Advancing Pressure Spinning Polymeric Fibre Manufacture for Enhanced Performance and Sustainability

A thesis submitted for the partial fulfilment of the requirements for transferring to the degree of

Doctor of Philosophy

by

Manul Amarakoon

Department of Mechanical Engineering

University College London

Torrington Place

London WC1E 7JE

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Declaration

I, Manul Amarakoon confirm that the work presented in this thesis is my own. Where information has been derived from other sources, I confirm that this has been indicated in the thesis.

Abstract

This thesis presents a comprehensive investigation into the environmental impact, energy consumption and production efficiency of polymeric submicrometre fibre manufacturing, developing a sustainable approach aligned with the principles of Green Chemistry and Green Engineering. The research explores pressure spinning, a novel method using centrifugal force and applied gas pressure, establishing it as a viable alternative to conventional techniques.

The study systematically optimises process parameters including applied gas pressure and polymer solution concentrations, using water-soluble Polyethylene Oxide (PEO) and Polyvinylpyrrolidone (PVP) to establish empirical relationships for efficient, sustainable manufacture. A major innovation is the successful production and characterisation of core-sheath fibres via pressure spinning, demonstrating the reproducible manufacture of dual-component fibres with high structural integrity for advanced applications like drug delivery and energy storage.

A comparative lifecycle assessment (LCA) focusing on the manufacturing phase demonstrated that pressure spinning can reduce energy consumption by up to several orders of magnitude compared to traditional methods and exhibits a markedly reduced dependency on hazardous solvents. Furthermore, analysis of previously neglected variables, vessel geometry (60 mm and 75 mm) and collector distance (100 mm to 200 mm), found that wider vessels and greater collector

distances enhance fibre uniformity, orientation and production efficiency with no additional energy consumption.

Ultimately, this thesis provides a roadmap for translating laboratory-scale innovation into scalable industrial practice, establishing pressure spinning as a transformative, green technology that mitigates environmental harm and promotes the circular economy.

Impact Statement

This research makes a significant contribution to advancing sustainable polymeric fibre production by optimising the pressure spinning technique, directly addressing global challenges through the principles of Green Chemistry and Green Engineering. The work differentiates itself by investigating previously neglected parameters, collector distance and rotary vessel geometry, demonstrating a pathway to enhanced fibre morphology, production efficiency and reduced energy consumption for industrial scalability. These insights provide a low-environmental-footprint roadmap for developing application-specific fibres for advanced technologies, thereby laying the groundwork for transforming industrial practices, minimising hazardous solvent dependency, and supporting the integration of sustainability into future engineering designs.

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Abbreviations

AFM – Atomic Force Microscopy

BPA – Bisphenol A

C–S – Core–Sheath

CV – Coefficient of Variation

DCM – Dichloromethane

DES – Deep Eutectic Solvents

DMF – Dimethylformamide

ECHA – European Chemicals Agency

EDC – Endocrine-Disrupting Chemicals

EPA – Environmental Protection Agency

FDA – Food and Drug Administration

FTIR – Fourier Transform Infrared Spectroscopy

H_2O – Water

J – Joules

LCA – Life Cycle Assessment

Micro-CT – Micro-Computed Tomography

mm – Millimetre

MPa – Megapascals

MW – Mega Watt

Mw – Molecular Weight

NIPS – Non-solvent Induced Phase Separation

PAH – Polycyclic Aromatic Hydrocarbons

PCL – Polycaprolactone

PEO – Polyethylene Oxide

PCB – Polychlorinated Biphenyls

PLA – Polylactic Acid

PVA – Polyvinyl Alcohol

PVP – Polyvinylpyrrolidone

REACH – Registration, Evaluation, Authorisation and Restriction of Chemicals

RPM – Revolutions per Minute

SEM – Scanning Electron Microscopy

TIPS – Thermally Induced Phase Separation

TSCA – Toxic Substances Control Act

UV – Ultraviolet

W – Watts

wt % – Weight Percent (Mass Fraction Percentage)

µm – Micrometre

Chapter 1 – Introduction

1.1 Background

Polymers are long repeating chains of molecules with distinctive material properties, determined by the type of molecules and their bonding. Polymers are utilised in almost every aspect of modern human life. For instance, in the making of kitchen utensils, wearables, vehicle components and furniture to name a very few. Synthetic polymeric fibres such as nylon and polypropylene possess advantageous physical features. For instance, they improve mechanical properties and provide high specific surface area.[1] Synthetic fibres are man-made fibres primarily derived from petroleum derivatives via chemical modification. There are mainly semi-crystalline polymers that are extruded and drawn in a diverse range of cross-sectional configurations.[2] More recently, polymeric fibres have become crucial constituents of many advanced technologies, finding applications in biomedical scaffolds, drug delivery systems, air and water filtration, energy storage and wearable electronics.[3, 4, 5, 6, 7] The versatility in manufacture and production processes, along with their broad range of material properties, significantly contributes to the increasing use of polymers. It is abstruse to visualise our everyday lives without polymers or plastics.

Plastics use polymers as their main ingredient. However, the application of plastics has a serious drawback with regards to their negative environmental effects. There are many research articles and scholarly forums on the environmental impact from plastics and polymers, which

is the production output.[8, 9] However, the environmental impact from the extraction of materials for polymers and solvents and the manufacturing processes of the final polymeric fibres is less discussed.

Figure 1. *Plastic nurdles on the shores of Sri Lanka's coast after disaster in 2021 (Photo credit: Mark De Silva)*

Plastic mediums commonly require hundreds of years to disintegrate in the natural environment.[10] At present, the world has shown many environmental issues mainly due to landfill and plastic pollution. Plastic pollution from a catastrophic disaster in 2021 resulted in 70-75 billion individual plastic pellets from the sinking X-Press Pearl ship flooding over 300km of Sri Lanka's coast.[11, 12] This resulted in the countless loss of rare endangered marine species, such as rare turtles. These pellets (Figure 1) are similar to those used in polymeric manufacture methods such as electrospinning and pressure spinning, thus it is useful to review and assess the environmental impact of manufacturing from these processes.

There has been a strong focus in the advancement of the use and manufacture of biodegradable polymers due to their ability to easily decompose in the natural environment, which is significantly less harmful to the environment.[13] The manufacture process of biodegradable plastics is similar to regular plastics, apart from the materials utilised. However, the use of biodegradable polymers is not sufficient to curb the environmental impact of polymer usage, as they have some drawbacks. For instance, biodegradable polymers such as polyvinyl alcohol (PVA) and polycaprolactone (PCL) are made from petrochemicals that play a significant role in global warming.[14] Thermoplastics are recyclable unlike thermosets, where polymeric fibres are mainly produced using thermoplastics.[15] The accumulation of plastics, along with other materials, is becoming a serious problem for all countries in the world. These materials occupy significant volume in

landfills and dumps today. Recently, the presence of huge amounts of plastic fragments in the oceans has been observed, where a considerable part of them come from the streets, going through the drains with the rain and then going into the rivers and lakes and then to the oceans.[16] Currently, more than 99% of plastic is made of fossil fuels and around 19% of greenhouse gases are produced from fossil fuel extraction.[17] As of 2019, the entire lifecycle of global plastic production (from cradle to grave) had a climate impact equivalent to that of 189 coal-fired power plants, each with a capacity of 500 MW.[17] By 2050, the impacts are predicted to rise to the effects of 615 coal power plants. As a result, there is a movement around the world for the use of materials that do not harm the planet.

Currently, humanity produces the largest amounts of waste in history, with plastics comprising a significant portion. In 2017, it was estimated that 91% of plastic was not recycled.[18] Plastic production has grown exponentially over the years and it is projected that by 2050, there will be more plastic in the ocean than fish, with microplastics contaminating 80% of drinking water.[19] In 2020, the global production of chemical fibres was estimated to reach 80.9 million metric tons, showing a consistent yearly increase.[20] These fibres, derived from both organic and synthetic polymers, consisted predominantly of synthetic fibres, which accounted for over 90% of total production in 2020.

Biodegradable polymers alone cannot solve these challenges due to their production costs, limited scalability and reliance on petrochemical feedstocks. Therefore, sustainable manufacturing practices, often

referred to as Green Production, are essential. These practices rely on the frameworks of Green Chemistry (the design of chemical products and processes to reduce hazardous substances) and Green Engineering (the design of systems and processes to minimise waste and environmental harm). These frameworks aim to reduce energy consumption, minimise material wastage and promote the use of non-toxic material.

This thesis addresses these urgent issues by exploring pressure spinning as a sustainable alternative for producing polymeric fibres. By optimising this technique, the research seeks to advance scalable, energy efficient fibre production that balances high performance with minimal environmental impact.

1.2 Focus of the Thesis

The environmental impacts of polymeric fibre production, from raw material extraction to end-of-life disposal, are an increasingly urgent concern. This research investigates the production of nanometre to micrometre-scale polymeric fibres with a focus on the manufacturing stage, where sustainability challenges are mostly unaddressed. It compares several widely used fibre manufacturing methods, such as electrospinning, phase separation and pressure spinning, evaluating their energy consumption, production efficiency and environmental hazards.

Particular emphasis is placed on pressure spinning (or pressurised gyration), a novel method that combines centrifugal force from a rotating vessel with internal applied gas pressure to extrude polymer solutions through fine orifices into fibres. This technique demonstrates significant potential for energy-efficient and sustainable fibre production due to its inherently high production rates, low energy requirements, and operational simplicity. Furthermore, pressure spinning minimises dependency on hazardous organic solvents, performing efficiently with water-soluble polymers, thereby enhancing worker safety and environmental exposure. By integrating these sustainable manufacturing practices, this thesis explores the optimisation of process parameters in pressure spinning to enhance energy efficiency and minimise environmental harm. This will serve as a foundation for developing greener polymeric fibre manufacturing technologies tailored to the needs of various advanced applications.

1.2.1 Research Aim

This aim of this research is to address the gap in existing research by evaluating the environmental impacts of polymeric fibre production and proposing sustainable solutions through Green Chemistry and Green Engineering, with a focus on pressure spinning as a model for developing energy-efficient and environmentally friendly manufacturing techniques.

1.2.2 Research Objectives

- **To review and assess the environmental impacts of submicrometre polymeric fibre manufacturing processes, from material extraction to final production.**

  This thesis begins by evaluating the environmental implications associated with the full lifecycle of submicrometre polymeric fibre manufacturing, including raw material extraction, polymer and solvent processing, fibre formation and end-of-life disposal. A detailed comparative analysis based on literature is conducted on existing fibre production methods such as electrospinning, phase separation, self-assembly and template synthesis. These methods are assessed not only in terms of their operational efficiency but also their energy demands, solvent toxicity, waste generation and overall environmental footprint. In particular, the study highlights the under-addressed impacts of solvent use and high energy consumption in fibre-forming processes, especially during the manufacturing stage. A lifecycle thinking approach is employed to quantify and contextualise these impacts, offering a foundational understanding of where current practices fall short in meeting environmental sustainability benchmarks. This critical review serves as the basis for identifying key opportunities for intervention, innovation and improvement in fibre manufacturing technologies.

- **To investigate the application of Green Chemistry and Green Engineering principles in pressure spinning for producing polymeric fibres with various properties.**

  In response to the environmental challenges identified in conventional nanofibre manufacturing, this research explores the integration of Green Chemistry and Green Engineering principles into the pressure spinning process. These principles are applied across material selection, solvent use, process design and operational conditions. The study evaluates pressure spinning using water-soluble polymers such as PEO and PVP, thereby reducing reliance on hazardous organic solvents. Additionally, the method's ability to form complex fibre architectures, such as core-sheath structures, under relatively low energy input and short processing times aligns with the green engineering goal of designing energy-efficient and scalable processes. The approach emphasises atom economy, reduced toxicity and energy conservation, offering a practical route toward sustainable fibre production. By demonstrating how environmentally conscious design can be harmonised with material performance, the research showcases pressure spinning as a model for sustainable polymer processing in alignment with circular economy objectives.

- **To optimise and understand the effects of process control parameters in pressure spinning for improved production efficiency, fibre quality and reduced environmental impact.**

To fully harness the benefits of pressure spinning, this thesis systematically investigates the effects of key process parameters, including applied gas pressure, vessel geometry, polymer concentration and collector distance, on fibre production outcomes. A series of controlled experiments are conducted to evaluate how these variables influence fibre diameter, uniformity, orientation, production rate and energy consumption. The results demonstrate that precise tuning of these parameters can significantly enhance fibre morphology while reducing energy input and material waste. For instance, increasing collector distance improves fibre uniformity and orientation, whereas optimised applied gas pressure enables finer diameter control without compromising throughput. Moreover, the study introduces the role of rotary vessel geometry as a previously unexplored but critical factor in shaping the efficiency and quality of fibre production. The findings culminate in a comprehensive framework for process optimisation that balances high-performance fibre output with minimal environmental burden, thereby contributing to the industrial scalability and ecological viability of pressure spinning.

The expected contribution of this work is the quantitative data and novel insights derived from these objectives, specifically pioneering the study of geometric parameters, validating a cleaner method for complex core-

sheath fibres and providing a foundational roadmap for the industrial scalability of this transformative, green technology.

1.3 Structure of the Thesis

This thesis is organised into seven chapters, each designed to systematically explore and address the research objectives. The structure ensures a logical progression, starting with the broader context of polymeric fibre production and narrowing down to the experimental work, analysis and future directions. Below is an outline of the content and purpose of each chapter:

Introduction

This chapter sets the stage by discussing the global environmental challenges posed by conventional polymeric fibre production methods. It introduces the concept of pressure spinning as a sustainable alternative, emphasising its alignment with the principles of Green Chemistry and Green Engineering (Section 2.2). The chapter outlines the research aim and objectives, providing a clear framework for the subsequent chapters.

Literature Review

A comprehensive review of existing polymeric fibre manufacturing techniques, including electrospinning, phase separation and pressure

spinning, is presented. The chapter critically evaluates their environmental and operational limitations, highlighting the knowledge gaps. Additionally, the potential applications of polymeric fibres in biomedical, filtration and energy sectors are explored, setting the foundation for this study's focus on sustainable and efficient production.

Methodological Framework

This chapter details the experimental setup, materials and methods used in the study. It explains the selection of Polyethylene Oxide (PEO) and Polyvinylpyrrolidone (PVP) as model polymers and provides a step-by-step description of the pressure spinning process. The methods of analyzing key parameters is outlined to establish a basis for understanding the forming of polymeric fibres. Characterisation methods used to evaluate the properties of the fibres formed in each study is detailed.

Sustainability of Submicrometric PEO and PVP Fibre Production

This chapter presents a detailed experimental investigation into the production of submicrometric fibres using PEO and PVP, both of which are water-soluble, biocompatible polymers frequently used in biomedical and filtration applications. The study systematically explores the impact of polymer concentration and applied gas pressure on production rate, fibre diameter and energy consumption within the pressure spinning process. By evaluating multiple concentrations of PEO and PVP

solutions, this work identifies the optimum viscosities required for stable fibre formation and maximum yield. Applied gas pressure is varied in fine increments to observe its direct influence on fibre diameter reduction and energy efficiency. Results are analysed using Scanning Electron Microscopy (SEM) and image analysis to quantify morphology and uniformity. The chapter highlights trade-offs such as viscosity and energy consumption and establishes optimum operational parameters that minimise environmental impact while maximising process efficiency. The outcomes provide foundational data for energy-efficient, scalable manufacturing of single-component polymer fibres in a green solvent system, setting the stage for more complex fibre structures.

Sustainability of Core-Sheath Fibre Production

Building on the understanding of single-polymer systems, this chapter investigates the feasibility and sustainability of producing core-sheath fibres using a dual-reservoir pressure spinning setup. Core-sheath architectures are particularly valuable for applications requiring multifunctional fibres, such as targeted drug release, wound healing and biosensing, where a functional outer sheath protects or modulates the inner core material. The experimental setup involves pressure spinning with concentric inner and outer vessel compartments designed to separately feed PVP and PEO solutions in different combinations and concentrations (e.g., PVP 50% in core and PEO 40% in sheath). The effects of varying applied gas pressure on the encapsulation efficiency,

structural continuity and energy consumption are systematically studied. Optical and SEM imaging, in conjunction with FTIR spectroscopy, are used to verify the core-sheath integrity and chemical composition. This chapter reveals that modest applied gas pressures yield robust core-sheath morphologies without fibre breakage or sheath collapse, while maintaining production efficiency and reducing material wastage. Furthermore, energy consumption per mass of fibres produced showed a decreasing trend overall with increasing applied gas pressure. The findings demonstrate that complex fibre architectures can be achieved through pressure spinning without additional processing steps or hazardous solvents, reinforcing its role as a sustainable and versatile manufacturing platform.

Exploring the Effects of Vessel Geometry and Collector Distance

This chapter delves into the mechanical and spatial dynamics of the pressure spinning process by evaluating how variations in rotary vessel geometry and collector distance influence fibre morphology, orientation and yield. Two custom-designed vessels with diameters of 60 mm and 75 mm are used to assess the influence of centrifugal force and fluid distribution across the vessel wall. Simultaneously, collector distances are varied from 100 mm to 200 mm to examine the effects of trajectory length and air resistance on fibre formation. The study finds that larger vessel diameters generate greater centrifugal force at the same angular speed, resulting in thinner and more uniformly distributed fibres,

particularly at higher polymer concentrations. Increased collector distances lead to improved fibre spreading and alignment due to extended flight time, reducing clumping and enhancing uniformity. SEM images and fibre diameter distribution graphs reveal lower coefficient of variation (CV) values in setups using larger vessels and longer collector distances. The chapter also explores the physical rationale behind these observations, referencing angular momentum and fluid jet dynamics to explain trends. From a sustainability perspective, these modifications enable more efficient fibre formation with fewer defects, reduced post-processing requirements and higher material utilisation. The insights gained offer actionable guidance for scaling up pressure spinning systems and standardising geometrical configurations for industrial applications.

Limitations and Future Work

The final chapter of this thesis provides a comprehensive reflection on the limitations of the research and outlines a detailed roadmap for advancing sustainable polymeric fibre production via pressure spinning. It begins by acknowledging practical and methodological constraints encountered during the investigation, such as limited material scope, reliance on manual process monitoring and the absence of real-time analytics, while also identifying areas where process assumptions and characterisation methods could be refined. These limitations are

contextualised in relation to each core experimental chapter, providing a foundation for targeted improvements.

Building on these reflections, the chapter proposes several future research directions that span materials, process engineering, environmental assessment and application-specific design. Key avenues include the incorporation of naturally derived and biodegradable polymers, broader evaluation of processing parameters (e.g. viscometer energy requirements) and the adoption of advanced characterisation tools such as atomic force microscopy (AFM), micro-CT and confocal microscopy. The chapter also introduces novel opportunities such as developing further hybrid fibre architectures, integrating real-time sensor monitoring systems and applying machine learning for predictive control.

Crucially, the chapter emphasises the importance of full life cycle assessment (LCA) to ensure that sustainability gains at the manufacturing stage are not offset by upstream or downstream impacts. It calls for a holistic systems approach to evaluating polymer sources, solvent use, energy consumption and end-of-life outcomes. In parallel, it advocates for industrial-scale implementation through automation, sensor integration and pilot testing in partnership with manufacturers.

By addressing these challenges through interdisciplinary research and practical scale-up, the insights presented in this thesis can support the transformation of pressure spinning into a commercially viable,

environmentally responsible method for producing next-generation polymeric fibres.

1.4 The Contemporary Research Environment

Polymeric fibre production has become an essential industry, supporting applications ranging from biomedical scaffolds and filtration systems to energy storage and wearable electronics.[21, 22, 23, 24] Traditional manufacturing methods, such as electrospinning, phase separation and melt spinning, have been widely adopted due to their ability to produce high-performance fibres with controlled properties. However, these techniques often come with significant drawbacks, including high energy consumption, reliance on toxic solvents and limited scalability.[25]

Electrospinning (described in Section 2.2.1), for example, is a popular method for producing submicrometric fibres which was first patented in the year 1900.[26] Since 1995, the number of publications about electrospinning has been increasing exponentially every year. This method requires high voltages and often hazardous organic solvents, posing risks to both human health and the environment.[27] Phase separation and self-assembly, while effective for certain specialised applications, involve lengthy and energy-intensive processes that limit their industrial viability.[25] Moreover, many of these methods rely on non-renewable resources, exacerbating the environmental impact of polymeric fibre production.

Despite growing interest in greener alternatives, there has been almost no investigation into the energy consumption associated with producing polymeric fibres in the nano to microscale. Understanding energy efficiency in these processes is crucial for advancing sustainable manufacturing practices. Additionally, in pressure spinning, which is a promising alternative that is scalable and eco-friendly, current research has predominantly focused on optimising rotary speed and gas pressure, with comparatively less attention given to other critical parameters such as the collector distance.[28] The influence of collector distance on fibre morphology, production efficiency and energy consumption remains underexplored, even though it may significantly impact the process.

Furthermore, in pressure spinning, the geometry of the rotary vessel, which could potentially affect fibre formation, energy efficiency and overall scalability, has not been sufficiently studied. Vessel design may hold the key to further optimising the process and achieving industrial-scale production.

Sustainable approaches, such as pressure spinning, offer promising solutions by reducing energy usage and minimising post processing requirements such as freeze drying. Pressure spinning has emerged as a scalable and eco-friendly technique capable of producing high-quality fibres rapidly and efficiently. However, challenges remain in optimising the process to achieve consistent fibre quality, reducing operational costs and scaling up for industrial production.

The integration of Green Chemistry and Green Engineering principles into fibre production processes is now a critical focus area for researchers and industries alike.[29] These principles emphasise the use of safer solvents, renewable feedstocks and energy-efficient processes, aligning with global efforts to reduce carbon footprints and promote sustainability.

1.5 Pressure Spinning

Originally developed as an extension of centrifugal spinning, pressure spinning emerged in 2013 from the need to provide additional control over the fiber production process.[30] This high-throughput method is the core technology of this thesis as it offers compelling advantages aligned with Green Engineering. It features high production rates, low energy consumption, and operational simplicity. Pressure Spinning is compatible with benign solvents like water and avoids the high-voltage systems used in electrospinning.

In its simplest form, the model used in this thesis, pre-processed polymer solutions are manually loaded into a cylindrical vessel using a syringe. The vessel is then sealed and mounted on a high-speed motor. Once spinning begins, the vessel is rotated at speeds typically exceeding several thousand revolutions per minute (RPM), generating strong centrifugal forces. Simultaneously, compressed gas (usually nitrogen or air) may be introduced into the sealed vessel to increase the

internal pressure, although this is not always necessary depending on solution properties.

The combined action of centrifugal force and internal applied gas pressure compels the polymer solution to extrude through uniformly distributed orifices along the vessel wall in the form of thin liquid jets. As these jets travel outward through ambient air, solvent evaporation occurs rapidly, solidifying the extruded solution into fine polymer fibres. These fibres are then collected on a stationary external collector, typically a frame or wall, which is strategically positioned at a selected distance from the vessel. This collector distance influences fibre alignment, spread and morphology due to the aerodynamic and gravitational forces acting on the fibres during flight. The simplicity of this setup, combined with the ability to produce large quantities of fibres in a short duration without complex auxiliary systems, makes pressure spinning a compelling alternative to conventional fibre manufacturing techniques.

Studies have demonstrated that the superimposed gas pressure not only enhances fibre ejection but also contributes to finer control over jet elongation and solvent evaporation, yielding fibres with narrower diameter distributions and greater structural integrity.[31] Furthermore, recent advances have extended pressure spinning to the fabrication of complex fibre morphologies, including core-sheath architectures, multilayered structures and nanoparticle-embedded fibres without requiring additional post-processing steps.[32]

Several variants of pressure spinning have since been developed to broaden its applicability. Pressurised melt gyration, for instance, replaces polymer solutions with molten polymers, thereby enabling the fibre production of polymers that are not soluble in conventional solvents and can only be processed via melting.[32] While this allows for solvent-free fibre formation, it also requires continuous thermal input to maintain polymers in a molten state, significantly increasing energy consumption. Additionally, the elevated temperatures involved may risk thermal degradation of temperature-sensitive polymers and the method demands more complex temperature control systems. Another variant, infusion pressurised gyration, integrates a syringe-pump-driven infusion mechanism to deliver polymer solution into the vessel at a controlled rate.[32] While this approach can improve flow stability and feed control, it introduces additional mechanical complexity and energy demands, particularly due to the need for continuous infusion and its synchronisation with spinning and applied gas pressure parameters. Considering these trade-offs, this study focuses exclusively on the original solvent-based pressure spinning model, which combines centrifugal force and applied gas pressure to process water-soluble polymers under ambient temperature conditions. This configuration not only maintains system simplicity but also significantly reduces energy usage, making it more compatible with the principles of Green Chemistry and Green Engineering. Moreover, by avoiding thermal processing or active infusion, it facilitates a clearer and more isolated investigation of process variables such as vessel geometry and collector distance,

parameters that have been largely overlooked in prior studies and are central to the sustainability and scalability objectives of this research.

Chapter 2 – Literature Review

The increasing environmental challenges posed by polymeric submicrometre fibre production and the use of solvents necessitate a reevaluation of traditional manufacturing methods. Synthetic polymers, derived largely from petroleum, have become indispensable in modern society due to their versatility and durability.[33] However, their production, coupled with the extensive use of hazardous solvents, poses significant threats to both human health and ecosystems. This chapter explores the lifecycle of polymeric fibres, focusing on the production of synthetic polymers and solvents and their associated environmental impacts.

The analysis begins by examining the effects on the environment due to the production of polymer-solvent solutions and the manufacture of polymeric fibres of thicknesses from a nanometre up to a millimetre using these solutions. Next, the role of solvents in polymeric fibre manufacturing is critically analysed. Solvents, integral to methods such as electrospinning and pressure spinning, often come with high toxicity and volatility, posing both immediate and long-term health risks. The environmental ramifications, including soil and water contamination, further underscore the need for safer alternatives.

A detailed comparison of polymeric fibre manufacturing techniques is then presented, with a focus on their energy consumption and production efficiency. Established methods such as electrospinning, phase separation and template synthesis are evaluated alongside

emerging green alternatives like pressure spinning. These comparisons provide a basis for identifying more sustainable and energy-efficient methods for fibre production.

This chapter also underscores the relevance of Green Chemistry and Green Engineering principles as frameworks for addressing sustainability challenges. These principles provide a framework for developing sustainable fibre manufacturing technologies.

Finally, the importance of fibre morphology and performance is discussed, highlighting how smaller diameter fibres (or finer fibres) with uniform diameters enhance efficiency and functionality in applications such as biomedical scaffolds, filtration systems and energy storage devices. In addition, core-sheath fibres, a specialised fibre structure with unique properties such as controlled drug release and enhanced mechanical strength, are examined for their potential in advanced applications and sustainable manufacturing.

2.1 Production of Synthetic Polymers and Solvents

2.1.1 Synthetic Polymers

Synthetic polymers are manufactured via chemical reactions called polymerisation, which occur in many forms where they consist of recurring chemical bindings of individual molecules (monomers).[34] Variations in parameters such as temperature and pressure result in different chemical bonds, which holds monomers together creating polymers. Combinations of monomers are used to create polymers that

show the characteristics of each component and addition polymerisation and condensation polymerisation are the two main methods of polymerisation.

In addition polymerisation, basic hydrocarbons such as ethylene and propylene are converted into polymers by adding one monomer to another in a sequence that continues to elongate.[34] This is due to the free radicals created as monomers bonding to the chain, which allows for yet another monomer to join resulting in a recurring procedure, which produces thousands of monomers that are bonded jointly. Ethylene and propylene are sort after due to its historical significance in the making of polymers.[35] They are both derived from petroleum and are often used in the textile industry and to make plastic bottles, from the resulting polymers, polypropylene and polyethylene, respectively.

Both the polymers mentioned above are called additions as every part of ethylene and propylene are shown in the final polymers, polyethylene and polypropylene. A chemical reaction that only utilises just a particular component of a monomer is known as a condensation polymer. This can only take place if the monomer under consideration possesses two or more reactive groups to result in the production of a chain.[36] In this procedure, the hydrogen in one monomer attaches to the oxygen of another monomer to produce water, which is considered a by-product (condensation). An example of this polymer is Nylon.

Apart from potential hazardous reactions, the polymerisation process itself does not significantly affect the environment directly in a negative

way, in comparison to the extraction of monomers or the disposal of non-biodegradable polymers.[37] Water is the only emission in condensation polymerisation. However, it can be hazardous in the instance where the reactions are out of control, causing fire or even explosions. Some materials are also known to strongly react with water to produce gases, such as cyanide, which are lethal at low airborne concentrations. Synthetic polymers mainly obtained from petroleum is a major environmental concern.[38] The drilling of petroleum can cause major disruptions to wildlands and habitats, along with potential pollution (such as leakage of toxic substances) from active wells and processing plants. Many polymers consist of various other chemical substances such as stabilisers and flame-retardants to enhance the polymer life and properties.[39] These additives may be released during disposal to contaminate soil, water, air and food.

Moreover, the environmental impact of synthetic polymers extends beyond initial production and use, particularly due to their persistence and tendency to fragment into microplastics.[40] These small plastic particles, often originating from the degradation of larger polymeric products through processes such as UV exposure, mechanical abrasion and oxidation, are increasingly found across terrestrial, marine and freshwater environments.[41] They pose serious threats to wildlife through ingestion, entanglement and disruption of natural behaviours. More concerningly, microplastics are now widely recognised as a pathway for indirect human exposure. These particles have been detected in drinking water, sea salt, seafood, fruits, vegetables and even

in the air, raising the possibility of chronic human intake through multiple routes including ingestion, inhalation and dermal contact.[42]

Emerging evidence suggests that microplastics may pose a risk to human health, not only due to their physical presence but also because of the chemicals they carry. Due to their hydrophobic surfaces and high surface-area-to-volume ratios, microplastics can adsorb persistent organic pollutants such as polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs) and heavy metals from the environment.[43] When ingested, these contaminants may desorb within the human gastrointestinal tract, potentially entering the bloodstream and bioaccumulating in tissues.[44] Laboratory studies have shown that microplastics can trigger inflammatory responses, oxidative stress and cellular damage in mammalian cells.[45] Smaller micro-plastics and nano-plastics (particles less than 1 μm) may penetrate biological barriers such as the gut lining or even cross the blood-brain and placental barriers, though long-term in vivo effects remain under investigation.[46] Additionally, plastic additives such as bisphenol A (BPA) and phthalates, many of which are endocrine-disrupting chemicals (EDCs), can leach from microplastics and interfere with hormonal function, reproductive development and metabolic processes.[47]

In addition to concerns over microplastics, the extraction and refining of petrochemical feedstocks required for monomer synthesis are associated with significant greenhouse gas emissions, water usage and the generation of hazardous waste.[48] These processes negatively

affect local air and water quality, disrupt ecosystems and contribute to long-term climate change. The energy-intensive nature of converting crude oil into usable monomers such as ethylene, propylene and styrene further compounds the carbon footprint of synthetic polymer production.[49]

Beyond stabilisers and flame retardants, modern synthetic polymers often contain additional additives such as plasticisers, pigments, UV blockers and antimicrobial agents, many of which have known or suspected toxicological profiles.[50] These substances can leach out during polymer use, degradation, or incineration, particularly under mechanical or environmental stressors such as heat and sunlight. When released, they have the potential to contaminate soil, water and air, entering food webs and potentially exposing humans and animals to chronic low-dose chemical mixtures. While the long-term health consequences of continuous exposure to microplastics and associated chemicals remain a subject of active research, their widespread presence in the environment underscores the urgent need for more sustainable polymer production pathways, improved regulatory frameworks and the development of safer material alternatives.

2.1.2 Solvents

Many of the polymeric fibre manufacture methods such as electrospinning and pressure spinning require solvents to form polymeric solutions to produce polymeric fibres.[51] The evaporation of

the solvents from the solutions in these methods results in polymeric fibres. The chemical grouping of solvents is dependent on its structure, where they are classified as hydrocarbons, oxygenated or halogenated solvents.[52] Hydrocarbons such as paint thinner consists of a ‘carbon skeleton’ in the molecules. Oxygenated solvents, such as esters and alcohols, are manufactured via chemical reactions from oil or natural gases. Halogenated solvents consist of halogens such as chlorine. With the exception of solvents, which are fermented alcohols, non-aqueous solvents are produced from fossil fuel sources such as oil and gas.[53] The extraction of these non-renewable sources has environmental concerns similar to drilling petroleum.

In the making of polymeric fibres, solvents are selected for polymeric solutions based on previous experience and literature. However, in recent times, strict safety precautions and regulations need to be considered when handling solvents to assess their volatilities, along with features such as boiling point. Organic solvents are effectively classed into either hydrocarbons, alcohols, ethers, or chlorinated solvents.[54] In general, many solvents are associated with health hazards due to its toxicity to the human body.[55] The lipophilic feature of some solvents such as hexane and toluene deeply enhances the absorption into humans immediately after dermal contact, inhalation or oral exposure.[56] These solvents can badly affect the nervous system along with other organs such as the kidneys and are known to be highly flammable based on their volatility. Metabolism and excretion can occur immediately with the liver and lungs depending on the type of exposure

of the solvent. Depending on the solvent, the unmetabolised substance can be deposited in human tissue to affect the human body on a long-term basis. Most organic solvents are categorised as flammable and when mixed with air some of these solvents are also known to explode. Solvent vapour is denser than air, hence the vapour will descend towards the ground and can move large distances whilst remaining concentrated.[57] When drawn in large amounts, many solvents cause an unexpected loss of consciousness, where halogenated hydrocarbon solvents such as chloroform have been used in medicine such as sedatives. Chloroform is commonly used to dissolve polymers such as polylactic acid (PLA) to produce polymeric nanofibre via manufacturing methods.[58] The necessity of using such highly toxic solvents highlights the fundamental health and environmental hazards that this research seeks to mitigate, by focusing on water-based systems for Pressure Spinning. The primary organ chloroform can affect is the liver in humans and which leads to necrosis from exposure.[59] It also affects the kidneys causing tubular necrosis and swelling of the organs, subsequently leading to the growth of tumours in these organs.[60] These solvents can also lead to long-term health effects such as cancer. Dichloromethane (DCM), which is commonly utilised to produce polymeric fibres, is a potential human carcinogen and can cause liver cancer when ingested.[61] Aromatic hydrocarbons such as toluene is also used in paints, hair dyes and cleansing agents, where the primary target organ is the central nervous system which can cause headaches and cardiac arrhythmia in humans over long-term exposure.[62] All

ethers such as tetrahydrofuran can cause toxicity to blood lymphocytes, carcinogenicity along with toxicity to the central nervous system in humans.[63] Alcoholic solvents such as methanol can also cause permanent blindness or death if accidently ingested. Chronic exposure to solvents in the work environment can also lead to many neuropsychiatric issues. For instance, a large number of painters are known to suffer from alcoholism due to their exposure to alcohol-based solvents in their work environment.[64] Many organic solvents are used in a wide variety of industries, from paint manufacture to engineering, are known or suspected to immensely increase the risk of blindness through the development of cataracts in the eye and also cause the loss of hearing.[65] Furthermore, environmental contamination is also a major risk from the use of toxic solvents, as solvents can readily move great distances, where widespread polluting or poisoning of the soil is not common.

In light of the significant health and environmental risks associated with traditional organic solvents, current research has increasingly focused on identifying safer, more sustainable alternatives. Green solvents including water, supercritical fluids (e.g., supercritical CO_2), ionic liquids and deep eutectic solvents, are being investigated as viable replacements for conventional toxic and volatile organic compounds[66].Among these, water-based systems are the most attractive for polymer fibre manufacture due to their non-toxicity, abundance and compatibility with biocompatible polymers such as PEO and PVP. However, not all polymers are water-soluble, limiting their

applicability. Deep eutectic solvents (DES), composed of biodegradable and non-volatile components such as choline chloride and urea, offer tunable physicochemical properties that may support fibre production while reducing ecological risk.[67] Supercritical CO_2, while requiring high pressure, is non-toxic and leaves no residue, making it an attractive option for solvent-free spinning techniques, although it remains cost-intensive.[68]

Furthermore, global regulatory bodies such as the European Chemicals Agency (ECHA) and the U.S. Environmental Protection Agency (EPA) are increasingly enforcing solvent substitution under frameworks like REACH (Registration, Evaluation, Authorisation and Restriction of Chemicals) and the Toxic Substances Control Act (TSCA).[69] In the UK, Control of Substances Hazardous to Health (COSHH) is a law that requires employers to control substances that are hazardous to health, encompassing both solvents and emerging materials like nanomaterials.[70] These policies are encouraging industry-wide shifts toward greener formulations, especially in medical, food-contact and textile applications. For fibre manufacturing, the adoption of greener solvents not only aligns with environmental goals but may also improve workplace safety, reduce regulatory compliance costs and simplify waste disposal protocols. Pressure spinning, which can be adapted for use with water-soluble polymers and requires minimal solvent use, therefore presents a forward-compatible manufacturing approach that aligns with both current scientific innovation and regulatory trends.

