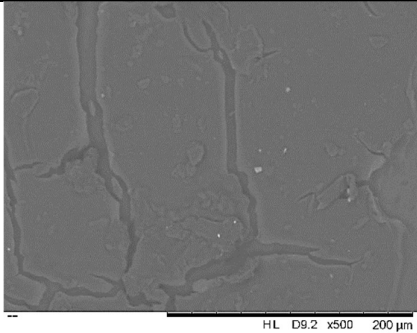
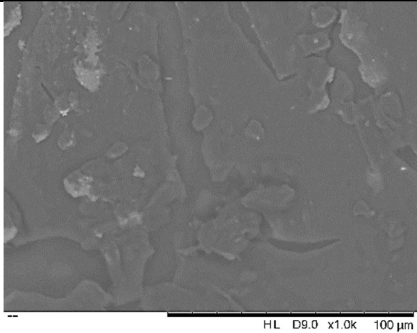
Sample	500x Magnification	1000x Magnification
A0 Pristine PVDF hollow fiber membrane		
PV01 Cellulose at 0.250g		
PV02 Cellulose at 0.500g		

Table 4.4 continued

PV03 Cellulose at 0.625g		
PV04 Cellulose at 0.725g		
PV05 Cellulose at 1.000g		

PV01 surface structure shows a webbing type structure and observably porous in comparison to the rest of the coated samples. PV02 shows no webbing type structure but a mildly porous surface structure. It should be noted that PV01 and PV02 has the highest water flux performance among all samples, with PV01 being the highest water flux, followed by

PV02. These SEM results do relate to a few studies with the incorporation of cellulose or PVA with PVDF membrane (Ma et al. 2017; O. Koriem et al. 2022; N. Li et al. 2010). The surface structure of PV03-PV05, shows visible thicker coating, with overlapping layer with cracking and lengthy voids. This is indicative of a thicker coating due to the increasing cellulose content (Ma et al. 2017; N. Li et al. 2010). In respect to the correlation between the SEM and water flux performances, it is likely that even with the thicker coating for PV03-PV05, due to the hydrophilic nature of both cellulose and PVA, the water flux performances are still higher in comparison to the control sample, especially for PV01 which is considered to be the best performing or optimal sample (Ma et al. 2017; O. Koriem et al. 2022; N. Li et al. 2010).

In regards to coating uniformity, samples PV01 and PV02 appear to showed more favourable surface coating in comparison to samples PV03–PV05. The web-like and mildly porous structures observed for PV01 and PV02 suggest that the cellulose/PVA coating was distributed across the surface thinly and developed during phase inversion, resulting in improved water transport and permeability. On the other hand, the thicker coating layers observed for PV03–PV05 indicate less coating uniformity, which may be due to increased coating solution density as cellulose weightage increases. Similar findings have been reported for membranes with polymer or cellulose increased in concentration influenced membrane morphology (coating uniformity), pore structure, and water permeability (Ma et al., 2017; Li et al., 2010; Koriem et al., 2022). The overlapping layers, cracks, and voids in present in PV03–PV05 suggests possible coating defects caused during phase inversion or by shrinkage during drying or excessive coating accumulation. These defects may reduce pore formation for enhanced water transport channels, hence explains the lower water flux performance for PV03–PV05 in comparison to PV01 and PV02, despite having better

performance in comparison to the control sample A0 for pristine PVDF hollow fiber membrane (Ma et al., 2017; Li et al., 2010; Koriem et al., 2022).

4.2.3 XRD Analysis for Cellulose/PVDF Coated PVDF Hollow Fiber Membrane

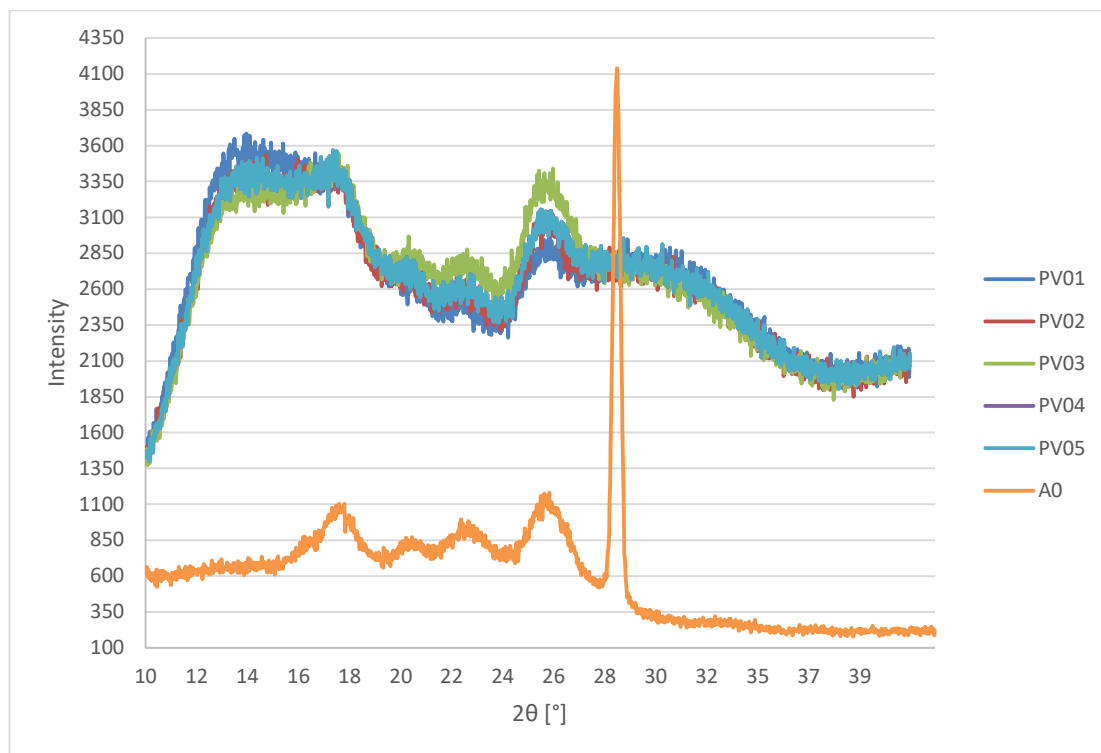


Figure 4.6: The combine XRD graph for Cellulose/PVA coated PVDF hollow fiber membrane and Control sample

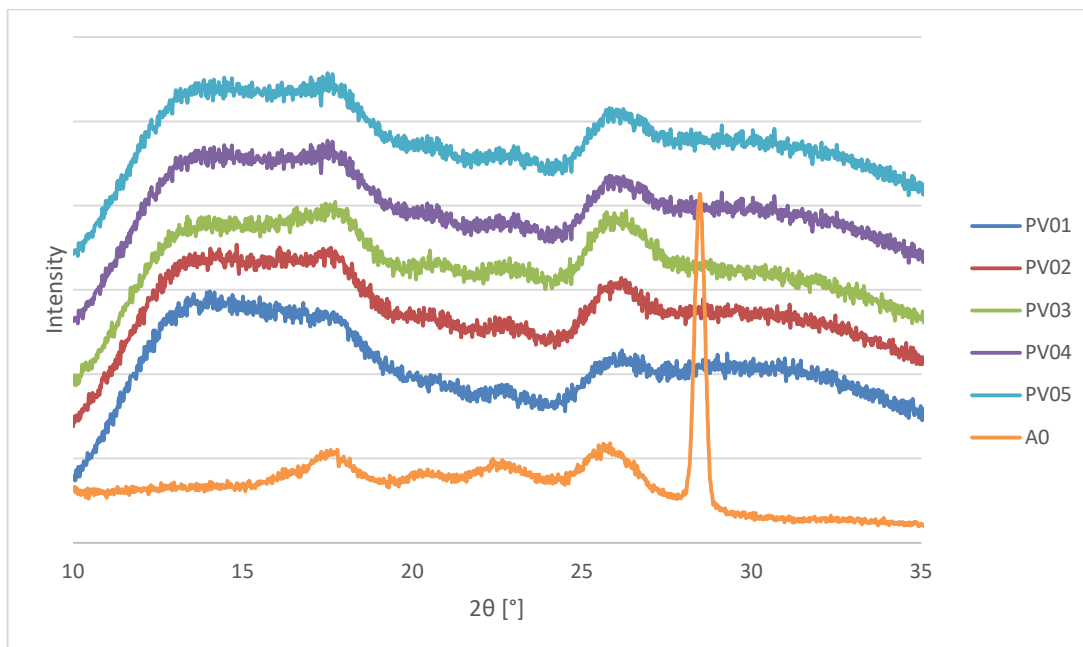


Figure 4.7: The stacking XRD graph for Cellulose/PVA coated PVDF hollow fiber membrane and Control sample

The XRD analysis is to identify the range of phases possibly between amorphous and crystallinity of membrane and additional evidence of the presence of cellulose and PVA powder incorporation as a coating. This will showcase evidence related to pure water flux performance, as amorphous regions for water filter membranes may be related to improved pure water flux performance, hence showing hydrophilic characteristics. The crystalline regions may be related to lower pure water flux performance hence showing hydrophobic characteristics (Yuan et al. 2020; Acarer-Arat et al. 2024). In Figure 4.6 and Figure 4.7, the combine XRD graph, the control neat PVDF hollow fiber membrane sample shows peaks and intensity indicative of a neat PVDF membrane with a semi crystalline region which is similarly shown in other XRD analysis studies regarding neat PVDF membranes, which is the same as in the 1st experiment (Acarer-Arat et al. 2024; Yuan et al. 2020; Ma et al. 2017). The key peaks “ 2θ (°)” of PVDF control sample are within the 18, 20, 26 and 28° mark which can be clearly seen in Figure 4.6 and Figure 4.7.