2.2 Sustainable Manufacture

The evolution of sustainable technologies has been tackled in multiple approaches, where utilising the principles of Green Chemistry is an advantageous approach, especially when dealing with polymer-based products.[71] As a developed and highly adaptable field, the polymer industry plays an important role, as polymers are ubiquitous in modern society. However, drawbacks such as the large-scale use of petroleum-based raw materials and vast quantities of reagents that are of environmental threat, along with the build-up of polymeric matter in the environment, bestows engineers and researchers the liability to re-evaluate the manufacture of polymers with regards to the 12 principles of Green Chemistry (Figure 10).[71]

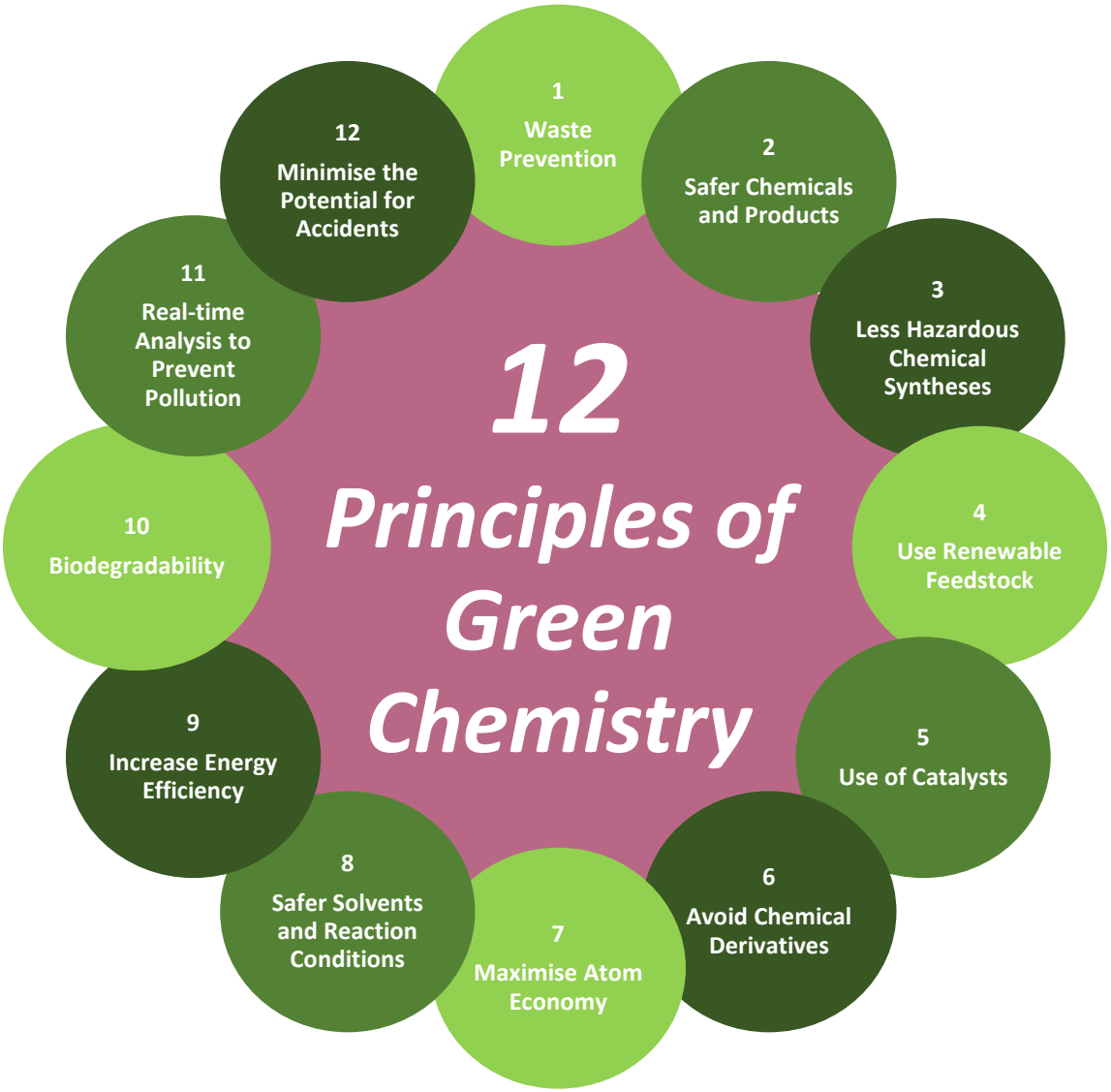

Figure 2. *The 12 principles of Green Chemistry.*

Overall, the 12 principles propose the design of chemical reactions and syntheses to promote safety, minimise waste and optimise energy efficiency. Arguably, principles 2, 3, 4, 8 and 9 can be deduced to have the capability to generate the most significant environmental benefit.[71] Principle 2, 3 and 8 emphasises the use of non-toxic alternatives and reaction conditions such as the use of non-toxic solvents in controlled conditions in the production of polymer products. Principle 4 is an

extensively researched area especially in the making of polymeric fibres, regardless of constraints in the supply of renewable feedstock for polymeric fibre. Principle 9 promotes energy efficiency which is currently an aspect in the production of polymeric fibres that is not as extensively researched. Similar to the 12 Principles of Green Chemistry, the 12 principles of Green Engineering (Figure 11) were determined to allow engineers to integrate features of sustainability in all areas of a project in a systematically comprehensive procedure.[72]

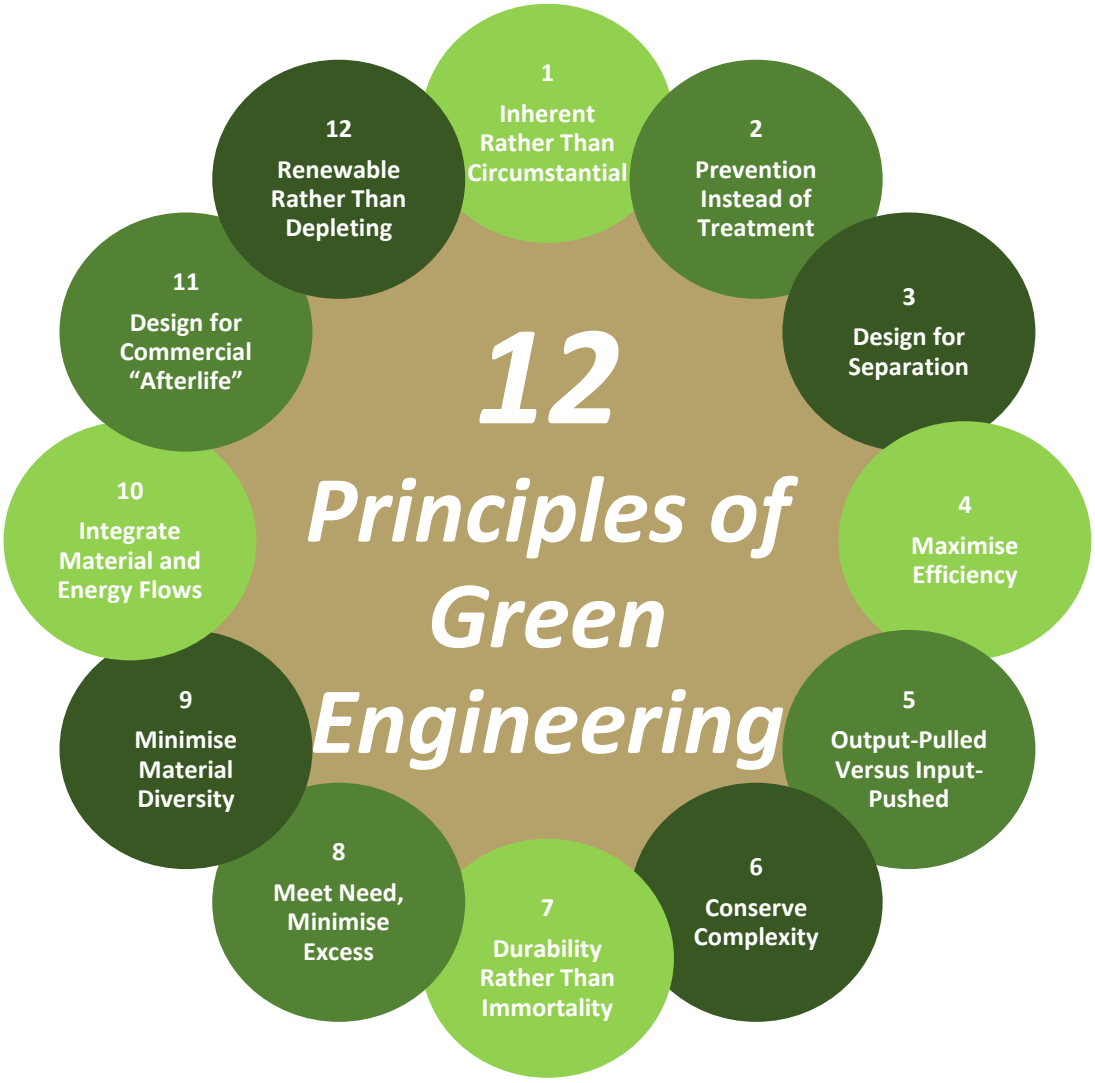

Figure 3. *The 12 principles of Green Engineering.*

Green engineering involves the development, marketing and utilisation of methods and goods with the aim of decreasing pollution, fostering sustainability and mitigating potential harm to human health and the environment, all while maintaining economic feasibility and effectiveness.[72] The main principles of Green Engineering promotes sustainability via design to enhance efficiency, simplicity, material efficiency and renewability. Seemingly, many of the principles of Green Chemistry and Green Engineering are integrated, where energy efficiency, minimisation of wastage and the use of sustainable materials are promoted. The application of the principle of Green Chemistry and Green Engineering in the production of polymeric submicrometre fibres is an appropriate foundation to promote sustainability.

2.3 Polymeric Fibre Manufacturing Techniques

This section compares the energy requirements of some of the most popular manufacturing techniques to produce polymeric fibre of submicrometre external diameter to assess environmental value. The evaluation criteria focus on the manufacture stage in the life cycle of polymeric fibres considering manufacturing processes of polymeric fibre.

Many mechanical and chemical methods of polymeric fibre production currently exist. Electrospinning is the most commonly utilised method for the manufacture of submicrometre polymeric fibres, whilst phase separation, self-assembly and template synthesis have been regularly

utilised to produce polymeric fibres.[51, 73] The environmental damage and health hazards vary according to each method. This section will review some of the significant methods of polymeric submicrometre fibre manufacture.

The various polymeric fibre manufacturing techniques reviewed fall into distinct categories based on their primary driving force, which fundamentally dictates their operational complexity, energy consumption, and reliance on hazardous solvents. Electric Field Driven methods, such as Electrospinning, utilise high-voltage electricity to elongate polymer jets, but often suffer from high-energy requirements and necessitate toxic organic solvents. Thermal/Chemical Driven techniques, including Phase Separation and Self-Assembly, rely on manipulating extreme temperatures or chemical gradients to induce solidification, making them highly energy-intensive due to extensive cooling and drying steps that lead to long processing times. In contrast, Mechanical/Pressure Driven methods, like Drawing, Template Synthesis and Gyration (Pressure Spinning), employ physical forces such as centrifugal force and gas pressure. These mechanical drivers generally lead to lower overall energy consumption and offer a clear, advantageous pathway to non-hazardous, water-based systems, aligning better with sustainable manufacturing goals.

Energy consumptions of each method is estimated considering the forming stages of each method. The data is representative of published literature referenced for each method. Power ratings of the actual equipment used in each method to produce fibres are assessed to

calculate energy consumption. However, in the instance where information on the equipment is lacking in literature, the energy requirements are estimated theoretically, as described in Tables 1-5. The primary purpose of quantifying energy consumption based on a consistent input volume (1 ml of polymer solution) is to establish a benchmark ratio for relative environmental impact between the technologies. While the absolute energy values are specific to the lab-scale parameters and necessary simplifying assumptions, the overall comparative hierarchy of energy efficiency between the methods is robust and is expected to be maintained at industrial scale. For a tangible, real-world translation, these values can be converted into the production rates for each method using the yield of fibres produced.

2.3.1 Electrospinning

There has been much progress in the development of electrospinning methods in the last decade, where methods such as co-axial and two-stream electrospinning evolved. Electrospinning (Figure 2) can be considered as a modified version of melt spinning, where it utilises an electric force to produce charged strains of polymer solutions or polymers melts.[74] In the melt spinning process the polymer is melted for extrusion and then directly solidified. However, electrospinning enhances the extrusion process of the method to obtain thinner polymeric fibres by incorporating electrostatic repulsion. Fibres of various configurations have been produced using over a hundred individual polymers.[75] However, the development of ‘green’

electrospinning methods remains a challenge mainly due to many polymers used not being water-soluble and a low supply of non-charged polymers that are water-soluble.[76] The majority of water-soluble polymers consists of charged polyelectrolytes, where the viscosities of polyelectrolyte solutions (where salt is not present) are known to be higher due to like charges repelling each other, in comparison to neutral polymer solutions consisting of the same polymer concentration.

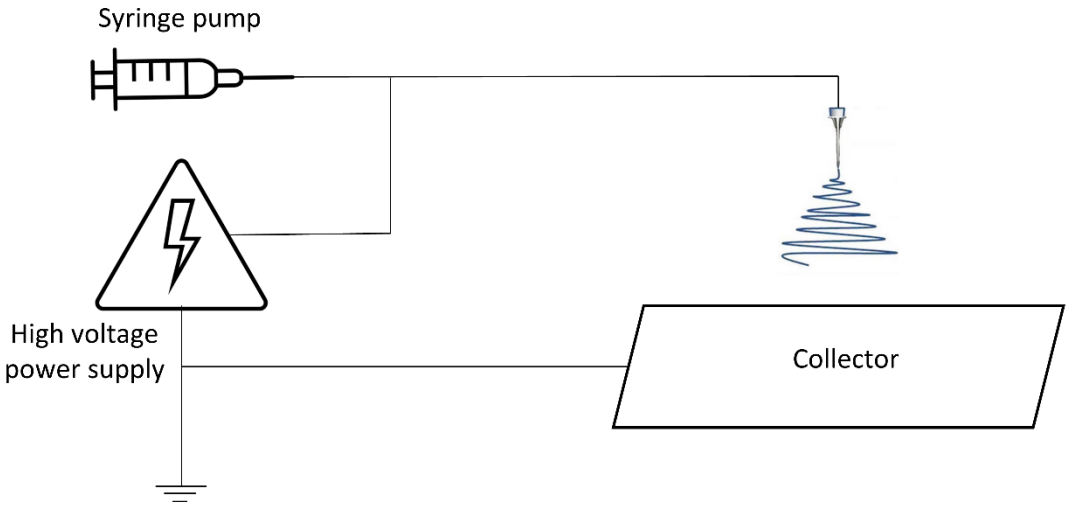

Figure 4. *Schematic illustration of a typical simple electrospinning set up to produce nanofibre*

Much of the published literature on electrospun polyelectrolytes (solutions), regardless of the presence of salt, utilises toxic solvents that are harmful to the environment in the spinning process.[77] The method requires vaporisation of the solvent to result in polymeric fibres, where the solvent vapour can cause harm, unless a non-hazardous solvent is utilised in the process. More research is required to be undertaken on either considering the choice of polymer for the selected application or the identification of a less harmful solvent and the optimisation of

electrospinning conditions. Although water-based polymers will have the lowest impact on the environment, these are mechanically weak and disintegrate rapidly in the environment.[78] Regardless, there are applications of the use of water-soluble polymers rather than solvent-based polymers. For instance, PVA has been electrospun to form filter media membranes for air filtration applications.[79] However, there is still a requirement to improve the efficiency and effectiveness of producing air purification filters using this method.[22] PVA and other water-soluble polymers have also been used to produce oral rapid drug release carriers via electrospinning.[80] Arguably the least toxic method of electrospinning is melt electrospinning, which does not utilise any solvents.[81] Instead, it makes use of heat to melt the polymer for electrospinning, which significantly reduces potential harm caused from toxicity. Electrospinning often requires high voltages via power units to produce fibres, which subsequently drives up energy consumption of the process. The use of heat to melt polymers may further add to the already high amount of energy required to produce fibres. There is also a challenge to produce fibres in the nanoscale using melt electrospinning in comparison to solution-based electrospinning where nanofibre can be more easily spun. More focus is required on the optimisation of the melt electrospinning process to produce fibres where there are some reports of success.[82]

A 2021 report by researchers at Columbia University elaborates a ‘green electrospinning’ technique that reduces the ejection of hazardous material into the environmental along with the mitigation of other

risks.[83] Electrospun fibres may also contain traces of solvent after production where the Food and Drug Administration (FDA) classes some solvents used for electrospinning such as DMF as restricted for use in pharmaceuticals.[84] According to the report, this green process is scalable and eco-conscious and uses acetic acid instead of traditional solvents like DMF and water-soluble polymers. These green electrospun fibres are incorporated with ceramic nanoparticles and the study concludes that this makes the fibres better than traditionally electrospun fibres in multiple ways. Seemingly, this would reduce negative manufacturing effects by up to six times and improve the mechanical properties of the resulting fibre itself. However, although less volatile, more concentrated acetic acid can also be harmful to humans. To assess the energy consumption of electrospinning, the use of acetic acid in the green electrospinning method described in the report by Columbia University was analysed to evaluate the overall energy consumption of the method as shown in Table 1. The incorporation of ceramic nanoparticles was ignored to evaluate the energy consumption of the green electrospinning process in its most basic form. The Vortex Genie 2 speed mixer utilised in this experiment used a voltage of 120 V along with a current of 0.95 A.[85] The solvent (acetic acid) was added to the polymer (PLGA/PCL) where it was vortexed for at least 1 hour. A standard syringe pump controller such as the Harvard Apparatus PHD 4400 has a power rating of 75 W.[86] It is assumed a device with a similar power rating is used to produce a flowrate of 0.75 mL/h in this step. The calculations in Table 1 estimate the energy consumption to

spin 1 ml of polymeric solution using this method. The solution in the study was exposed to a voltage of 10 kV. A typical high voltage power supply would carry a current of around 3 mA to produce an output voltage of 10 kV.[87]

Step of method	**Energy consumption**
Spinning of solutions	$\text{Power(P)} = \text{Voltage (V)} \times \text{Current(I)}$ $\text{P} = 120 \times 0.95$ $\text{P} = 114\ \text{W}$ $\text{Energy(E)} = \text{Power(P)} \times \text{time(t)}$ $\text{E} = 114 \times (60 \times 60)$ $\text{E} = 410{,}400\ \text{J}$
Syringe pump infusion	$\text{Flowrate (ml/s)} = 0.75/(60 \times 60)$ $\text{Flowrate} = 2.0833 \times 10^{-4}\ \text{ml/s}$ $\text{Time (t)} = 1/(2.0833 \times 10^{-4})$ $\text{t} = 4{,}800\ \text{s}$ $\text{E} = \text{P} \times \text{t}$ $\text{E} = 75 \times 4{,}800$ $\text{E} = 360{,}000\ \text{J}$
Power unit	$\text{P} = \text{V} \times \text{I}$ $\text{P} = 10{,}000 \times 0.003$ $\text{P} = 30\ \text{W}$ $\text{E} = \text{P} \times \text{t}$

	$\mathrm{E} = 30 \times 4{,}800$ $\mathrm{E} = 144{,}000\ \mathrm{J}$
	$\mathrm{Total} = 9.14 \times 10^5\ \mathrm{J}$

Table 1. *Energy consumption estimate of electrospinning to produce fibres from 1 ml of polymer solution*

2.3.2 Phase Separation

The phase separation method involves the production of two phases from a homogeneous mixture. Phase separation methods such as the non-solvent induced separation (NIPS) method and the thermally induced phase separation (TIPS) method have been used to produce polymeric fibres. Phase separation is more commonly utilised to produce membrane technology such as hollow fibre membranes.[88] In NIPS, the polymer-solvent solution is placed in a coagulation bath containing a nonsolvent. Once immersed, mutual diffusion between the solvent and nonsolvent leads to phase separation due to the change in composition which results in polymer precipitation and membrane formation. TIPS involves the manipulation of the solubility of polymers by lowering the temperature as the polymer separates out of their solvent. Initially the polymer is required to be dissolved into a solvent at a high temperature before being solidified via freezing (gelation). The dissolving is typically done at a temperature higher than the melting point of the polymer to produce a homogenous mix.[89] The gelation is considered to be the most difficult step in the process as the porosity

and morphology needs to be controlled.[90] The polymer solution also needs to be cast in fibre form when cooling to obtain fibres whilst the solvent is extracted. Freeze-drying is often utilised to obtain optimum porosity where this step can last up to 1 week.[91] Due to the requirement of gelation and freeze drying to obtain porosity, this method requires a considerable amount of energy for fibre manufacture as it involves the manipulation of temperature. Toxic solvents such as dimethylformamide (DMF) and dimethylacetamide (DMA) are commonly used to dissolve petroleum-based polymers using this method.[92] However, only a few polymers such as PLA have been utilised to produce fibres with phase separation.[93]

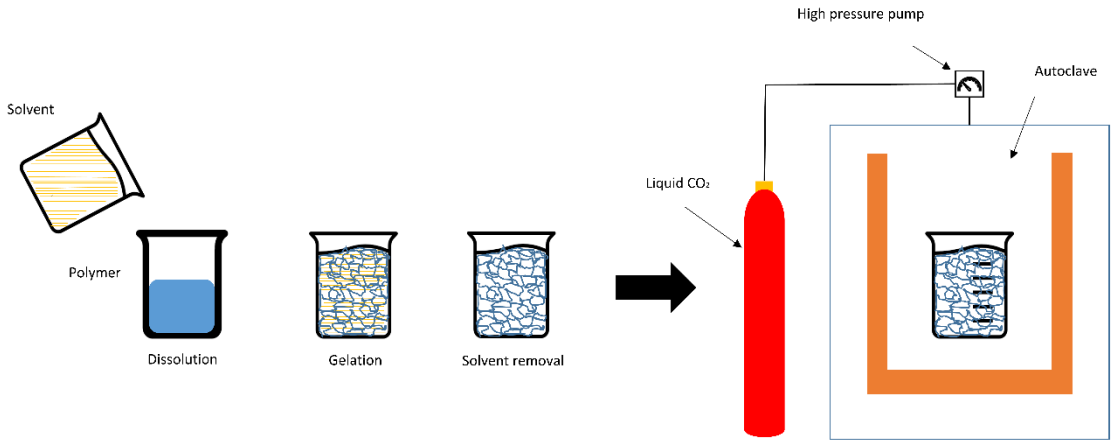

Figure 5. *Schematic illustration of phase separation process including the use of supercritical carbon dioxide*

A French report presents an environmentally improved phase separation method that makes use of supercritical carbon dioxide for drying instead of freeze-drying.[94] Figure 3 portraits a schematic diagram of the method in this report, which incorporates the use of supercritical CO_2. A Life Cycle Assessment of the traditional method in

comparison to this new method has shown a 50% reduction in environmental impact according to the study. To assess the energy consumption of the method described in this report, the processes of the technique are tabulated as shown in Table 2. The study utilised PLA which was magnetically stirred with 1,4-dioxane at 50°C to obtain a concentration between 5%wt to 10%wt.[94] Due to the requirement of heat to mix the solution, it assumed that a hot stirrer is used for this step rather than a vortex speed mixer. It is also assumed the solution was stirred for at least an hour, similar to the electrospinning method using a standard 500W magnetic hot stirrer.[95] Water was then added to the solution for the phase separation process, where 1 ml samples of the solution was then cooled to -20°C, -80°C and -196°C. 1,4-dioxane has molar heat capacity of $150.65\ \mathrm{J/molK}$. Since the solution is relatively dilute, where the solvent dominates the solution, it can be assumed that $c_p \approx c_{p,solve}$, where $'c_p'$ is the (molar) heat capacity of the mixture and $'c_{p,solve}'$ is the molar heat capacity of the solvent. 1,4-dioxane has a density of 1030 kg/m³ and it is assumed that the polymeric solution has a density close to this value considering the solution is relatively dilute. Therefore, a volume of 1 ml of the polymeric solution should have a mass of around 1.03 g. After cooling the samples were left at the above conditions in cold storage for 12 hours, before being soaked in liquid nitrogen for at least 5 minutes. Ultra-low temperature (ULT) freezers are typically utilised for biomedical processes such as cold storage. Typical ULT freezers have a power consumption of around 8 kWh per day at a temperature of -80°C.[96] The samples were then submerged in ethanol

precooled to -20°C, to extract the solvent in a 2-hour cold storage at 4°C. Domestic fridge power consumption is typically between 100 and 250 W.[97] To conclude, the samples were left in an autoclave at 35°C and pressurised with CO_2 at 15 MPa for 4 hours. The energy consumption of this step of the process is estimated considering the specific heat capacity of PLA. It is assumed that the samples were immediately placed in the autoclave after refrigeration. The liquid CO_2 was preheated with the use of a heat exchanger before being continuously pumped with the use of a high-pressure membrane pump at a rate of 1 kg/hr. The high-pressure pump used in this study (Milton Roy Europe) has a maximum motor power of 75 kW.[98] According to the specifications of the pump, the pump would have to perform on maximum motor power to deliver a liquid CO_2 flowrate of 1 kg/hr.

Step of method	**Energy consumption**
Mixing of solution	$\mathrm{E} = \mathrm{P} \times \mathrm{t}$ $\mathrm{E} = 500 \times (60 \times 60)$ $\mathrm{E} = 1{,}800{,}000\ \mathrm{J}$
Sampling cooling	1,4-dioxane has a heat capacity of $150.65\ \mathrm{J/molK}$ and a molar mass of $88.11\ \mathrm{g/mol}$. $\mathrm{c_p} = \frac{150.65\ \mathrm{J/molK}}{88.11\ \mathrm{g/mol}}$ $\mathrm{c_p} = 1.71\ \mathrm{J/gK}$ To cool samples to -80°C from 50°C:

	$Q = mc_p\Delta T$ $Q = 1.03 \times 1.71 \times 130$ $Q = 229\ J$
Cold storage	$E = P \times t$ $E = 8{,}000 \times (12 \times 60 \times 60)$ $E = 345.6 \times 10^6\ J$
Cold storage after ethanol submersion	Assuming minimal power consumption (at 100 W): $E = P \times t$ $E = 100 \times (2 \times 60 \times 60)$ $E = 720{,}000\ J$
Autoclave	PLA has a specific heat capacity of $1800\ J/kgK$. $Q = mc\Delta T$ $Q = 1.03 \times 10^{-3} \times 1800 \times 31$ $Q = 57.5\ J$
Application of pressurised CO_2	$E = P \times t$ $E = 75 \times 1{,}000 \times (4 \times 60 \times 60)$ $E = 1.08 \times 10^9\ J$
	$Total = 1.43 \times 10^9\ J$

Table 2. *Energy consumption estimate of phase separation to produce fibres from 1 ml of polymer solution*

2.3.3 Self-Assembly

As the name suggests, this involves molecules arranging into patterns via non-covalent forces such as electrostatic reactions and is also one of the bottom-up material production processes (Figure 4).[51] It is

considered to be a good method to produce fibres lower than 100 nm where the primary mechanism depends on intermolecular forces. It also makes use of gelation where the polymer solution is maintained at the gelation temperature before the solvent is removed resulting in the formation of polymeric fibres. The main limitation of this process is the complexity along with low productivity and lack of control of fibre dimensions.[93] It is also limited to fibres that can be formed from active molecules that can self-assemble spontaneously.[99] Therefore, the material choices to produce fibres using this method is limited. Its environmental effects are similar to phase separation due to the similarity of the gelation process and the use of solvents in both methods.

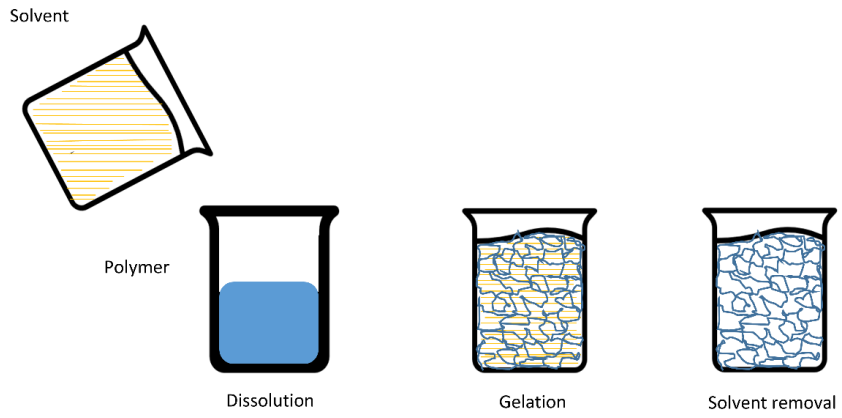

Figure 6. *Self-assembly process*

When estimating the energy consumption of this process, it can be assumed that the magnetic stirring, gelation and solvent removal steps of the process will have a similar energy consumption in the cooling steps as shown in the phase separation method. Assuming the same polymer and solvent with the same consistency as were used as in Table

2 an estimated total energy consumption of 348.12×10^6 J is deduced for the self-assembly method. The types of interactions dictate the intermolecular forces. Van der Waals forces, electrostatic interactions, hydrophobic and hydrogen bonding are the primary drivers of self-assembly. Intermolecular forces are ubiquitous in nature. However, these forces can be obtained by other means. For instance, to obtain electrostatic interactions, ions can be produced by methods such as electron ionisation to charge atoms or molecules in preparation for self-assembly. Considering such methods used to produce intermolecular forces may significantly further drive up the total energy consumption of the self-assembly method. For example, when using field-directed assembly, an electric field is used to promote interactions between nanoparticles into long continuous chains.[100] Depending on the equipment used, along with the time and magnitude the voltage is maintained for during this step of the process, the total energy consumption will be larger than 348.12×10^6 J.

2.3.4 Template Synthesis

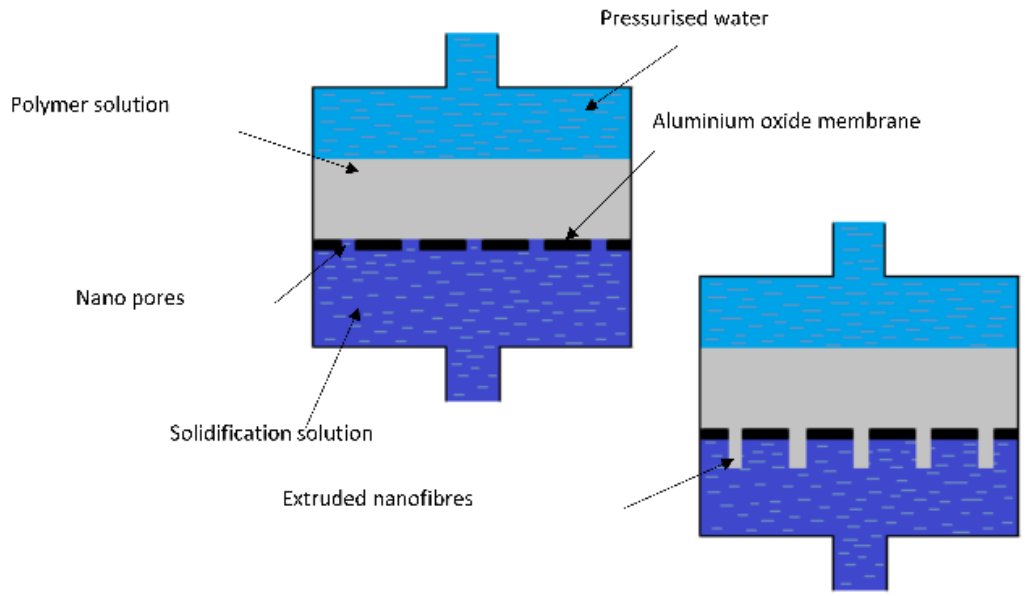

Figure 7. *Template synthesis process*

This process (Figure 5) incorporates the use of chemical or electrochemical oxidative polymerisation to produce fibres of different materials such as metals and polymers.[51] It employs the use of a template (or cross-sectional mould) of the required material and structure to produce polymeric fibres. When producing polymeric nanofibres, a metal oxide template or membrane with submillimetre scaled pores is utilised to extrude fibres by passing the polymeric solution through one side of the membrane to get in contact with the solidifying solution on the other side (as shown in Figure 5). A drawback of this method is that it is incapable of achieving long fibre lengths, although multiple diameters are feasible to obtain by changing the templates. Regardless of this drawback, template synthesis is the a commonly utilised method to manufacture fibres and hollow carbon

fibres.[101] The water pressure is the primary driver of extrusion to form fibres in this process. Therefore, it is a relatively less energy consuming method in comparison to most of the other methods listed in this section. However, the need for solvents remains.

A 2002 expeirment to produce polyacrylonitrile (PAN) fibres using template synthesis is analysed in Table 3 below to estimate its overall energy consumption.[102] PAN (10.976 g, M_w=120 000) was dissolved in DMF (50 mL) with stirring at 70 °C to form an 18 wt % precursor solution. A mixture of 40 wt % DMF and 60 wt % deaerated Milli-Q water was used as the solidifying solution. It is assumed a standard 500 W magnetic hot stirrer was utilised in this process, to stir both solutions at the same time in an hour. The fibres were synthesised by subjecting the polymeric solution to a water pressure of 0.1 MPa with the use of a water pump. An anodic aluminium oxide membrane with a pore diameter of about 102 nm was used as template. A standard pressure booster pump to produce a pressure of 0.1 MPa requires a wattage of 90 W.[103] It was assumed that a similar device was utilised in the experiment and the solution was subject to a pressure for no more than half a minute.

Step of method	**Energy consumption**
Preparation of polymeric solutions	$\mathrm{E} = \mathrm{P} \times \mathrm{t}$ $\mathrm{E} = 500 \times (60 \times 60)$ $\mathrm{E} = 1{,}800{,}000\ \mathrm{J}$
Synthesis of fibres	$\mathrm{E} = \mathrm{P} \times \mathrm{t}$

	$E = 90 \times 30$ $E = 2{,}700\ J$
	$Total = 1.80 \times 10^6\ J$

Table 3. *Energy consumption estimate of template synthesis to produce fibres from 1 ml of polymer solution*

2.3.5 Drawing

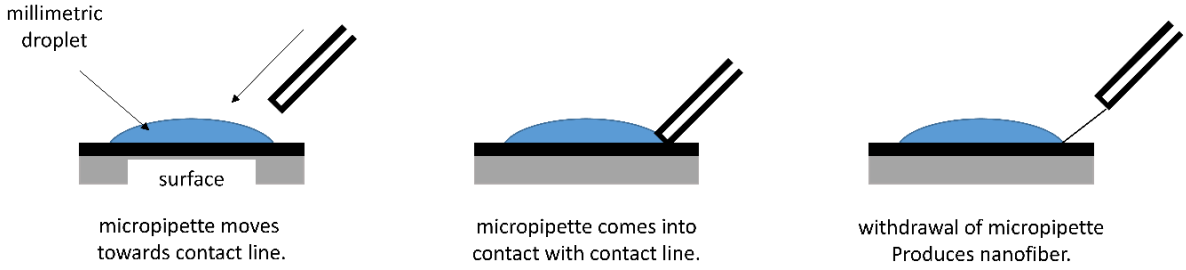

Figure 8. *Drawing method to produce fibres*

This process has the ability to produce long lengths of fibres in a single process, where the process just needs a micropipette to draw out polymeric fibres from either a polymer solution or melted polymer (Figure 6).[104] The solvent is evaporated by extrusion which causes high surface area, however, a cooling step might be necessary in the case of melt extrusion. The process can be slow as the micropipette gently pulls the liquid at a very low speeds of around 10^{-4} m/s to extrude fibres one at a time.[51] This is repeated several times to draw fibres from the same droplet of polymeric solution. Another limitation is that only materials that are viscoelastic and can withstand stresses and strains of drawing can make use of this process. Regardless of these drawbacks, this

technique is a very energy efficient method in that a polymeric solution is used instead of melt extrusion. Table 4 estimates the energy consumption of an experimental study from 2011, where the finest PVA fibres were drawn at a rate of 0.03 m/s from a polymeric solution concentration of 20%.[105] Due to the water solubility of PVA, it is assumed a magnetic stirrer is used for 24 hours to produce the solution for this process. A standard 6 W magnetic mixer with no heating is assumed to have been used for this part of the process.[106]

Step of method	**Energy consumption**
Production of 20% PVA solution for drawing.	$\mathrm{E} = \mathrm{P} \times \mathrm{t}$ $\mathrm{E} = 6 \times (24 \times 60 \times 60)$ $\mathrm{E} = 518{,}400\ \mathrm{J}$
	$\mathrm{Total} = 5.18 \times 10^5\ \mathrm{J}$

Table 4. *Energy consumption estimate of drawing method to produce fibres from 1 ml of polymer solution*

2.3.6 Gyration Based Spinning Methods

Gyration based spinning methods such as centrifugal spinning and pressure spinning primarily makes use of the centrifugal force from the rotation of the vessel to extrude polymeric fibres out of the orifices in the walls vessel (Figure 7).[107] In pressure spinning, the simultaneous application of gas pressure in to the vessel supplements the extrusion of fibres.

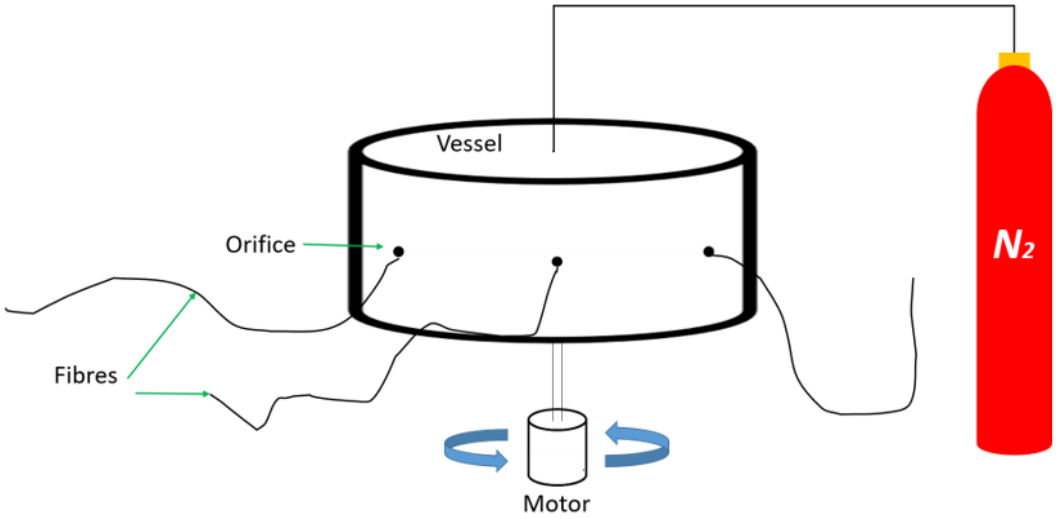

Figure 9. *Energy consumption estimate of drawing method to produce fibres from 1 ml of polymer solution*

Pressure spinning (and centrifugal spinning) is one of the few methods that has the ability to mass-produce submicrometre diameter polymeric fibres swiftly.[31] This is subsequently a more energy efficient technique in comparison to most of the other processes currently utilised. This method can make use of all of the polymeric solution in the vessel to convert to fibres within a minute of the system starting to run. This also contributes to making its power usage considerably lower in comparison

to other methods. A 2019 study in fluid behaviour during the use polyethylene oxide (PEO) and deionised water to manufacture via pressure spinning is analysed as shown in Table 5.[108] The application of pressure is ignored in this energy estimation, as nitrogen gas from a cylinder is manually released via a pressure control valve, which does not require any energy input. The study claims a magnetic stirrer at ambient temperature was utilised in this process for 24 hours. A standard 6 W magnetic mixer with no heating is assumed to have been used for this part of the process. A typical electric motor capable of producing enough torque to spin the vessel of the pressure spinning system up to 10,000 RPM has a power rating of 21.2 W.[109] A 2019 study claims that the method produces a yield of 3.2 kg of fibre an hour (0.89 g per second) when running at full speed.[110] PEO is utilised to produce solutions of multiple weight percentages, where 21%wt. was the largest percentage. This would equate to 0.21 g of PEO in 1 ml of solution at 21% concentration. In theory, 0.21 g of polymer requires less than 1 second for extrusion into fibres, as the paper suggests a yield of 3.2 kg h^{-1} was achieved. However, it is assumed that the motor is run for around 30 seconds for all of the polymeric solution to be used during the gyration process.

Step of method	**Energy consumption**
Preparation of polymeric solution	$\mathrm{E} = \mathrm{P} \times \mathrm{t}$ $\mathrm{E} = 6 \times (24 \times 60 \times 60)$

	$\mathrm{E} = 518{,}400\ \mathrm{J}$
Gyration of the vessel	$\mathrm{E} = \mathrm{P} \times \mathrm{t}$ $\mathrm{E} = 21.2 \times 30$ $\mathrm{E} = 636\ \mathrm{J}$
	$\mathrm{Total} = 5.19 \times 10^5\ \mathrm{J}$

Table 5. *Energy consumption estimate of pressure spinning*

2.4 Comparison of Fibre Manufacture Techniques

According to published literature, there are more attempts to improve the environmental effects in electrospinning than in any other polymeric fibre manufacturing method. Perhaps this is due to this method having the most room for improvement, along with it being the most commonly used method to fabricate polymeric fibres. Table 6 reviews the potential environmental effects considering the hazards, energy consumption and efficiency of each method discussed. To enable a structured comparison between polymer fibre production methods, qualitative scores for Hazard and Efficiency were assigned on a scale of 1 to 5, with 1 representing the least favourable outcome and 5 the most. The Hazard score reflects environmental, health and safety considerations, including solvent toxicity, energy demands, emissions and operational risks, based on data from peer-reviewed literature described in Section 2.2.1 to Section 2.2.6. The Efficiency score takes into account factors such as production rate, material yield, process scalability and equipment complexity, drawn from reported performance metrics in academic and

industrial studies. These numerical scores represent a relative ranking of the five manufacturing methods examined. In cases where direct quantitative comparisons were unavailable, scores were determined using relative assessments. This scoring framework provides a simplified yet informative means of evaluating the overall sustainability and practicality of each technique.

Production Method	Energy	Analysis	Hazard	Efficiency	Score
Electrospinning	9.14×10^5 J	The use of very high voltages, along with potentially hazardous solvents can be highly dangerous, unless the correct safety precautions are followed.[111] Due to flowrates being small, the method takes time to produce polymeric fibres from the same amount of solution in comparison to methods such as pressurised gyration.[112] In comparison, this subsequently results in high power consumption. Other issues also include solution clogging the needle.[113] Scaling up this method for mass production	3	4	12

		can cause the overall equipment to be more complex and expensive.[93]			
Phase Separation	1.43×10^9 J	The use of solvents, along with extreme temperatures and pressures can be highly hazardous. Furthermore, this method remains a laboratory scale process despite its simplicity.[114] It is only limited to a few polymers.[115] Temperatures need to be varied which subsequently require more energy to do so.	1	2	2
Self-Assembly	348.12×10^6 J	Similar to phase separation, the use of solvents, along with extreme temperatures and pressures can be highly hazardous. The necessity of a cooling procedure in the instance of melt spinning may require more energy usage that in the case of dry spinning. Complexity of the procedure influences the low efficiency score.[104]	2	2	4
Template Synthesis	1.80×10^6 J	The requirement of solvents for the extrusion process can be hazardous. Regardless, it can	4	3	12

		be argued that this method is a simple and environmentally friendly technique of producing fibres, as water pressure is the primary driver of producing the fibres via extrusion. However, the method cannot produce long continuous fibres.[115]			
Drawing	5.18×10^5 J	The requirement of solvents for the drawing process is hazardous. However, it has the potential to draw fibres from a polymer melt rather than a polymer solution, although the melting of polymers will cause the overall energy consumption due to manufacture to rise.[104] However, drawing is probably the simplest method of producing long single polymeric fibres that require the least energy, as all it requires is a micropipette to draw fibres.[115] However, the very low productivity is a major disadvantage.[51]	4	1	4
Gyration methods	5.19×10^5 J	The requirement of solvents to process fibres can be environmentally hazardous. It	4	5	20

		can be argued that the mass producing features and fast processing of polymeric solutions into fibres can be make this the more environmentally advantageous method.[116] The pressurised gyration method works best with water soluble polymers. This method can deliver a very high quantity of fibres within a minute whereas other methods discussed can take up to days.			

Table 6. *Comparative evaluation of common polymer fibre manufacturing techniques based on hazard and efficiency. Scores are assigned on a scale of 1 (least favourable) to 5 (most favourable), where hazard reflects environmental, health and safety concerns and efficiency reflects production scalability, material throughput and process speed.*

The energy consumed only during the actual manufacture of fibres including the dissolving of polymer into solvents for each method was evaluated. The analysis explicitly focused on the energy consumption necessary to transform the prepared polymer solution into final fibres. Energy in dissolving polymers to solvents can also take up to days for some polymeric solutions, depending on concentration. However, the assumption was made that this step of the method was the same in terms of energy consumed for each of the methods, to maintain an

unbiased analysis. Due to lack of information, humidity and temperature control was neglected when evaluating energy consumption of all methods, however these can be two major variables in controlling fibre morphology and properties.

Electrospinning required an estimated total of 9.14×10^5 J. This however can vary depending on the parameters, such as voltage, volumes of polymeric solution and flowrate used to spin fibres. The investigation made use of relatively high voltages, whereas some polymers are spun at lower voltages and may also require lower flowrates. These parameters will affect exactly how much energy is consumed in these methods. The time under which the solutions are exposed to electricity, during manufacture, is an important factor for the energy consumption in this process. The amount of solution used along with the flow rates considered for electrospinning will primarily dictate the amount of time required. The method was judged to be moderately hazardous but reliable overall regardless of the difficulties associated with the use of the technique due to its efficiency.

Phase separation was shown to be the most energy consuming method. Table 6 shows that this method was the least scoring considering its hazards and overall efficiency. The energy estimation of the phase separation method also only took into consideration the actual energy required theoretically to cool the samples to -80°C, considering the specific heat. The information regarding the appliance used for this step of the process was not indicated. In reality, this step of the process will consume more energy when taking the power rating of the equipment of

the cooling process are taken into account. Energy consumption was heavily influenced by the length of the freeze-drying step, which can last up to a week depending on the temperature and settings used during processing. In addition, the use of the autoclave along with pressurised CO_2 drastically increased the power consumption. However, according to the 2018 study, the use of supercritical CO_2 is said to have up to a 50% reduction on the effect of the environment than the conventional method. This indicates that the traditional method of phase separation may produce an even higher environmental impact than the use of supercritical CO_2. Supercritical fluids are considered green solvents as it is used in extraction processes and abide by the principles of green chemistry.[117] However, the use of supercritical CO_2 seems to require a vast amount of energy due to the use of a high-pressure membrane pump and is also not the simplest route due to the other complications.[118, 119] Besides this, the actual energy consumption estimation for this method is most likely higher, mainly due to the evaluation of thermal energy to increase or decrease temperature of only the polymeric solution. The use of the equation heat energy = (mass × specific heat × change in temperature), estimates the energy required to change the temperature of the polymeric solution in an ideal 100% efficient scenario. A more accurate measure is to assess the power rating of the equipment used in this step of the process for the phase separation method, where instead thermal energy was assessed using the heat energy equation in Table 2. Similar to phase separation, the self-assembly method also requires changes in temperature and

storage at low temperatures, which makes this method more energy consuming. An estimated 348.1×10^6 J of energy is required to produce polymeric fibres using this method, where the estimation did not include the energy required for potential methods to promote the interaction between atoms and molecules. Considering such processes for the self-assembly method would most likely show a higher estimation of the energy consumed.

At 1.8×10^6 J and 5.19×10^5 J respectively, template synthesis and drawing require low amounts of energy to produce fibres. However, in the instance that only ambient temperatures are required to stir polymeric solutions, these methods are likely to require even lower amounts of energy than electrospinning and pressure spinning. Pressure spinning and template synthesis are probably more convenient to produce fibres, due to its relatively quick production of fibres in comparison to drawing or electrospinning. However, electrospinning scores more than template synthesis in the efficiency scale on Table 6 as template synthesis cannot produce long fibres. Considering the drawbacks of the methods evaluated in this study, gyration-based methods show the most promise in delivering fibres efficiently and for an environmentally friendly bias. This is mainly due to its fast-processing features and relatively simple procedure in producing fibres.

Figure 8 displays an energy comparison pie chart to produce polymeric fibres from each method listed previously (Tables 1 to 5). It is shown that that self-assembly, template synthesis, drawing and gyration-based methods combined only require a very small fraction (less than 0.3%) of

the amount of energy in comparison to phase separation. However, considering the total lifecycle for polymeric fibres manufacturing methods may show different results in terms of energy consumed in their total cycle. For instance, when considering the process of obtaining nitrogen gas via fractional distillation of liquid air, the overall energy consumption of producing polymeric fibres using pressure spinning may be significantly higher.[120] However, nitrogen is not essential and pressure can be imparted using compressed air. Taking into consideration parameter control techniques, such as a dehumidifier for humidity control (which can be critical to produce polymeric fibres with desired characteristics), can further elevate energy consumption during manufacture.

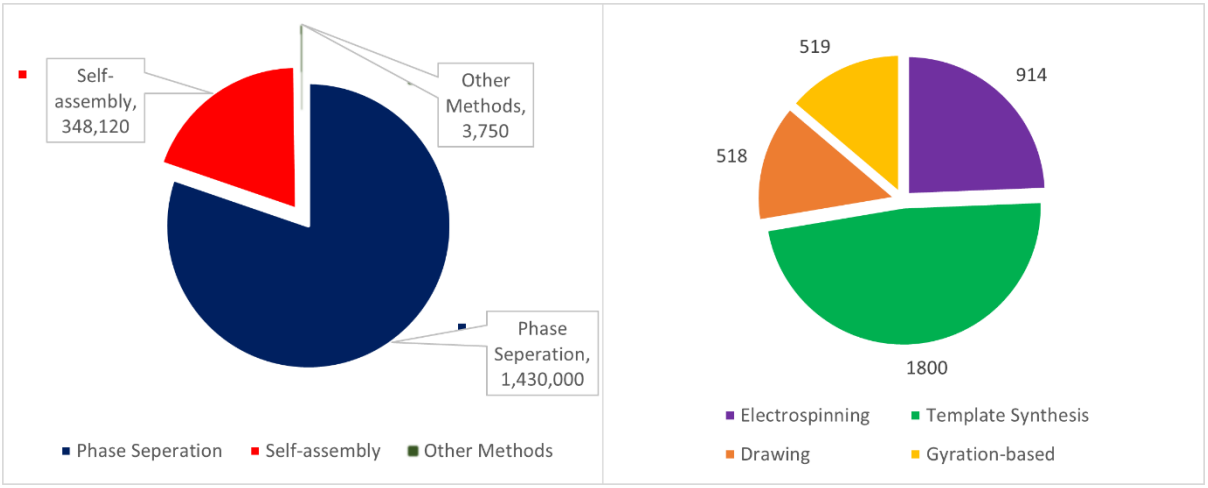

Figure 10. *Energy consumption comparison to produce fibres from 1ml of polymeric solution (in KJ)*

2.5 Financial Cost of Polymeric Fibre Manufacture Methods

The average electricity costs in the UK are at 24.86 p/kWh as of January 2025.[121] Considering this and the amount of time taken to complete each step of the polymeric fibre manufacture methods, the energy cost to produce fibres are estimated as shown in Table 7. 1 kWh equates to 3.6×10^6 J, therefore, the costs are estimated by dividing the energy values in Joule by 3.6×10^6 and multiplying this value by the unit cost electricity in the UK (£0.249/kWh).

Method	Step of the process	Cost calculations
Electrospinning	Spinning of solution	$410{,}000 \div 3.6 \times 10^6 = 0.114\ \text{kWh}$ $£0.249 \times 0.114 = £0.0283$
	Syringe pump infusion	$360{,}000 \div 3.6 \times 10^6 = 0.1\ \text{kWh}$ $£0.249 \times 0.1 = £0.0249$
	Power unit	$144{,}000 \div 3.6 \times 10^6 = 0.04\ \text{kWh}$ $£0.249 \times 0.04 = £0.0100$
	Total	$\mathbf{£0.0632}$
Phase separation	Hot stirrer	$1{,}800{,}000 \div 3.6 \times 10^6 = 0.5\ \text{kWh}$ $£0.249 \times 0.5 = £0.1245$
	The cooling step cost calculations excludes the energy required to cool the polymeric solution before storage in an Ultra-Low Temperature freezer.	$345.6 \times 10^6 \div 3.6 \times 10^6 = 96\ \text{kWh}$ $£0.249 \times 96 = £23.904$
	Ethanol cold storage	$720{,}000 \div 3.6 \times 10^6 = 0.2\ \text{kWh}$ $£0.249 \times 0.2 = £0.0498$
	Use of supercritical CO_2, where the energy used to maintain a	$1.08 \times 10^9 \div 3.6 \times 10^6 = 300\ \text{kWh}$ $£0.249 \times 300 = £74.7$

	temperature of 35°C is ignored in the calculation.	
	Total	£$\mathbf{98.78}$
Self-Assembly	The self-assembly process requires a total energy cost of **£24.078**, as previously assumed stirring, gelation and solvent removal steps of the phase separation process used for this technique.	
Template synthesis	Hot stirrer	$1{,}800{,}000 \div 3.6 \times 10^6 = 0.5$ kWh £$0.249 \times 0.5 =$ £0.1245
	Pressure booster pump	$2{,}700 \div 3.6 \times 10^6 = 0.00075$ kWh £$0.249 \times 0.00075 =$ £0.0002
	Total	£$\mathbf{0.1247}$
Drawing	Hot stirrer	$518{,}400 \div 3.6 \times 10^6 = 0.144$ kWh £$0.249 \times 0.144 =$ £$\mathbf{0.0359}$
Gyration-based	Hot stirrer	$518{,}400 \div 3.6 \times 10^6 = 0.144$ kWh £$0.249 \times 0.144 =$ £$\mathbf{0.0359}$
	Motor	$636 \div 3.6 \times 10^6 = 0.00018$ kWh £$0.249 \times 0.00018 =$ £0.000045
	Total	£$\mathbf{0.0359}$

Table 7. *Cost evaluation of polymeric fibre manufacturing methods to produce fibres from 1ml of polymeric solution.*

The cost estimations are likely to be higher in real situations when considering other parameters involved, such as controlling temperature and humidity. In addition, other factors such as method of stirring polymeric solutions can play a significant role in determining the energy and cost requirements. Regardless, the overall cost ratios of the methods would be more or less the same as shown in Figure 9. Considering the cost estimations are to produce polymeric fibres from 1 ml of solution, it can be shown that pressure spinning is the most cost effective and efficient technique to mass-produce polymeric fibres.

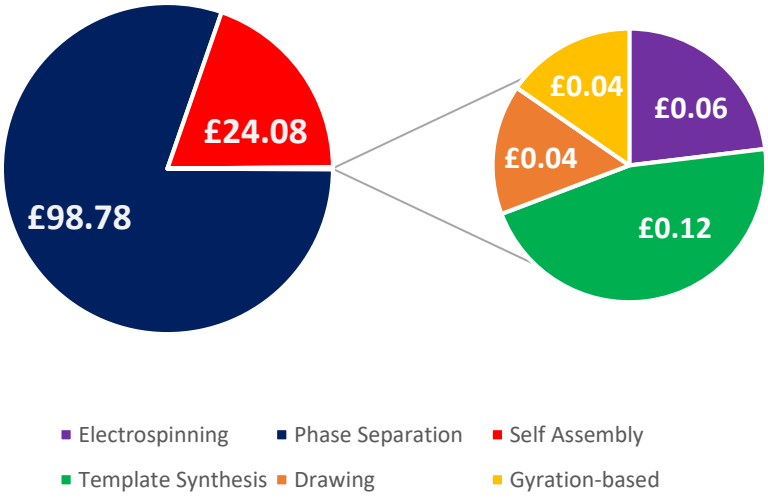

Figure 11. *Cost comparison of fibre production methods (based on the results calculated in Table 7).*

Although electricity prices are expected to stabilise from 2025 onwards, energy costs remain a critical consideration in manufacturing due to long-term economic and environmental pressures.[122] As of 2020, the majority of energy to generate electricity is sourced from gas which has shown an exponential rise price since 2020 and accounted for 80% of

the total electricity cost in the UK.[123] According to the 2021 energy brief, 56.9% of the UK's power grid is generated by non-renewable energy sources such as fossil fuels and nuclear energy.[124] However, the use of non-renewable energy sources has shown a decline in recent years in the UK, although, on a global scale, renewable energy is still dwarfed by fossil fuel power. Considering recent world events, the energy costs are estimated to further increase globally. This further justifies the need to deliver energy efficient means of mass-producing polymeric fibres.

2.6 Future Perspectives for Green Alternatives

While biodegradable plastics are often presented as environmentally benign alternatives to traditional petrochemical-derived polymers, recent findings highlight significant limitations in their real-world performance, especially in uncontrolled environments like oceans or landfills. Many biodegradable plastics, such as PLA, require industrial composting facilities with specific temperature and humidity profiles to degrade effectively. In cold marine environments, these conditions are rarely met, leading to persistent materials that behave much like conventional plastics, fragmenting into microplastics and contributing to marine pollution rather than alleviating it.[125] Furthermore, when considering all aspects of their lifecycle, bioplastics reveal a more complex environmental picture. While these materials may reduce fossil carbon inputs, they often entail high land and water use, reliance on agricultural pesticides and comparable, if not greater, greenhouse gas

emissions during production.[126] The sourcing of bioplastics from crops like corn, sugarcane, or cassava raises concerns around food security, biodiversity loss and deforestation, echoing the critiques faced by biofuels a decade earlier.

As researchers are focused on developing greener methods of manufacture, biopolymers itself may not hold promise to support the reduction of plastic pollution, along with the reduction of the carbon footprint. Therefore, it can be suggested that biodegradable polymers cannot resolve all pollution issues. Hence, to refine polymeric fibre production, the application of naturally derived polymers, additives and other solutions need to be taken into consideration in the manufacture of fibres.[127] Naturally derived polymers such as alginate, chitosan, gelatin, silk fibroin, cellulose, starch and lignin are renewable, often biodegradable under ambient conditions and can be extracted from agricultural or industrial waste streams. Alginate, for example, derived from seaweed, has shown promise in pressure spinning due to its low toxicity and ionic gelation capabilities.[128] Despite their promise, these materials face challenges such as batch-to-batch variability, low mechanical performance and limited compatibility with high-pressure or high-temperature processing techniques, which may restrict their broader application in fibre manufacturing.[129]

In addition to polymer selection, the choice of solvent also plays a crucial role in defining the environmental impact of fibre production technologies. Solvent choice plays a critical role in determining the sustainability of polymer fibre production. As covered in Section 2.1.2,

many traditional solvents used in electrospinning and phase separation are toxic, volatile and derived from fossil fuels. A future-forward strategy involves replacing these with green solvents such as ethanol, water, deep eutectic solvents (DES) and ionic liquids where possible. Water-soluble polymers such as PEO and PVP are already compatible with aqueous systems and have been demonstrated in pressure spinning platforms with minimal hazard potential.[28]

Beyond materials, reimagining the entire process chain through the lens of circular economy principles is essential. This includes integrating closed-loop manufacturing systems that recover and reuse solvent, recycle process water and upcycle fibre waste. In pressure spinning, energy consumption can be minimised through vessel design optimisation and by refining collector geometries to maximise fibre throughput while maintaining morphology.[28]

Technological innovation alone, however, will not be sufficient. A supportive policy environment is critical to enable industry-wide shifts towards sustainable practices. The shift towards greener alternatives is not purely technological, it must be supported by policy frameworks and standardisation bodies. Regulatory incentives such as extended producer responsibility (EPR) and eco-labelling can drive industry adoption of sustainable fibre production practices.[130, 131] Likewise, standards for biodegradability, compostability and toxicity must evolve to reflect real-world degradation conditions, not just idealised laboratory environments.

To unlock the full potential of green alternatives in polymeric fibre production, several critical research directions must be pursued. Comparative life cycle assessments (LCAs) are urgently needed to evaluate the true environmental trade-offs between natural and synthetic polymers when processed under standardised industrial conditions. In parallel, advances in biopolymer modification are required to enhance the mechanical strength, stability and processability of naturally derived materials through scalable and non-toxic techniques. Attention must also be directed toward the development of high-throughput green processing methods, such as pressure spinning and electrospinning, which are compatible with bio-based polymers and enable scalable fibre production. Furthermore, the viability of end-of-life options such as industrial composting, anaerobic digestion and chemical recycling must be evaluated to ensure circularity at scale. Ultimately, the success of green polymeric fibre technologies will depend on strong cross-disciplinary collaboration, bridging material science, chemical engineering, environmental science and policy to ensure that innovation aligns with ecological and regulatory goals.

2.7 Bioplastics and Biodegradable Plastics

There are various origins for natural polymers, where as far as the biomaterials field is concerned, natural polymers are sourced from microorganisms, plants and animals. Due to the benefits natural polymers offer, especially environmentally, they are a considered good substitute for fibre production, especially for biomedical related

applications. Some of the major benefits of utilising natural polymers are the fast integration on to wounded sites and resemblance to the host tissue, along with its biocompatibility and biodegradation. It also has the potential to degrade by enzymes and the capacity to react with biological structures in a controlled method.[132] At present, there are a large number of natural polymers that are compatible with current polymer fibre manufacturing methods, which have been extensively researched in the recent past.[73]

In general, biopolymers are thought to be eco-friendlier than traditional polymers. However, a 2010 study by the University of Pittsburgh concluded that this is not particularly factual when the entire material life cycle was analysed.[133] The research contrasted seven traditional plastics, along with four bioplastics and a single plastic produced from both fossil fuels and renewable resources. The investigators discovered that bioplastics manufacture actually produced a larger quantity of pollutants when fertilisers and pesticides were taken into account. These chemicals are utilised in growing crops and chemical processing which are required to convert organic material into bioplastic. It was also found that bioplastics promote ozone depletion more than regular plastics and also require large-scale land use. However, bioplastics do emit a remarkably lower amount of greenhouse gas emissions in comparison to traditional plastics. There is no resulting increase in carbon dioxide when bioplastics breakdown as they are produced from the same quantity of carbon dioxide. A 2017 report concluded that substituting traditional plastics with corn-based PLA would reduce greenhouse gas

emissions by 25% in the USA.[126] This reduction is primarily attributed to the bio-based origin of PLA, which relies on carbon fixation through photosynthesis during corn cultivation, rather than fossil fuel extraction and refinement. The report also determined that the production of traditional plastics from only renewable energy sources can reduce emission by 50-75%. Although the biodegradability of biopolymers is beneficial, most of these biopolymers require high temperature industrial compositing provisions, which is uncommon and only a few cities seem to possess such provisions. Subsequently, these biopolymers culminate in landfills, where there is risk of methane being released from them, a greenhouse gas known to be 23 times more potent than carbon dioxide. Solar UV radiation is necessary to trigger the photo-oxidation degradation process of most polymers, which occurs through a radical chain mechanism that result in bond cleavage and a reduction in molecular weight.[10] Polymers discarded in landfills are unlikely to have enough access to UV radiation. Bioplastics (and biodegradable) when discarded incorrectly can contaminate recycled plastics and can impair recycling infrastructure. For instance, if PET (polyethylene terephthalate) which is common plastic utilised to produce bottles, is exposed to bioplastics, the whole lot may be declined and end up in a landfill. Therefore, there is need for separate streams to appropriately discard both biodegradable plastics and bioplastics. Bioplastics are produced from natural materials and hence, composting of bioplastic components can make soil fertile, due to the absence of artificial chemicals.[134] The land need for the production of bioplastics may

hinder food production, as the crops that are used to manufacture these plastics can by utilised for food which is known to be in a shortage. In 2017, a joint report of European environmental organisations, estimated that the area of land required to keep up with global demand would equate to more than 1.3 million acres of land by 2019.[135] This is an area larger than Denmark, the Netherlands and Belgium put together. It can also be noted that the petroleum required to manoeuvre farming machinery will also emit greenhouse gases. Bioplastics can also be relatively costly. For instance, PLA can be up to 50% more expensive in comparison to similar traditional plastics due to the intricate method utilised to convert corn or sugarcane into the building blocks for PLA.[127] Regardless, costs of bioplastics have slowly reduced as businesses and researchers evolve more greener and efficient techniques for manufacturing bioplastics. The production of bioplastics such as PLA, commonly used to make nanofibres for various applications, does not require the process of discovery, acquisition and transportation of hydrocarbons. This results in the use of fewer fossil fuels and produces up to 67% less greenhouse gases during manufacture.[136]

The manufacture of biodegradable plastics can be costly. However, considering the clean-up costs along with the lower negative environmental effects, biodegradable polymers are the better option. They also require less energy for production in comparison to regular plastics. Crude oil (petroleum) is a major element in the production of traditional plastics. The extraction and refinement of crude oil

significantly affects the environment. According to the British Plastic Federation, it is believed that 4-6% of oil is used in the manufacture of plastics in Europe alone.[137] Biodegradable plastics use up to 20% renewable materials in its production when compared to traditional plastics. Current research is being undertaken on biodegradable plastics, where in the future, these plastics will only release the same quantity of energy used in manufacture.[138] Nonpetroleum-based plastics such as PLA, that are made from plants such as corn and sugarcane also curtail carbon dioxide as they do not emit an excess amount of CO_2 during decomposition.[139] The use of plant oil as renewable feedstock for the synthesis of polymers has also been recently researched for development.[140]

The global plastic production reached 381 million tonnes in 2015.[141] Assuming that a majority of these plastics are for the making of plastic bags and bottles, the manufacture of such quantities of plastic would emit a staggering 2.3 billion tons of CO_2 into the earth's atmosphere.[142] This is equivalent to the emissions from 87 million vehicles every year. They also release an inebriating greenhouse gas when burnt at landfills. These calculations are likely to be underestimated as new research suggests CO_2 emissions have risen considering the plastic production in countries that utilise large amounts of coal.[143] Moving to the use of bioplastics can significantly lower the emission of greenhouse gasses and subsequently reduce the impact from its effects, such as extreme flooding and desertification. In terms of recycling, biodegradable polymers are quick to decompose and can be

rather easily broken down via an organic procedure. These are also non-toxic and help with the reduction of landfill related issues. The recycle waste product can also be utilised as compost or for biogas. Biodegradable polymers disintegrate in the space of a few months, based on the materials utilised to produce the polymer and method of disposal. It also requires less space for disposal in the event where it does not fully decompose. The disposal of traditional plastics result in the release of other toxic chemicals and various other pollutants to the environment. These chemicals are known to easily harm marine life, ecosystems and also affect human health. For instance, Bisphenol A, which is an important ingredient in the production of plastics, is linked with the endocrine disruption, which is immensely damaging to the human reproductive cycle.[144] Bisphenol A is commonly utilised in many polymer products such as baby products and food containers.[71] Other chemicals in the production of regular plastics have also been associated with diseases such as cancers. The transfer to biodegradable plastics will significantly reduce the release of such lethal by-products into the natural environment and support in the delivery of a ‘greener’ future. Biodegradable components are closer to nature than traditional plastics as they do not emit harmful products and produce manageable amounts of waste when decomposed by bacteria in the soil. This natural decomposition process subsequently means that the energy consumption during this process is zero and is highly cost effective.

One of the major benefits of biodegradable products are its flexibility. After the required material is converted into a polymer, the polymer can be effortlessly integrated with the traditional components that are utilised in producing traditional plastics. There is no need for the production of completely new products to generate biodegradable plastics. Biodegradable plastic products are expected to become a flourishing industry on this generation. It is expected to make a significant impact in the export industry and in the marketing industry. China in 2016 witnessed a 13% increase in local business sales resulting from the manufacture of 290,000 tons of biodegradable plastic products.[145] It is also stated that the country only used 130,000 tons of the manufactured products but accounted for an increase in sales of $350 million. As the awareness and willingness to reduce carbon footprint increases, it can be strongly anticipated that biodegradable plastic products will see a surge in demand very soon. Many companies such as Coca Cola have already proceeded to target this area of awareness by marketing bottles made from biodegradable plastics.[146] This not only increases their company sales with this marketing strategy, but also positively helps the environment. These strategies can potentially be utilised in industries associated with polymeric fibre applications to positively impact its business. Another desirable quality of biodegradable plastics is its ability to decompose under certain desired conditions. For instance, corn-starch which is a significant ingredient used in the manufacture of biodegradable plastics, can be easily broken down when in contact with water in a matter of weeks.[147]

Regardless of the many benefits of using biodegradable plastics over traditional plastics, there are multiple disadvantages that need to be taken into consideration. Biodegradable plastics are produced from natural materials such as soybean and corn. However, there are potential risks of contamination via pesticides, which can be easily conveyed to the end-product. Another drawback of biodegradable plastics is the requirement for expensive industrial processors and composters, mainly those that need high industrial-magnitude temperatures to be broken down.[148] The availability of equipment may also cause issues. Furthermore, it is an obstacle to distinguish between biodegradable and non-biodegradable polymers, as they should not be assorted when discarded. This would result in bioplastics being contaminated, which subsequently means that they cannot be easily recyclable and add up to the waste volume.[149] Methane is produced from some biodegradable polymer during decomposition in landfills. Methane is 84 times more potent than CO_2 (in the time frame of 20 years) and also absorbs heat faster which significantly drives climate change.[150] Biodegradable polymers do not decompose in ocean waters due to the overall cold temperature of ocean waters. Sufficient manufacture of biodegradable plastics will need the use of cropland to supply material instead of growing food. With food shortage and hunger striking 1 in 5 families in developed countries and significantly more in developing regions, there is an ethical debate on the justification to expand this industry.[151]

At present, it costs around 20-50% to manufacture bioplastic in comparison to regular plastics.[152] However, with the implementation of new technology which is currently being researched like the pressure spinning system for the manufacture of polymeric fibres, these costs can be seriously reduced. Some biodegradable plastics products are known to contain certain metals, which may evolve during decomposition.[13] For instance, high quantities of cobalt and lead in a certain brands of biodegradable plastic bag, which raises the question about its potential toxicity during decomposition. Regardless of the fact that biodegradable polymers can effortlessly decompose quickly, these materials require to follow a very specify disposal method without exception as the process can be hindered. Disposing these plastics directly into a landfill will produce methane and it is essential to verify they are recycled or that other waste reduction procedures are met. Water is necessary for the timely decomposition of biodegradable polymers made using corn starch.[153] Rain can easily support decomposition. However, issues arise when there is no rain present and managing waste during such instances. In spite of recent achievement, economic feasibility of composting plastic waste in conventional waste facilities is still some distance away. It is also noteworthy that biodegradable polymers in general are functionally second to traditional polymers. There is a requirement to move consumer behaviour to incomputerate and accept less durable biodegradable plastic products, which will eventually lower the commencement of biodegradable polymers being a commercial reality.[154] There have been rising concerns over greenwashing within

industries, where the UK government is aiming to develop standards for bioplastics and biodegradable plastics to curb this.[155] However, regulations that are more stringent are also essential on a global scale to set high threshold standards for sustainability.[156]

2.8 Toward Sustainable Polymeric Fibre Production

The use of nanotechnology methods and materials have been proven to pose some damage to the environment. However, it can also be argued that the use of these methods and materials have also positively reflected on the environment. For instance, pollution of ground and surface water around the globe is now a significant problem. Regardless of the environmental effects of most of the fibre production methods, nanotechnology has considerably progressed water treatment development.[89] Material selection plays a very crucial role in the design and manufacture of sustainable, eco-friendly products in the field of engineering design.[157] Materials are utilised to make use of their physical and mechanical properties depending on the application of the product. Polymer composite materials are an example of this, for instance it provides ease of manufacture, productivity and is cost effective.[158] Composites are bespoke materials where it possesses distinctive attributes, thus properties can be changed by altering the reinforcement and matrix phase.[159] In comparisons to synthetic fibres, natural fibres can be advantageous due to their availability and abundance, along with their cost effectiveness.[160] These fibres have

been introduced to composites in place of synthetic fibres to make composites lighter.

The use of work-related nanomaterials is managed by the Control of Substances Hazardous to Health (COSHH). COSHH is a law that is requisite of employers to control substances that are hazardous to health, which also includes nanomaterials and solvents.[70] In the USA, the Toxic Substance Control act, curtails the use of chemical substances that present unreasonable dangers to human health and to the environment.[161] To keep up with this, manufacturing methodologies have progressed to adhere with these regulations. For instance, this is reflected in the textile industry where firms have taken on eco-friendly manufacturing and management techniques, such as greener fabrication processes, circular supply chains and recycling. It is also shown in the automotive industry where vehicles emissions are controlled by regulations along with new technologies such as hybrids and electric vehicles. However, in comparison the biomaterials and medical devices industry where the application of polymeric fibres has advanced significantly, greener polymeric nanofibre production practices remain nascent.[83]

2.9 Sustainable Polymers and Solvents

Natural polymers also known as biopolymers may also be another potential solution in controlling environmental effects from polymeric fibres production. They have already been utilised in a diverse range of

biomedical applications such as in tissue engineering and pharmaceuticals.[162] These polymers offer a significant contribution in curtailing the need for fossil fuels, which subsequently reduces carbon dioxide emissions. Natural polymers occur naturally or are produced by living organisms. Although, there is usually a requirement for these naturally occurring materials to be processed to obtain natural polymers and their mediocre transformation into polymers is yet a significant challenge.[163] It was previously mentioned that polymeric fibre manufacture is only just recently seeing some research focused on how to make the processes greener. However, green chemistry supported by green engineering has been seeking to improve efficiency and reduce health and environmental effects throughout the chemical manufacturing process.[164] Green polymers, on the other hand, are manufactured utilising green chemistry, which takes into consideration the sustainability of the entire process to produce the final polymer product. Natural polymers are not necessarily green polymers. A few principles the production process of green polymers encompass are wasteless manufacture processes, along with a high content of raw material, low carbon footprint, high-energy efficiency and the use of renewable energy. In recent times, PEO has shown a lot of attention due to non-volatility and very low toxicities.[165] They are currently in use in many pharmaceutical and cosmetic applications. Food chemistry and food processing are known to be ample sources to generate ideas on how to use renewable sources and apply green chemistry to create environmentally friendly polymers.[166] Regardless, synthetic polymers

such as PEO are still commonly utilised over natural polymers as they are functionally superior. The use of such low toxic synthetic polymers can still be more environmentally beneficial in comparison to other synthetic polymers due to features such as water solubility. Similarly, fibre production from water soluble cyclodextrin have also recently been successful and the use of such oligosaccharide polymeric fibres in a variety of functional applications such as in healthcare can enhance sustainability goals.[167]

Green solvents are a substitute for organic solvents. Organic solvents are classed as synthetic or natural, whereas similar to natural polymers, natural solvents are derived from living organisms. The global bio-based chemical market is growing in size and importance. Bio-based solvents such as glycerol and 2-methyltetrahydrofuran are often discussed as important introductions to the conventional repertoire of solvents.[53] One of the primary aims of green chemistry is the use of renewable sources over non-renewable feedstock.[71] Bio based solvents are considered green solvents as they take green chemistry into consideration as they are generally derived from agricultural crops. Green solvents help in contributing to making a chemical reaction green, to support green chemistry.[168] Green solvents are safe, generally biodegradable and very low in toxicity, throughout its production to its final product. Water is the greenest solvent taking into consideration the principles of green chemistry.[169] However, the use of a non-green polymer would cancel out the environmental benefits of this solvent in the production of polymeric fibres. Healthcare industry giants such as

Pfizer and GSK have adapted the use of green chemistry when making solvent selections in the recent past.[170] The use of very low toxic solvents or zero toxic solvents such as water is ideal for medical applications due to minimal hazards to humans. It is highly important that there is a strong focus on green chemistry in the making of polymeric fibres to further support the confidence in large-scale production and use in industries.

2.10 Fibre Morphology and Performance in Advanced Applications

Polymeric fibres are crucial constituents of many advanced technologies, finding applications in biomedical scaffolds, drug delivery systems, air and water filtration, energy storage and wearable electronics. The versatility of these fibres arises from their ability to be tailored to meet specific performance criteria. For many of these applications, finer fibre diameters (nanometre scale up to a few micrometres) are especially desirable as they optimise structural and functional properties. Finer fibres provide greater surface area, enhanced permeability and improved mechanical performance, making them indispensable in numerous fields.

In biomedical applications, finer fibres mimic the extracellular matrix (ECM) more effectively, promoting cell adhesion, proliferation and tissue regeneration.[171, 172] For example, scaffolds designed for

regenerative medicine benefit from the structural integrity of nanoscale fibres, which closely resemble the fibrous structures found in natural tissues.[173] In drug delivery, the fine diameters of fibres allow for controlled release profiles, enabling precise dosing and extended therapeutic effects.[174] Similarly, in air and water filtration, fibres with reduced diameters offer a larger surface area, facilitating the efficient capture of microscopic contaminants such as bacteria, viruses and fine particulates.[175] This increased filtration efficiency is critical in applications ranging from cleanroom environments to water purification systems.

Finer fibre diameters also enhance the performance of energy storage and conversion devices. In batteries and super-capacitors, for example, the high surface-to-volume ratio of fine fibres improves charge storage and facilitates ion transport.[176, 177] In catalytic applications, such as pollutant breakdown and water purification, smaller fibres allow for better catalyst immobilisation and increased reaction rates.[178] Moreover, in sensors, the enhanced sensitivity and rapid response times enabled by fine fibres are critical for detecting chemical or biological agents, along with mechanical properties such as strain with high precision.[179, 180]

In addition to fibre diameter, uniformity is another critical parameter that influences the performance of polymeric fibres. Fibre uniformity is often measured by the coefficient of variation (CV) of fibre diameters. Low CV values indicate high uniformity, a property that is essential for applications requiring consistent mechanical, structural, or functional characteristics. For instance, sensors made from conductive polymers

such as polyaniline (PANI) demonstrate more reliable performance when the fibres have uniform diameters, ensuring repeatability and precision in sensor responses.[181] However, not all applications demand strict uniformity. In some cases, higher CV values ranging from 20% to 40% can be acceptable or even beneficial. For instance, in filtration, fibres with a higher CV can create a more complex structure, increasing surface irregularities and promoting the separation of particles of different sizes.[182]

From a sustainability perspective, optimising fibre morphology aligns closely with the principles of Green Chemistry and Green Engineering by reducing energy consumption, minimising material waste and facilitating easier recycling and reuse. Additionally, achieving uniformity in fibre production ensures that fewer defective fibres are produced, minimising the waste generated during manufacturing and lowering the need for post-production reprocessing or discarding substandard materials.