The sharper and intense peaks are signs of the membrane crystallinity phase (Acarer-Arat et al. 2024; Yuan et al. 2020; Ma et al. 2017; H. Bai et al. 2012). It could be observed in Figure 4.6 and Figure 4.7 for PV01 to PV05, all shows a broad and flatten peak from 13° to 18° indicating amorphous regions likely to be from the Cellulose/PVA coating. The peaks observed for PV01, shows higher intensity in comparison to the other samples, which may be the results of better coating distribution due to the x-ray diffraction is prominent and there are observable boarder peaks, indicating more amorphous regions (Karim et al. 2020; Mohamat et al. 2023).

It should be noted that the XRD analysis in this study was limited to qualitative interpretation supported by semi-quantitative comparison of 2θ peak position, relative peak intensity, and peak broadness. Quantitative peak analysis, such as peak deconvolution, peak area integration, and crystallinity percentage calculation, was not conducted. Hence, the XRD results should not be interpreted as exact crystallinity values, but as relative structural trends between cellulose/PVA coated PVDF hollow fiber membranes (PV01-PV05) and the control (A0).

However, despite this limitation, the observed reduction in PVDF peak intensity and the appearance of broader peaks between 13° and 18° provides valuable evidence of cellulose/PVA coating contributed to an amorphous phase surface structure. This interpretation is sufficient to support the coating evaluation and relate the structural changes observed by XRD with enhanced pure water flux performance for the coated membranes (Yuan et al., 2020; Acarer-Arat et al., 2024; Ma et al., 2017).

4.2.4 Water Flux Analysis for Cellulose/PVDF Coated PVDF Hollow Fiber Membrane

The water flux performance involves the analysis of pure water flux, rainwater flux, river water flux, and peat water flux. All water flux performance test with Cellulose/PVA coated PVDF hollow fiber membranes and control membrane was conducted at 1 bar functioning pressure for the total time of 1 hour per sample.

Pure Water Flux Analysis:

Table 4.5: Pure water flux performance data for control and coated samples

Sample (0.25g PVA Powder)		Pure water flux (L/m ² ·h·bar)
A0	PVDF (control)	157.89 ± 1.57
PV01	cellulose 0.25g	860.79 ± 4.14
PV02	cellulose 0.5g	741.02 ± 3.32
PV03	cellulose 0.625g	414.20 ± 2.54
PV04	cellulose 0.725g	239.12 ± 2.28
PV05	cellulose 1.0g	253.38 ± 1.81

Rainwater Flux Analysis:

Table 4.6: Rainwater flux performance data for control and coated samples

Sample (0.25g PVA Powder)		Rainwater flux (L/m ² ·h·bar)
A0	PVDF (control)	125.80 ± 1.73
PV01	cellulose 0.25g	732.52 ± 3.79
PV02	cellulose 0.5g	626.07 ± 3.30
PV03	cellulose 0.625g	414.13 ± 2.50
PV04	cellulose 0.725g	201.18 ± 1.88
PV05	cellulose 1.0g	238.10 ± 2.03

River water Flux Analysis:

Table 4.7: River water flux performance data for control and coated samples

Sample (0.25g PVA Powder)		River water flux (L/m ² ·h·bar)
A0	PVDF (control)	121.53 ± 1.53
PV01	cellulose 0.25g	631.55 ± 3.89
PV02	cellulose 0.5g	392.17 ± 2.68
PV03	cellulose 0.625g	258.48 ± 1.96
PV04	cellulose 0.725g	147.70 ± 1.34
PV05	cellulose 1.0g	183.35 ± 1.67

Peat water Flux Analysis

Table 4.8: Peat water flux performance data for control and coated samples

Sample (0.25g PVA Powder)		Peat water flux (L/m ² ·h·bar)
A0	PVDF (control)	98.81 ± 1.38
PV01	cellulose 0.25g	421.45 ± 3.10
PV02	cellulose 0.5g	385.80 ± 2.72
PV03	cellulose 0.625g	213.91 ± 2.19
PV04	cellulose 0.725g	128.98 ± 1.86
PV05	cellulose 1.0g	156.61 ± 1.88

From analysing Table 4.5 to Table 4.8, a pattern can be identified, where all coated samples have improved water flux performance regardless of the type of water being filtered (pure, rain, river, peat water). Consistently PV01 has proven to be the best performing coated membrane, followed by PV02 as the second best performing coated membrane. The

pattern of improved performance for PV01 and PV02, followed by a steep decline in performance for PV03 to PV05, corroborates with the porosity analysis.

The incorporation of increasing cellulose content and PVA as polymer binder, has its merits for improving water flux performance, especially for test real water sources such as rain, river and peat water. This improved water flux performance is likely to due to both Cellulose and PVA has an affinity for water (Choi and Nam 2025). If comparing all water flux performance (pure, peat, river and rain water) for PV01 and PV02 against the control A0, the water flux performance for PV01 and PV02 has increased between four to five folds. Some studies share a similar finding of modifying a PVDF membrane by utilizing cellulose (acetate, nanocrystals) or PVA doped, and/or blended, has increased water flux performance two to three folds in comparison to the control samples. It was also considered by these researchers that the increase porosity, pore size and hydrophilic behaviour of cellulose and PVA may help modify and improve the PVDF hollow fiber membrane surface structure hence improving water flux performance (M. Li et al. 2019; Choi and Nam 2025; X. Bai et al. 2025; N. Li et al. 2010).

In regards to real world applications, the improved water flux of the cellulose/PVA coated PVDF hollow fiber membranes is relevant for rural water treatment in Borneo, where rainwater, river water and peat water are the available water sources. The higher water flux allows more water to be filtered in a shorter span of time at relatively low pump operating pressure (1bar). This is important for small scale and decentralized systems with limited energy and maintenance capacity. The improved performance of PV01 and PV02 suggests that cellulose/PVA coating has potential to enhance pristine PVDF hollow fiber membrane filtration for rural water supply applications, however further investigation for long term

usage, fouling resistance and field stability may still be required (Li et al., 2010; Kamaludin et al., 2022; Akhondi et al., 2017; He et al., 2017).

4.2.5 Porosity and Mean Pore size Analysis for Cellulose/PVDF Coated PVDF Hollow

Table 4.9: Characteristic of porosity and mean pore size of control and coated samples via gravimetric method

Sample (0.25g PVA Powder)		Porosity (%)	Mean Pore Size (μm)
A0	PVDF (control)	69.73 ± 0.91	2.0208 ± 0.0098
PV01	cellulose 0.25g	79.58 ± 0.71	4.3311 ± 0.0220
PV02	cellulose 0.5g	78.82 ± 0.58	4.6049 ± 0.0265
PV03	cellulose 0.625g	78.00 ± 0.61	2.4475 ± 0.0191
PV04	cellulose 0.725g	77.10 ± 0.71	3.0976 ± 0.0236
PV05	cellulose 1.0g	77.10 ± 0.66	2.3745 ± 0.0199

In Table 4.9 showcases the porosity and mean pore size analysis via Gravimetric method for Cellulose/PVA coated PVDF hollow fiber membrane samples PV01-PV05 and the control A0. All Cellulose/PVA coated samples PV01-PV05 have increased in porosity and mean pore size in comparison to the control A0. In correlation to the water flux performance, the best performing coated membrane PV01, has increased porosity from $69.73 \pm 0.91\%$ for A0 to $79.58 \pm 0.71\%$ for PV01, for the second best performing membrane $78.82 \pm 0.58\%$ for PV02. In regards to the mean pore size, for PV01 and PV02, it increased from $2.0208 \pm 0.0098\mu\text{m}$ for the control A0 to $4.3311 \pm 0.0220 \mu\text{m}$ and $4.6049 \pm 0.0265\mu\text{m}$ respectively. The porosity and mean pore size do corroborate with the water flux performance, as PV01 and PV02 increased in water flux performance along with the increasing porosity and mean pore size. PV03- PV05 show signs of slight decrease in porosity and a significant decrease in mean pore size, in which may lead to a drop in water

flux performance. The decreasing mean pore size may influence the drop in water flux performance.

As the membrane is in contact with the water, it swells in size, in combination with a reduced mean pore size, a blockage may occur reducing the available spaces for water to permeate through the membrane (Trisnawati, Suryanti, and Pramono 2024). As the more cellulose content is being used, the blockage is likely to occur, as observed with the reduction in mean pore size as the cellulose content is increased to the maximum content of 1.0g. Even though the Cellulose/PVA has a hydrophilic behaviour, the denser the coating the porosity and mean pore size are affected due to fewer macrovoids (Trisnawati, Suryanti, and Pramono 2024; Hossein Razzaghi et al. 2014).

4.2.6 AAS Analysis for Cellulose/PVDF Coated PVDF Hollow Fiber Membrane

The filtered and raw unfiltered rain, river and peat water samples undergo an Atomic Absorption Spectrometry (AAS) Analysis for heavy metals, utilizing iron (Fe) as a key marker for water treatment capabilities of the samples. As raw rainwater is rather clean from the start of the experiment with a negative ppm reading, it indicates rainwater is free of heavy metal iron, hence no further AAS analysis is required.

Table 4.10: Atomic Absorption Spectrometry (AAS) for Raw (Unfiltered) and Filtered Rainwater, River water, and Peat water for Control A0 and Coated samples PV01 to PV05

Sample	Iron (Fe) Concentration	Percentage Filtered
Raw Rainwater	-0.1153 ppm	-
Raw River water	0.3108 ppm	-
Raw Peat water	0.7840 ppm	-
Control Filtered Rainwater A0	-	-

Table 4.10 continued

Filtered Rainwater PV01	-	-
Filtered Rainwater PV02	-	-
Filtered Rainwater PV03	-	-
Filtered Rainwater PV04	-	-
Filtered Rainwater PV05	-	-
Control Filtered River water A0	-0.0353 ppm	100%
Filtered River water PV01	-0.1153 ppm	100%
Filtered River water PV02	-0.0282 ppm	100%
Filtered River water PV03	-0.0212 ppm	100%
Filtered River water PV04	-0.0706 ppm	100%
Filtered River water PV05	-0.0565 ppm	100%
Control Filter Peat water A0	0.6169 ppm	23.31%
Filtered Peat water PV01	0.4733 ppm	39.64%
Filtered Peat water PV02	0.4238 ppm	45.94%
Filtered Peat water PV03	0.4356 ppm	44.43%
Filtered Peat water PV04	0.5439 ppm	30.63%
Filtered Peat water PV05	0.4215 ppm	46.23%

From analysing Table 4.10, river water was successful at filtering the 0.3108ppm of iron from raw river water, this result is consistent with the control and all coated sample. Hence the river water is not sufficient to showcase the coated samples capabilities. However, for peat water there is a clear pattern emerges in correlation with increased cellulose content. The control A0 manages to filter 23.31% of iron, followed by PV01 with 39.64%, PV02 with 45.49%, PV03 with 44.43%, PV04 with 30.63%, and PV05 with 46.23%. Overall, as

cellulose content increases, the concentration of iron being filtered also increases (Çifci and Kaya 2010; Arthanareeswaran et al. 2007; Fakhfekh et al. 2017).

However, in respect to the water flux performance results, higher cellulose content shows a decline in water flux performance. Hence it is suggested that coated samples could be utilized for the purpose of rain, river and peat water filtration applications via separate modular units. For example, using the percentage filtered data, if one is using the control sample A0, it may take up 5 times the modular unit in comparison to the coated samples PV01-PV05 which may utilize up to 2.5 times the modular units to successfully remove iron from raw peat water. A study on the preparation of PVA/Cellulose composite membranes for metal removal from aqueous solutions showcases the removal of iron Fe (III) in terms of retention %. It was found that the PVA/Cellulose composite membrane is capable of 82% retention, meaning 18% of iron Fe (III) was able to pass through the membrane. This showcases the capability and potential of the variation of PVA/Cellulose membranes, even as a coating, for water treatment applications (Çifci and Kaya 2010; Fakhfekh et al. 2017).

As the AAS analysis in this research only focused on the iron removal aspect, natural water sources such as river water and peat water also contain other contaminants and dissolved metals which could influence the membrane performance. Hence, in future research could include multi-metal analysis to better evaluate the broad spectrum of the filtration capability of the cellulose/PVA coated PVDF hollow fiber membranes. Previous studies show that cellulose/PVA composite membranes and other polymeric membrane systems could be used for metal ion or heavy metal removal from aqueous solutions (Çifci & Kaya, 2010; Arthanareeswaran et al., 2007). In addition, long term heavy metal removal efficiency should be investigated through extended filtration cycles, since membrane

performance may alter over time due to overall age, usage, fouling and pore blockage. According to Fakhfeh et al., (2017), the relevance of membrane based iron removal for drinking water treatment has also been demonstrated through oxidation microfiltration systems (Fakhfekh et al., 2017). Hence, the present research demonstrates the potential of cellulose/PVA coating on pristine PVDF hollow fiber membranes for iron removal from real water sources, further multi metal removal and long term membrane stability testing may be required to validate their practical suitability for prolonged real world water treatment applications.

4.2.7 2nd Experiment Conclusion

The phase inversion method of coating as a surface modification technique using Cellulose/PVA on neat PVDF hollow fiber membrane is successful as observe in the SEM. The coating distribution and surface roughness give indicative signs of improved water flux performance. The FTIR analysis showcases evidence of availability of Cellulose/PVA on the surface of the PVDF hollow fiber membrane, from observing the O-H and C-H stretching regions present. The XRD analysis showcases evidence of hydrophilic behaviours via peaks which illustrates an amorphous phase for the presence of Cellulose/PVA and semi-crystalline phase for PVDF. In comparison to the control, the coated samples have improved pure water, rainwater, river water and peat water flux performance up to four to five folds the value for sample PV01, which is the best performing membrane. The correlation between cellulose content, porosity, mean pore size and water flux (pure water, rainwater, river water and peat water) performance is apparent as there an optimal range (PV01-PV02) and the limit (PV03-PV05) of adding cellulose, where there is a decline in performance. The AAS analysis showcases the filtering ability for iron (Fe) in rain, river and peat water. The results

yield the higher the cellulose content to more iron (Fe) the membrane can filter, however it will sacrifice the water flux performance.

4.3 Characterization of Graphene Oxide Powder Effect on Cellulose/PVDF and Cellulose/PVA Coated PVDF Hollow Fiber Membrane – 3rd Experiment

4.3 Overview for 3rd Experiment

This section covers the characterization for 3rd experiment for Graphene oxide Cellulose/PVDF and Graphene oxide Cellulose/PVA coating onto a pristine stock PVDF hollow fiber membrane. The 3rd experiment repeats the 1st and 2nd experiment methodology and characterization, but with an additional graphene oxide as additive was included in the respective coating solution. The characterization for 3rd experiment includes FTIR, SEM, XRD, Water flux performance, and Porosity analysis.

4.3.1 FTIR Analysis for Graphene Oxide Cellulose/PVDF and Cellulose/PVA Coated PVDF Hollow Fiber Membrane

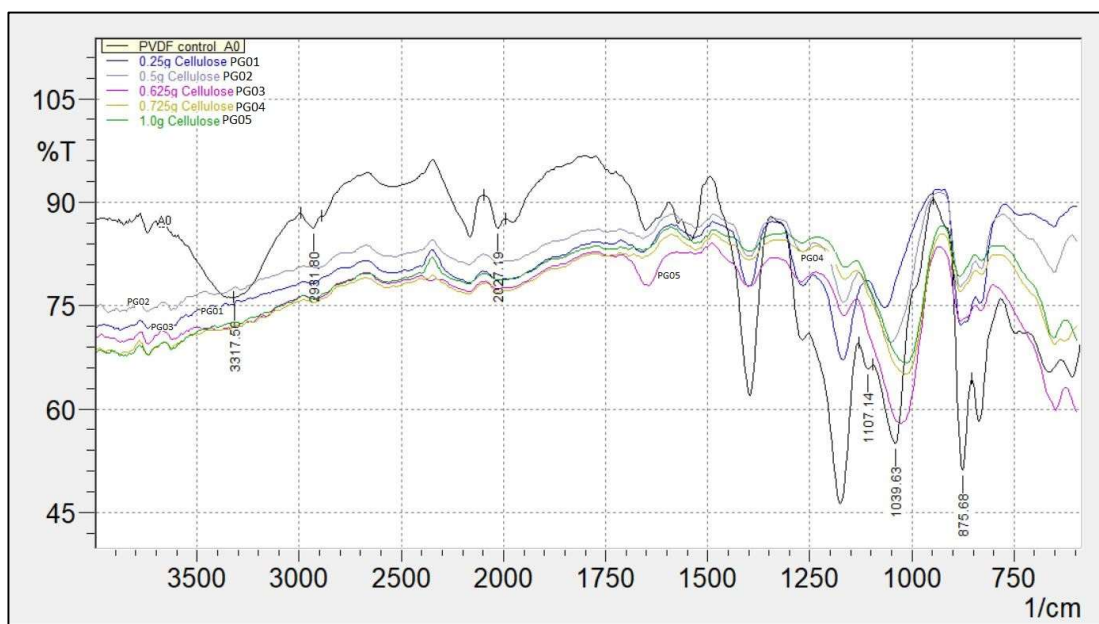


Figure 4.8: FTIR Analysis for Graphene Oxide Cellulose/PVDF Coated PVDF Hollow Fiber Membrane

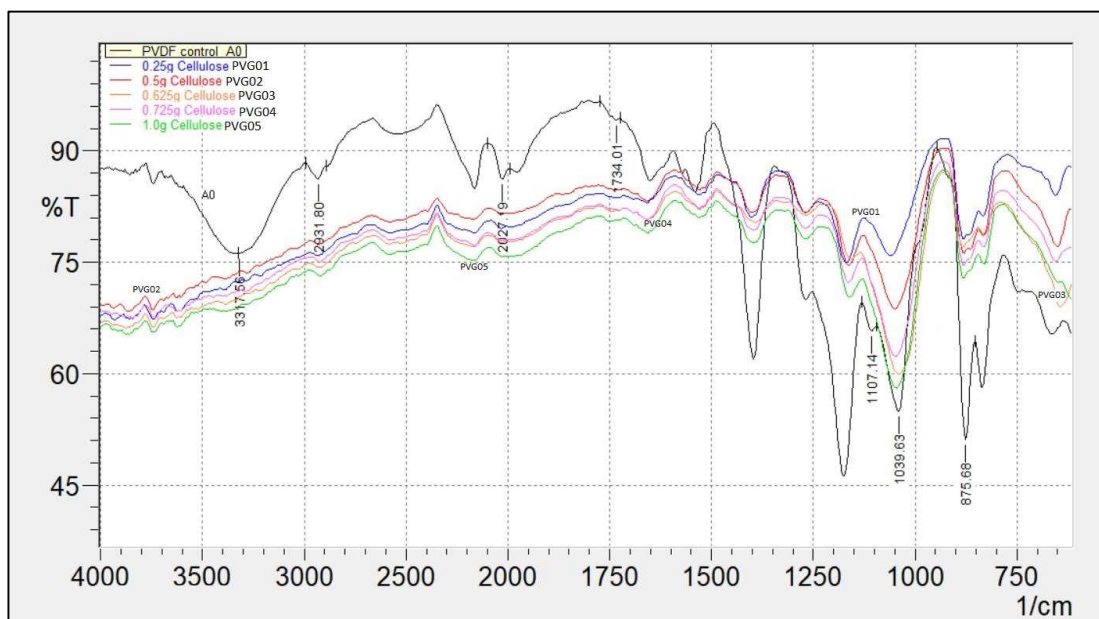


Figure 4.9: FTIR Analysis for Graphene Oxide Cellulose/PVA Coated PVDF Hollow Fiber Membrane