2.11 Core-Sheath Fibres

In the area of polymeric fibre production, the evolution of core-sheath fibres offers a paradigm shift towards multifunctional materials with diverse applications.[183] The core-sheath structure of polymer fibres refers to forms of fibre where a ‘core’ polymeric fibre material is coated by another polymeric material forming the ‘sheath’ of the overall fibre. Depending on the application, the advantages of this fibre structure

include enhancing mechanical properties of the overall fibre, improving electrical conductivity in electronic applications and revamping electrochemical performance for efficient energy storage.[184, 185, 186, 187] These fibres are also used in filtration, for instance, the reusability and longevity of core-sheath structures are the subjects for research in the field of water treatment.[188]

Core-sheath fibres are often used in scaffolds and drug delivery systems.[189, 190, 191] A typical case is when the core can contain drugs or therapeutic agents, while the sheath provides a protective barrier. The core-sheath design also enables controlled release kinetics, enhancing the efficacy and duration of the therapeutic effect.[192, 193] Furthermore, the structure allows for biocompatibility where the use of biocompatible materials in the core-sheath fibre design ensures compatibility with biological systems.[194] This is essential for applications such as tissue engineering and implantable medical devices.[195]

However, the traditional techniques utilised in the fabrication of these fibres often entail inefficiencies leading to material wastage and high-power consumption.[25] Core-sheath polymeric fibres in the micrometer and nanometer scale are produced using multiple methods where each has its advantages along with limitations. The methods include template assisted methods, layer by layer assembly, solution blowing, gyration-based methods and electrospinning methods such as coaxial electrospinning and emulsion electrospinning.[32, 184, 196, 197, 198, 199]

2.12 Process Parameters of Pressure Spinning

Much of the research on pressure spinning has focused on optimising parameters such as the rotational speed and applied gas pressure to influence fibre morphology and production rate. However, the effects of collector distance, which is the distance from the orifice to the collector wall is comparatively understudied. This is a critical gap in understanding, as collector distance can significantly affect fibre formation, production efficiency and energy consumption. Figure 12 illustrates the primary parameters influencing fibre formation in pressure spinning. The pressure difference between the inside and outside of the vessel drives the polymer solution through the orifices. This pressure difference is generated by the combination of centrifugal force due to the vessel's rotation and the applied gas pressure. Specifically, the high-pressure region inside the vessel results from the combined effects of the applied gas pressure and the centrifugal force acting on the polymer solution as the vessel rotates. Outside the vessel, the pressure is atmospheric creating a significant gradient (pressure difference) that propels the solution through the orifices. The polymer solution's properties, particularly its viscosity, determine the flow rate through the orifices and influence the resulting fibre diameter and uniformity. Environmental conditions such as the temperature and relative humidity contribute to fibre properties such as morphology and production rate as they influence solvent evaporation.

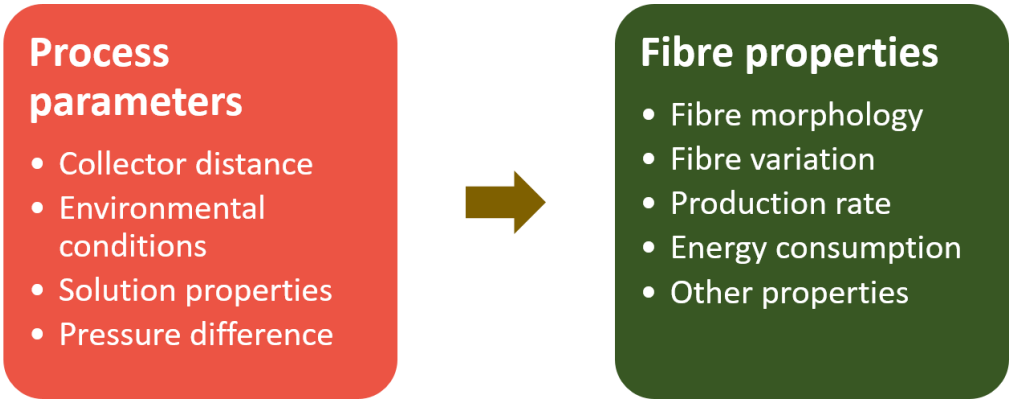

Figure 15. *Processing parameters and fibre output*

Collector distance is an essential parameter in fibre production techniques, such as pressure spinning and electrospinning.[32, 200] It determines the flight time of polymer jets from the orifice to the collector and, consequently, solvent evaporation, the extent of fibre thinning and solidification during this flight. Variations in collector distance can influence fibre diameter, uniformity and production rate. For instance, shorter collector distances may result in incomplete solvent evaporation, leading to thicker and less uniform fibres.[201] Conversely, longer collector distances provide more time for jet stretching and solvent evaporation, potentially resulting in finer and more uniform fibres. However, excessively long distances are known to lead to fibre breakage or reduced production efficiency. The effects of collector distance extend beyond fibre morphology. Fibre production rate and energy consumption are also influenced by this parameter. Optimising the collector distance can reduce material wastage during production by alleviating wasted polymer solution and minimise energy requirements, aligning with the principles of green engineering and sustainable manufacturing.

Similarly, the diameter of the vessel in pressure spinning plays a crucial role in influencing the centrifugal force, which consequently affects the pressure difference in the polymer solution when moving through the orifices of the vessel. The diameter of the vessel also remains an unexplored parameter in comparison to other parameters that affect the pressure difference in the pressure spinning process. According to Newton's second law of motion, the centrifugal force in the pressure spinning method increases with a larger vessel diameter and a larger rotational speed. A greater centrifugal force results in a higher-pressure gradient across the orifices, enhancing the extrusion of the polymer solution. This increased pressure difference can influence the jetting behaviour of the polymer solution, potentially leading to a higher production rate and variations in fibre morphology. Larger vessel diameters will allow for the generation of finer fibres due to the greater force driving the solution through the orifices. Conversely, smaller vessel diameters result in reduced centrifugal forces, potentially producing thicker fibres or limiting the production efficiency. Thus, vessel diameter is a critical parameter for tailoring fibre properties and optimising the process.

Fibre formation behaviour in pressure spinning is effectively governed by centrifugal forces acting on the polymer, which vary as a function of the vessel radius.[28] The centrifugal force generated in pressure spinning is governed by Newton's second law for rotational motion, defined as

$$F_c = mr\omega^2 \quad [1]$$

where 'F_c' is the centrifugal force, 'm' is the mass of the polymer solution, 'r' is the radial distance from the axis of rotation and 'ω' is the angular velocity. This relation indicates that for a fixed rotational speed, increasing the vessel radius results in a proportional increase in centrifugal force.

2.13 Concluding Remarks

The detailed literature review established that conventional fibre manufacturing methods present significant environmental and safety hazards due, critically, to their extreme energy demands and reliance on toxic organic solvents. This validated Pressure Spinning as the optimal low-energy manufacturing model, aligning with Green Engineering principles. Crucially, the move to sustainable production mandates safer chemistry, which led to the selection of Polyethylene Oxide (PEO) and Polyvinylpyrrolidone (PVP) for the experimental work. These synthetic polymers are favored because they are water-soluble (eliminating hazardous solvents) and possess very low toxicity, offering a superior functional middle ground compared to many mechanically weaker natural polymers.

Chapter 3 – Methodological Framework

This chapter outlines the experimental methods used throughout the research, which is structured around three core investigations presented in Chapters 4, 5 and 6. Each of these studies focuses on a specific aspect of polymeric fibre production via pressure spinning: (i) optimising pressure spinning parameters for efficiency and sustainability, (ii) developing sustainable core-sheath fibres via pressure spinning and (iii) assessing the influence of vessel geometry and collector distance on fibre morphology. While each chapter presents independent experiments, they share foundational methods and equipment, adapted to the unique aims of each study.

The chapter is therefore organised into three main sections, each aligned with the corresponding experimental focus of the subsequent chapters. However, due to methodological overlap (particularly in polymer selection, solution preparation and core process control parameters) relevant procedures may appear more than once, with necessary modifications explained in context. This structure was chosen to ensure clarity and continuity between method and analysis and to maintain traceability of experimental decisions across the different research strands.

Key considerations for selecting primary polymers, solution preparation and processing conditions were informed by preliminary testing and prior research. All tests were conducted under ambient conditions, with room temperatures maintained between 22–24 °C and relative humidity

ranging from 35–55%. The details herein ensure a clear understanding of the experimental workflow and the rationale behind the chosen methodologies, which aim to optimise fibre production while adhering to sustainability and efficiency.

The polymers, Polyethylene oxide (PEO) and Polyvinylpyrrolidone (PVP) were used in all studies to produce fibres via pressure spinning. Both PEO and PVP are polymers with very low toxicity in comparison to other synthetic polymers and water-soluble which as a consequence compliments the principles of Green Chemistry.[202] Natural polymers are more ‘greener’ however, synthetic polymers are functionally superior to natural polymers. Therefore, PEO and PVP was selected as a middle ground for this research as the polymers are both functional and less hazardous.

3.1 Sustainability of Submicrometre PEO and PVP Fibre Production

This study investigates how variations in the process control parameters, applied gas pressure and polymer solution concentration, influence energy consumption. The morphology of the fibres produce, specifically the fibre diameter is analysed to maintain the relevance of the produced fibres for applications, whereas the production rate is evaluated to understand material efficiency.

Changes in processing parameters significantly affect fibre properties such as diameter, morphology and internal structure, which in turn affect their suitability for applications in filtration, biomedical scaffolds and energy storage. While extensive research has focused on the physical and chemical properties of submicrometre polymeric fibres, including their morphology and functional applications, energy consumption and resource efficiency have received comparatively less attention. This study addresses this gap by evaluating the sustainability of submicrometre PEO and PVP fibre production, incorporating insights from Green Chemistry and Green Engineering to reduce environmental impacts.

3.1.1 Materials

Polyvinylpyrrolidone (PVP, Mw ≈ 1 300 000 g mol^{-1}, CAS: 9003-39-8) and polyethylene oxide (PEO, Mw ≈ 200 000 g mol^{-1}, CAS: 25322-68-3) were obtained from Sigma-Aldrich (Gillingham, UK) and used as received. The solvent used was distilled water.

3.1.2 Solution Preparation and Characterisation

PEO and PVP were dissolved separately in distilled water to produce polymer solutions of concentrations of 30 wt %, 35 wt %, 40 wt % and 50 wt %. Homogeneity was achieved by magnetically stirring all eight solutions for 24 hours at the ambient temperature (°C) and relative humidity (%). The PEO-H_2O and PVP-H_2O solution viscosities used in

this study were characterised using a calibrated Brookfield Viscosity-meter where a SC4-18 spindle model was used. The viscosity readings of PEO 30%, PEO 35%, PEO 40% and PEO 50% were 9418 mPa s, 19576 mPa s, 29544 mPa s, 59387 mPa s, respectively. Whereas the viscosity of PVP 30%, PVP 35%, PVP 40% and PVP 50% were, 3879 mPa s, 4255 mPa s, 7483 mPa s and 14983 mPa s, respectively. To obtain a valid measurement, the torque value of the rotational viscometer obtained must be between 10 % and 100 %, where higher torque values indicate better accuracy.[203] Hence, the all viscosity readings were captured at 100% torque for best accuracy.

3.1.3 Fibre Production and Characterisation

The pressure spinning device was set up with a laser tachometer aimed at the rotating vessel, a power meter connected to the motor along with a video recorder which was set up to obtain real-time readings of the rotary speed of the vessel and power usage during each experiment (Figure 13). Each concentration of PVP and PEO solution utilised in this experiment was subjected to an applied pressure of 0 MPa (no gas flow), 0.1 MPa and 0.2 MPa, which makes a total of 24 experimental samples of 4 ml each. The video recorder was used during each experiment to obtain real-time footage of the tachometer and power meter readings whilst evaluating the time the vessel was spun during the extrusion of fibres from the orifices of the vessel.

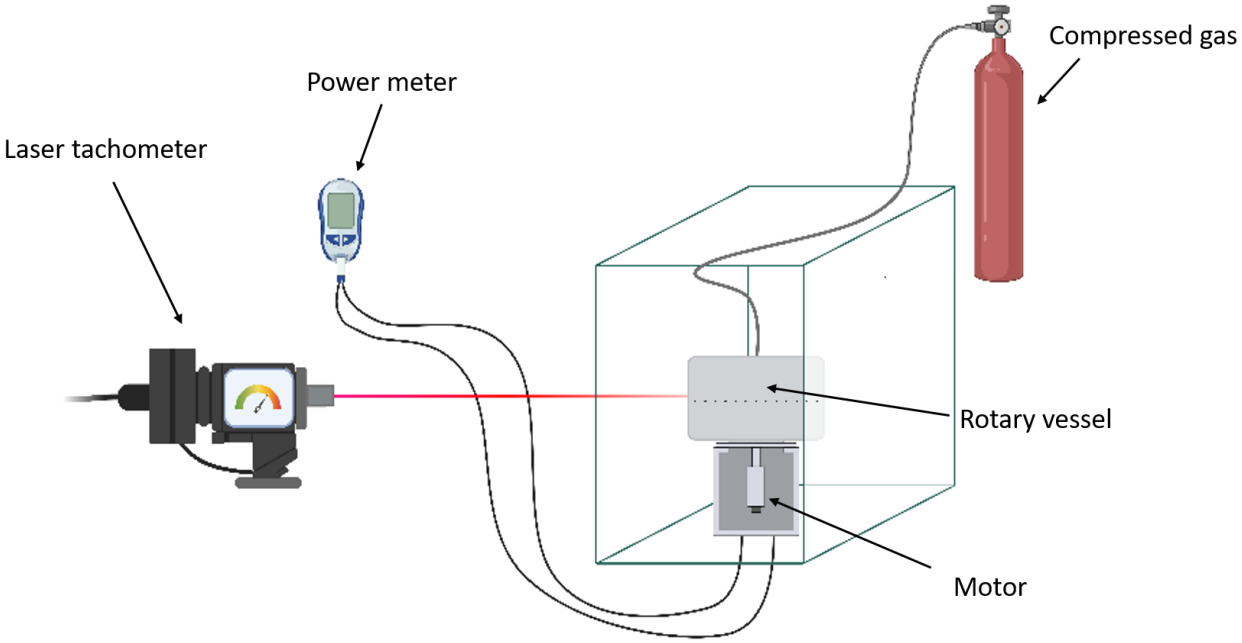

Figure 16. *Experimental setup*

The collector distance was set at 100 mm where the laser tachometer readings show that the vessel reached average rotatory speeds of 12,000 RPM, 11,400 RPM and 10,900 RPM at applied pressures of 0 MPa, 0.1 MPa and 0.2 MPa, respectively.

The production rate of the resulting fibres were calculated by measuring the mass of the fibres produced using a very sensitive scale and evaluating the time the fibres took to spin by reviewing video footage. The fibre diameter was evaluated using a scanning electron microscope (SEM). The micrographs acquired via SEM imaging were analysed using Image J software to obtain the average fibre diameter. Energy consumption was calculated using a power meter connected to the power socket which displays the power drawn by the motor when used.

The time taken to spin the fibres along with the average power meter reading was used to estimate the energy consumption.

3.2 Sustainable Production of Core-Sheath Polymer Fibres

The aim of this study is to systematically examine the impact of varying gas pressures and polymer concentrations on the production rate, fibre morphology and energy efficiency of core-sheath fibre configurations produce using pressure spinning. Core-sheath pressure spinning is a relatively simple and efficient process of producing core-sheath fibres where the operation involves rapidly extruding polymer solutions through a spinneret that consists of two reservoirs to hold the polymeric solutions of the core and the sheath of the fibres produced through co-axial orifices in the outer wall of the spinneret.

The spinning process of core-sheath pressure spinning can use excess energy to produce fibres if the required process control parameters are ill-understood. Forming fibres from polymer solutions using this method can require high rotary speeds, applied pressure magnitudes or changes to the environment such as temperature and humidity control, where achieving these parameters is typically associated with increasing power consumption. Attaining specific temperatures and humidity control typically requires energy-intensive heating or cooling systems, contributing to elevated power consumption. However, process optimisation with a focus on varying solution characteristics

rather than varying other parameters can augment energy efficiency in pressure spinning.

To enhance sustainability by maintaining minimal material wastage in core-sheath pressure spinning, there is a need to ensure all fibres formed have a core-sheath structure. Hence, it is pivotal to understand the fluid dynamics through the coaxial orifices that jet out core-sheath fibres. The cross-section of the orifices of the vessel shows that the solution used for the core travels through a tube, whereas the solution to form the sheath moves through an annulus to jet out core-sheath fibres out of the vessel (Fig. 1). Assessing the pressure difference at both ends of the core tube and sheath tube allows for the determination of the volumetric flow rates using Poiseuille's Law.[204]

3.2.1 Materials

Polyethylene Oxide (PEO) (Mw = 200,000 g mol^{-1}), Polyvinylpyrrolidone (PVP) (Mw = 1,300,000 g mol^{-1}) and Rhodamine B were obtained from Sigma Aldrich. Deionised water was used as a solvent for both PEO and PVP.

3.2.2 Solution Preparation

The solution for the core of the fibres used two concentrations of PVP dissolved in deionised water at concentrations of 50 wt.% and 60 wt.%. The solutions for the sheath of the fibres involved two concentrations of PEO 40 wt.% and 50 wt.%, along with Rhodamine 1 wt.% dissolved in

deionised water. All solutions were magnetically stirred for 24 hours to ensure homogeneity. The solution viscosities used in this study were characterised using a calibrated Brookfield Viscosity-meter along with a LV-4 (64) spindle attachment. The viscosities of the PEO solutions with PEO 40% and PEO 50% both mixed with Rhodamine 1% were evaluated to be 146 Pa s and 405 Pa s, respectively and PVP 50% and PVP 60% were shown to have viscosities of 124 Pa s and 240 Pa s.

In previous work the water-soluble polymers, PEO and PVP, were shown to effectively produce fibres via pressure spinning at the selected concentrations.[205] Specifically, these polymers demonstrated excellent solubility in the chosen solvent (deionised water), appropriate viscosity levels and compatibility with the process parameters, thus enabling the successful production of fibres. Hence, based on experience and the formability of the fibres, along with the range of the magnitudes of process control parameters that were available, PEO was selected for the sheath and PVP was selected for the core. PEO was deemed suitable for the sheath component, due to its excellent film-forming properties which contributes to the functionality of the resulting core-sheath fibres.[206] Conversely, PVP, recognised for its mechanical stability, was selected for the core to enhance structural integrity.[207]

3.2.3 Core-Sheath Pressure spinning

Figure 14 depicts the experimental setup along with a closer internal look at the open rotary vessel that shows the inner reservoir and the

outer reservoir that hold solution to produce the core and the sheath, respectively. The largest internal diameter of the inner reservoir (which is on the same plane as the connecting tubes to the orifices) is 36 mm. The largest internal diameter of the outer reservoir is 70 mm whereas the diameter of the external wall of the vessel up to the end of the orifices from opposing ends is 80 mm. The system includes a pressurised nitrogen gas inlet connected to the top of the vessel, controlled by a pressure gauge that can deliver up to 0.3 MPa of applied gas pressure into the vessel. The fibres produced for characterisation were sampled from the wall of the collector, which is 150 mm away from the external wall of the vessel.

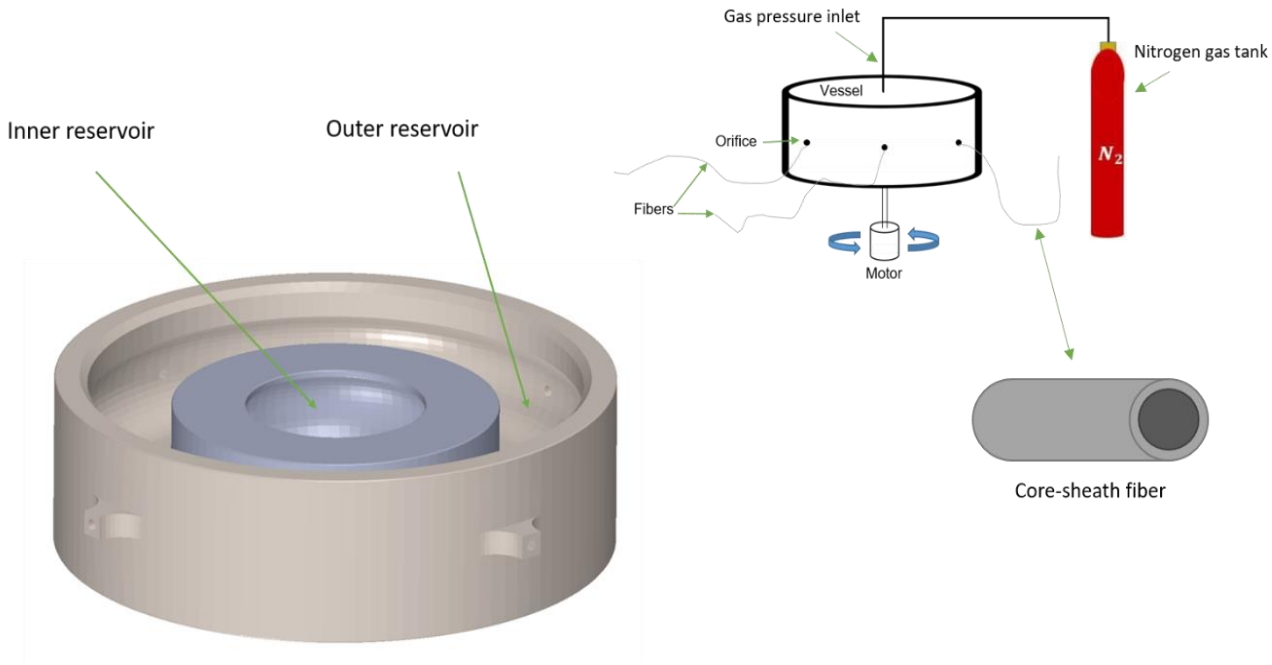

Figure 17. *Schematic of the core-sheath pressure spinning method (right), along with an internal view of the core-sheath vessel (left)*

The connecting tube that moves solution from the inner reservoir through the orifice to form the core of fibre is reinforced at the external

wall of the inner reservoir by fillets. Similar fillets are shown in Figure 14 in the outer wall of the vessel where the orifices are protruding 3 mm from the outer wall of the vessel. This allows the annulated tube of the orifice which forms the sheath of fibres to be of length 5 mm. Previous work has shown that the incorporation of a deeper orifice stabilises the flow state of the solution and better promotes the formation of polymer jets parallel to the axis of the tubes of the orifice.[208] This is paramount in the design of the core-sheath vessels as the sheath travels in a tube that is significantly shorter than the tube which forms the core. Hence, the addition of protruding orifices allows for better formation of both the core and the sheath polymer jets. Furthermore, the incorporation of fillets to the design maintains the concentricity of the orifice. It is important for the tubes in the orifice to have a common center to support a uniform width of the sheath around the core of the fibres produced.

The vessel also uses fillets in the internal walls of both the outer and inner reservoirs. The fluid behaviour within the reservoir of vessel when spun is known to have a parabolic shape as it reaches the tubes in the inner walls of gyration based fibre manufacture vessels.[32] Therefore, the fillets within the internal walls of the reservoirs support the movement of solution and its necessity is further exemplified when dealing with high viscous polymer solution as in this study.

3.2.4 Fibre Production

Core-sheath fibres were generated by loading both reservoirs with 2 ml of solution and in all experiments, spinning was carried out for 30 s to maintain consistency throughout the analysis. However, for each core-sheath polymer configuration, the spinning process was commenced with the extrusion of PVP solution loaded in the core leaving the outer reservoir for the sheath empty, to produce PVP fibres. The process was then repeated by loading both PVP solution in the core and PEO solution mixed with Rhodamine 1% in the sheath leading to the formation of core-sheath fibres characterised by a visible pink-dyed sheath surrounding an undyed PVP core. Hence, two samples were obtained at the same magnitudes of effecting parameters, where the first is 'core-only' PVP fibres and the second sample is core-sheath fibres. The mass of fibre samples was measured using a microscale and the production rates were evaluated by dividing the mass of the samples by the spin time. The difference of the two production rates of the samples was used to evaluate the production rate of the sheath in the core-sheath sample.

3.2.5 Evaluation of Process Parameters

The overall experimental results are categorised according to the configuration of the core-sheath configurations as shown in Tables 1, 2, 3 and 4. The Nichibo DC motor connected to the vessel has an unloaded power rating of 21.2 W, which was seen to draw a power of 28 W when run with the vessel attached. The power increased to 28.7 W, 30.7 W and 33.7 W at applied gas pressure magnitudes of 0.1 MPa, 0.2 MPa

and 0.3 MPa, respectively. The power readings were captured using a Maxcio Energy Monitor which measured real-time power drawn by the motor, whereas the rotational speed of the vessel was measured using a laser tachometer.

3.2.6 Fibre Characterisation

Optical microscopy was used to confirm the formation of a sheath around the core to result in core-sheath fibres as shown in Figure 15. The overall fibre dimensions were evaluated using a ZEISS Gemini SEM 360 Scanning Electron Microscope (SEM). The difference in the fibre diameter of the two samples (core only and core-sheath) was used to evaluate the width (or radius) of the sheath of the resulting core-sheath fibres. Fibre dimensions were calculated using Image J analysis where a 100 randomly selected fibres from SEM images were averaged.

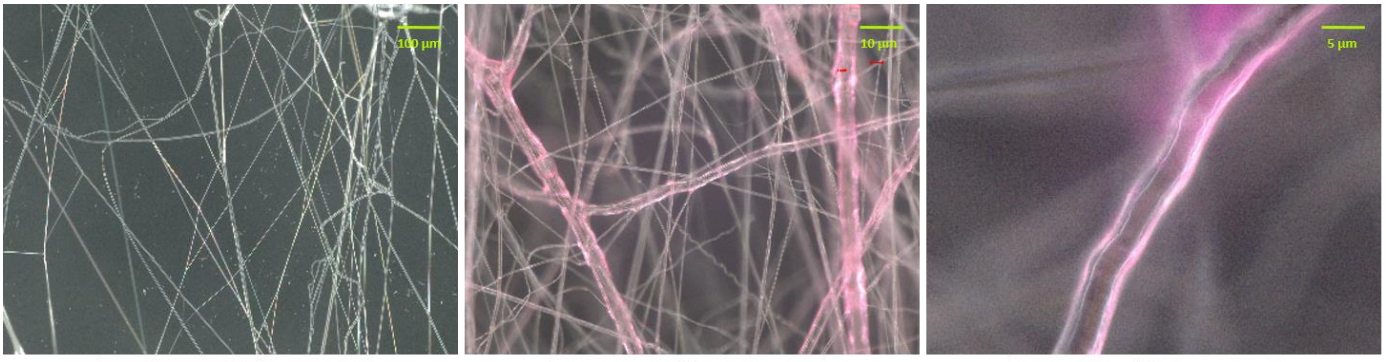

Figure 18. *(a) Core only PVP fibres. (b) Core-sheath PEO-PVP fibres. (c) Close-up of core-sheath fibre*

To further confirm the presence of PEO and PVP when producing core-sheath fibres, Fourier Transform Infrared Spectroscopy (FTIR) was

undertaken using a Thermo Fisher Scientific, Nicolet iS50 FTIR. 2 mg of PEO, PVP and core-sheath fibre samples were placed on the ATR crystal and evaluated over 10 rounds in the range of 4000–1000 cm^{-1} at a resolution of 4 cm^{-1} to record the measurements.

3.2.7 Analytical Modelling

The volume flow rates of the polymer solutions used in this study were evaluated by considering the dimensions of the rotary vessel (which has an external diameter of 80 mm) and the magnitude of rotary speed, fluid viscosities and pressure differences. The dimensions of the vessel are shown in Figure 16 where a close-up cross-sectional schematic of the vessel is included (within the red box). It is assumed that flow within the coaxial system of the vessel is laminar, considering relatively higher viscosities of the polymeric solutions used in this study.[209]

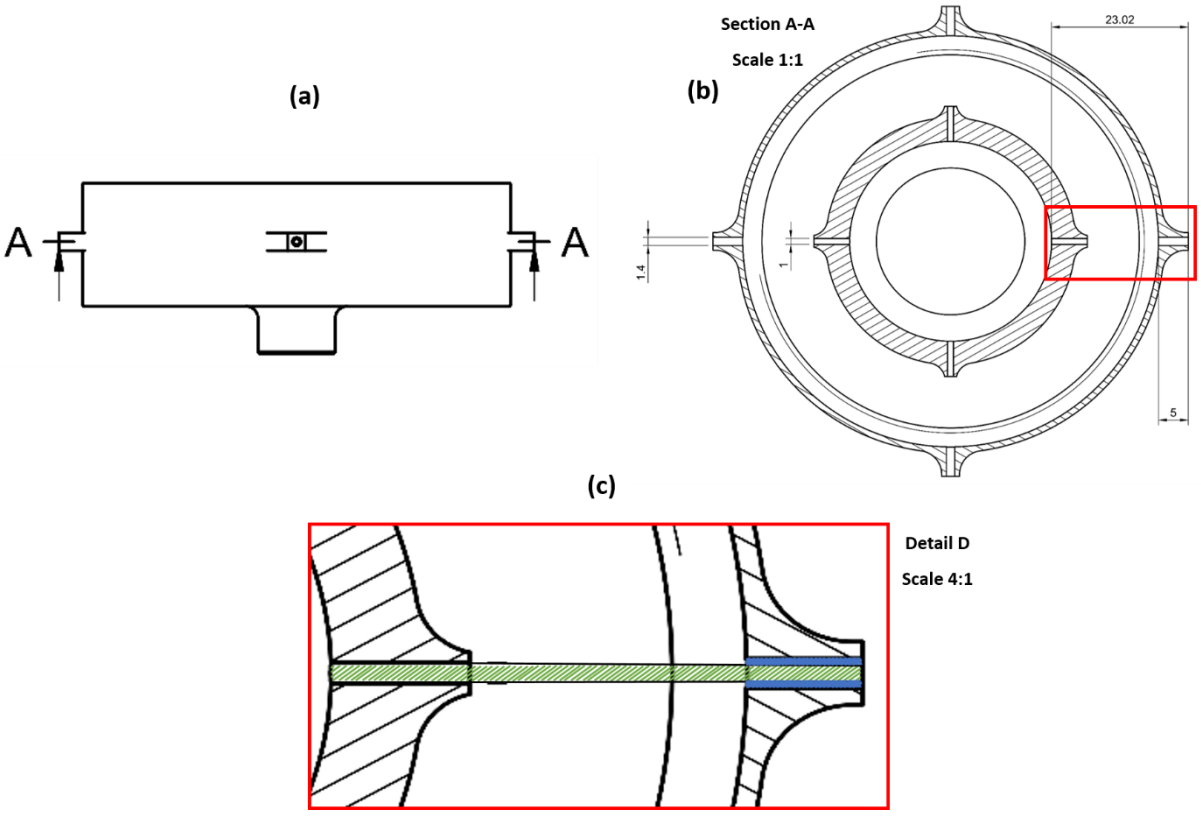

Figure 19. *(a) External view of the rotary vessel. (b) Cross-sectional view of section A-A. (c) Close up view of detail D, portraying the simple tube (green) and annular tube (left in blue) connecting the inner reservoir and outer reservoir, respectively, to the external wall*

Using the dimensions of the simple tube of radius r (r = 0.34 mm) that connects the inner reservoir to the orifice, the volumetric flowrate of the polymeric solution used to produce the core of the core-sheath fibres formed is calculated as follows.[210]

$$Q_{core} = \frac{\pi(\Delta P)r^4}{8 \times \text{pipe length} \times \text{viscosity}}$$

The volumetric flow rate of the solution to produce the sheath is calculated as follows considering the solution moves through an annular tube (indicated in Figure 16) to reach the orifice. If r_2 and r_1 are the external radius (0.7 mm) and internal radius (0.5 mm) of the annular tube.

$$Q_{sheath} = \frac{\pi({r_2}^2 - {r_1}^2)}{8 \times \text{viscosity}}\left(({r_2}^2 + {r_1}^2) - \frac{({r_2}^2 - {r_1}^2)}{\ln\left(\frac{r_2}{r_1}\right)} \right)\frac{\Delta P}{\text{annular tube length}}$$

The effective pressure difference in both the simple tube and the annular tube is calculated as follows.

$$\Delta P = P_1 + \frac{1}{2}\rho\omega^2(R^2 - {R_0}^2)$$

'P_1' is the magnitude of applied gas pressure, 'R' is the distance from the center of the vessel to the end of the orifice (tube length), 'R_0' is the inner radius of the reservoir, 'ρ' is the density of the fluid and 'ω' is the angular velocity.

3.3 Optimising Fibre Morphology and Production Efficiency in Pressure Spinning through Vessel and Collector Design

This study aims to investigate how variations in collector distance, vessel diameter and other parameters influences production rate, energy consumption, fibre diameter and fibre uniformity. A higher production rate ensures the scalability of fibre production, making it feasible for industrial applications. Lower energy consumption aligns with sustainability goals, reducing the environmental impact of the process. Finally, achieving smaller fibre diameters is particularly desirable for applications requiring high surface area-to-volume ratios, such as filtration, energy storage and biomedical scaffolding. By systematically studying the role of collector distance, this research seeks to optimise pressure spinning for both performance and sustainability. The findings will contribute to a deeper understanding of the process, paving the way for the efficient production of high-quality polymeric fibres with application-specific characteristics. By addressing the influence of collector distance and vessel diameter, this study

contributes to the broader effort of making fibre production more sustainable and environmentally friendly.

3.3.1 Materials

Polyethylene Oxide (PEO) (Mw = 200,000 g mol^{-1}) and Polyvinylpyrrolidone (PVP) (Mw = 1,300,000 g mol^{-1}) were obtained from Sigma Aldrich (Gillingham, UK). Deionised water was used as a solvent for both PEO and PVP.

3.3.2 Experimental Setup

Two rotary vessels of diameters 60 mm and 75 mm were used in this study. The vessel is connected to a motor (RS Pro 238-9759, Corby, United Kingdom) which is fixed to a laboratory scissor jack. This sub-assembly is surround by a conical shaped collector, where the scissor jack can be adjusted to move the vessel vertically to obtain the desired perpendicular distance from the orifices of the vessel to the collector wall. The system has two conical shaped collector walls where the internal wall can be removed to obtain larger collector distances (Figure 17). To enhance safety and minimise interference from external air drafts, the top of the collector is covered with a screen. This screen also features slits or gaps to facilitate airflow generated by the vessel's rotation, ensuring stable fibre deposition. This screen is removed in Figure 17 to show the primary components within.

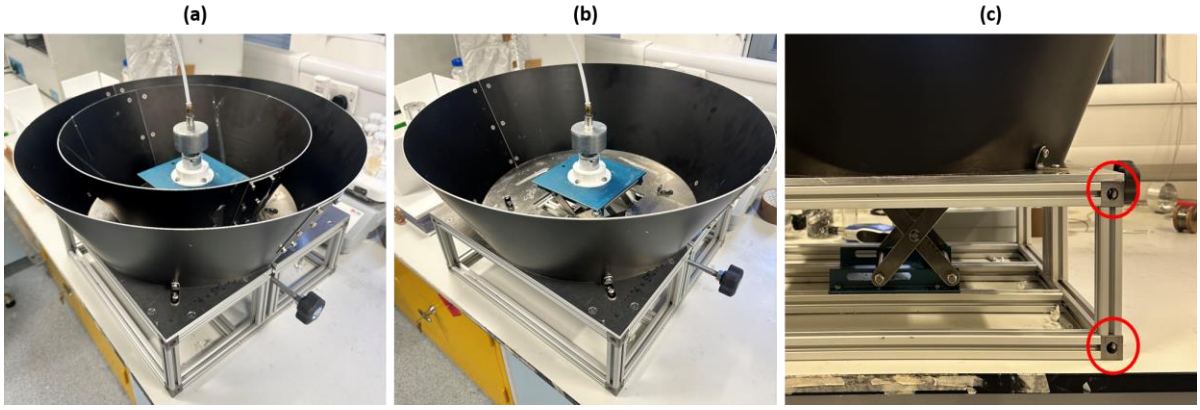

Figure 20. *(a) Pressure spinning set up with inner wall. (b) Experimental set up where fibres are deposited in the outer wall. (c) Aluminium clips (circled in red) designed to hold the frames of the platform in places.*

Anodised aluminum sheets were cut into arc shapes to match the collector's surface area using a water-jet cutter (iCUTwater SMART, Eiterfeld, Germany). These sheets were subsequently rolled into a conical shape to form the collector walls using a sheet metal rolling machine. The same water-jet cutter was used to shape the aluminum components for the platform frame, which serves as the foundation for the collector walls and supports the laboratory scissor jack and motor subassembly. The platform is assembled using custom-designed aluminum cubic clips, as shown in Figure 17c. These clips were produced via additive manufacturing using a metal 3D printer (Markforged Metal X, Billerica, MA, USA). Post-printing, the clips were processed in a washer (Markforged Wash 1, Billerica, MA, USA) for 8 hours and then sintered in a furnace (Markforged Sinter Furnace 2, Billerica, MA, USA) for 30 hours to achieve the final form.

3.3.3 Evaluation of Processing Parameters

PEO and PVP were dissolved separately in distilled water to produce polymer solutions of concentrations of 40 wt% and 50 wt%. Homogeneity was achieved by magnetically stirring all solutions for 24 h at ambient temperature (~20°C) and relative humidity (~50%).

The solution viscosities were characterised using a calibrated Brookfield Viscosity-meter (AMETEK Brookfield, Harlow, UK) along with a LV-4 (64) spindle attachment (similar to Section 3.2.2). The viscosities of the PEO solutions with PEO 40% and PEO 50% were evaluated to be 146 Pa s and 405 Pa s, respectively, whereas PVP 40% and PVP 50% were shown to have viscosities of 39 Pa s and 146 Pa s, respectively.

The RS PRO Geared DC Motor connected to the vessel has an unloaded power rating of 21.2 W. The power readings were captured using a Maxcio Energy Monitor (Shenzhen, China), which measured the real-time power drawn by the motor. The ⌀60 mm vessel drew 32 W with the vessel attached and the power increased to 36 W and 38 W at applied gas pressures of 0.1 MPa and 0.2 MPa, respectively. For the ⌀75 mm vessel, the power usage was 27 W, increasing to 33 W and 38 W at the same applied pressures.