The FTIR graphs for Graphene oxide Cellulose/PVDF and Graphene oxide Cellulose/PVA coated PVDF hollow fiber membrane and the control sample, it could be observed that there is a visible difference in peak intensity between coated samples and the control. Peaks labelled for the control illustrates the control is truly the pristine PVDF hollow fiber membrane sample. The control sample shows an identifiable stretching at C-H stretching region with bands located at 3371.56 and 2931.80 cm^{-1} , this is indicative of $-\text{CH}_2$ groups for PVDF. Another identifiable stretching region at the C-F stretching region with band located at 1174.65 and 1107.14 cm^{-1} , the intense stretching and peaks at the C-F stretching region is one of the key areas to identify the existence of PVDF of the PVDF hollow fiber membrane control sample tested. This is a match to the control sample for previous 1st and 2nd experiment results.

Graphene oxide Cellulose/PVDF coating on PVDF hollow fiber membrane main identifiers could be observed at the O-H stretching region a continuously increasing broad peaks from $3700\text{-}2500\text{ cm}^{-1}$. These broad and gradual bands this is indicative of hydroxyl groups showcasing the addition of cellulose and graphene oxide available on the sample. This could be also observed the same with Graphene oxide Cellulose/PVA coated PVDF hollow fiber membrane samples. In the 3rd experiment, the addition of graphene oxide has a significant effect on the transmittance observed in both FTIR graphs, when compared to 1st and 2nd experiment FTIR results.

According to a study on Graphene oxide/PVA membrane, features of graphene oxide are observed in the FTIR results peaks at 1720 cm^{-1} , 1235 cm^{-1} , 1090 cm^{-1} and 1050 cm^{-1} . These peaks functional groups are categorized as C-O stretching, C-OH stretching, O-H deformation and C-O-C stretching, respectively (Lin et al. 2025; Rapisarda, Malfense Fierro, and Meo 2021). In respect to both Graphene oxide Cellulose/PVDF and Graphene

oxide Cellulose/PVA FTIR results, these regions could similarly be observed (Lin et al. 2025). The masking effect is observable due to the available increasingly broad peaks from 3700-2500 cm^{-1} , which is a significant functional group for hydroxyl groups, however this also indicates the samples hydrophilic behaviour. A study on graphene oxide/cellulose nanocrystals PVDF membrane, showcases similar FTIR result with their sample having a broad peak at the region 3342 cm^{-1} onwards and a sharp peak at 1660 cm^{-1} . According to the researchers FTIR results, it was suggested that the results showcase a strong water adsorption capacity and hydrophilic nature of their graphene oxide/cellulose nanocrystal PVDF membrane (Lv, Zhang, Zhang, and Yang 2018).

To assess graphene oxide incorporation, the FTIR spectra were compared based on peak position, relative intensity, and peak broadening. In comparison to the control, graphene oxide cellulose/PVDF and graphene oxide cellulose/PVA coated membranes showed broader absorption in the 3700–2500 cm^{-1} region, indicating increased O–H contribution from cellulose, PVA and graphene oxides oxygen containing groups. In addition, features around 1720, 1235, 1090, and 1050 cm^{-1} are associated with C=O, C–OH, C–O, and C–O–C groups commonly reported in graphene oxide-based membranes (Lin et al., 2025; Rapisarda et al., 2021). Hence, the increased broadness and relative intensity of those oxygen containing functional group regions supports the successful incorporation of graphene oxide into the coating layer. However, the FTIR analysis is limited to relative FTIR comparison, as peak area integration or intensity ratio calculation was not performed.

4.3.2 SEM Analysis for Graphene Oxide Cellulose/PVDF and Cellulose/PVA Coated PVDF Hollow Fiber Membrane

Table 4.11: SEM Analysis for Graphene Oxide Cellulose/PVDF and Cellulose/PVA Coated PVDF Hollow Fiber Membrane

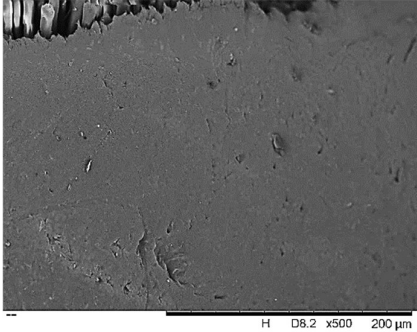
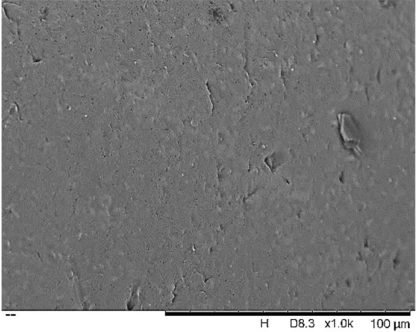
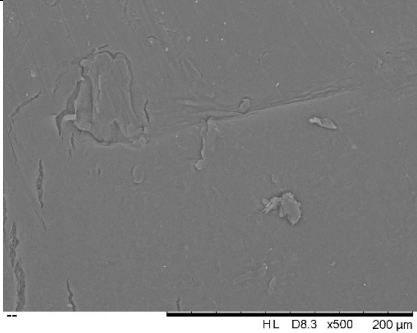
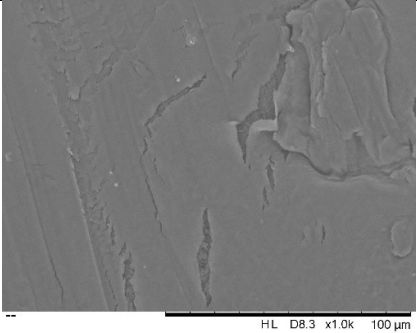
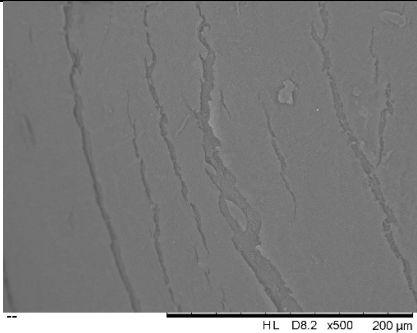
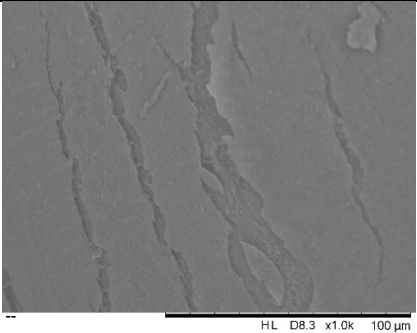
Sample	500x Magnification	1000x Magnification
A0 Pristine PVDF hollow fiber membrane		
PG01 PVDF powder, Cellulose at 0.250g		
PG02 PVDF powder, Cellulose at 0.500g		

Table 4.11 continued

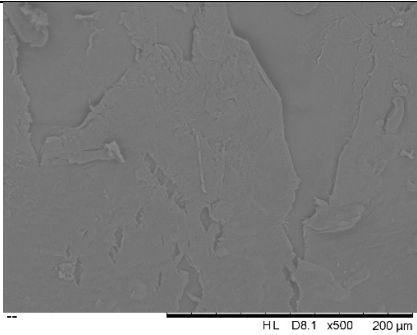
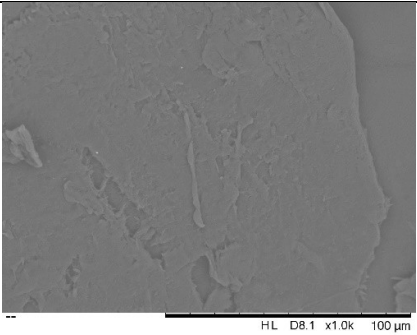
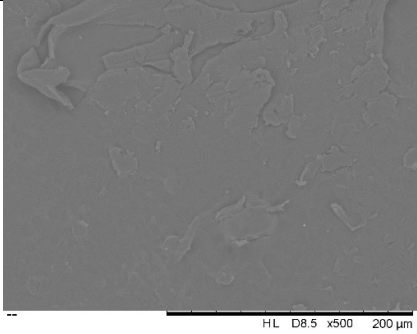
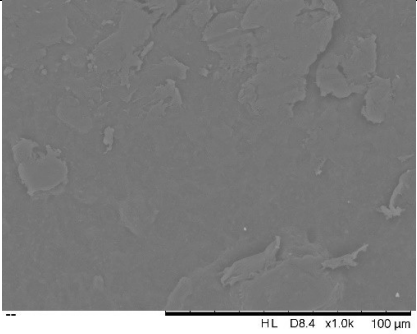
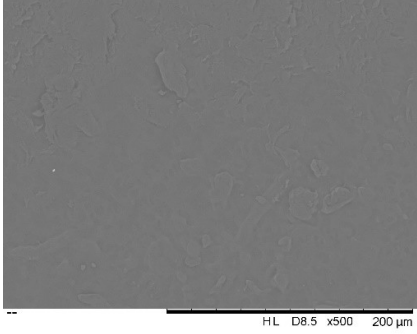
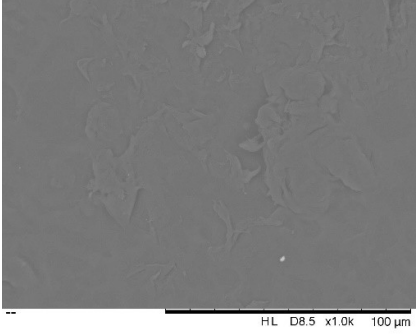
PG03 PVDF powder, Cellulose at 0.625g	 HL D8.1 x500 200 µm	 HL D8.1 x1.0k 100 µm
PG04 PVDF powder, Cellulose at 0.725g	 HL D8.5 x500 200 µm	 HL D8.4 x1.0k 100 µm
PG05 PVDF powder, Cellulose at 1.000g	 HL D8.5 x500 200 µm	 HL D8.5 x1.0k 100 µm

Table 4.11 continued

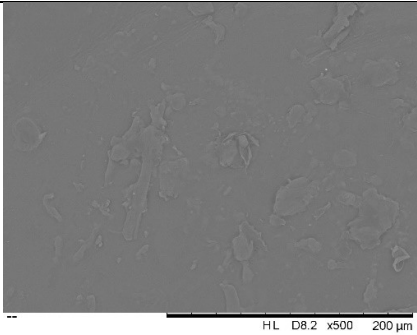
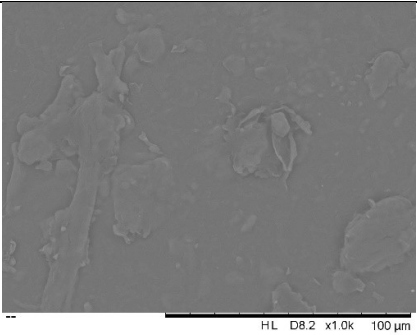
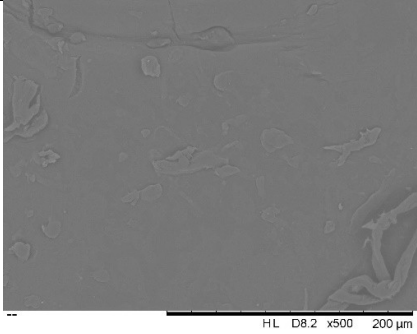
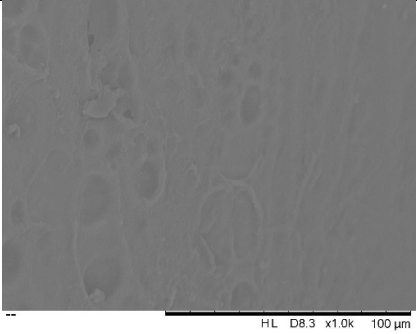
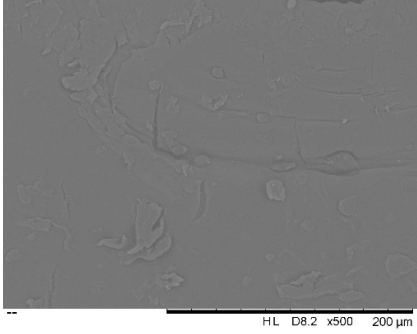
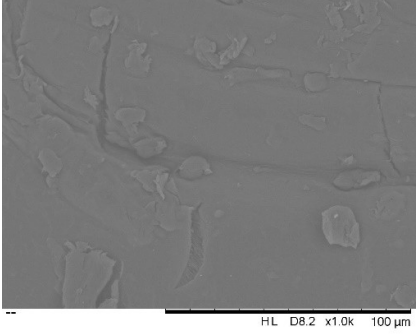
PVG01 PVA powder, Cellulose at 0.250g	 HL D8.2 x500 200 µm	 HL D8.2 x1.0k 100 µm
PVG02 PVA powder, Cellulose at 0.500g	 HL D8.2 x500 200 µm	 HL D8.3 x1.0k 100 µm
PVG03 PVA powder, Cellulose at 0.625g	 HL D8.2 x500 200 µm	 HL D8.2 x1.0k 100 µm

Table 4.11 continued

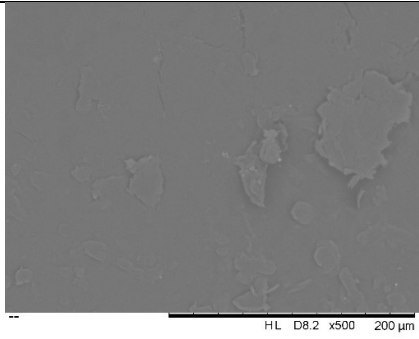
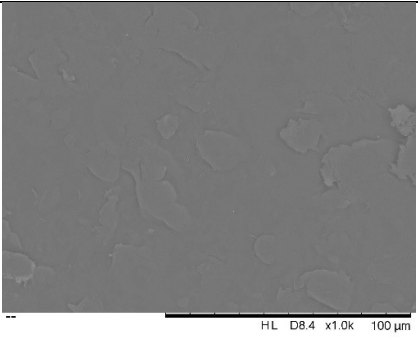
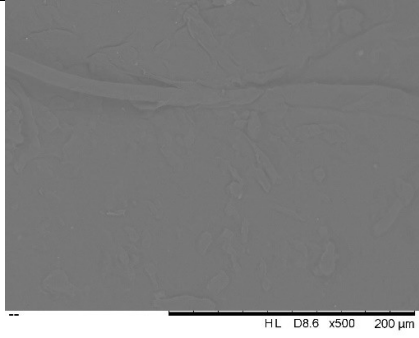
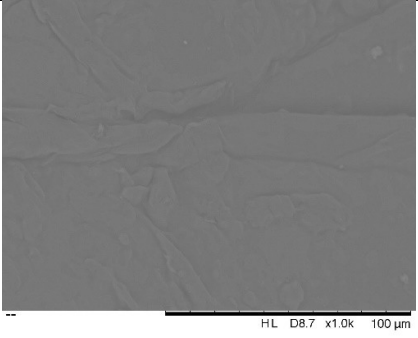
PVG04 PVA powder, Cellulose at 0.725g		
PVG05 PVA powder, Cellulose at 1.000g		

Table 4.11 showcases the SEM imagery at 500x and 1000x magnification for the control PVDF hollow fiber membrane: A0. Coated Graphene oxide Cellulose/PVDF samples: PG01, PG02, PG03, PG04, PG05 and coated Graphene oxide Cellulose/PVA samples: PVG01, PVG02, PVG03, PVG04, PVG05.

It could be observed that A0 has a surface structure which is generally smooth and compact with minimal visible pores. This result matches few studies in regard to neat PVDF, where water flux or permeability of the membrane is lower as compared to modified PVDF membrane (Ma et al. 2017; H. Bai et al. 2012). The addition of graphene oxide to the coating solution seems to give a thicker distributed layer along the surface of the PVDF hollow fiber membrane samples. This observation could be seen in both Graphene oxide Cellulose/PVDF (PG01-PG05) and Graphene oxide Cellulose/PVA coatings (PVG01-PVG05). In

comparison to without graphene oxide in 1st and 2nd experiment, the SEM imagery shows a rougher and larger porous structure on the samples.

It is possible that cellulose in addition with graphene oxide contributes to a thicker distributed layer along the surface of the PVDF hollow fiber membrane samples (El MIRI et al. 2015). One study does support this case, where the researchers showcase their samples with dense and hierarchical layered sheet-like structure on the surface of the PVDF sample and suggests its due to the aggregation of their cellulose nanocrystals (Lv, Zhang, Zhang, and Yang 2018; Sun et al. 2022).

Similar case wise to the 1st and 2nd experiment, the addition of graphene oxide may accelerate the thickening of the coating solution even more with the increased in cellulose content. This is indicative of a thicker and denser coating due to the increasing cellulose content (Ma et al. 2017; N. Li et al. 2010). In respect to the correlation between the SEM and water flux performances for the 3rd experiment it is fairly similar to the previous 1st and 2nd experiment. However, due to the graphene oxide accelerating the thickening of the coating on the surface alongside the increasing cellulose content, the water flux performance only improved for the smallest cellulose content (PG01 and PVG01) and drop significantly for the rest of the samples (Ma et al. 2017; O. Koriem et al. 2022; N. Li et al. 2010).

Based on the SEM results, it is suggested that graphene oxide may contribute to aggregation and coating defects as cellulose weightage is increased as formulated. However, as graphene oxide has oxygen functional groups which could improve hydrophilic properties, the graphene oxide structural sheets may influence stacking or uneven dispersion within the coating. As observed for PG01 and PVG01, at lower cellulose weightage, graphene oxide may improve surface roughness and water transport channels. In contrast, as cellulose weightage is increased, graphene oxide aggregation, alongside denser and thicker coating

formation may reduce pore accessibility and increase hydraulic resistance which explains lower water flux performance for PG02–PG05 and PVG02–PVG05 samples (Lv et al., 2018; Sun et al., 2022; Ma et al., 2017; Li et al., 2010).