The rotational speed of the vessel was measured using a laser tachometer. Where both vessels maintained an approximate rotational speed of 13,000 RPM throughout all experiments. A total of 72 experiments were conducted using the 4 polymer solutions, 3 magnitudes of applied gas pressure, 3 collector distances and 2 vessels.

The spinning process lasted for 30 seconds from the time fibres began extruding from the orifices.

3.3.4 Fibre Characterisation

The fibre depositions were weighed using a high-precision microscale balance (A&D FZ-300i-WP-EC, Abingdon, United Kingdom) to determine the mass of fibres produced. The average of three samples were taken under each experimental condition. The overall fibre dimensions were then assessed using a Scanning Electron Microscope (GeminiSEM 360, Carl Zeiss Microscopy GmbH, Oberkochen, Germany). Prior to imaging, the fibre samples were coated with a thin layer of gold using a sputtering coater (Leica EM ACE600, Wetzlar, Germany)), with a coating thickness of approximately 3 nm. Gold coating was performed to enhance the conductivity of the samples, thereby reducing charging effects and improving image resolution and contrast during SEM analysis. Fibre dimensions were subsequently calculated using ImageJ software (version 1.49) and Orientation J. For this analysis, 300 fibres were randomly selected from the SEM images and their diameters were averaged to ensure statistical reliability.

Chapter 4 - Sustainability of Submicrometre PEO and PVP Fibre Production

This chapter explores the influence of varying polymer concentrations and applied pressures on the energy consumption, production efficiency and fibre morphology in the pressure spinning of PEO and PVP polymer solutions. By systematically analysing power consumption, production rate and fibre characteristics under consistent rotary speeds and applied gas pressures, the study aims to identify the optimal operational parameters for energy-efficient fibre production. A key observation is the minimal impact of applied pressure increases from 0.1 MPa to 0.2 MPa on production efficiency and fibre morphology compared to the pronounced effects observed between 0 MPa and 0.1 MPa. Additionally, the role of polymer viscosity in dictating energy requirements is examined, underscoring the proportional relationship between viscosity and energy consumption. Overall, the findings offer a basis into balancing efficiency, sustainability and fibre quality in the pressure spinning process.

4.1 Overall Findings

A notable change in the rotary speed of the vessel was not identified with the use of the different concentrations of PEO and PVP. On average, under the same effecting parameters (RPM, concentration and Pressure), the power consumption was very similar, where an average reading of 31.5 W is shown at 0 MPa for each sample, which increased

by 5 W to an average of 36.4 W when a pressure of 0.1 MPa is applied and an average of 37.8 W at an applied pressure of 0.2 MPa. However, the actual power readings for each sample is utilised in this study as shown in Table 8 to maintain the accuracy of energy consumption findings.

Polymer-Solvent	% (w/v)	Pressure (MPa)	Speed (RPM)	Power (W)	Fibre mass (g)	Spin time (s)	Production rate (g/hr)	Diameter (μm)	Energy (J)
PEO-H_2O	30	0	12000	30.7	0.057	35	5.9	0.615 ± 0.204	1075
PEO-H_2O	30	0.1	11400	35.7	0.088	30	10.6	0.387 ± 0.119	1071
PEO-H_2O	30	0.2	10900	38	0.089	30	10.7	0.316 ± 0.132	1140
PEO-H_2O	35	0	12000	31	0.079	45	6.32	0.627 ± 0.231	1395
PEO-H_2O	35	0.1	11400	35.1	0.126	40	11.34	0.371 ± 0.132	1404
PEO-H_2O	35	0.2	10900	37.1	0.138	40	12.42	0.321 ± 0.127	1484
PEO-H_2O	40	0	12000	32	0.148	60	8.88	1.215 ± 0.427	1920
PEO-H_2O	40	0.1	11400	37	0.174	50	12.528	0.631 ± 0.292	1850
PEO-H_2O	40	0.2	10900	38	0.176	50	12.672	0.578 ± 0.134	1900
PEO-H_2O	50	0	12000	32	0.187	75	8.976	2.385 ± 0.714	2400
PEO-H_2O	50	0.1	11400	37	0.226	60	13.56	1.089 ± 0.455	2220
PEO-H_2O	50	0.2	10900	38	0.233	60	13.98	0.889 ± 0.389	2280
PVP-H_2O	30	0	12000	31	0.019	10	6.84	0.640 ± 0.286	310
PVP-H_2O	30	0.1	11400	35	0.031	8	13.95	0.455 ± 0.145	280
PVP-H_2O	30	0.2	10900	37	0.037	8	16.65	0.451 ± 0.159	296
PVP-H_2O	35	0	12000	32	0.021	10	7.56	0.704 ± 0.266	320
PVP-H_2O	35	0.1	11400	37	0.044	8	19.8	0.532 ± 0.205	296
PVP-H_2O	35	0.2	10900	38	0.049	8	22.05	0.501 ± 0.214	304
PVP-H_2O	40	0	12000	32	0.075	15	18	1.869 ± 0.780	480
PVP-H_2O	40	0.1	11400	37	0.089	12	26.7	0.941 ± 0.518	444
PVP-H_2O	40	0.2	10900	38	0.093	12	27.9	0.627 ± 0.197	456
PVP-H_2O	50	0	12,000	32	0.087	15	20.88	2.642 ± 0.833	480
PVP-H_2O	50	0.1	11,400	37	0.117	13	32.04	1.232 ± 0.652	481
PVP-H_2O	50	0.2	10,900	38	0.121	13	33.51	1.086 ± 0.503	494

Table 8. *Experimental results of solutions*

Under the same magnitudes of affecting parameters, the energy consumption to produce PVP fibres is shown to be lower than the energy consumption to produce PEO. This is primarily due to the lower spin time required to produce PVP fibres using the same volume of polymeric solution as PEO, where the spin time is attributed to the viscosity of the solutions along with the applied pressure magnitudes. The production rate of PVP fibres was evaluated to be higher than PEO fibres under the same magnitudes of effecting parameters, which is attributed to the molecular weight of PVP (Mw ≈ 1,300,000) used in the study being higher than that of PEO (Mw ≈ 200,000). This is due to the viscosity and subsequently the spin time of PVP fibres being significantly lower than that of PEO fibres using the same volume of polymeric solution results in a higher production rate for producing PVP fibres.

4.2 Effect of Applied Gas Pressure on Fibre Diameter

Figure 18 illustrates that the application of pressure decreased the fibre diameter of both PEO and PVP at all concentrations. However, this reduction is less significant when the applied gas pressure magnitude is increased from 0.1 MPa to 0.2 MPa, in comparison to the reduction of fibre diameter caused by the application of a pressure magnitude of 0.1 MPa from no pressure.

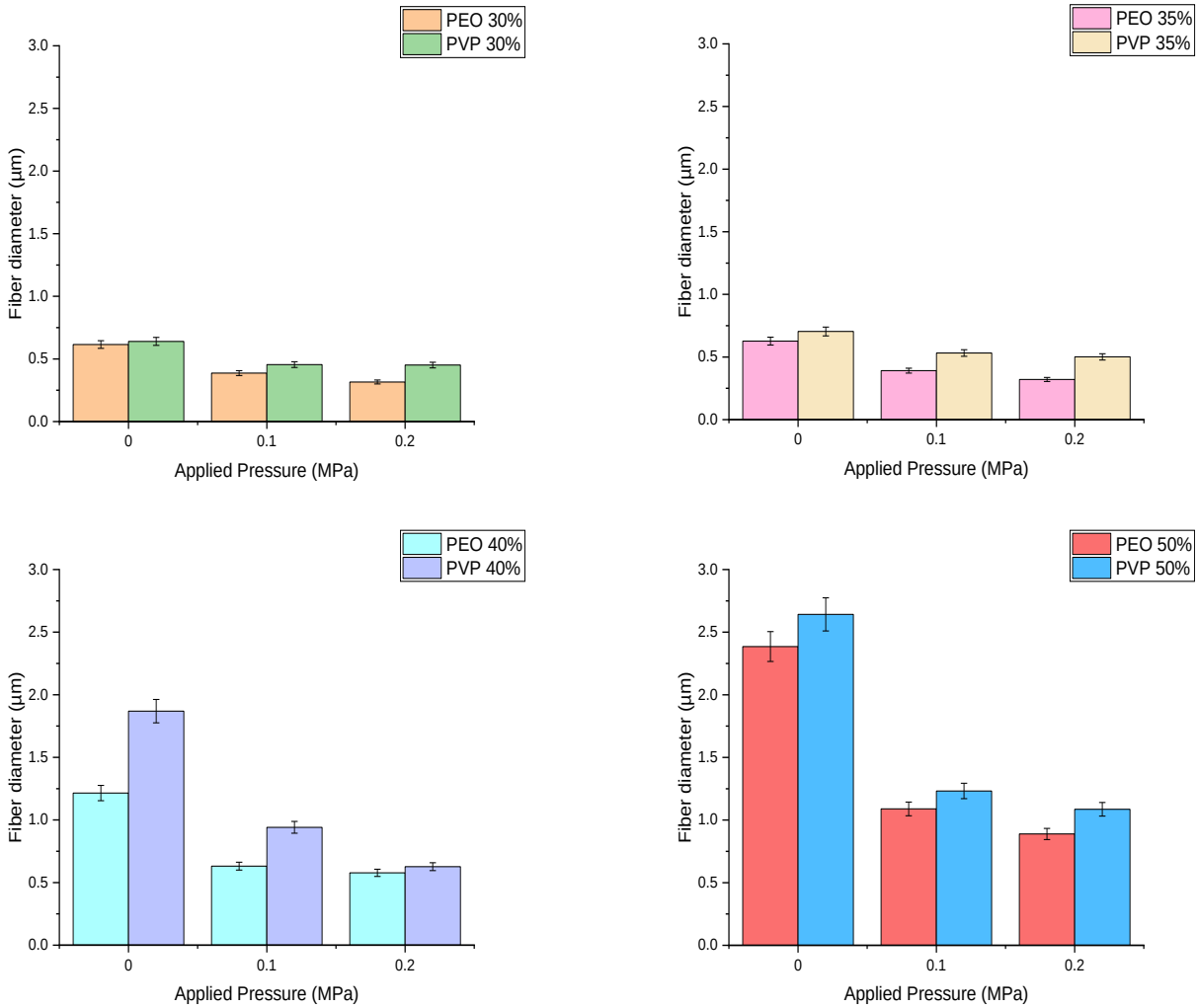

Figure 21. *The effects of applied pressure magnitude on fibre diameter for PEO-H2O and PVP-H2O samples (where the error bars represent the standard deviation)*

PEO fibres were shown to have a relatively smaller diameter in comparison to PVP fibres under the same magnitudes of the three effecting parameters taken into consideration in this study (rotary speed, pressure and the concentration of the polymeric solution). For instance, at a concentration 40% under an applied pressure of 0.1 MPa, PEO fibres resulted in an average diameter of 0.631 µm, whereas PVP fibres resulted in an average diameter of 0.941 µm as depicted in Figure 19.

The larger diameter of PVP fibres is attributed to the higher molecular weight of the polymer used, which affects the ability of the polymer solution to flow and form thin fibres.

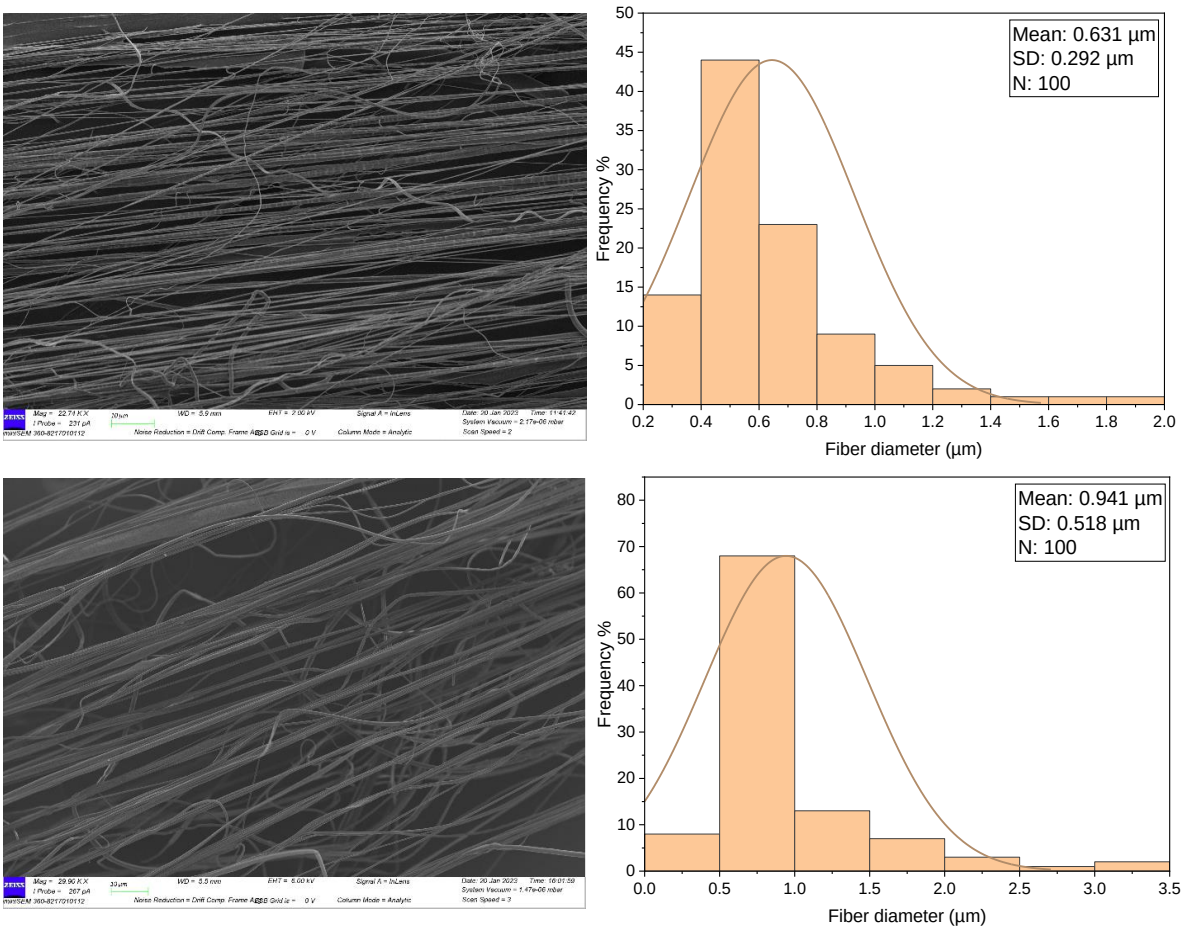

Figure 22. *Scanning Electron Microscope Image of PEO 40% (top left) and PVP 40% fibres (bottom left), along with corresponding fibre distribution graphs (right) produced under an applied pressure of 0.1 MPa (where the error bars represent the standard deviation)*

The results in Table 8 show that for all four concentrations of PEO and PVP, the difference in the mass of fibres collected was significantly less than the difference in the mass of fibres collected at 0 MPa and 0.1 MPa. Regardless, a small increase in the mass of fibres collected with the

application of pressure from 0.1 MPa to 0.2 MPa is still seen. The spin time taken for fibres to be extruded also showed that the application of a pressure of 0.2 MPa did not show a noticeable difference in comparison to an applied pressure of 0.1 MPa for both PEO and PVP at all concentrations. This lack of variation in spin time indicates that while increased pressure facilitates fibre formation, it does not necessarily translate into faster extrusion rates.

The findings suggest that while higher pressures (up to 0.2 MPa) contribute incrementally to fibre mass, they do not provide significant gains in production efficiency. Instead, the results highlight the importance of identifying an optimal pressure range that maximises fibre yield without unnecessarily increasing energy consumption or compromising fibre quality.

4.3 Effect of Applied Gas Pressure on Production Rate and Energy Consumption

Figure 20 indicates that both PVP and PEO fibres experienced a decrease in fibre diameter along with an increase in production rate under increasing pressure magnitudes. However, the increase in production rate is less significant from 0.1 MPa to 0.2 MPa in comparison to the increase from 0 MPa to 0.1 MPa. In general, the production rate for PVP fibres was lower than that of PEO fibres, particularly at higher concentrations and pressures. For instance, at a concentration of 40%, the PEO fibres had a production rate of 12.5 g/hr

at 0.1 MPa pressure, while the PVP fibres had a production rate of 26.7 g/hr under the same conditions. This is attributed to the differences in the solution properties of the two polymers as seen in Table 8, which can affect their ability to flow and form fibres under pressure.

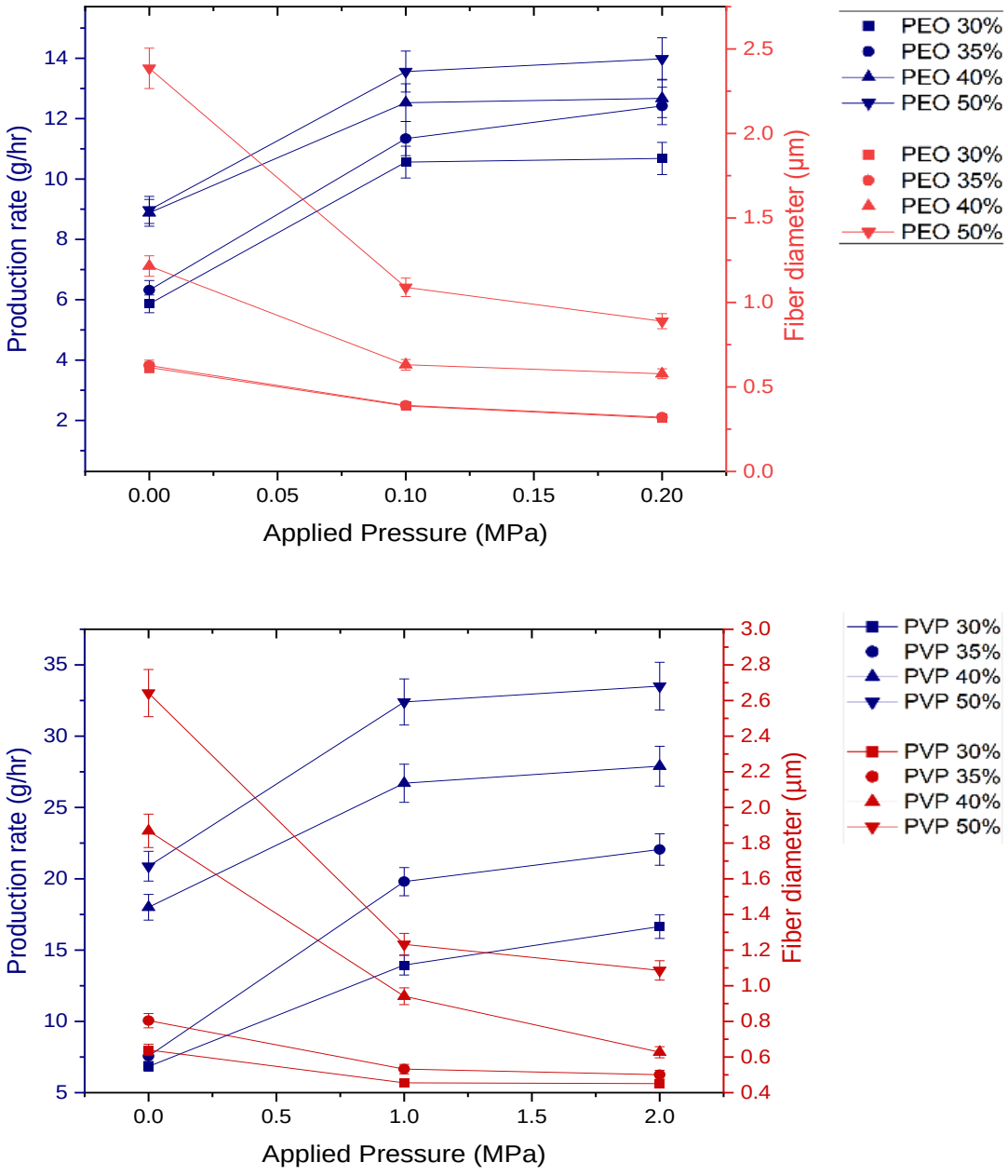

Figure 23. *The effects of applied pressure magnitudes on fibre production rate (blue) and fibre diameter (red) for PVP-H2O (right) and PEO-H2O (left) samples (where the error bars represent the standard deviation)*

Although the magnitudes of the energy consumption were comparable at the same magnitudes of the effecting parameters, a notable difference in the total energy to produce both PVP and PEO fibres is comprehended (Figure 21). The energy consumption is directly proportional to the time taken for each 4 ml sample to produce fibres.

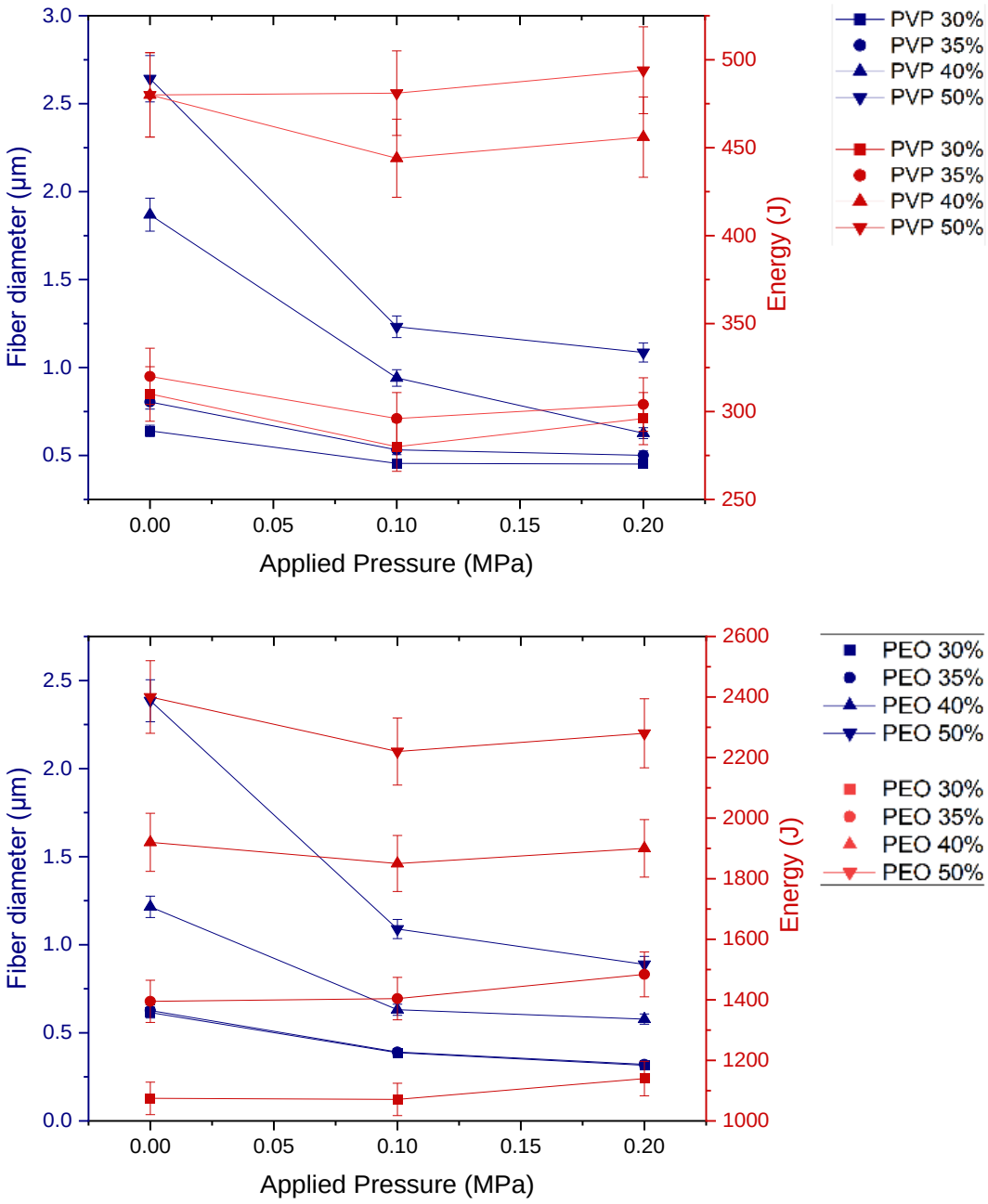

Figure 24. *The effects of applied pressure magnitudes on fibre diameter (blue) and energy consumption (red) for PVP-H2O (left) and PEO-H2O*

PEO used a significantly longer time in comparison to PVP to produce fibres at the same concentrations and magnitudes of pressures as seen in Table 8. Hence, the energy consumption for PVP fibres was lower than that of PEO fibres at all concentrations and pressures tested. This is due to differences in the rheological properties of the two polymer solutions as well as the differences in their ability to form fibres under pressure.

The increase of applied gas pressure from 0.1 MPa to 0.2 MPa does not show a better performance in energy consumption to produce PEO and PVP fibres in comparison to an increase in applied pressure from 0 MPa to 0.1 MPa. This is due to the result of an application of pressure of 0.2 MPa which did not show a perceptible improvement in the time taken to form fibres but increased the power consumption to an average of 37.4 W across all samples as seen in Table 8. The experimental video footage did not identify a significant decrease in spin time due to the increase in applied gas pressure from 0.1 MPa to 0.2 MPa. Hence the energy consumption was evaluated to be higher at an applied gas pressure magnitude of 0.2 MPa in comparison to an applied gas pressure of 0.1 MPa for most of the samples in the study as elucidated in Figure 22.

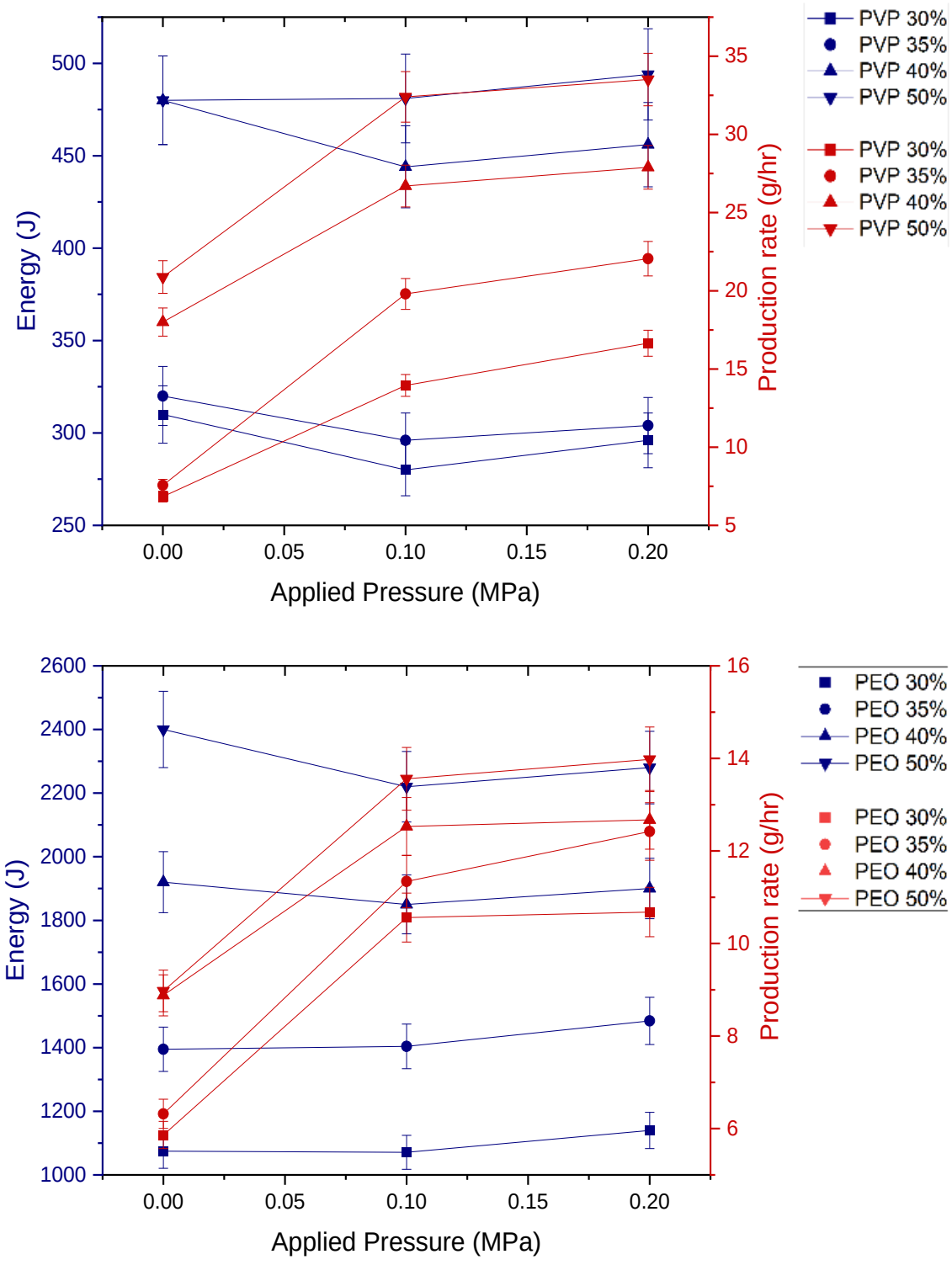

Figure 25. *The effects of applied pressure magnitudes on fibre production rate (red) and energy consumption (blue) for PVP-H2O (left) and PEO-H2O (right) samples (where the error bars represent the standard deviation)*

Overall, it can be established that to result in optimum efficiency in the range of magnitudes used in this study is to produce PVP and PEO fibres, utilising a pressure magnitude of 0.1 MPa is ideal taking into consideration the range of magnitudes of affecting parameters used in this study. Optimum efficiency considers the highest production rate and lowest fibre diameter (depending on applications) using the lowest energy consumption.

4.4 Role of Viscosity in Fibre Formation and Energy Demand

Figure 23 shows that as the viscosity of a specific polymer solution increases, the energy consumption required for the pressure spinning process also tends to increase. This is particularly evident when comparing the data for PEO 30%, 35%, 40% and 50%, where the highest viscosity polymer solution (PEO 50%) required the most energy to produce fibres with the desired characteristics. One reason for this trend is that higher viscosity solutions require higher pressures and speeds to achieve the desired fibre diameter and production rate, which in turn require more energy.[108] Furthermore, highly viscous solutions also require more power to overcome the increased resistance to flow through the spinning equipment.

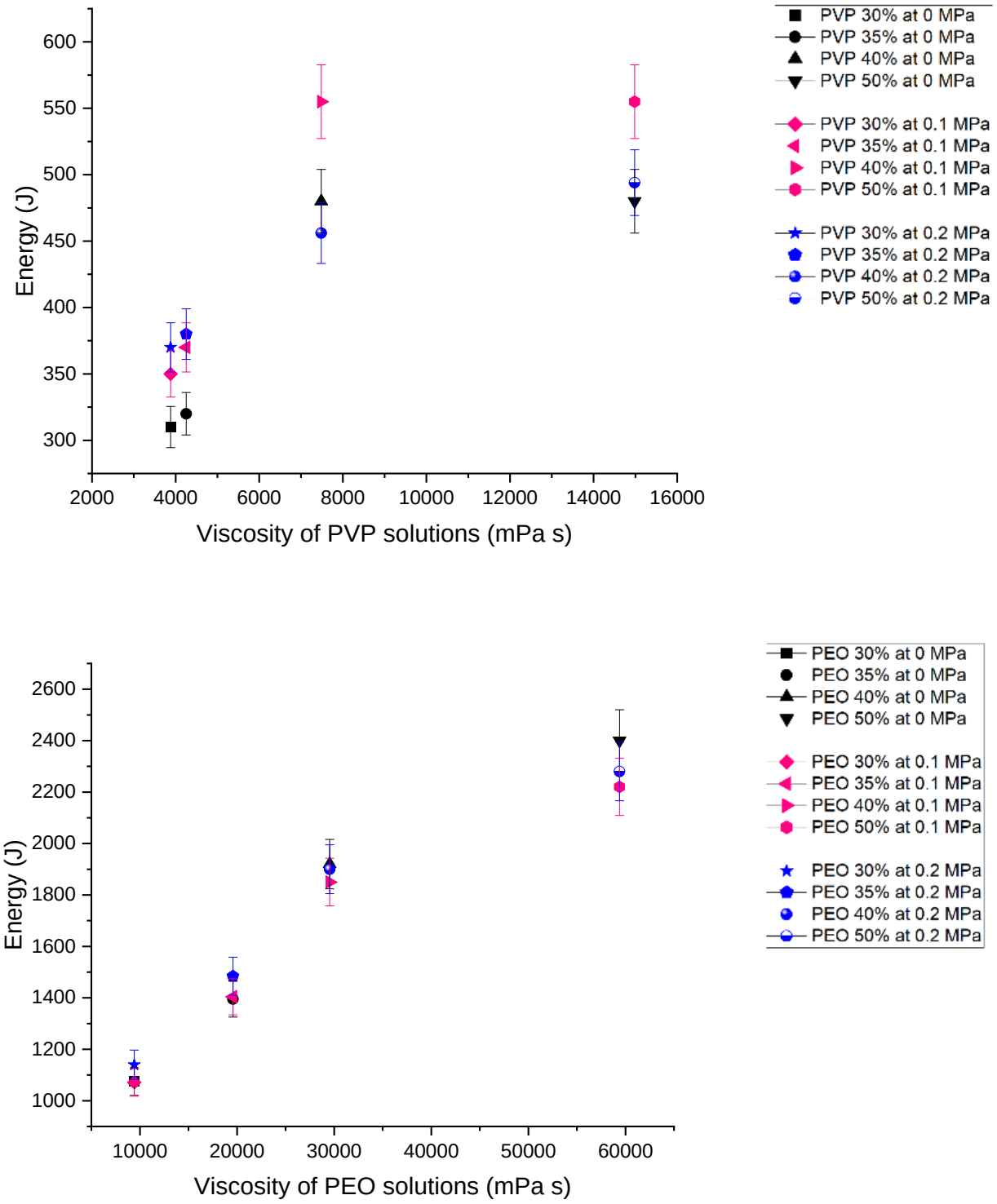

Figure 26. *Energy consumption to produce fibres at viscosities associated with each concentration of PVP (left) and PEO (right) (where the error bars represent the standard deviation)*

In this study, the optimal concentration that balances efficiency and performance for both polymers was established as the 35% solution under an applied pressure of 0.2 MPa. This concentration represents the ideal compromise in the process parameters. For PEO H_2O, the 35% solution at 0.2 MPa achieves a fine fibre diameter of 0.321 µm while maintaining a high production rate of 12.42 g/hr. Similarly, for PVP H_2O, the 35% concentration at 0.2 MPa delivers a high production rate of 22.05 g/hr while keeping energy consumption 304 J near the lowest point recorded for this polymer. This optimal 35% concentration successfully avoids the significant high-viscosity energy penalty observed in the 50% solutions (where PEO consumption, for example, reached up to 2400 J at 0 MPa). Simultaneously, it ensures maximum mass yield and production rate compared to the lower 30% concentration, thus achieving the maximum overall efficiency for the experimental conditions used.

4.5 Sustainability Analysis of the Fibre Formation Process

Overall the results show that PVP performed better than PEO in terms of energy efficiency and production rate under the same magnitudes of effecting parameters. However, PEO was superior in terms of obtaining lower fibre dimeters under the same magnitudes of effecting parameters. Hence, it is inconclusive if PEO or PVP is preferable over the other to produce fibres via pressure spinning considering both efficiency and application.

With respects to the resulting production rate, energy consumption and fibre diameter due to the range of magnitudes of the affecting parameters in this study, it can be judged that both PEO and PVP performed best under an applied pressure of 0.2 MPA at a concentration of 35%. PEO fibres produced under these magnitudes of the affecting parameters the fibre diameter were only 5 nm larger from value of the smallest PEO fibre diameters produced in this study, whereas the production rate was only 1.5 g/hr less than highest production reached at a value of 13.98 g/hr at a concentration of 50% and applied pressure of 0.2 MPa. The energy consumption under for PEO 35% at an applied pressure of 0.2 MPa was calculated to be 1,484 J which is less than the median value for energy to produce PEO fibres in this study. In the case of PVP fibres at an applied pressure of 0.2 MPa and concentration of 35%, the diameter of the fibres and the energy to produce them were only 50 nm and 9 J respectively, more than the value of the fibre produced at a concentration of 30% under 0.2 MPa. The production rate was calculated to be 22.05 g/hr was over the median value for PVP fibres.

It has been concluded in previous literature that the rotary speed of the gyrating vessel along with the applied pressure are the primary parameters that dictate the morphology such as the fibre diameter of resulting fibres in the pressure spinning method.[31] There has been a focus on how various rotational speeds affect fibres when using pressure spinning, where higher speeds are associated with lower diameters of fibres which are idealised for many applications due to the

resulting higher surface area to volume ratio.[211] High rotational speeds supplemented with high applied pressure aid the extrusion process of fibres by producing higher centrifugal force along with force due to applied pressure to obtain diameters in the nanoscale. However, higher speeds may not be as energy efficient, due to the requirements of motors to draw in larger currents to reach such speeds and hence larger output power magnitudes. In comparison, this study shows that increasing the pressure does not drastically increase the power consumption of the motor. However, it was also summarised that increasing the pressure from 0.1 MPa to 0.2 MPa did not result in a significant decrease in fibre diameter and increase in production rate, in comparison to increasing the pressure from 0 MPa to 0.1 MPa. Regardless, the effects of higher magnitudes of applied pressure over 0.2 MPa can be explored and may be an effective approach to obtain fibres of lower diameters and higher production rates more energy efficiently.

Furthermore, the results of this study proves that the viscosity of the polymeric solution is directly proportional to energy consumption to form fibres using the pressure spinning method. Nevertheless, there is a need to also consider the surface tension of the solvent in the polymeric solution as gas pressure acting as the primary driving force can cause a rapid loss of solvent from the polymeric solution.[212] However, this study used water which is considered the ‘greenest’ solvent. The surface tension of water is around 72.8 mN/m at room temperature which is high in comparison to other commonly used solvents such as acetone which

has a surface tension of 24.5 mN/m.[213] Hence, the effects of applied gas pressure magnitudes and the rapid loss of solvents when using water as the solvent is comparatively insignificant.

During the study, it was observed that increasing the pressure immediately caused solution and polymers to spray out of the orifices rather than in fibre form, in comparison to applying pressure more gradually to the required magnitude once the rotary speed of the vessel is about to reach its critical speed for fibre formation. To abide by the principles of green engineering, more timed control of the application of pressure promotes less wastage of solution to optimise fibre yield as an immediate application of applied pressure causes the polymeric solution to spray or jet out of the orifices. Minimal wastage of polymeric solution subsequently increases the production rate of the system as the mass of fibres formed will be optimal.