4.3.3 XRD Analysis for Graphene Oxide Cellulose/PVDF and Cellulose/PVA Coated PVDF Hollow Fiber Membrane

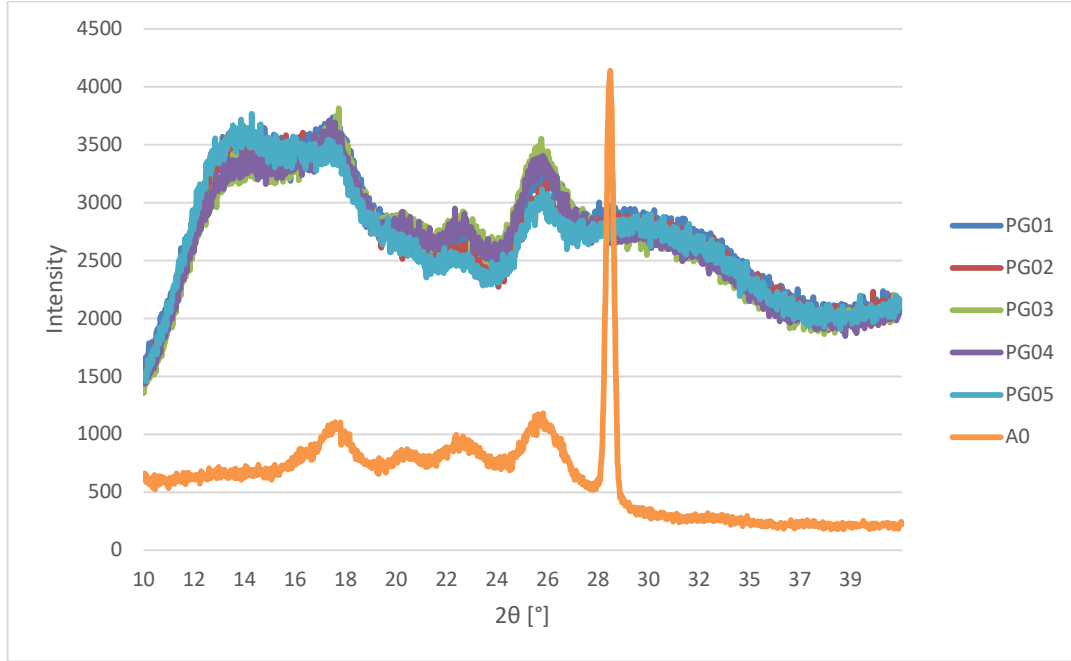


Figure 4.10: The combine XRD graph for Graphene Cellulose/PVDF coated PVDF hollow fiber membrane (PG01-PG05) and Control sample (A0)

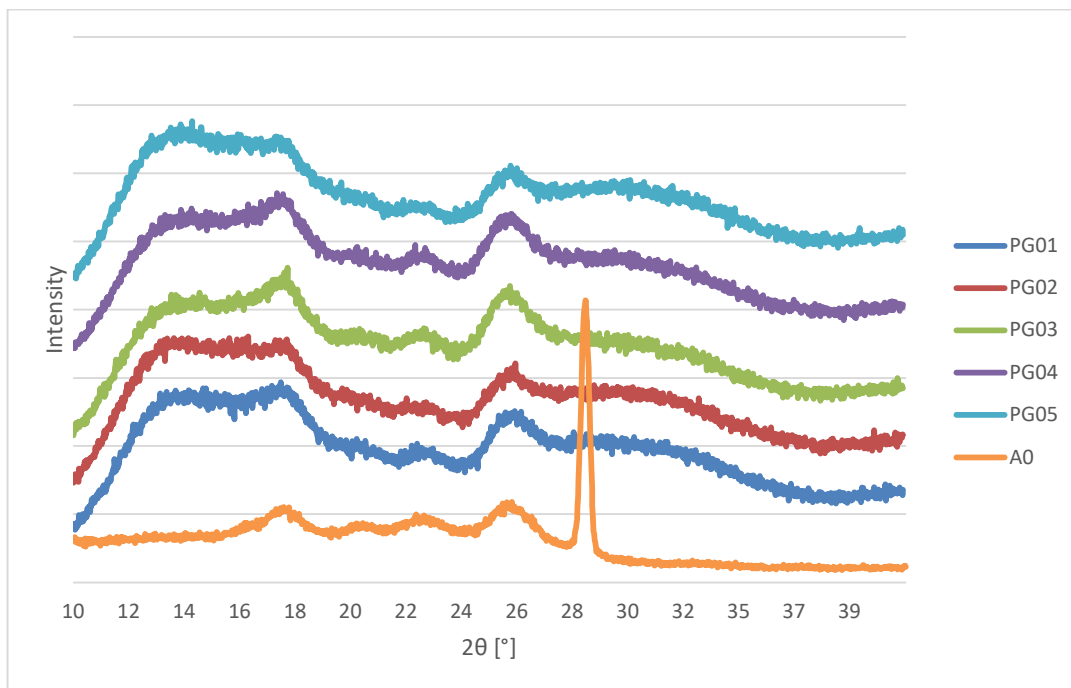


Figure 4.11: The stacked XRD graph for Graphene Cellulose/PVDF coated PVDF hollow fiber membrane (PG01-PG05) and Control sample (A0)

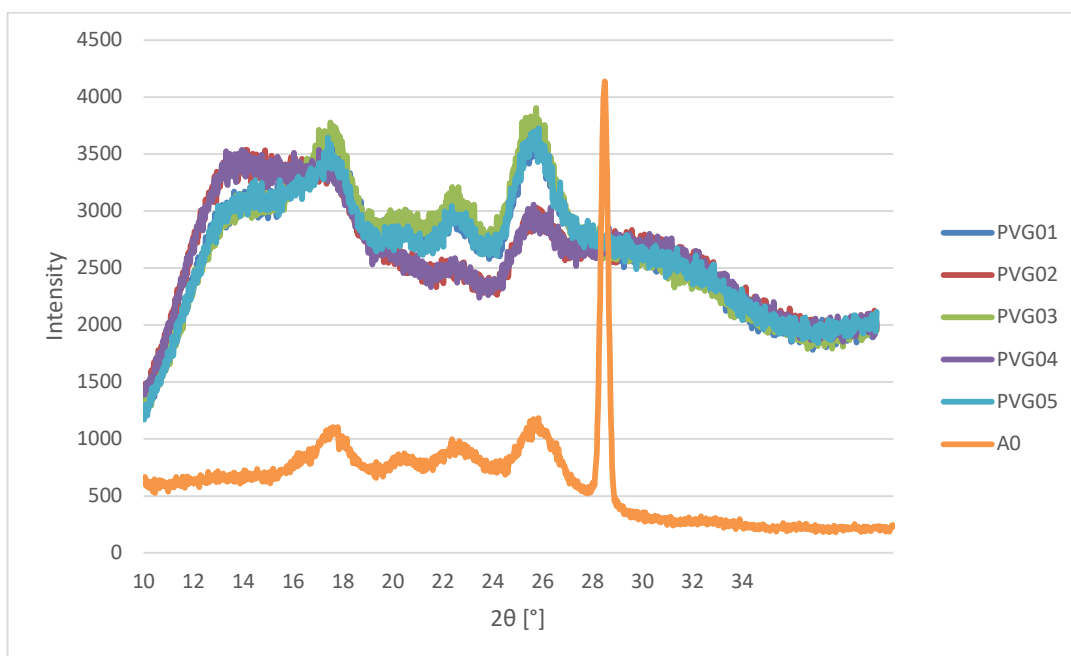


Figure 4.12: The combine XRD graph for Graphene Cellulose/PVA coated PVDF hollow fiber membrane (PVG01-PVG05) and Control sample (A0)

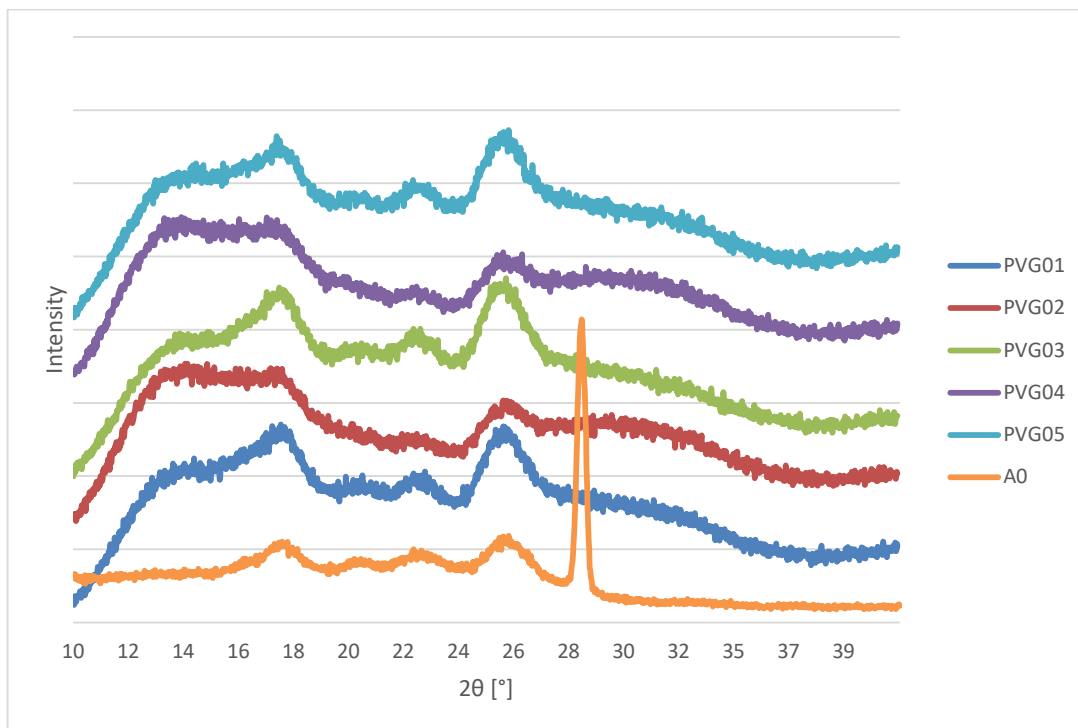


Figure 4.13: The stacked XRD graph for Graphene Cellulose/PVA coated PVDF hollow fiber membrane (PVG01-PVG05) and Control sample (A0)

The XRD analysis is to identify the range of phases possibly between amorphous and crystallinity of membrane and additional evidence of the presence of graphene oxide, cellulose, PVA or PVDF powder incorporation as a coating. This will showcase evidence related to pure water flux performance, as amorphous regions for water filter membranes may be related to improved pure water flux performance, hence showing hydrophilic characteristics. The crystalline regions may be related to lower pure water flux performance hence showing hydrophobic characteristics (Yuan et al. 2020; Acarer-Arat et al. 2024).