It is comprehended that the overall load on the motor dictates the power drawn by the motor. This is not only seen in the application of pressure but also when the motor is turned on when it is loaded with the vessel and when the motor is turned on when unloaded. The power drawn when the motor is turned when unloaded is equal to the power rating for the Nichibo motor used in this study, which is 21.2 W.[109] When the motor is run under load, the power drawn will be higher than the rated power of the motor. However, lesser loads on the motor will show that the power drawn will be closer to the rated power of the motor.

The choice of vessel material plays a critical role in the design and performance of pressure spinning systems, influencing energy efficiency, chemical compatibility and thermal stability. While metal vessels are commonly used due to their mechanical robustness, they impose a higher load on the spinning motor because of their weight, which can reduce overall energy efficiency.[32] As a potential alternative, carbon fibre-reinforced composites present a promising solution. In their rigid form, carbon fibre is typically embedded in a polymer matrix to create lightweight, high-strength components. The epoxy resin used in these composites provides excellent chemical resistance to a wide range of alcohols, acids and other processing solvents.[214] Additionally, the low thermal expansion of carbon fibre makes it well-suited for pressure spinning vessels, as it can withstand the high temperatures and stresses involved in gyration-based fibre manufacturing.

4.6 Concluding Remarks

Based on the findings of this study, there were notable differences between the performance of PEO and PVP used in the pressure spinning process. Overall, these differences highlight the importance of selecting the appropriate polymer for a given application in pressure spinning manufacture. Both PVP and PEO performed best under an applied pressure magnitude of 0.2 MPa and a concentration of 35% and it is notable that these findings are in keeping with the magnitudes of the affecting parameters considered in this study.

The choice of polymer can affect the production rate, fibre diameter and energy consumption of the process, which can in turn impact the quality and cost of the final product. An increase in concentration or viscosity of PEO and PVP solutions subsequently increased the diameter of the fibres produced and production rate, along with an increase in total energy consumption to manufacture these fibres. An increase in applied pressure increased production rate and a decrease in fibre diameter was seen. Improvements in the effects of the application of increasing pressure on energy consumption cannot be legitimately concluded for both PEO and PVP. However, energy consumption was seen to be lower with the application of 0.1 MPa of pressure, relative to no applied pressure.

Chapter 5 – Sustainability of Core-Sheath Fibre Production

This chapter presents a detailed investigation into the formation and sustainability performance of core-sheath polymeric fibres produced via pressure spinning. Unlike single-component fibres, core-sheath architectures consist of two concentric polymer phases. Depending on the design, either the core or the sheath may serve as the mechanically supportive phase, while the other delivers chemical functionality, biodegradability, or biological activity. This structural versatility enables spatial compartmentalisation of properties, such as mechanical strength, chemical reactivity and degradation behaviour, within a single fibre, offering multifunctionality and broad adaptability for applications in drug delivery, biosensing, tissue engineering and advanced materials development.[215, 216]

The aim of this chapter is to understand how applied gas pressure and polymer concentration affect the successful formation of these coaxial structures and how these parameters influence key sustainability indicators such as material utilisation, energy consumption and production efficiency. Building upon the findings of single-polymer fibre studies (Chapter 4), the core-sheath configuration introduces new complexity in terms of fluid dynamics and flow synchronisation between two distinct polymer solutions. To explore this, a series of experiments were conducted using Polyvinylpyrrolidone (PVP) as the core-forming polymer and Polyethylene Oxide (PEO) as the sheath-forming polymer.

Both polymers are water-soluble, non-toxic and widely used in green processing systems.

Rhodamine B dye was incorporated into the PEO sheath solution to visually confirm sheath formation through optical microscopy, as the appearance of a pink coating provided direct evidence of PEO deposition. The concentric nature of core-sheath fibres was further characterised using Scanning Electron Microscopy (SEM) to quantify fibre diameter and sheath thickness and Fourier Transform Infrared Spectroscopy (FTIR) to confirm chemical integrity.

The chapter systematically analyses how combinations of PVP (50% and 60% wt) and PEO (40% and 50% wt) perform under varying applied pressure conditions (0 to 0.3 MPa). Special attention is given to the volumetric flow rate ratios of the two solutions, which are critical in ensuring uniform and continuous fibre formation. Flow synchronisation is crucial because deviations can result in sheath-only or core-only fibres, leading to material wastage and inconsistencies in functional performance. In this context, sustainability is evaluated not only in terms of energy input and production yield, but also by assessing wastage minimisation and uniformity of the resultant fibres.

Tables 9 to 12 report detailed measurements of fibre diameter, sheath thickness (where applicable) and production yield for each material pairing and pressure condition. The presence of core-sheath fibres was not evident when gas pressure was not applied using a core-sheath configuration of PEO 40% in the sheath, indicated as ‘no core-sheath’ in

Table 9 and Table 10. However, the formation of core-sheath fibres with PEO 40% was seen when pressure was introduced and it was seen to further improve when the magnitude of the applied pressure was increased. Regardless, not all of the produced fibres were core-sheath and hence, the core-sheath results for fibre mass and production rates were ignored as indicated in Table 9 and Table 10. Therefore, the mass estimates along with the production rate estimates for the core-sheath fibres that used PEO 40% in the sheath were not considered. The core-sheath fibre dimensions when using PEO 40% were evaluated by distinguishing the fibres that showed a pink dye coating in the optical images of samples, as a pink coating demonstrates that the fibre is core-sheath. The area of samples produced using PEO 40% that showed a full sheath under optical microscopy was analysed using SEM to obtain fibre dimensions.

	PVP 50/ PEO 40							
	Core	C-S	Core	C-S	Core	C-S	Core	C-S
Pressure (MPa)	0		0.1		0.2		0.3	
Speed (RPM)	12 000		12 000		11 800		11 600	
Fibre mass (g)	0.15 ± 0.01	no c-s	0.17 ± 0.01	not all c-s	0.19 ± 0.01	not all c-s	0.25 ± 0.01	not all c-s
Production rate (g/hr)	18 ± 0.9	no c-s	21 ± 1.0	not all c-s	23 ± 1.2	not all c-s	30 ± 1.5	not all c-s
Diameter (µm)	2.6 ± 1.2	no c-s	2.3 ± 1.2	3.8 ± 1.8	2.1 ± 1.1	3.4 ± 1.8	2.0 ± 1.0	3.2 ± 1.7
Sheath thickness (µm)	N/A		0.8 ± 1.1		0.7 ± 1.1		0.6 ± 1.0	

Table 9. *Experimental results of core-sheath (c-s) configuration of PVP 50% and PEO 40%.*

	PVP 60/ PEO 40							
	Core	C-S	Core	C-S	Core	C-S	Core	C-S
Pressure (MPa)	0		0.1		0.2		0.3	
Speed (RPM)	12 000		12 000		11 800		11 600	
Fibre mass (g)	0.25 ± 0.01	no c-s	0.28 ± 0.01	not all c-s	0.32 ± 0.02	not all c-s	0.35 ± 0.02	not all c-s
Production rate (g/hr)	30 ± 1.5	no c-s	34 ± 1.7	not all c-s	39 ± 2.0	not all c-s	42 ± 2.1	not all c-s
Diameter (µm)	2.8 ± 1.3	no c-s	2.4 ± 1.3	4.0 ± 1.9	2.2 ± 1.2	3.8 ± 1.9	2.1 ± 1.2	3.5 ± 1.8
Sheath thickness (µm)	N/A		0.8 ± 1.2		0.8 ± 1.1		0.7 ± 1.1	

Table 10. *Experimental results of core-sheath (c-s) configuration of PVP 60% and PEO 40%.*

	PVP 50/ PEO 50							
	Core	C-S	Core	C-S	Core	C-S	Core	C-S
Pressure (MPa)	0		0.1		0.2		0.3	
Speed (RPM)	12 000		12 000		11 800		11 600	
Fibre mass (g)	0.15 ± 0.01	0.21 ± 0.01	0.17 ± 0.01	0.25 ± 0.01	0.19 ± 0.01	0.32 ± 0.02	0.25 ± 0.01	0.41 ± 0.02
Production rate (g/hr)	18 ± 0.9	25 ± 1.3	21 ± 1.0	30 ± 1.5	23 ± 1.2	39 ± 2.0	30 ± 1.5	49 ± 2.45
Diameter (µm)	2.6 ± 1.2	4.2 ± 1.9	2.3 ± 1.2	3.9 ± 1.9	2.1 ± 1.1	3.7 ± 2.0	2.0 ± 1.0	3.5 ± 2.1
Sheath thickness (µm)	0.8 ± 1.1		0.8 ± 1.1		0.8 ± 1.1		0.8 ± 1.2	

Table 11. *Experimental results of core-sheath configuration of PVP 50% and PEO 50%.*

	PVP 60/ PEO 50							
	Core	C-S	Core	C-S	Core	C-S	Core	C-S
Pressure (MPa)	0		0.1		0.2		0.3	
Speed (RPM)	12 000		12 000		11 800		11 600	
Fibre mass (g)	0.25 ± 0.01	0.33 ± 0.01	0.28 ± 0.01	0.41 ± 0.02	0.32 ± 0.02	0.49 ± 0.02	0.35 ± 0.02	0.53 ± 0.03
Production rate (g/hr)	30 ± 1.5	40 ± 2.0	34 ± 1.7	50 ± 2.5	39 ± 2.0	58 ± 2.9	42 ± 2.1	64 ± 3.2
Diameter (µm)	2.8 ± 1.3	4.6 ± 2.0	2.4 ± 1.3	4.1 ± 2.0	2.2 ± 1.2	4.0 ± 2.1	2.1 ± 1.2	3.8 ± 2.1
Sheath thickness (µm)	0.9 ± 1.2		0.9 ± 1.2		0.9 ± 1.2		0.9 ± 1.2	

Table 12. *Experimental results of core-sheath configuration of PVP 60% and PEO 50%.*

FTIR spectrum for PEO fibres show an absorption at 2875.64 cm^{-1} corresponding to molecular stretching of the methylene group CH_2, whereas the peaks at 1097.01 and 961.32 cm^{-1} are caused by stretching of the ether group in PEO which is further indicated as the C–O–C absorption complex.[217] The spectrum for PVP indicated a peak at 1646.01 cm^{-1} proved the stretching of C-O, whilst the C-H bending and CH_2 wagging were observed at 1420.79 cm^{-1} and 1287.19 cm^{-1}, respectively.[183] The presence of these peaks in the core-sheath sample is evident and this corresponds to the presence of both PEO and PVP as shown in Figure 24.

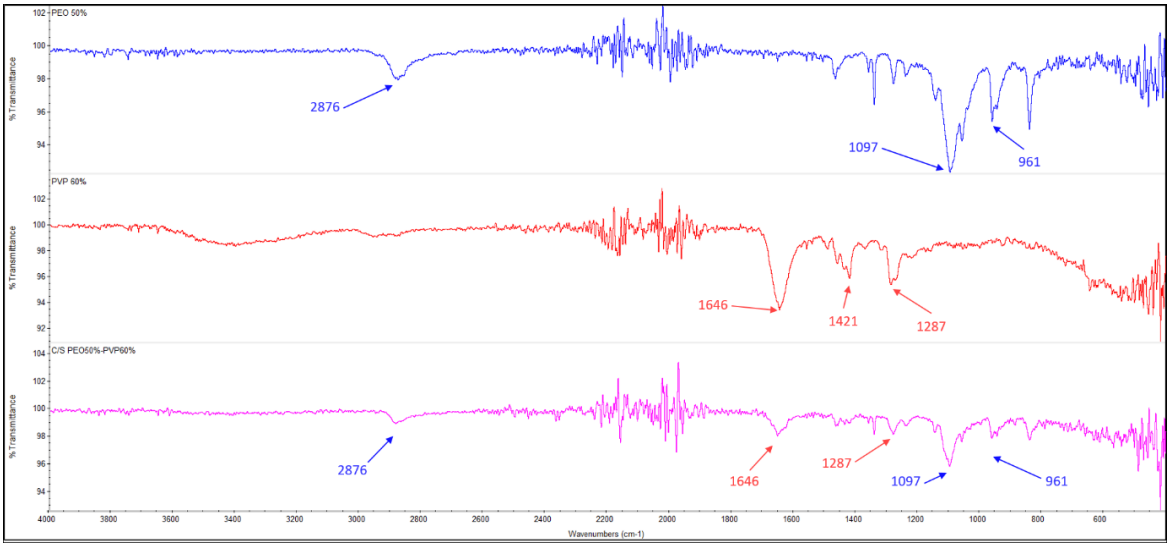

Figure 27. *FTIR spectra PEO fibres, PVP fibres and core-sheath PEO50%-PVP60% fibres*

Applying the theory of volumetric flow rate, the optimum parameters to form core-sheath fibres with minimal material wastage, using the same volume of solution in both the inner and outer reservoir of the vessel was calculated to be PEO 40% in the sheath and PVP 60% in the core with no applied gas pressure. Minimal wastage in this case means that once both solutions travel through the orifice at the same time. For instance, if the time taken for a specific quantity of solution to move from the outer reservoirs to form the sheath is half of the time taken for same quantity of solution to move from the inner reservoir to form the core, this would mean that only half of the fibres produced are core-sheath. The time taken for the volume of solution in the two reservoirs to eject through (either the simple tube or annulated tube depending on core or sheath) to the orifices was calculated based on the number of orifices (4) and flowrates through either tube.

$$\text{Time} = \left(\frac{\text{Volume in reservoir}}{\text{Number of orifices}}\right) \div \text{Flowrate}$$

The theoretical time to spin identical volumes of PVP 60% in the core and PE0 40% in the sheath results in the best 'core:sheath' volumetric flow rate ratio that is closest to a ratio of 1:1.

When considering fibre quality, production rate and energy consumption, experimental validation of the polymer configurations used in this study, the best core-sheath fibres resulted when PVP 60% is used in the core and PEO 50% is used in the sheath (see section 5.2). For the PVP 60% by PEO 50% core-sheath configuration, the theory of volumetric flow rate show that the most desirable 'core:sheath' volumetric flow rate ratio is achieved at an applied pressure magnitude of 0.3 MPa which resulted in a volumetric flowrate ratio of 0.52. This is considering the magnitudes of process control parameters available and when the same volumes of solutions are used in the core and the sheath. To maintain minimal material wastage, it is rational to utilise the theory of volumetric flow rate to estimate the amounts of polymeric solution to use in the inner reservoir and the outer reservoir, once the optimum core-sheath fibre forming parameters are experimentally validated. Hence, using a 2:1 core to sheath volume ratio to form core-sheath fibres using PVP 60% and PEO 50% at an applied pressure magnitude of 0.3 MPa will equate to a core to sheath volumetric flowrate ratio of 1.06. This can be translated directly into real-world production

terms by defining the conditions necessary to maximise material efficiency and throughput for a continuous manufacturing run.

5.1 Production Rate

The results obtained when using PEO 40% as the sheath were ignored in the production rate analysis as it is not feasible to separate the fibres that are core-sheath from the fibres that are not. Nevertheless, optical microscopy proved that the application of pressure improved the forming of core-sheath fibres using PEO 40% in the sheath as seen in Figure 25. In Figure 25a, where no gas pressure (0 MPa) was applied, no core-sheath fibres were observed. Instead, distinct pink droplets of unspun PEO 40% solution are visible, indicating that sheath extrusion was insufficient to initiate fibre formation. In Figure 25b (0.1 MPa), a small number of core-sheath fibres appear alongside residual pink droplets, suggesting partial extrusion and unstable sheath flow. However, in Figures 25c and 25d (0.2 MPa and 0.3 MPa respectively), continuous fibres with a uniform pink coating are observed, clearly indicating successful and consistent formation of core-sheath structures. The presence of the pink-coloured sheath surrounding the fibre core at these higher pressures confirms that both polymer streams are being simultaneously ejected and stabilised during spinning.

These results suggest that a minimum pressure threshold (~0.2 MPa) is required to generate sufficient flow of PEO-H_2O solution at a concentration of 40% through the nozzle system to achieve a stable

sheath layer. This highlights the importance of optimising pneumatic input not only for yield but also for structural integrity of core-sheath fibres.

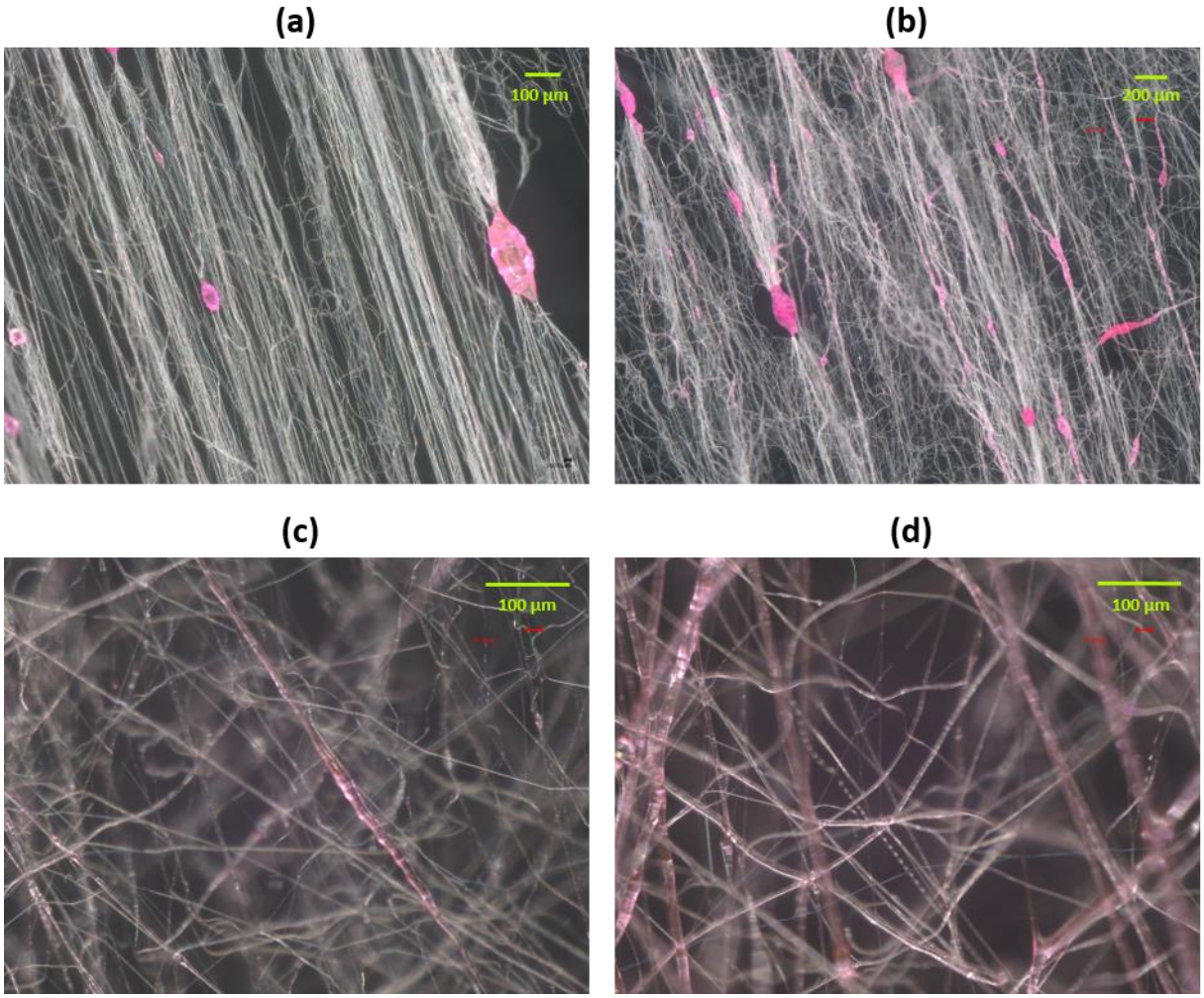

Figure 28. *Optical images of attempt to produce core-sheath fibre using PEO 40% in the sheath at applied pressure magnitudes of (a) 0 MPa, (b) 0.1 MPa, (c) 0.2 MPa and (d) 0.3 MPa*

The production rates increased with the application of pressure for all configurations of core-sheath fibre sample. This is seen in the core and in the sheath of the fibre separately and in the fibre as a whole (Figure

26). The production rate also has an increasing trend as polymer concentration is increased. This shows that although the viscosity is paramount in terms of the volumetric flow rate through the orifices of the vessel, it does not necessarily mean this reflects on the actual fibres produced, as the resulting solid fibres that are studied are not of liquid state. In theory, the volumetric flow rate of a solution is inversely proportional to it viscosity. However, in this study, the production rate is a measure of the mass of fibres formed per unit time, rather than a measure of the mass of solution jetted through the orifices.

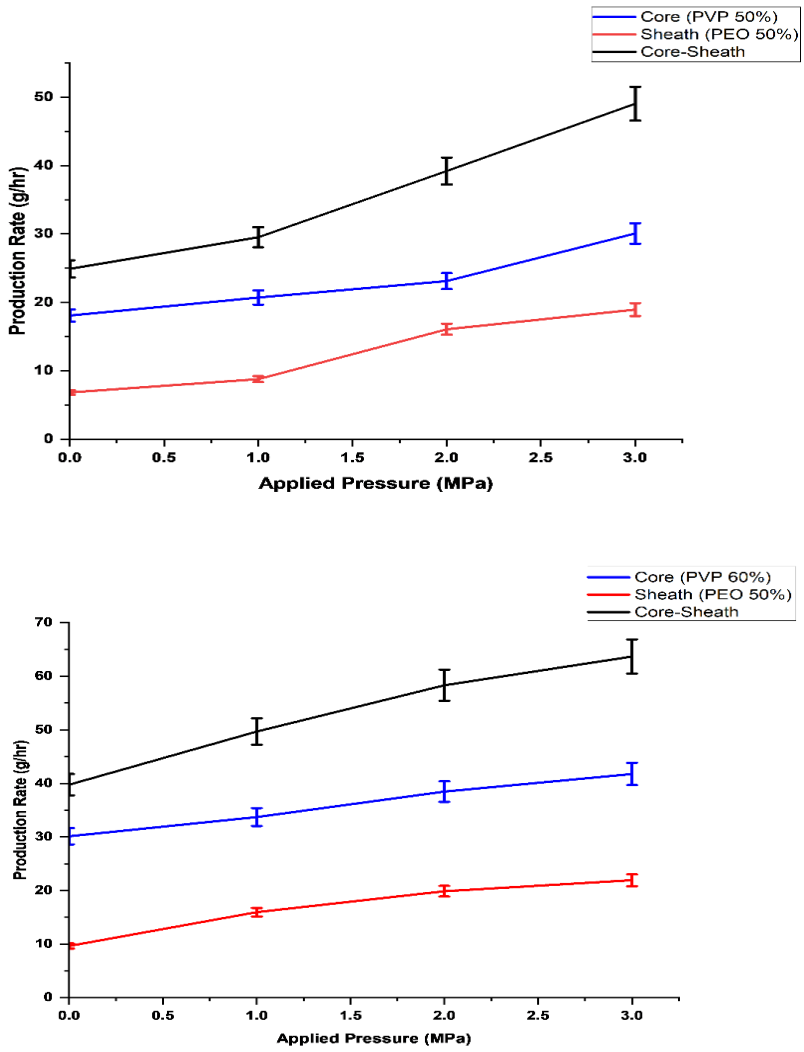

Figure 29. *Production rate against applied pressure magnitudes.*

5.2 Energy

The necessity of applied pressure magnitudes to produce core-sheath fibres were signified when using PEO 40% in the sheath. However, the increasing addition of applied pressure magnitudes result in the increase of power consumption by the motor, along with a slight decrease in the rotary speed of the vessel. The decrease in rotary speed at specific magnitudes of applied gas pressures were taken into consideration in the volumetric flow rate analysis. The change in rotary speed of the

spinning vessel with addition of gas pressure is attributed to the need for more torque to maintain rotational speed caused by the additional load on the motor. In a DC motor, the output torque is directly proportional to the current. Hence, an increase in power drawn is witnessed, as power is the product of voltage and current.

Figure 28 depicts the energy per mass of core-sheath fibre produced at applied pressure magnitudes. The graph depicts only the forming of fibres using PEO 50% in the sheath, as not all fibres formed using PEO 40% in the sheath were characterised to be fully core-sheath. A predominantly decreasing energy consumption is seen with the application of higher-pressure magnitudes to produce fibres. It should be noted that the energy usage associated with the pressurisation of the gas is not included, as this gas pressurisation is preprocessed. The pressure spinning process involves the release of gas from a tank to obtain desired applied gas pressures. Hence, there is no energy requirement to apply gas pressure magnitudes during fibre production.

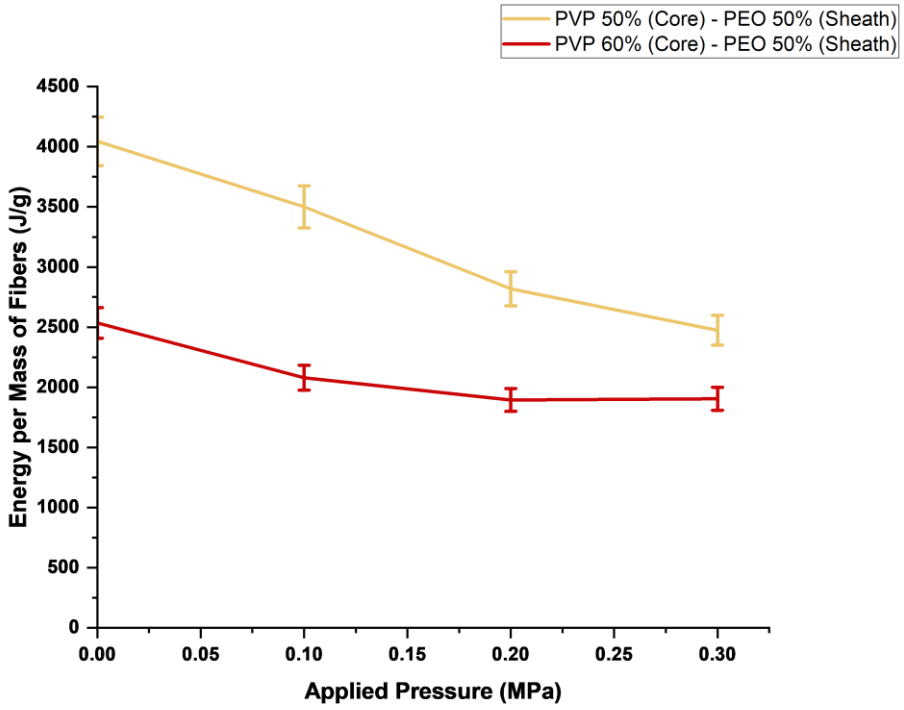

Figure 30. *Energy consumption per mass of fibres produced.*

Figure 27 shows that using PVP 60% over PVP 50% for the core of the fibres result in a more energy efficient production with respect to the mass of fibres produced. However, it is seen that when using PVP 60% in the core at an applied pressure magnitude of 0.3 MPa, the energy required to produce a specific mass of fibres is higher than that to produce the same mass of fibres with PVP 60% at 0.2 MPa, due to the increase in power consumption due to higher applied pressure magnitudes. Regardless, increasing applied pressure magnitudes is shown to improve processing energy efficiency and the application of pressure to produce sustainable fibres in a gyration-based process is exemplified.

5.3 Fibre Dimensions

Under the same magnitudes of effecting parameters, the diameter of the core-only fibre is likely to be smaller than the core of the core-sheath configuration, as the core of the core-sheath fibre is not exposed to the atmosphere during jetting. Hence, the core of a core-sheath fibre experiences less solvent evaporation in comparison to a core-only fibre. Regardless, the evaluation of the fibre dimensions assumed that the diameter of the core in the core-sheath configuration will be the same as in the core-only fibre. Figure 28 depicts the effects of applied pressure magnitudes on fibre dimensions that includes the radius of the core, the thickness of the sheath and the radius of the overall core-sheath fibres.

The decrease in cross-sectional dimensions under increasing pressure magnitudes associated with polymeric fibres that are produced via pressure spinning is evident. An increase in concentration of the solutions is also shown to increase the overall diameter of the core-sheath fibres produced. This is attributed to the higher quantity of polymers that is in the polymeric solution of higher concentrations. Hence, when the solvent is evaporated, polymeric fibres formed are larger.

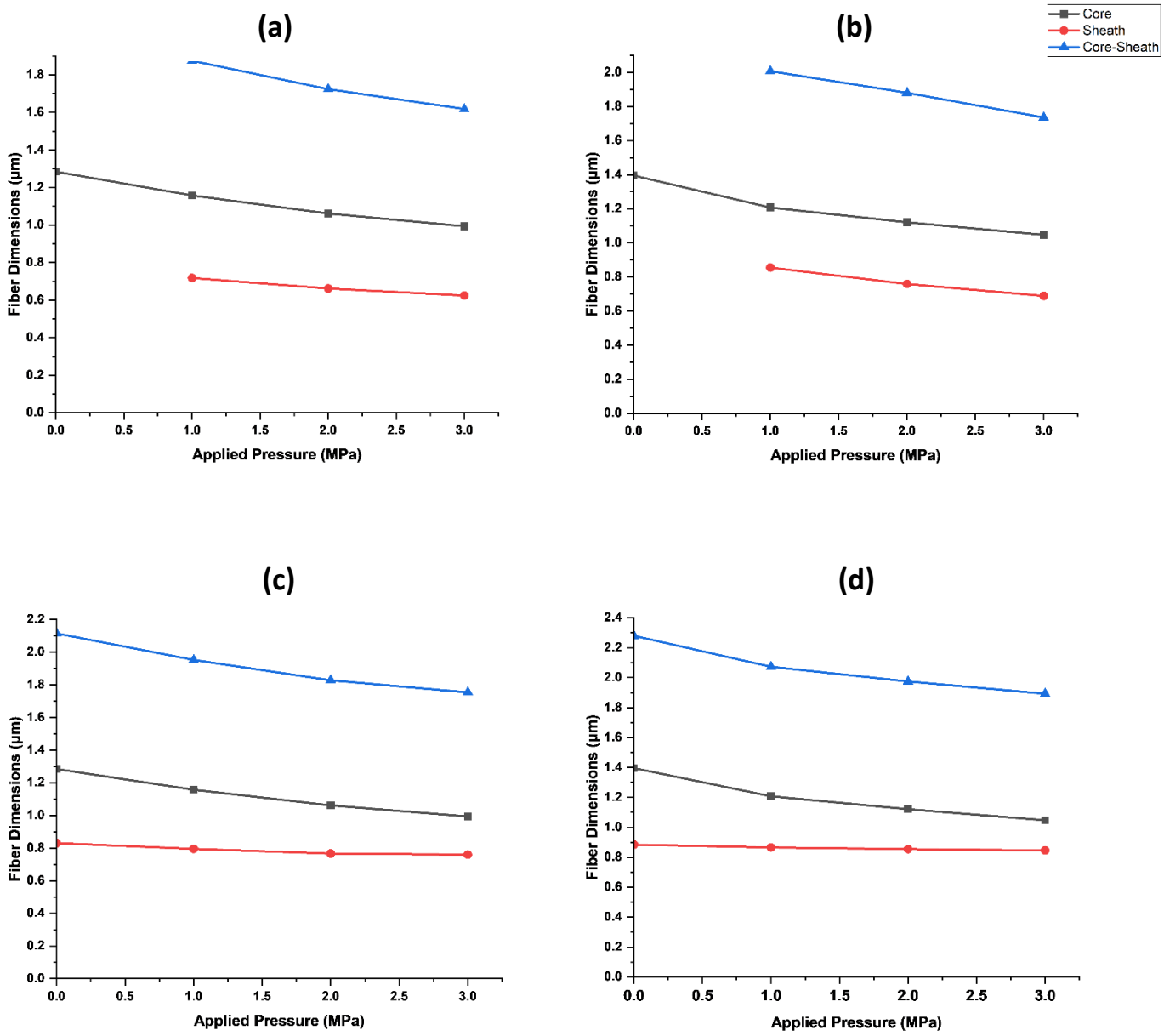

Figure 31. *Fibre dimensions against applied pressure magnitudes. (a) PVP 50% (core) – PEO 40% (sheath). (b) PVP 60% (core) – PEO 40% (sheath). (c) PVP 50% (core) – PEO 50% (sheath). (d) PVP 60% (core) – PEO 50% (sheath). The standard deviation of the results are*

Figure 29 show that for a specific configuration, the radius of the core decreases with the increase in applied pressure magnitudes. However, the width of the sheath seems to exhibit a lower decreasing trend in comparison to the radius of the core with the application of pressure.

Moreover, using PEO 50% in the sheath shows that the width of the sheath remains almost the same with the application of increasing pressure magnitudes. This is attributed to the effective pressure difference which drives solution through the inner tube of the orifice to core being 2.4 (at 0.3 MPa applied pressure) to 3.5 times (at no applied pressure) larger than the pressure difference created along the comparatively shorter annular tube that produces the sheath. Therefore, the core of the fibres produced will experience more driving force in comparison to the sheath, which promotes more elongation in the core in comparison to the sheath. This along with the difference in the solution characteristics where PEO 50% has a higher viscosity than the PVP solutions justifies the fibre dimensions obtained, as the lower viscous PVP solutions are easier to elongate than the higher viscous PEO 50% solution.

In all core-sheath polymer solution configurations, a decrease in fibre diameter along with an increase in variance was seen with the application of increasing pressure magnitudes. Figure 29 portrays the scanning electron micrographs along with fibre diameters distributions for PVP 60% and PEO 50% core-sheath configuration at minimum and maximum applied pressure magnitudes (no applied pressure and 0.3 MPa).

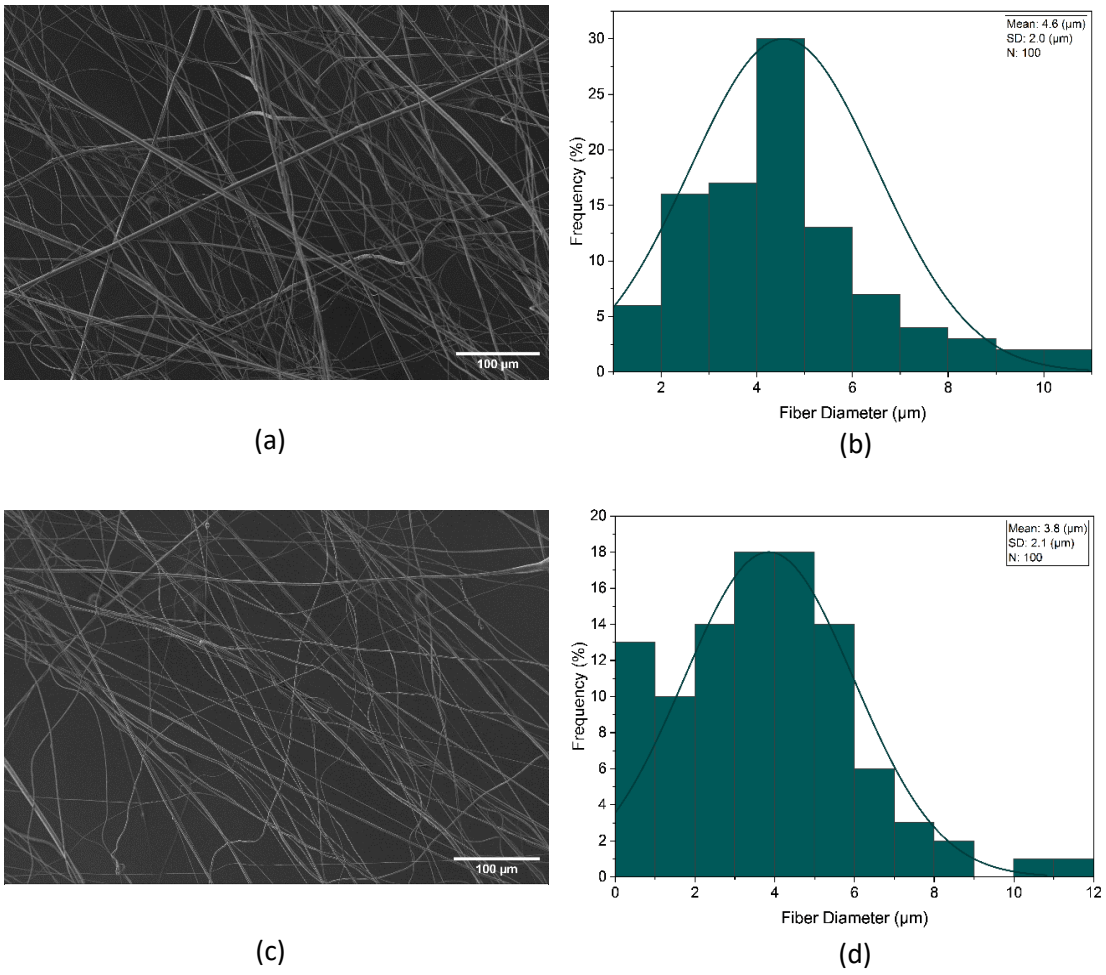

Figure 32. *SEM image at no applied pressure (a) along with fibre distribution (b) and SEM image at 0.3 MPa applied pressure (c) along with fibre distribution (d) for PVP 60% and PEO 50% core-sheath fibre configuration.*

In comparison to the PVP 60% by PEO 50% core-sheath configuration, the PVP 50% by PEO 50% configuration shown in Figure 30 shows that the fibre diameters are smaller at the same magnitudes of applied pressure. Both configurations show an increase in variance with applied gas pressure as the standard deviation increases with the application of gas pressure. However, this increase in standard deviation associated with the application of pressure is not very significant in comparison to previous studies that used lesser viscous polymer solutions to produce

fibres via pressure spinning.[208, 210] Regardless, higher viscosity serves to enhance flow stability, as solutions exhibit greater resistance to deformation and flow fluctuations. This increased resistance makes them less susceptible to jet instabilities, ultimately leading to the production of more consistently sized fibres. Hence, in the instance where fibre uniformity is significant, using higher viscous polymeric solutions is a more sustainable approach.