The pristine PVDF hollow fiber membrane control sample shown in Figure 4.10, 4.11, 4.12 and 4.13 has peaks and intensity indicative of a neat PVDF membrane with a semi crystalline region which is similarly shown in other XRD analysis studies regarding neat

PVDF membranes, the same as in 1st and 2nd experiment (Acarer-Arat et al. 2024; Yuan et al. 2020; B. Ma et al. 2017).

It could be observed in Figure 4.10 and Figure 4.11 for graphene oxide Cellulose/PVDF samples, shows an increasing broad trend from 13°-16° followed by a small peak at 17° and a bigger peak at 25°-26°. indicating amorphous regions likely to be from the cellulose present in the coating. These observable boarder trends could indicate amorphous regions on the samples (Karim et al. 2020; Mohamat et al. 2023). Some studies utilizing graphene oxide and PVDF for membrane development has shown peaks at 18°, 20° and 26.5° to showcase the evidence of graphene oxide and PVDF present (Sun et al. 2022; Kumar et al. 2023). The presence of cellulose is quite similar to the 1st experiment XRD results where the peaks are noticeably broader with moderate peaks, which shows evidence of the coating on the membrane has modified showcases an amorphous phase (Acarer-Arat et al. 2024; Yuan et al. 2020; Ma et al. 2017; Kumar et al. 2023).

The same could be observed in Figure 4.12 and Figure 4.13 for graphene oxide Cellulose/PVA samples. In comparison to the 2nd experiment, the addition of graphene oxide to the Cellulose/PVA samples has shown an increasing trend from 10° to 13° which may indicate the presence of the graphene oxide(El MIRI et al. 2015; Sun et al. 2022). The broad trend and peaks at 17°, 23° and 26° do matches similarly to the 2nd experiment of only Cellulose/PVA hence indicate the presence of cellulose on the surface of the sample (Kumar et al. 2023).

To clarify the identification of graphene oxide incorporation, the graphene oxide related diffraction features could be highlighted around the 10° to 13° region, which is more prominent at Figure 4.10 for the combined graph, when compare to the previous 1st experiment XRD results for cellulose/PVDF coating. For cellulose/PVA at Figure 4.13 for

the combined graph these features are masked and overlapped due to the contribution of cellulose as well as PVA as binders. Studies shows that this region is generally associated with increased interlayer spacings of graphene oxide sheets due to oxygen containing functional groups and adsorbed water content (El Miri et al., 2015; Sun et al., 2022).

4.3.4 Water Flux Analysis for Graphene Oxide Cellulose/PVDF and Cellulose/PVA Coated PVDF Hollow Fiber Membrane

Table 4.12: Pure Water Flux Analysis for Graphene Oxide Cellulose/PVDF (PG01-PG05) and Graphene oxide Cellulose/PVA (PVG01-PVG05) coated PVDF hollow fiber membrane and contral sample (A0)

Sample (0.25g PVDF Powder), (0.105g Graphene oxide)		Pure water flux (L/m ² ·h·bar)
A0	PVDF (control)	157.89 ± 1.57
PG01	cellulose 0.25g	207.67 ± 1.21
PG02	cellulose 0.5g	81.36 ± 1.31
PG03	cellulose 0.625g	72.70 ± 1.38
PG04	cellulose 0.725g	32.47 ± 0.99
PG05	cellulose 1.0g	43.17 ± 1.03
PVG01	cellulose 0.25g	215.57 ± 1.73
PVG02	cellulose 0.5g	186.28 ± 1.76
PVG03	cellulose 0.625g	111.28 ± 1.66
PVG04	cellulose 0.725g	99.70 ± 1.39
PVG05	cellulose 1.0g	52.84 ± 1.11

Table 4.12 shows the pure water flux performance of both graphene oxide Cellulose/PVDF and graphene oxide Cellulose/PVA, which seem to have a similar trend. The pure water flux for graphene oxide Cellulose/PVDF samples increased in performance for only PG01 at 207.67±1.21 L/m²·h·bar, which is the smallest iteration of cellulose content,

which follows by a sharp decline in pure water flux performance for the rest of the samples. This could be caused by the thick layered coating evidently seen in the SEM imagery from the combination of graphene oxide and the increased cellulose content. The swelling of cellulose and graphene oxide nano size structure maybe contributed to the decreasing pure water permeability by blocking the channels and pores of the membrane sample. In comparison to the 1st experiment for Cellulose/PVDF samples without graphene oxide, the pure water flux results also showed a decrease in performance with increased cellulose content, however it still maintained an overall higher pure water flux compared to the control (An et al. 2017; Majumder and Gangopadhyay 2022; Perreault, Fonseca de Faria, and Elimelech 2015; Sahu et al. 2023).

The pure water flux for graphene oxide Cellulose/PVA samples increased in performance for PVG01 at $215.57 \pm 1.73 \text{ L/m}^2 \cdot \text{h} \cdot \text{bar}$ and PVG02 at $186.28 \pm 1.76 \text{ L/m}^2 \cdot \text{h} \cdot \text{bar}$, which follows by a sharp decline in pure water flux performance. It seems that PVA has some hydrophilic properties which enables water permeability. If compared to the 2nd experiment for Cellulose/PVA samples without graphene oxide, the pure water flux increased multiple folds from the control and outperform the 1st experiment for Cellulose/PVDF (An et al. 2017; Majumder and Gangopadhyay 2022; Perreault, Fonseca de Faria, and Elimelech 2015; Sahu et al. 2023).

The graphene oxide nanosized particles, even at a 0.105g weightage, has tremendous effect on the coating solution development and coating of the neat PVDF hollow fiber membrane. The graphene oxide nanosized particles are clogging the areas for water permeability, in addition to the swelling of the cellulose being in contact with the pure water, further disabling the membranes water permeability (Sahu et al. 2023; Perreault, Fonseca de Faria, and Elimelech 2015).

4.3.5 Porosity and Mean Pore Size Analysis for Graphene Oxide Cellulose/PVDF and Cellulose/PVA Coated PVDF Hollow Fiber Membrane via gravimetric method

Table 4.13: Porosity and Mean Pore Size Data for Graphene Oxide Cellulose/PVDF and Cellulose/PVA Coated PVDF Hollow Fiber Membrane via Gravimetric Method, A0 (Control), PG01-PG05 (Graphene Oxide Cellulose/PVDF), PVG01-PVG05 (Graphene Oxide Cellulose/PVA)

Sample Name	Porosity %	Mean Pore Size (μm)
A0	69.73 ± 0.91	2.0208 ± 0.0098
PG01	65.04 ± 1.00	2.8883 ± 0.0188
PG02	65.48 ± 0.77	1.7989 ± 0.0172
PG03	65.04 ± 0.98	1.7093 ± 0.0189
PG04	67.65 ± 0.86	1.1065 ± 0.0171
PG05	63.93 ± 0.84	1.3367 ± 0.0179
PVG01	69.73 ± 0.91	2.7755 ± 0.0153
PVG02	64.61 ± 0.81	2.7505 ± 0.0148
PVG03	70.21 ± 0.88	1.9886 ± 0.0138
PVG04	66.50 ± 0.92	1.9657 ± 0.0153
PVG05	67.46 ± 1.05	1.4138 ± 0.0154

Table 4.13 shows the porosity and mean pore size analysis via gravimetric method. These results showcase the data which may support the experimental results shown in the SEM, FTIR, XRD and Pure water flux performance analysis for both graphene oxide Cellulose/PVDF and graphene oxide Cellulose/PVA membrane samples. It could be observed that the porosity for PG01-PG05 and PVG01-PVG05 maintained a 97% porosity. However, the mean pore size steadily decreases as the cellulose content increases. This could explain the reduction in water permeability in the pure water flux performance, as the high porosity combined with the smaller mean pore size may block available passages by the

swelling of the membrane when in contact with water. In comparison to the 1st and 2nd experiment without graphene oxide, even though porosity increased to around 70-79%, their mean pore size is much bigger in comparison. With the data showing a decrease in mean pore size, it could mean that graphene oxide is latching onto the cellulose. The higher the cellulose content, the more graphene oxide can latch onto and distribute along the membrane coating (An et al. 2017; Majumder and Gangopadhyay 2022; Perreault, Fonseca de Faria, and Elimelech 2015; Sahu et al. 2023).

Table 4.14: Summary table showcasing observable trends for Graphene Oxide in both Cellulose/PVA and Cellulose/PVDF samples and the control A0 for Pure water flux, Porosity and Mean Pore Size

Sample	Pure water flux (L/m ² ·h·bar)	Porosity (%)	Mean pore size (μm)	Observable pure water flux trends
A0	157.89 ± 1.57	69.73 ± 0.91	2.0208 ± 0.0098	Baseline Control
PG01	207.67 ± 1.21	65.04 ± 1.00	2.8883 ± 0.0188	Improved (Highest flux for Cellulose/PVDF + Graphene Oxide)
PG02	81.36 ± 1.31	65.48 ± 0.77	1.7989 ± 0.0172	Decreased Flux
PG03	72.70 ± 1.38	65.04 ± 0.98	1.7093 ± 0.0189	Lower flux
PG04	32.47 ± 0.99	67.65 ± 0.86	1.1065 ± 0.0171	Lowest flux
PG05	43.17 ± 1.03	63.93 ± 0.84	1.3367 ± 0.0179	Slight recovery
PVG01	215.57 ± 1.73	69.73 ± 0.91	2.7755 ± 0.0153	Improved (Highest flux for Cellulose/PVA + Graphene Oxide)
PVG02	186.28 ± 1.76	64.61 ± 0.81	2.7505 ± 0.0148	Flux decreased
PVG03	111.28 ± 1.66	70.21 ± 0.88	1.9886 ± 0.0138	Lower flux

Table 4.14 continued

PVG04	99.70 ± 1.39	66.50 ± 0.92	1.9657 ± 0.0153	Lowest flux
PVG05	52.84 ± 1.11	67.46 ± 1.05	1.4138 ± 0.0154	Slight recovery

The Table 4.14 is the summary of the relationship between pure water flux, porosity and mean pore size for the control A0, PG01-PG05 and PVG01-PVG05 samples. The results demonstrates that at the lowest cellulose weightage at PG01 and PVG01, improved pure water flux and achieved the highest for the graphene oxide containing formulation in comparison to the control A0. As some coated samples showed comparable or higher porosity, the reduction in mean pore size when cellulose weightage is increased relates to the lower water flux performance. This suggests that water flux was not controlled by porosity alone, but also by pore accessibility, water transport channels and possible aggregation from graphene oxide and cellulose loading formulation.

4.3.6 3rd Experiment Conclusion

The phase inversion coating as a surface modification technique using graphene oxide Cellulose/PVDF and graphene oxide Cellulose/PVA on neat PVDF hollow fiber membrane is successful, where a thick layered and cracked surface structure in the SEM imagery was observed for both coating types. The FTIR analysis showcases evidence of availability of graphene oxide and Cellulose/PVDF and Cellulose/PVA on the surface of the PVDF hollow fiber membrane, from the C–OH stretching, O–H deformation and C–O–C stretching regions present. The XRD analysis showcases evidence of hydrophilic behaviours via peaks which illustrates an amorphous phase for the presence of cellulose. The graphene oxide availability seems to show at the 10-13° increasingly rising trend followed by a flat and broad stretch trend. In comparison to the control, the coated graphene oxide

Cellulose/PVDF and graphene oxide Cellulose/PVA samples have improved their pure water flux performance for PG01, PVG01 and PVG02, followed by a steep decline in performance with increased cellulose content. In comparison to 1st and 2nd experiment, the 3rd experiment with the additional graphene oxide shown the least amount of improvement in pure water flux. These results are supported by the porosity and mean pore size analysis, where the high porosity of 97% and reducing mean pore size explain why the pure water flux decreased in performance. Passages, channels with large enough mean pore size allows water permeability, which in return improves pure water flux performance.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusions

In conclusion this research successfully developed bamboo cellulose/polymer coated PVDF hollow fiber membranes using PVA and PVDF as polymer binders, with and without graphene oxide as additive. Addressing the first objective, bamboo cellulose/polymer coating formulations were successfully developed with PVDF and PVA polymers as binders and with and without incorporation of graphene oxide as additives. The 1st experiment involved with cellulose/PVDF coating, 2nd experiment involved cellulose/PVA coating, and the 3rd experiment involved formulation of graphene oxide as additive added into cellulose/PVDF and cellulose/PVA coatings. The successful deposition of the coatings was supported by SEM imagery observations, which showed clear surface morphological changes after coating, while FTIR and XRD results further confirms evidence of chemical and structural changes on the surface of coated PVDF hollow fiber membrane.

To address the second objective, based on characterization tests conducted, the effects of the coating formulations on surface chemistry, morphology, structural characteristics, porosity, and pure water flux were successfully characterized. SEM analysis showed coating presences influence surface morphology such as coating development and distribution, voids and cracks with possible coating defects. The FTIR analysis further supports SEM analysis with confirmed presence of cellulose related functional groups, particularly O–H and C–O bands, while the addition of graphene oxide as additives showed additional oxygen containing functional groups. The XRD analysis demonstrates changes in peak intensity and broadness related to the amorphous phase, suggesting that the coatings

modified the surface characteristics of the pristine PVDF membrane improved hydrophilicity, hence supports the subsequent pore related and water flux results. The porosity and mean pore size analysis showed that coating composition influencing porosity and mean pore size, while pure water flux results demonstrated that coating formulation had a significant effect on membrane permeability.

Lastly for the third objective, the optimal performing formulation was determined to be from the second experiment, with cellulose/PVA coating without graphene oxide. At the lowest cellulose weightage for cellulose/PVA coating, PV01 showed the highest pure water flux performance at $860.79 \pm 4.14 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$, followed by PV02 at $741.02 \pm 3.32 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$. PV01 also demonstrated improved water flux performance for rainwater, river water and peat water. Showcasing the filtration performance regarding water flux output. The AAS analysis showed that filtered rainwater had no detectable iron content and the filtered river water iron was completely removed. For filtered peat water, the iron removal improved from 23.31% (control) to the maximum filtration percentage of 46.23% for PV05. The results show that the optimized cellulose/PVA coated samples improved both water flux performance and iron removal potential in comparison the control.

Overall, this research demonstrated that bamboo derived cellulose/PVA coating has the highest potential for developing improved water flux performance of pristine PVDF hollow fiber membrane. The use of PVA as a hydrophilic polymer binder produced better coating performance in comparison to PVDF binder, while the addition of graphene oxide hinders performances due to coating formulation density (thickens), aggregation and reduced water transport channels from pore development.

5.2 Recommendations

From the collection of results from 1st, 2nd and 3rd experiments, the optimal sample was found to be from the 2nd experiment with Cellulose/PVA as coating, without graphene oxide added. The future recommendations may regard in further studies include:

- Deeper investigation regarding the optimal sample formulation, utilizing specialized statistical software for Design of Experiments (DOE). This enables the comparison between theoretical and experimental results for further optimization and streamline the research and development process towards real world applications.
- Comparing different coating methods (e.g. spraying, electrospinning) other than phase inversion technique to deposit cellulose/PVA (optimal formulation) onto the surface of pristine PVDF hollow fiber membrane.
- Determine possible chemical modification techniques as pretreatments which could improve coating deposition onto the surface of pristine PVDF hollow fiber membrane.
- Determine the extent of fouling occur on the coated membrane and investigate antifouling properties of coated membranes for long term application using real water sources (e.g. rain, river and peat water).

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APPENDICES

Appendix A: Journal Publications

1. **Nyuk Khui, Perry Law.,** Md Rezaur Rahman., Mohamad Khairul Anwar Bin Said Al-Khalid Othman., Jamal Uddin., & Kuok King Kuok. (2024). Nanocarbon from pine wood sawdust and its biocomposites applications. *Woodhead Publishing in Materials*, 17-47. <https://doi.org/10.1016/b978-0-443-13981-9.00002-8>.
2. **Nyuk Khui, Perry Law.,** Md Rezaur Rahman., Khairul Anwar Bin Mohamad Said., Jehan Y. Al-Humaidi., Muhammad Khusairy Bin Bakri., King Kuok Kuok., Raed H. Althomali., Muhammad Rafiq Mirza Bin Julaihi., & Mohammed M. Rahman. (2024). Enhancing Water Treatment with Polyvinylidene Fluoride (PVDF)/Cellulose Composite Membranes: A Comprehensive Review. *Polymer-Plastics Technology and Materials*, 64(2), 119–37. <https://doi.org/10.1080/25740881.2024.2391349>.
3. **Nyuk Khui, Perry Law.,** Md Rezaur Rahman., Khairul Anwar Bin Mohamad Said., Murtala Namakka., M. Shahabuddin., Muneera S. M. Al-Saleem., Jehan Y. Al-Humaidi., Mohammed M. Rahman., & Kuok King Kuok. (2025). Influence of bamboo cellulose coating on the flux performance of polyvinylidene fluoride hollow fiber membrane. *BioResources*, 20(4), 8737–8754. <https://doi.org/10.15376/biores.20.4.8737-8754>.
4. **Nyuk Khui, Perry Law.,** Md Rezaur Rahman., Mohamad Khairul Anwar bin Said., Murtala Namakka., M. Shahabuddin., Muneera S. M. Al-Saleem., Jehan Y. Al-Humaidi., & M. Rahman Mohammed. (2025). Enhanced flux and surface modification of PVDF hollow fiber membranes using bamboo cellulose/PVA composite coatings. *Polymer Bulletin*, 0170-0839. <https://doi.org/10.1007/s00289-025-06023-8>.

5. **Nyuk Khui, Perry Law., Rahman Md. Rezaur., & Bakri Muhammad Khusairy Bin.** (2025). Polymer nanocomposite membranes for oil removal. *Woodhead Publishing in Materials*, 125-139. <https://doi.org/10.1016/B978-0-443-23879-6.00004-X>.