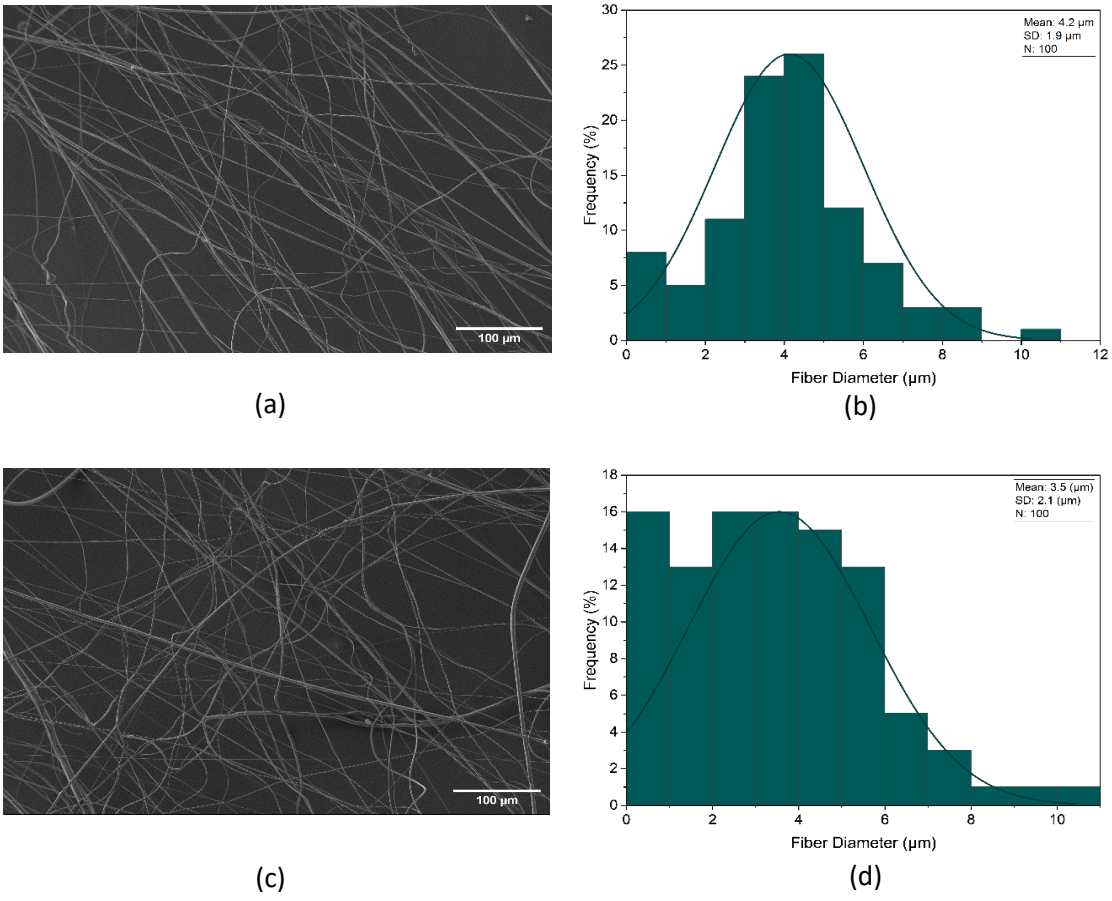

Figure 33. *SEM image at no applied pressure (a) along with fibre distribution (b) and SEM image at 0.3 MPa applied pressure (c) along with fibre distribution (d) for PVP 50% and PEO 50% core-sheath fibre configuration.*

In comparison to water-soluble polymers, non-water-soluble polymers are less sensitive to variations in relative humidity.[218, 219] This is attributed to the higher interaction of water-soluble polymers with moisture in the air, leading to changes in their physical properties and processing behaviour.[220] In comparison, non-water-soluble polymers are less affected by humidity fluctuations. Regardless, water-soluble polymers are preferred as they abide by the Principles of Green Chemistry, as they do not require hazardous solvents.

Optimising the polymer concentration of non-water-soluble polymer solvents can mitigate the impact of environmental conditions as higher concentration promote solidification. This study showed that higher polymer concentrations provide better control over the fibre formation process and the sensitivity to environmental conditions. Achieving a sufficient viscosity when using non-water-soluble polymers allowed for effective fibre production, without the need for strict control over temperature and humidity. Overall, this can be advantageous in practical applications where maintaining precise environmental conditions can be both, economically and environmentally challenging.

The limitations of this study such as the use of Pouseillie's Law to determine the flow rates of solution based on effective pressure difference assumes that polymer solution used in this study is incompressible and the flow is laminar. Furthermore, it is also acknowledge that the uncertainty associated with the width of the sheath may be large considering it is calculated by evaluating the difference of the mean and standard deviations of the core and the core-sheath fibre

dimensions. Regardless this study provides a useful approximation, where despite the challenges, the insights gained from this study contribute to our understanding of sustainable production of fibres via pressure spinning.

5.4 Concluding Remarks

This investigation into core-sheath fibre production via pressure spinning unravels the intricate relationship between process control parameters, fluid dynamics and resultant fibre dimensions, along with energy and production efficiency. Core-sheath fibres produced using higher concentrations of polymer solution under higher applied pressures were more energy and production efficient. The findings indicate that applied gas pressure plays a critical role in enabling dual-material flow and stable sheath formation. While energy demand increases with pressure, the efficiency of material usage and reduction in wasted polymer justify operating at moderate to high pressures (0.2-0.3 MPa) for core-sheath applications. Furthermore, to enhance fibre uniformity, optimising the choice of solution is paramount: future efforts should prioritize the use of higher viscosity polymer solutions for both the core and sheath, as these systems demonstrate greater resistance to flow fluctuations, resulting in more consistently sized fibres.

Empirical validation considering all process control parameters proved to be more effective than relying solely on theoretical volumetric flowrate calculations, guiding the selection of optimal polymer concentrations for

tailored core-sheath fibre production. Before large-scale production, this flow synchronization can help determine the optimal core-to-sheath volumetric ratio for the chosen polymer concentrations, ensuring both materials are consumed simultaneously in long-run production.

This study shows how varying pressure magnitudes and polymer concentrations influence fibre dimensions and uniformity, with higher viscosity polymer solutions yielding more consistently sized fibres, whilst mitigating the need for strict control over temperature and humidity. Despite inherent limitations, these findings offer valuable insights into advancing sustainable core-sheath fibre production methods.

Chapter 6 – Optimising Fibre Morphology and Production Efficiency in Pressure Spinning through Vessel and Collector Design

The production rate, fibre diameter and energy consumption were recorded at three different collector distances (100 mm, 150 mm and 200 mm) and applied pressures (0 MPa, 0.1 MPa and 0.2 MPa). As shown in Table 13 (PEO 40%), Table 14 (PEO 50%), Table 15 (PVP 40%) and Table 16 (PVP 50%), the production rate decreased with increasing collector distance and applied pressure, while fibre diameters generally decreased with applied pressure. The energy consumption per gram of fibre showed a trend of decreasing with greater collector distances. The two rotary vessel diameters were selected based on the 60 mm vessel diameters used in previous studies and a larger diameter of 75 mm which would not alter the load on the motor whilst maximising the resultant centrifugal force.

		60 mm vessel			75 mm vessel		
Collector Distance (mm)	**Applied Pressure (MPa)**	**Production Rate (g/hr)**	**Fibre Diameter (µm)**	**Energy (KJ/g)**	**Production Rate (g/hr)**	**Fibre Diameter (µm)**	**Energy (KJ/g)**
100	0	7.0 ± 0.4	1.3 ± 0.4	16.0	6.8 ± 0.3	1.2 ± 0.4	13.5
	0.1	11.2 ± 0.6	0.7 ± 0.3	12.0	11.8 ± 0.6	0.6 ± 0.2	9.9
	0.2	11.9 ± 0.7	0.7 ± 0.3	11.4	14.5 ± 0.7	0.5 ± 0.2	9.0
150	0	7.0 ± 0.4	0.9 ± 0.3	16.0	7.4 ± 0.4	0.9 ± 0.3	13.5
	0.1	10.2 ± 0.5	0.7 ± 0.3	12.0	10.7 ± 0.5	0.6 ± 0.3	11.0
	0.2	12.1 ± 0.6	0.6 ± 0.3	11.4	13.6 ± 0.7	0.5 ± 0.2	9.8
200	0	6.1 ± 0.3	0.4 ± 0.1	16.0	6.5 ± 0.3	0.4 ± 0.1	16.2
	0.1	10.1 ± 0.5	0.4 ± 0.2	13.5	10.9 ± 0.5	0.4 ± 0.2	11.0
	0.2	12.2 ± 0.6	0.4 ± 0.2	11.4	12.7 ± 0.6	0.4 ± 0.1	9.8

Table 13. *Experimental results of PEO 40% when spun for 30 seconds*

		60 mm vessel			75 mm vessel		
Collector Distance (mm)	**Applied Pressure (MPa)**	**Production Rate (g/hr)**	**Fibre Diameter (µm)**	**Energy (KJ/g)**	**Production Rate (g/hr)**	**Fibre Diameter (µm)**	**Energy (KJ/g)**
100	0	8.5 ± 0.4	2.3 ± 0.6	13.7	8.4 ± 0.4	1.7 ± 0.4	11.6
	0.1	20.9 ± 1.0	1.1 ± 0.4	6.4	20.3 ± 1.0	1.1 ± 0.3	5.8
	0.2	22.6 ± 1.1	0.9 ± 0.3	6.0	23.6 ± 1.2	0.8 ± 0.3	5.4
150	0	8.2 ± 0.4	1.0 ± 0.3	13.7	9.0 ± 0.5	0.9 ± 0.3	10.1
	0.1	24.1 ± 1.2	0.9 ± 0.3	5.4	23.8 ± 1.2	0.9 ± 0.3	5.0
	0.2	26.9 ± 1.3	0.8 ± 0.3	5.2	26.3 ± 1.3	0.7 ± 0.3	4.9
200	0	8.8 ± 0.4	0.6 ± 0.2	13.7	9.5 ± 0.5	0.5 ± 0.2	10.1
	0.1	22.7 ± 1.1	0.5 ± 0.2	5.7	24.1 ± 1.2	0.5 ± 0.2	5.2
	0.2	26.6 ± 1.3	0.5 ± 0.2	5.2	27.8 ± 1.4	0.4 ± 0.2	4.9

Table 14. *Experimental results of PEO 50% when spun for 30 seconds*

		60 mm vessel			75 mm vessel		
Collector Distance (mm)	**Applied Pressure (MPa)**	**Production Rate (g/hr)**	**Fibre Diameter (μm)**	**Energy (J/g)**	**Production Rate (g/hr)**	**Fibre Diameter (μm)**	**Energy (J/g)**
100	0	17.6 ± 0.9	1.9 ± 0.8	6.4	18.9 ± 0.9	1.4 ± 0.6	5.4
	0.1	23.9 ± 1.2	0.9 ± 0.4	5.4	25.7 ± 1.3	0.8 ± 0.4	5.0
	0.2	26.0 ± 1.3	0.7 ± 0.3	5.4	27.8 ± 1.4	0.7 ± 0.3	5.1
150	0	18.1 ± 0.9	1.2 ± 0.5	6.4	18.5 ± 0.9	1.1 ± 0.5	5.4
	0.1	22.7 ± 1.1	0.8 ± 0.4	5.7	23.4 ± 1.2	0.7 ± 0.3	5.0
	0.2	24.8 ± 1.2	0.7 ± 0.3	5.4	24.7 ± 1.2	0.6 ± 0.3	5.1
200	0	18.5 ± 0.9	0.6 ± 0.3	6.4	19.2 ± 1.0	0.6 ± 0.2	5.4
	0.1	23.9 ± 1.2	0.6 ± 0.3	5.4	23.0 ± 1.2	0.5 ± 0.3	5.2
	0.2	24.5 ± 1.2	0.5 ± 0.3	5.7	25.1 ± 1.3	0.4 ± 0.2	5.1

Table 15. *Experimental results of PVP 40% when spun for 30 seconds*

		60 mm vessel			75 mm vessel		
Collector Distance	**Applied Pressure**	**Production Rate (g/hr)**	**Fibre Diameter (μm)**	**Energy (KJ/g)**	**Production Rate (g/hr)**	**Fibre Diameter (μm)**	**Energy (KJ/g)**
100 mm	0 MPa	17.9 ± 0.9	2.7 ± 0.9	6.4	20.8 ± 1.0	2.0 ± 0.6	5.4
	0.1 MPa	28.2 ± 1.4	1.3 ± 0.6	4.5	31.1 ± 1.6	1.2 ± 0.5	3.8
	0.2 MPa	28.8 ±1.4	1.1 ± 0.5	4.8	31.3 ± 1.6	0.9 ± 0.4	4.2
150 mm	0 MPa	18.1 ± 0.9	1.1 ± 0.4	6.4	19.1 ± 1.0	1.0 ± 0.4	5.1
	0.1 MPa	28.3 ± 1.4	1.0 ± 0.4	4.5	30.6 ± 1.5	0.8 ± 0.3	3.8
	0.2 MPa	29.0 ±1.5	0.9 ± 0.4	4.8	31.1 ± 1.6	0.7 ± 0.3	4.2
200 mm	0 MPa	17.4 ± 0.9	0.7 ± 0.2	6.4	19.6 ± 1.0	0.6 ± 0.2	5.4
	0.1 MPa	29.0 ± 1.5	0.6 ± 0.3	4.5	31.0 ± 1.6	0.6 ± 0.3	3.8
	0.2 MPa	29.3 ± 1.5	0.6 ± 0.3	4.8	31.6 ± 1.6	0.4 ± 0.2	4.0

Table 16. *Experimental results of PVP 50% when spun for 30 seconds*

6.1 Production Rate

No significant change in production rate is seen as the collector distance increases from 100 mm to 200 mm, while applied gas pressure and polymer concentration are constant (Figure 31). This suggests that the production rates are more influenced by parameters that affect the pressure difference in the vessel and solution properties, while collector distance has a minimal effect on the fibre production rate under these specific conditions. Since the collector distance doesn’t significantly impact the production rate, the choice of collector distance might be based on other factors, such as fibre morphology or energy efficiency rather than production rate optimisation.

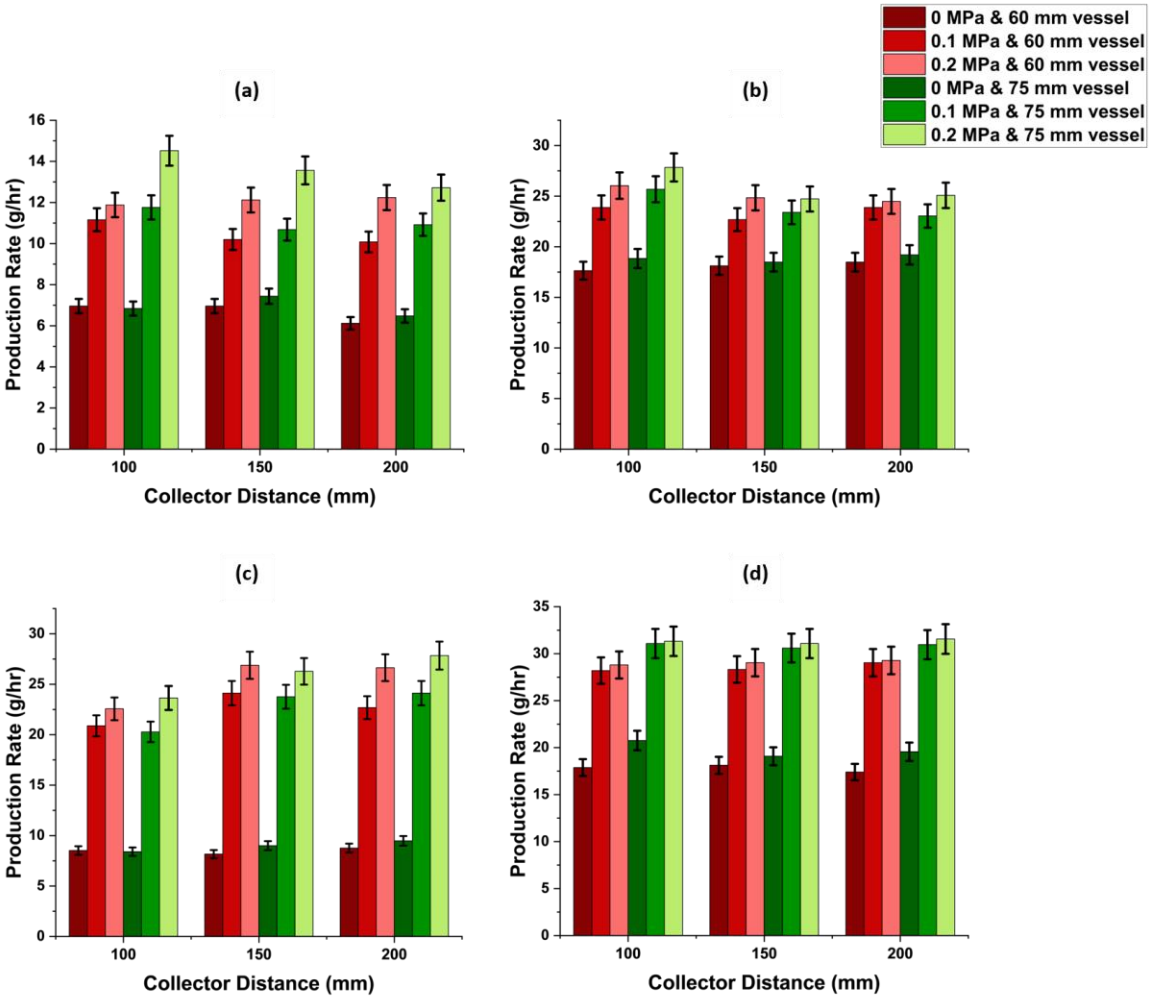

Figure 34. *Production rate against collector distance. (a) PEO 40% (b) PVP 40% (c) PEO 50% (d) PVP 50%. The error bars show the standard deviation of the results.*

Increasing the applied gas pressure leads to significant increases in production rates for all polymer solutions and both vessel sizes. Higher applied gas pressures drive the polymer solution more forcefully through the orifices, resulting in a greater volume of fibres being extruded per unit time. This effect is most pronounced at 0.2 MPa, where production rates are at their highest across all conditions. At each gas pressure magnitude and solution concentration, the 75 mm vessel consistently exhibits slightly higher production rates than the 60 mm vessel. Additionally, higher polymer concentrations (50%) yield significantly

greater production rates compared to 40% concentrations in both vessels. This is attributed to the increased polymer content per unit volume at higher concentrations, leading to a larger mass of fibres being produced per unit time.

6.2 Energy

Across different polymer concentrations, applied pressures and collector distances, energy consumption per gram of fibre produced is on average lower for the 75 mm vessel than for the 60 mm vessel. The 75 mm vessel demonstrates a more efficient fibre production process, as it produces a larger fibre output for the same magnitudes of other affecting parameters. Hence, larger diameter vessels appears to be more energy-efficient, making it preferable for applications where minimising energy costs is a priority.

Both PEO-H_2O and PVP-H_2O solutions at 40% concentration, energy consumption is higher at lower applied gas pressures and decreases as applied gas pressure increases. This is especially evident at collector distances of 100 mm and 150 mm. For instance, PEO- H20 at 40% with the 60 mm vessel, energy decreases from 16,000 J/g at no applied gas pressure to 11,400 J/g at 0.2 MPa applied gas pressure. For both polymer solutions at 50% concentration, energy consumption is significantly lower across all pressures and distances compared to the 40% concentrations. Higher polymer concentration (50%) contributes to

production energy efficiency, due to the production of relatively thicker fibres.

As applied pressure increases, energy consumption tends to decrease significantly. For instance, PEO- H20 at 50% concentration and a 150 mm collector distance with the 60mm diameter vessel, energy decreases from 13,714 J/g at no applied gas pressure, to 5,400 J/g at 0.1 MPa and further to 4,500 J/g at 0.2 MPa (Figure 32). Higher applied pressures likely facilitate faster and more efficient fibre production, resulting in lower energy costs per gram of fibre. This implies that higher pressures are impactful for reducing energy consumption, making them beneficial for scaling up the process in an energy-efficient manner. However, it should be noted that the energy usage associated with the pressurisation of the gas is not included, as this gas pressurisation is pre-processed. The pressure spinning process involves the release of gas from a tank to obtain the desired applied gas pressures. Hence, there is no energy requirement to apply gas pressure magnitudes during fibre production.

Similar to the production rate, increasing the collector distance did not show a significant change in energy consumption for both vessel sizes. Energy consumption does not show to consistently increase or decrease as the collector distance changes when applied gas pressure, polymer concentration and vessel diameter remain constant.

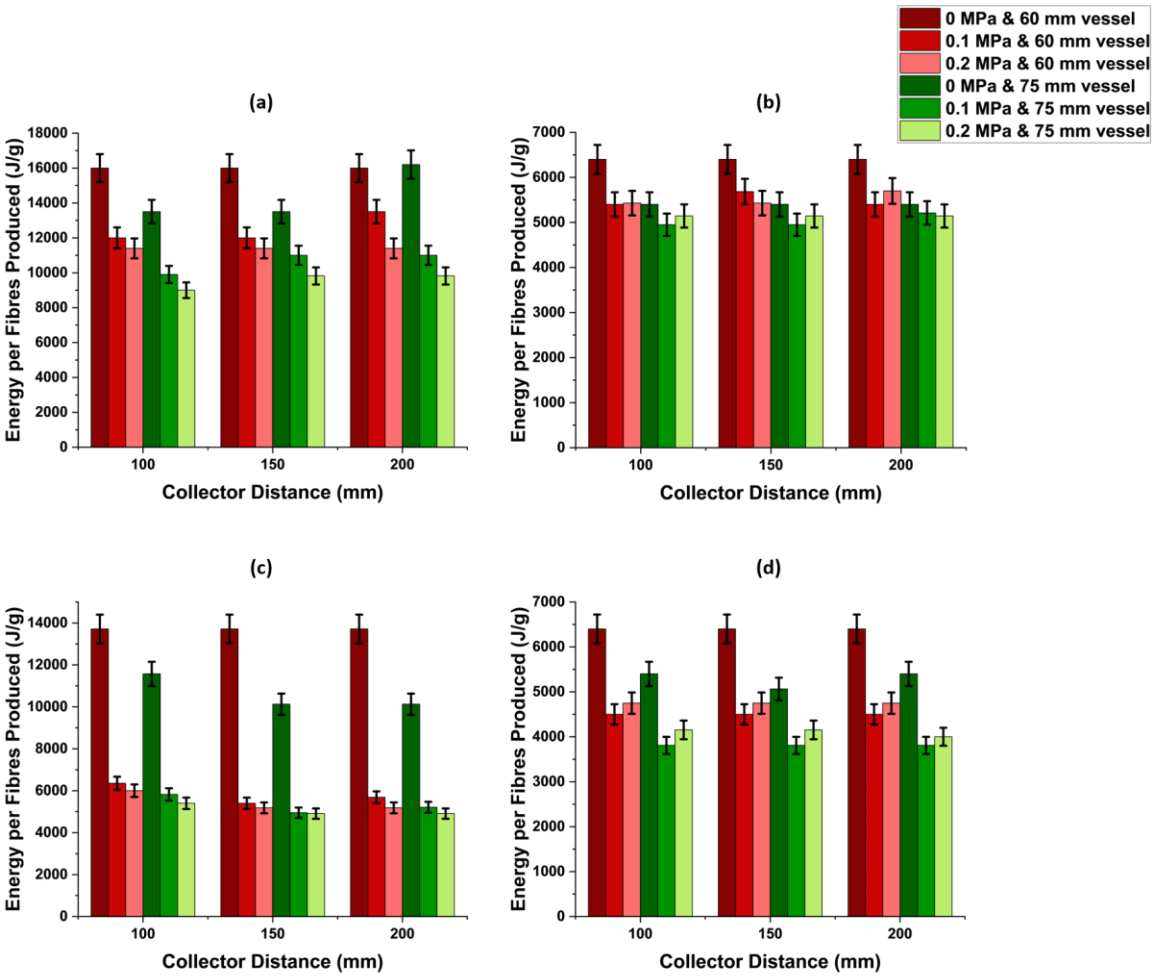

Figure 35. *Energy per mass of fibres formed against processing magnitudes. (a) PEO 40% (b) PVP 40% (c) PEO 50% (d) PVP 50%. The error bars show the standard deviation of the results.*

6.3 Fibre Morphology

Increasing applied gas pressure from no applied gas pressure to 0.2 MPa consistently reduces fibre diameter for both PEO-H_2O and PVP-H_2O solutions at specific collector distances. Applied gas pressure increase the force exerted on the polymer solution as it exits the orifices, leading to finer fibre formation due to enhanced jet stretching and thinning. This is particularly evident at 0.2 MPa, where diameters are generally at their lowest for a given collector distance and polymer concentration (Figure 33). The 75 mm vessel consistently produces

smaller fibres than the 60 mm vessel under the same magnitudes of processing parameters, where this is more evident with no applied gas pressure and at a collector distance of 100 mm for all polymer solutions. This suggests that vessel diameter can be an important parameter to obtain finer fibres when there are limitations in obtaining higher magnitudes of applied gas pressure, rotary vessel speed and collector distance.

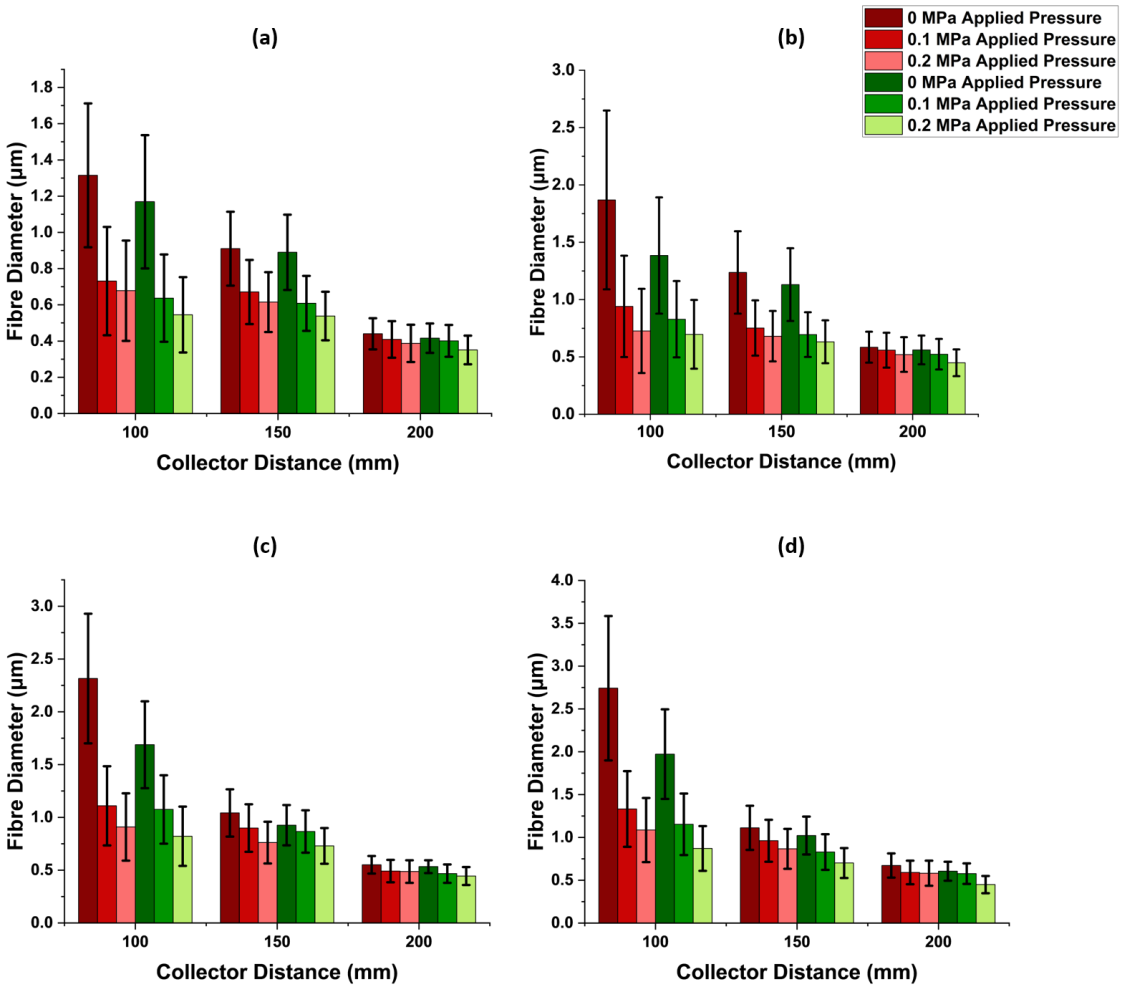

Figure 36. *Fibre diameter against processing parameters. (a) PEO 40% (b) PVP 40% (c) PEO 50% (d) PVP 50%. The error bars show the standard deviation of the results.*

Increasing polymer concentration from 40% to 50% leads to larger fibre diameters for both PEO-H2O and PVP-H2O solutions, regardless of other parameters. Higher concentration solutions have greater viscosity, which makes them more resistant to deformation and stretching, resulting in thicker fibres. This is a common observation in fibre spinning, where higher viscosities tend to correlate with larger fibre diameters.

As the collector distance increases from 100 mm to 200 mm, there is a noticeable reduction in fibre diameter across both vessels (60 mm and 75 mm). A larger collector distance allows the polymer jets more time to stretch and thin out before reaching the collector surface, contributing to the formation of finer fibres. Furthermore, for each polymer solution, the variation in fibre diameters at a collector distance of 200 mm is lower than at shorter collector distances, as evidenced by the relatively uniform heights of the stacked columns in Figure 33 at 200 mm in comparison to the other collector distances. This suggests that the impact of parameters such as vessel geometry and gas pressure (within the magnitudes used in this study) diminishes as the collector distance increases. Therefore, optimising the collector distance is shown to be a sustainable method of producing finer fibres while minimising the energy-intensive adjustments to other parameters such as gas pressure or vessel speed.

The standard deviation of fibre diameter generally increases with higher applied pressure, indicating a loss in uniformity. Higher applied gas pressures are associated with more turbulent jet behaviour, resulting in greater fluctuations in fibre diameter as the jet exits the spinning vessel.

This suggests that, while increased pressure helps reduce fibre diameter, it also induces greater instability in the jet, making it more challenging to achieve consistent diameters. To quantify fibre uniformity, the coefficient of variation (CV) was calculated by normalising the standard deviation to the mean fibre diameter and expressing it as a ratio. The CV provides a direct comparison of fibre uniformity across different experimental conditions, where lower CV ratios correspond to more consistent fibre diameters.[221]

At higher concentrations (50%), CV is lower compared to lower concentrations (40%) under same conditions, meaning the variation is relatively smaller in relation to the average diameter. This suggests that fibres produced at higher concentrations have a more consistent structure relative to their thickness. For instance, when using the 75 mm vessel, the CV ratio for PEO 50% at no applied gas pressure and at a collector distance of 100 mm provides a value of 0.24 (Figure 34d). Under the same parameters, PEO 40% is shown to have a CV ratio of 0.31 (Figure 34b).

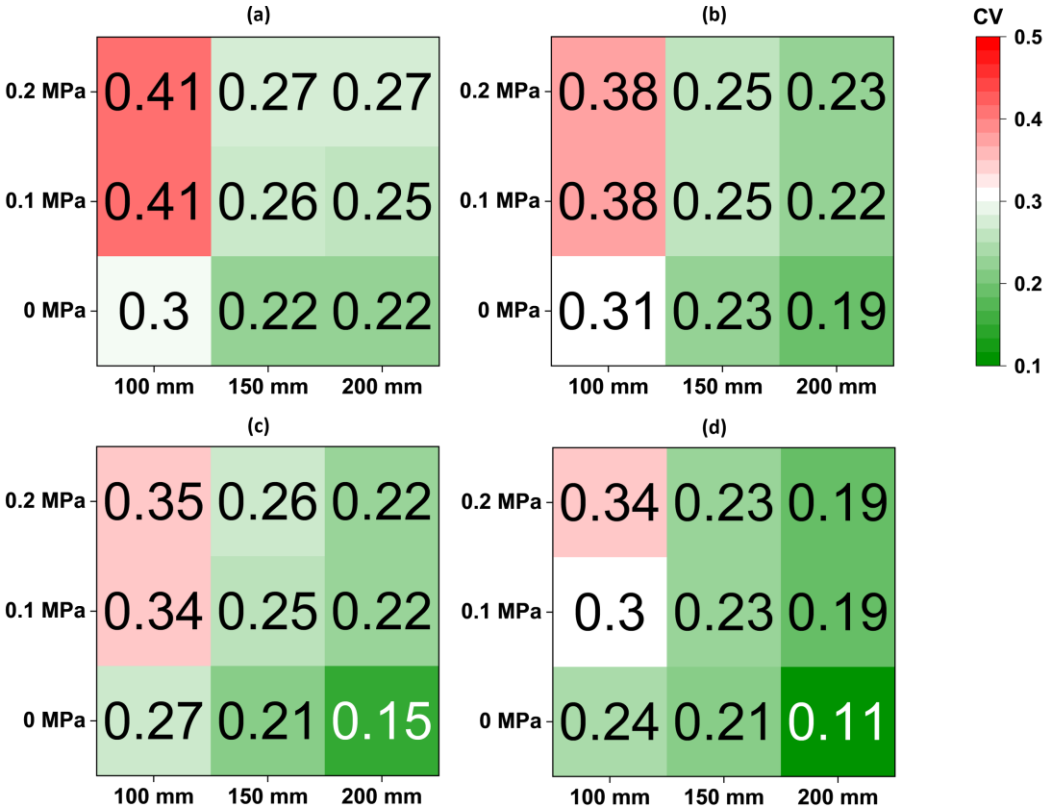

Figure 37. *CV ratio (standard deviation divided by mean fibre diameter) at (a) PEO 40% & 60 mm vessel, (b) PEO 40% & 75 mm vessel, (c) PEO 50% & 60 mm vessel, (d) PEO 50% & 75 mm vessel*

Similarly, PVP 50% shows lower CV values than PVP 40% under some conditions (Figure 35). Overall, PEO 50% tends to yield fibres with lower CV ratios. This aligns with the expectation that high viscous polymer solutions offer more stable and uniform fibre formation. This effect of viscosity on CV ratios is also evident when evaluating all for four polymer solutions, as shown by the comparatively higher red shading in Figure 35 (PVP) in comparison to Figure 34 (PEO) where both graphs use the same colour scale.

However, for all solutions greater collector distances are shown to be associated with reduced CV ratios across all polymers and

concentrations, indicating an improvement in uniformity. For instance, increasing the collector distance from 100 mm to 200 mm when using PVP 50% with the 60 mm vessel under no applied gas pressure improved the CV from 0.31 to 0.21 (Figure 35c).

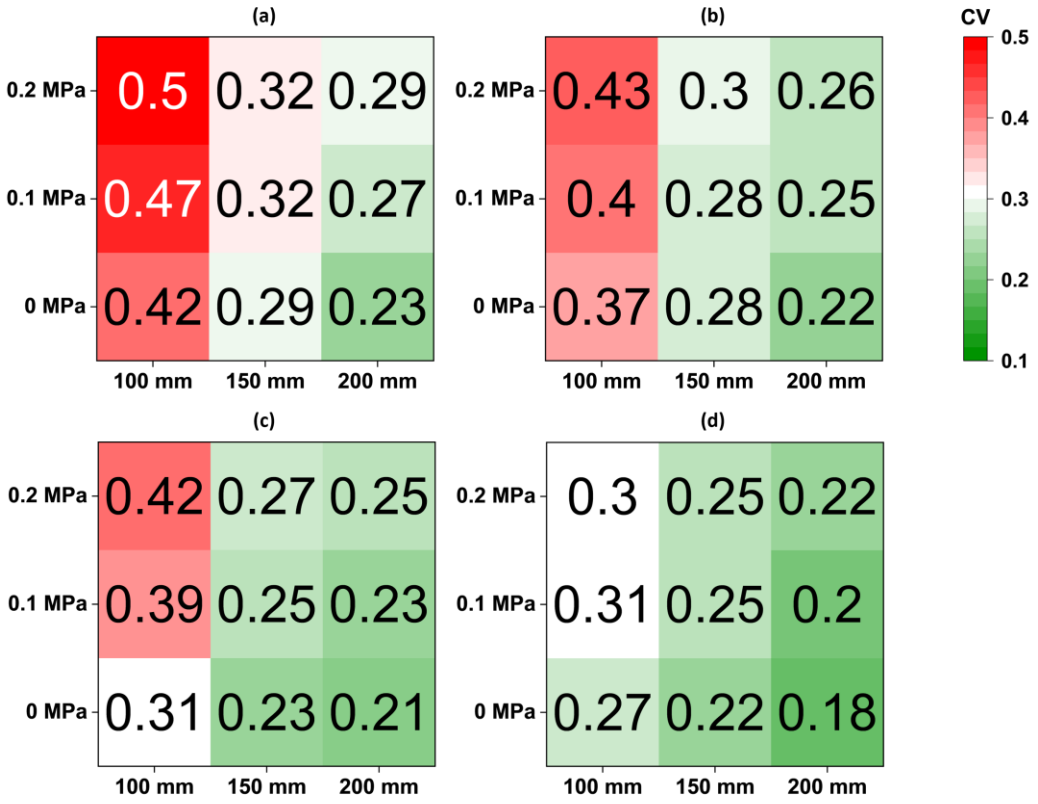

Figure 38. *CV ratio (standard deviation divided by mean fibre dimeter) at (a) PVP 40% & 60 mm vessel, (b) PVP 40% & 75 mm vessel, (c) PVP 50% & 60 mm vessel, (d) PVP 50% & 75 mm vessel*

Comparable to the effects of the collector distance, the 75 mm vessel exhibited superior fibre uniformity and alignment compared to the 60 mm vessel. This enhancement was particularly evident with PVP 50% at 0.2 MPa of applied gas pressure and a collector distance of 200 mm, where the CV value improved from 0.21 to 0.18 (Figure 35c and Figure 35d).

Fibre alignment showed a similar trend to fibre uniformity, where a greater coherency was observed with the 75 mm vessel across all processing parameters. This shows that the greater centrifugal forces generated by the larger vessel results in more aligned fibres as the polymer jets experience more tension and are drawn in a consistent direction. The improvement in fibre alignment was most pronounced at the shortest collector distance and highest applied gas pressures used in this study, along with the lowest-viscosity solution (PVP 40%), as shown in Figure 36.

Figure 36a-c show the scanning electron micrograph, distribution and orientation graph, respectively, for PVP 40% using the 60 mm vessel at a collector distance of 100 mm under an applied gas pressure of 0.2 MPa. A coherency of 0.235 was achieved under these conditions. The fibre alignment was shown to improve under the same conditions using the 75 mm vessel as indicated in Figure 36d-f where a coherency of 0.322 was achieved. Fibre alignment further improved when using the two vessels with PVP 40% under an applied gas pressure of 0.2 MPa at a collector distance of 200 mm. The coherency improved to 0.470 and to 0.481 for the 60 mm and 75 mm vessel respectively when increasing the collector distance from 100 mm to 200 mm as shown in Figure 36i and Figure 36l. These findings further validate how the vessel geometry can be optimised to improve fibre morphology, at the expense of no additional energy requirements during fibre production.

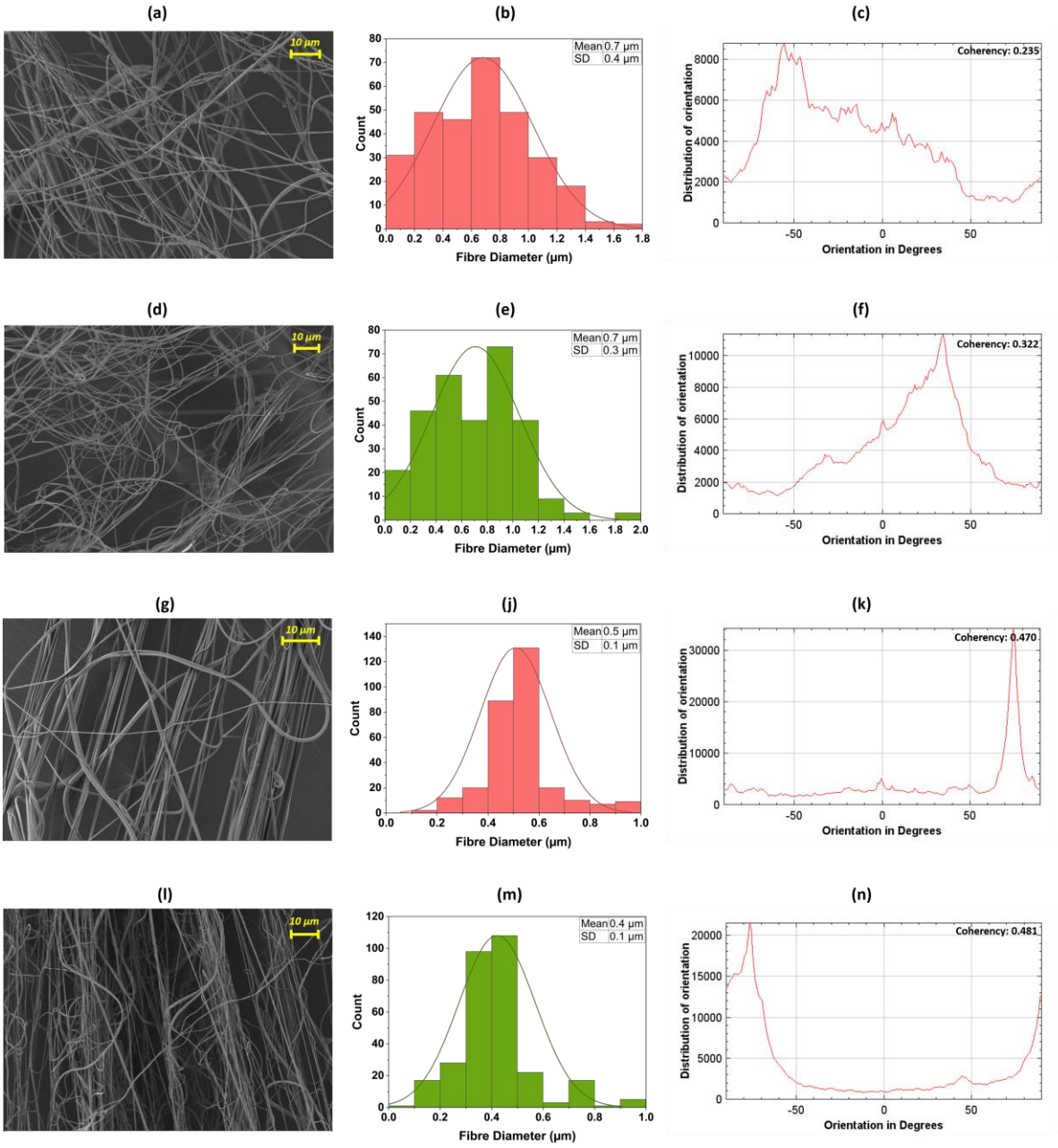

Figure 39. *Figure 36a-f is at a collector distance of 100mm and 0.2 MPa applied gas pressure. (a) Micrograph of PVP 40% & 60 mm vessel, (b) Fibre distribution of PVP 40% & 60 mm vessel, (c) Fibre orientation of PVP 40% & 60 mm vessel, (d) Micrograph of PVP 40% &*

Similar to the vessel geometry, the collector distance is identified to be a critical parameter in sustainably enhancing both fibre uniformity and fibre alignment. This effect was consistent across all solutions used in this study. Figure 37 demonstrates this effect for PEO 50% which

produced the most favourable results in achieving finer fibre, with the best alignment and uniformity under all conditions of other parameters in this study. For instance, at a collector distance of 100 mm using the 75 mm vessel with no applied pressure produced a coherency of 0.620 (Figure 37f). Whereas, PVP 40% (which produced the least favourable fibre characteristics in this study) under the same parameters produced a coherency of 0.322 (Figure 36f). These metrics are shown to further improve with a collector distance of 200 mm, where the best coherency in this study of 0.733 was achieved with PEO 50% using the 75 mm vessel at a collector distance of 200m under no applied gas pressure (Figure 37i).

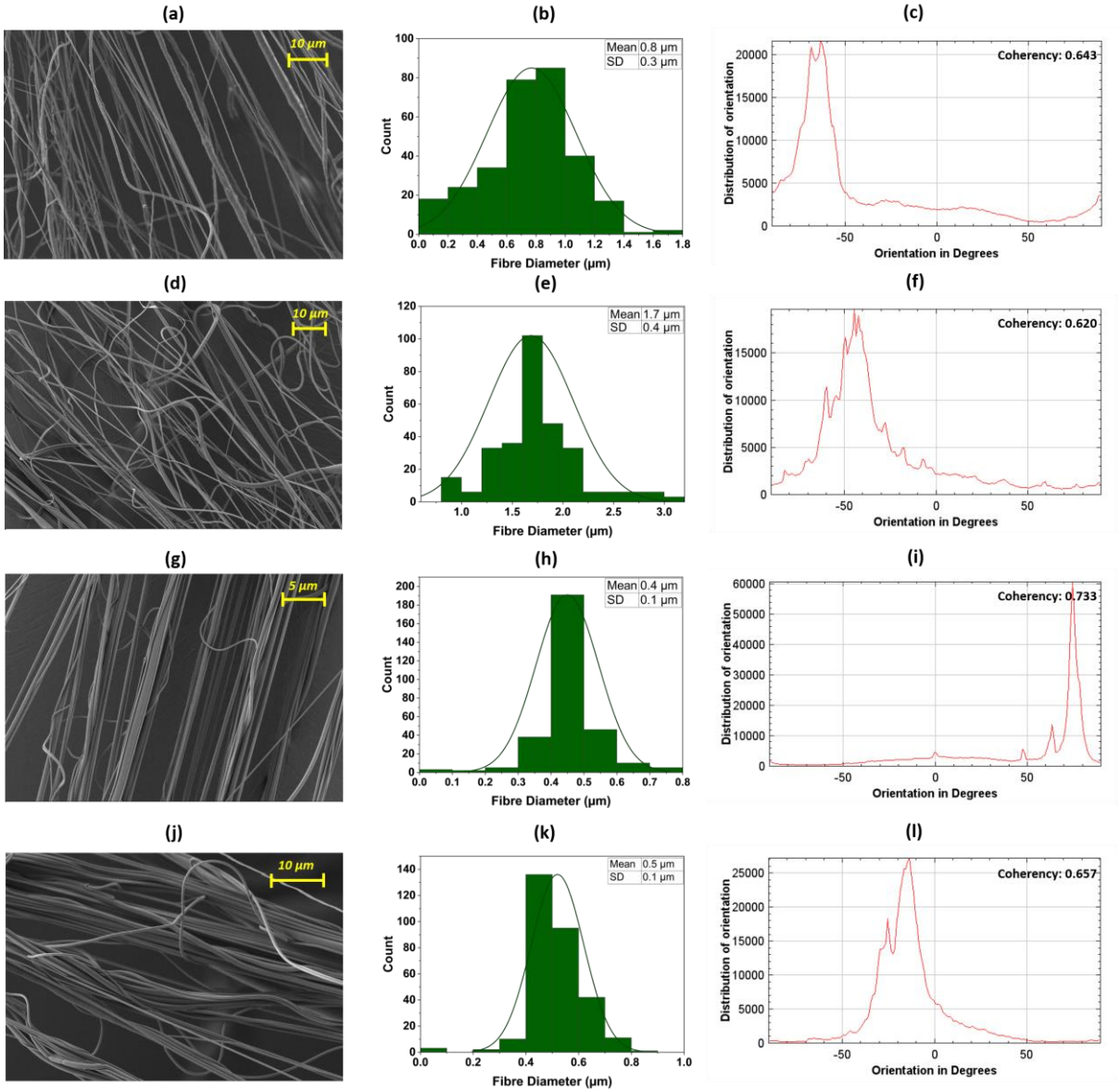

Figure 40. *Scanning electron micrographs, fibre diameter distribution graphs and orientation distribution graphs of (a-c) PEO 50%, no applied gas pressure & 100 mm collector distance, (d-f) PEO 50%, 0.2 MPa applied gas pressure & 100 mm collector distance, (g-i)*

6.4 Physical Interpretation of Vessel Diameter Effects

According to Newton's second law of rotational motion, for the same mass and concentration of polymer solution, the 75 mm vessel generates a centrifugal force that is 25% higher than the 60 mm vessel when spinning at the same rotational speed. This equates to the 60 mm

vessel needing to rotate at 14,534 RPM to produce a centrifugal force in of the 75 mm vessel rotating at 13,000 RPM. However, the motor used in this study has a maximum achievable speed of 13,000 RPM.

For instance, considering 4ml of a 50% concentration of a polymer dissolved in water equates to a mass of 0.006 kg. Whereas, 13000 RPM equates to an angular velocity of 1361.4 rad/s. Inputting this into the equation of rotational motion (equation 2), for the 60 mm vessel and the 75 mm vessel, results in a centrifugal force of 0.33 N and 0.42 N, respectively. Dividing the centrifugal forces obtained from the 75 mm vessel by the centrifugal force of the 60 mm vessel shows a 25 % increase. This effect is the same for any mass of a specific solution used in the two vessels.

This theoretical understanding of the relationship between centrifugal force, vessel diameter and rotational speed establishes a foundation for practical design considerations, particularly in selecting geometry that optimises fibre formation while maintaining energy efficiency.

6.5 Design Implications for Pressure Spinning Systems

The results of this study offer practical design strategies for pressure spinning setups aiming to enhance fibre morphology and energy efficiency. A key feature of the experimental system is its modularity: rather than altering the collector geometry, the vessel itself is repositioned vertically within a fixed conical collector. This approach enables rapid tuning of collector distance without requiring structural

modifications. Such a setup may be particularly advantageous in industrial applications where space constraints or material uniformity demands require quick adjustment of jet travel distance.

Although increasing vessel diameter offers a sustainable and energy-efficient means of increasing centrifugal force, owing to the direct proportionality between radius and force as defined by Newton's second law of rotational motion (equation 1) the impact of increasing rotational speed is significantly greater due to its quadratic relationship with centrifugal force. For instance, doubling the radius from 60 mm to 120 mm would only double the centrifugal force from 0.33 N to 0.67 N. In contrast, doubling the RPM from 13,000 to 26,000 would result in four times more centrifugal force, which is an increase from 0.33 N to 1.33 N, due to the 'ω^2' dependence.

A maximum rotational speed of 36000 RPM has been experimented with pressure spinning to date.[29] To match the centrifugal force of a 60 mm vessel spinning at this speed, a vessel operating at 13,000 RPM would need to be approximately 460 mm in diameter. While theoretically feasible, such a large vessel would be mechanically and spatially impractical, requiring extensive structural support and oversized collector walls to preserve jet path length and fibre alignment. These limitations highlight that while increasing vessel diameter is effective for enhancing fibre morphology without increasing power demand, it may be best suited as a supplementary strategy when high rotational speeds are not feasible due to motor constraints or energy limitations.

From a design standpoint, pressure spinning systems can benefit from adjustable platforms, interchangeable vessel sizes and modular collector configurations. In addition to these features, orifice geometry such as nozzle diameter and shape also presents an opportunity to manipulate fibre formation by influencing jet initiation, stability and thinning. These findings reinforce the value of geometric optimisation as a powerful and sustainable lever for tailoring fibre characteristics in scalable manufacturing environments. However, extending collector distance in industrial-scale systems poses challenges, including spatial limitations, increased turbulence and potential fibre entanglement. These factors may offset alignment benefits, necessitating a balance between morphological quality and process scalability in future system designs.

6.6 Concluding Remarks

A key design feature in this study was the ability to manipulate the placement of the rotary vessel within a conical-shaped collector rather than adjusting the collector walls to achieve the desired collector distance. This approach offers greater flexibility in tuning processing parameters without altering the overall structure of the collector and minimising structural modifications. Additionally, this design simplifies experimental setup adjustments while maintaining reproducibility, making it a practical and scalable method for fibre production. This study examined the influence of collector distance and vessel diameter on polymeric fibre production via pressure spinning, highlighting their

effects on fibre characteristics, production rate and energy consumption. Larger collector distances consistently resulted in thinner fibres due to increased jet elongation, with fibres being more aligned and uniform at 200 mm compared to 100 mm. The 75 mm vessel outperformed the 60 mm vessel in producing finer fibres and achieving greater energy efficiency, due to its enhanced radial dispersion and distribution capabilities. While production rates were slightly higher with the 75 mm vessel, both vessels showed reduced energy consumption per unit mass of fibre formed at larger applied gas pressure magnitudes and enhanced fibre morphology at larger collector distances. This demonstrates the interplay between process parameters and production efficiency in producing fibres with minimal environmental impact using pressure spinning. Furthermore, the demonstrated increase in fibre alignment, especially from increasing collector distance, can be necessary for advanced applications like biomedical scaffolds and promoting conductivity. By achieving these superior morphologies through passive geometric tuning, the process minimises reliance on high-energy parameter inputs, directly fulfilling the mandate for sustainable and energy-efficient manufacture. These findings underscore the critical role of collector distance and vessel geometry in optimising fibre morphology and energy efficiency, providing valuable insights for scaling up sustainable fibre production systems.

Chapter 7 – Limitations and Future Work

This thesis demonstrates the critical role of Green Chemistry and Green Engineering in advancing sustainable polymeric fibre production. By focusing on Pressure Spinning**,** this study validated its potential to significantly reduce environmental impacts compared to conventional, energy-intensive methods like electrospinning and phase separation. The findings directly extend existing knowledge and yield three primary novel contributions to the field:

- **Pioneering Geometric Optimisation:** This research, unlike previous Pressure Spinning studies which primarily focused on rotational speed and applied gas pressure, systematically introduced and quantified the unaddressed parameters of rotary vessel geometry and collector distance. This provided novel, non-energy-intensive controls for achieving enhanced fibre morphology, uniformity and alignment, thereby establishing a critical geometric roadmap for industrial design.
- **Validation of Green Core-Sheath Architectures:** The work pioneered a clean, sustainable route for the production of core-sheath fibres using water-soluble, low-toxicity polymers (PEO/PVP). This demonstrated the viability of manufacturing complex, multifunctional structures without reliance on hazardous solvents or highly complex equipment, a necessary step for advanced applications.

- **Quantitative Energy Benchmarking:** Key findings revealed that optimising process parameters enhances fibre quality and production efficiency while rigorously minimising energy consumption and material waste. This contributes critical quantitative data that validates Pressure Spinning as an energy-efficient platform capable of achieving superior performance and industrial sustainability goals.

7.1 Limitations

While this thesis has demonstrated the significant potential of pressure spinning as a sustainable and high-throughput fibre manufacturing technique, it is important to acknowledge the methodological, analytical and practical limitations encountered during the course of the research. These limitations span instrumentation constraints, scope of materials tested, characterisation techniques and scale of implementation. Recognising these boundaries is essential not only for contextualising the findings, but also for shaping the direction of future investigations aimed at industrial translation and environmental benchmarking.

Each of the experimental chapters, focusing on submicrometre single-polymer fibres, core-sheath fibres and process optimisation via vessel and collector design, provided valuable insights but was limited by certain simplifying assumptions, equipment availability and experimental design decisions. The subsections below detail the specific limitations encountered in each R&D chapter (Chapters 4, 5 and

6), highlighting opportunities for methodological refinement and broader application in subsequent studies.

7.1.1 Sustainability of Submicrometre PEO and PVP Fibre Production

One of the most significant limitations in the investigation of submicrometre PEO and PVP fibre production was the absence of real-time monitoring technologies during the spinning process. In this study, the onset and termination of fibre formation were determined manually through visual (video) inspection and timing. This approach introduces a degree of uncertainty in accurately measuring the spin duration, which directly affects the reliability of calculated production rates and associated energy consumption figures. Without precise temporal resolution, slight variations in when fibres begin to form or cease production may lead to over or underestimation of throughput and energy efficiency. The integration of real-time monitoring tools, such as high-speed cameras, photodetectors or fibre tracking sensors, would significantly improve the resolution of spin event data.[222] These tools could capture the exact moment when fibre jets initiate and terminate, allowing for more rigorous and repeatable quantification of operational metrics and process transitions.

The polymer selection in this chapter, while well-controlled, was limited in scope to two synthetic, water-soluble polymers, PEO and PVP. Although these materials are biocompatible and commonly used in biomedical and filtration contexts, their synthetic origin and specific

rheological properties restrict the generalisability of the findings. In particular, the study does not explore the behaviour of naturally derived or biodegradable polymers such as alginate, chitosan, cellulose derivatives or starch-based materials. Many of these bio-origin polymers can be sourced from agricultural or industrial waste streams, enhancing their appeal for sustainable manufacturing. Their renewable origin, biodegradability and potential for upcycling make them highly relevant to green material development. As such, their exclusion from the current study limits the ecological scope and broader applicability of the results, particularly in the context of advancing environmentally conscious manufacturing strategies aligned with circular economy principles.

Additionally, while this chapter made meaningful strides in assessing the energy efficiency of pressure spinning, it did not include a comprehensive Life Cycle Assessment (LCA). The environmental evaluation focused primarily on energy consumption during the fibre formation phase, omitting other critical stages such as raw material extraction, polymer and solvent synthesis, equipment manufacturing and end-of-life disposal. Without this cradle-to-grave perspective, the broader sustainability performance of pressure spinning relative to conventional fibre production methods such as electrospinning or phase separation remains partially understood. A full LCA incorporating material sourcing, emissions, toxicity and recyclability would offer a more holistic and comparable assessment of environmental impacts.

Finally, the method of applying gas pressure in this study presents an additional limitation. A decompressed nitrogen cylinder was used to

provide pressurised gas into the vessel, which offers convenience but does not accurately reflect the energy demand or cost implications of generating that pressure. In practical or industrial contexts, compressed gases must often be produced on-site or transported and stored under high pressure, both of which carry energy and infrastructure penalties. Moreover, this setup does not account for real-time energy inputs associated with pressure delivery. A more representative system would involve a gas pump or compressor that actively generates and maintains the required pressure, thereby enabling a more accurate accounting of the energy associated with applied pressure and its contribution to the overall environmental footprint of the process.

7.1.2 Sustainability of Core-Sheath Fibre Production

A key limitation of the core-sheath fibre study lies in the absence of application-specific dimensional optimisation. Although this chapter demonstrated the sustainable feasibility of producing concentric core-sheath fibres using a dual-reservoir pressure spinning setup, the dimensions of the fibres, such as the ratio between the core and sheath, the total fibre diameter and the degree of alignment, were not tailored for any particular end-use application. In practical contexts, fibre architecture plays a critical role in determining functional performance. For example, in drug delivery, a thicker sheath may be required to control release kinetics, while in tissue scaffolding, uniform diameters and aligned fibres are essential for mimicking the extracellular matrix and guiding cell growth.[223] Similarly, in biosensing applications, the

core may contain conductive or responsive materials that must be precisely dimensioned to ensure signal fidelity.[224] However, the current study did not seek to define such relationships or performance thresholds. As a result, while the general viability of core-sheath fibre production was established, the findings remain limited in terms of translation to real-world application settings.

Another constraint in this study is the limited range and resolution of the characterisation techniques used to evaluate fibre structure. The analysis of core-sheath morphology relied primarily on optical microscopy, scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR). While these methods confirmed the presence of distinct core and sheath phases, they lack the spatial resolution and three-dimensional insight required to thoroughly assess the internal architecture of multilayer fibres. Optical microscopy, although convenient, provides only surface-level visualisation. SEM offers better resolution but is limited to surface imaging and often requires conductive coating, which can obscure fine structural features. FTIR spectroscopy, while useful for confirming chemical composition, cannot resolve spatial distribution or interface quality within the fibre.

To fully evaluate core-sheath fibre integrity, particularly interface uniformity, layer thickness distribution and potential defects, more advanced imaging modalities are required. Confocal laser scanning microscopy, for instance, can provide optical sectioning and 3D reconstruction of transparent or fluorescent-labelled fibre systems, enabling detailed assessment of the internal structure.[225] X-ray

microtomography (micro-CT) could also offer non-destructive volumetric imaging to reveal the core and sheath interfaces across an ensemble of fibres.[226] Without such tools, the structural fidelity of the produced fibres, particularly under varying pressure conditions remains partially understood. This limitation restricts the ability to draw robust conclusions about their reliability, scalability and suitability for precision-demanding applications such as drug encapsulation or multifunctional wearable systems.

7.1.3 Optimising Fibre Morphology and Production Efficiency in Pressure Spinning through Vessel and Collector Design

A notable limitation of this chapter is the scale of the experimental setup. The two vessel diameters tested, 60 mm and 75 mm, are representative of benchtop or lab-scale systems and may not fully capture the potential mechanical or process complexities encountered in industrial-scale fibre production. In real-world manufacturing environments, larger vessel diameters and more extended collector distances maybe required to meet throughput demands and integrate with automated systems. However, the current study did not investigate vessels larger than 75 mm or collector distances beyond 200 mm, which restricts the extrapolation of the findings to potential full-scale production scenarios. As a result, the scalability of the identified trends, such as improved fibre uniformity with wider vessels and increased separation with longer collector distances, remains to be validated under high-volume manufacturing conditions.

Another important consideration that was not fully addressed involves the interplay between polymer solution viscosity and energy input across different stages of the process. High-viscosity polymer solutions typically require prolonged pre-processing using vortex mixers or magnetic stirrers to achieve full homogenisation, which contributes to energy consumption prior to the spinning phase. Conversely, lower-viscosity solutions, while easier to mix, often demand higher rotational speeds (up to 36000 RPM) to generate sufficient centrifugal force for stable fibre extrusion.[116] This dynamic introduces a trade-off between the energy required for solution preparation and that required during the spinning process itself. However, this study did not undertake a detailed comparative analysis of total energy expenditure across different viscosity regimes. Without such a holistic energy balance, it is difficult to determine the most sustainable or cost-effective combination of solution formulation and processing conditions. Optimising this trade-off could play a crucial role in reducing the overall environmental footprint and improving the energy efficiency of pressure spinning systems.

Furthermore, while the chapter introduces promising design variables for improving fibre yield, uniformity and morphology, it stops short of addressing key elements of industrial implementation. In practice, pressure spinning systems intended for commercial use may need to incorporate features such as continuous-feed (infusion) reservoirs to eliminate manual reloading, automated collection platforms to streamline post-processing and scalable vessel fabrication methods to ensure cost-efficiency. These components are critical for transitioning

from experimental setups to production-ready systems capable of operating reliably over long durations. Their omission from the present work means that the proposed design improvements, though promising at a proof-of-concept level, remain disconnected from the operational realities of fibre manufacturing at scale. Addressing these engineering integration challenges will be essential for translating the demonstrated lab-scale efficiencies into viable industrial solutions.

7.2 Future Work

The findings presented in this thesis provide a solid foundation for advancing the sustainability, efficiency and functionality of pressure spinning as a fibre manufacturing technique. However, as outlined in the preceding limitations, several critical avenues remain unexplored or underdeveloped. These represent both technical challenges and opportunities for innovation that could significantly enhance the scientific and industrial relevance of pressure spinning technology.

Future research should focus on extending the scope of materials, improving the precision and scalability of the spinning process and aligning fibre properties more closely with specific application requirements. In parallel, greater emphasis is needed on real-time process monitoring, advanced characterisation, lifecycle environmental assessment and integration with industrial systems. By addressing these interconnected areas, future work can accelerate the transition of pressure spinning from a promising laboratory method to a widely

adopted, sustainable manufacturing platform for high-performance polymeric fibres.

7.2.1 Biobased and Functional Polymers

The research demonstrated the benefits of using water-soluble polymers like PEO and PVP in polymeric fibre production. Future work could explore incorporating biodegradable and naturally derived polymers, such as polylactic acid (PLA), chitosan and alginate to further minimise environmental impact. For instance, alginate, a polysaccharide derived from brown seaweed, is a particularly attractive candidate for sustainable fibre production due to its renewable origin, biocompatibility and biodegradability.[128] Its unique ability to form hydrogels in the presence of divalent cations, such as calcium, allows for straightforward fibre formation via spinning techniques. Additionally, investigating polymer blends or composite fibres with bio-based fillers such as cellulose nanocrystals or clay nanoparticles could enhance mechanical and thermal properties while maintaining sustainability.[227] Developing non-toxic additives to improve fibre conductivity, strength, or biocompatibility will also broaden the range of potential applications.

7.2.2 Broader Parameter Optimisation

To further refine fibre production, a comprehensive exploration of additional process parameters is essential. One critical area is understanding and mitigating fibre uniformity loss when applying gas

pressure. While increased pressure aids in polymer extrusion, it can introduce instabilities in the polymer jet, leading to variations in fibre diameter and morphology. Future work could focus on optimising pressure application techniques, such as controlled gas pressure ramping or pulsation, to maintain uniform fibre formation. Additionally, investigating the interplay between gas pressure and solution rheology could offer insights into stabilising the fibre jet, ensuring consistency across production batches.

Equally important is the consideration of how polymer solution viscosity influences energy demand throughout different stages of the process. High-viscosity solutions may typically require prolonged mixing or vortexing, supplemented with additional heating to achieve solution homogeneity during the preparation phase, increasing pre-processing energy input. Conversely, lower-viscosity solutions may reduce mixing times but often necessitate higher rotational speeds or greater applied pressures during spinning to achieve sufficient centrifugal and pressure-driven extrusion. This introduces a trade-off between energy consumed during solution preparation and energy used in the spinning phase. Future studies should focus on quantifying this interplay, enabling the identification of viscosity "sweet spots" that minimise total energy input while maintaining optimal fibre quality and production rate. Such optimisation could be further supported through process modelling and life cycle analysis to inform environmentally conscious design choices.

In addition to pressure and viscosity-related parameters, physical aspects of the spinning setup such as internal vessel geometry and

orifice configuration play a vital role in fibre quality. Adjusting vessel dimensions, such as diameter and height, or incorporating internal features like contours, could aid fluid flow and enhance uniformity. Similarly, optimising orifice size and spacing can strike a balance between fibre fineness and production efficiency, while advanced surface coatings may reduce blockages and ensure smooth extrusion.[228] Environmental factors, including temperature and humidity, also warrant systematic investigation, as they directly affect solvent evaporation and fibre integrity. Leveraging computational fluid dynamics (CFD) simulations could streamline these optimisations, enabling precise control over process conditions and minimising trial-and-error experimentation. Collectively, these efforts will strengthen pressure spinning as a reliable, scalable and versatile manufacturing process for advanced applications.

7.2.3 Lifecycle Assessment

It can be argued that the design of current submicrometre polymeric fibre manufacturing methods has been focused on quality and safety specifications. Consideration of green engineering principles and green chemistry principles in the design of polymeric fibre manufacturing methods can be utilised to enhance or consider environmental, social and economic factors.[138] It is beneficial to visualise these principles as parameters where the application of one parameter may enhance one or more other principles of green engineering and/or green chemistry. Regardless, two of the most important concepts that

designers are pushed to endeavour are considering the lifecycle and the first principle of green engineering, which promotes the minimal use of hazardous materials and energy obtained from hazardous means.[138]

A life cycle analysis (LCA) is a method that can be used to evaluate environmental impacts such as energy consumption at all stages of polymeric fibre. The materials and energy inputs at every section of the life cycle of a specific product and process fully captures their life cycle. If a product is environmentally friendly but is made using hazardous or non-renewable materials, the impacts are merely transferred to another part of the overall life cycle. Polymer and solvent selection are vital when considering the lifecycle. In the case of pressure spinning, regardless of the method's energy efficiency in comparison to other processes, if the extraction and manufacture of certain polymers and solvents offset any energy savings, there is no net sustainable advantage. This research evaluated how the environmental impact can be improved in terms of energy used in the 'manufacture' stage of the LCA of PEO and PVP polymeric nanofibres produced using pressure spinning.

Synthetic polymers are produced via polymerisation, which is an exothermic process and is derived from fossil fuels. Therefore, it is useful to not only consider the functional and safety aspect of polymers but also the life cycle of the polymer material itself. Similarly, for the solvent used (distilled water), the distillation process of water requires the liquid to be heated until evaporation, along with nitrogen gas compression methods and energy to produce various concentrations of polymeric solutions can compromise overall energy efficiency. However,

natural water is likely to suffice equally well in case commercial manufacturing is pursued. This study also proved that highly viscous polymeric solutions consumed more energy to produce polymeric fibres. This is also the case during the preparation of polymeric solutions of the same volume, where the high viscous solutions used in this study require more time to reach homogeneity on the magnetic stirrer. Hence, it is necessary to undertake a full life cycle analysis, including energy consumption during the application or use of polymeric fibres along with the distribution and end of life. However, the scope for this is too wide when considering the range of polymers, solvents and the number of applications polymeric fibres are utilised for. An approach may be to identify the most popular application and most popular polymer used to produce fibres for this application and evaluate its life cycle considering the most efficient method of manufacture.

7.2.4 Application-Specific Tailoring

The versatility of pressure spinning allows for precise control over fibre morphology, enabling tailored production for specific advanced applications. By optimising process parameters such as rotational speed, gas pressure and collector distance, critical fibre characteristics such as diameter, porosity and mechanical properties can be fine-tuned. For instance, in biomedical applications, the production of ultra-fine, porous fibres can be optimised to mimic the extracellular matrix, promoting cell adhesion and tissue regeneration.[229] Moreover, the

use of sustainable polymers like PEO and PVP enhances biocompatibility while reducing environmental impact.

In energy storage, optimising fibre diameter and uniformity increases surface area, improving ion transport and charge storage efficiency in batteries and supercapacitors.[230] Similarly, in filtration systems, controlling fibre porosity and diameter distribution enhances contaminant capture, while sustainable production methods minimise environmental harm. For wearable electronics, the alignment and uniformity of fibres can be adjusted to improve conductivity and mechanical flexibility.[231] By integrating sustainable practices and fine-tuning process parameters, pressure spinning offers a pathway for developing environmentally responsible, application-specific fibres with high performance and efficiency.

7.2.5 Hybrid Fibre Structures

Hybrid fibre structures present an exciting avenue for developing multifunctional materials by integrating the distinct properties of different polymers, fillers, or coatings within a single fibre. These structures address the limitations of traditional polymeric fibres, such as limited mechanical strength, low functionality, or poor conductivity, while expanding their potential applications. Building upon the core-sheath fibres explored in this research, further fibre structures can further enhance the versatility of pressure spinning, enabling the production of

fibres tailored for specific advanced applications in fields like biomedical engineering, energy storage and filtration.

Various types of hybrid fibres offer unique benefits. Core-sheath fibres, for example, allow for independent tuning of core and sheath properties, enabling applications such as conductive polymer cores with biodegradable sheaths for bioelectronics or temporary implants.[232] Coaxial multilayer fibres extend this concept by introducing additional functional layers, such as insulating or biocompatible coatings, making them suitable for advanced sensors or neural interfacing.[233] Multicore fibres, which embed multiple discrete cores within a single sheath, enable parallel functionalities such as multi-analyte sensing or simultaneous electrical and optical transmission.[234] Similarly, side-by-side (Janus) fibres combine two distinct materials in a parallel configuration, creating anisotropic chemical, mechanical, or surface properties useful in stimuli-responsive textiles, directional drug delivery and filtration systems.[235, 236, 237] These complex architectures expand the functional design space of polymeric fibres, supporting advanced use cases in biomedical, wearable and environmental technologies.

Future research on hybrid fibre structures should prioritise process optimisation, particularly in refining pressure spinning techniques to achieve precise control over layer thickness, composition and uniformity, along with efficiency related properties. Sustainability remains a key focus, with an emphasis on incorporating biodegradable or bio-based materials into hybrid fibres to align with green chemistry

and engineering principles. Furthermore, functional testing under real-world conditions, such as mechanical stress, thermal cycling, or chemical exposure, will provide valuable insights into the practical performance of these fibres.

By advancing hybrid fibre structures, pressure spinning can transcend traditional fibre production methods, paving the way for innovative materials that address contemporary technological and environmental challenges. This evolution not only enhances the utility of polymeric fibres but also aligns with global efforts to promote sustainable and high-performance materials.

7.2.6 Advanced Characterisation

Application-specific tailoring requires specific characterisation methods to ensure that the fibres meet the distinct functional demands of their intended applications. Further studies should employ advanced techniques such as atomic force microscopy (AFM) for surface morphology analysis, in situ monitoring to observe fibre formation dynamics and X-ray diffraction (XRD) to evaluate crystallinity.[238] Advanced spectroscopic techniques, like Raman and nuclear magnetic resonance (NMR), can provide further insights into the chemical interactions within fibres.[239] Additionally, using 3D imaging technologies, such as micro-computed tomography (micro-CT), can enable a deeper understanding of fibre porosity and internal structures, crucial for applications like filtration and tissue scaffolds.

These characterisation techniques are not only essential for understanding material properties but are also pivotal in promoting sustainability in polymeric fibre production. Crystallinity, for example, can significantly influence the mechanical strength and biodegradability of fibres, which is critical for applications in biodegradable packaging and biomedical scaffolds. Enhanced control over crystallinity can reduce the environmental impact by tailoring fibres to degrade efficiently under specific conditions, thus aligning with the principles of Green Chemistry.

Chemical interactions within fibres, as revealed by Raman and NMR, help in assessing the compatibility of fibres with various functional additives or coatings. This insight is vital for developing fibres that require minimal post-processing while achieving desired application-specific properties, such as conductivity in energy storage devices or bioactivity in medical applications. By optimising chemical interactions, the need for additional, potentially harmful, chemical treatments can be reduced, contributing to a more sustainable production process.

Fibre porosity and surface morphology play a critical role in applications like filtration, where higher porosity increases efficiency by enhancing permeability while reducing the material's weight.[240] Understanding and optimising porosity can lead to fibres that achieve the same functional performance with less raw material, reducing resource consumption and waste. Similarly, surface morphology impacts cell adhesion and proliferation in biomedical scaffolds and a deeper understanding of these parameters can help design fibres that mimic

natural tissues more effectively, thereby reducing the trial-and-error phase of production and its associated waste.[241]

The integration of these advanced characterisation methods facilitates the precise tailoring of fibre properties to meet specific application requirements while minimising environmental impacts. This approach not only aligns with the principles of Green Chemistry and Green Engineering but also enhances the scalability and economic viability of sustainable polymeric fibre production.

7.2.7 Real-time Process Analytics

A major opportunity for advancing the precision and scalability of pressure spinning lies in the incorporation of real-time process analytics. In the current study, key operational parameters such as the start and stop points of fibre formation were estimated manually, which introduced variability in measurements of spin duration, production rate and energy efficiency. Future setups should integrate real-time monitoring tools, such as high-speed cameras, thermal sensors and fibre flowrate sensors, to provide continuous, accurate feedback on system behaviour during spinning. High-speed imaging, for instance, could capture the dynamic evolution of fibre jets at the orifice, allowing researchers to determine precisely when fibre extrusion begins, stabilises, or ceases. Similarly, thermal sensors could be used to track temperature gradients that may affect solvent evaporation, solution viscosity, or phase transitions in real time.[242]

The implementation of such real-time analytics would not only enhance measurement accuracy but also open the door to adaptive process control. By feeding sensor data into control algorithms or machine learning models, the system could automatically adjust parameters such as rotational speed, gas pressure, or flow rate in response to fluctuations in fibre formation behaviour. This feedback-driven optimisation would ensure consistent fibre quality and production efficiency, especially in long-duration or industrial-scale runs. Moreover, real-time data acquisition is essential for building predictive models of process performance, enabling the development of digital twins or simulation tools that can accelerate design and scale-up. Overall, integrating real-time process analytics represents a key step toward transforming pressure spinning into a fully digitised, intelligent manufacturing platform.

7.2.8 Industrial Scale-Up

To fully commercialise pressure spinning, a systematic approach is needed to scale up production while ensuring product consistency and cost-effectiveness. Future research should explore methods of achieving continuous production at industrial scales. These systems can integrate automation technologies for precise control over operational parameters such as rotational speed, gas pressure and temperature, thereby ensuring uniform fibre characteristics.

Additionally, the design of larger spinning vessels, tailored to accommodate higher polymer throughput, will be critical. This requires careful evaluation of material properties and mechanical stability to prevent operational inefficiencies or downtime during prolonged industrial use. Incorporating sensor technology can also aid real-time monitoring of process control parameters of fibre formation processes, providing feedback to optimise system performance dynamically.

Energy optimisation is another essential factor for scalability. Developing energy recovery systems, such as regenerative braking in motors, can significantly reduce operational costs and environmental impact. Life cycle assessments should accompany these developments to quantify and mitigate environmental footprints across production stages.

Collaboration with industrial partners will also be pivotal. Industry insights can identify practical challenges such as material handling, waste management and compliance with regulatory standards. Prototyping and pilot-scale trials in industrial settings can bridge the gap between laboratory research and full-scale implementation.

Lastly, integrating machine learning and computational modeling will enhance scalability by enabling predictive maintenance and process optimisation.[243] These tools can identify potential bottlenecks and propose solutions to maximise throughput while maintaining fibre quality. Scaling up pressure spinning, therefore, not only requires

technological advancements but also a multidisciplinary effort to address the complexities of industrial operations comprehensively.

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