Assessing alternative textile recycling methods: Combining chemical processing with electrospinning technology for life-cycle circularity

Courtney Jo Barbour

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Assessing alternative textile recycling methods:

Combining chemical processing with electrospring technology for life-cycle circularity

by

Courtney Barbour

A thesis submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Apparel Merchandising & Design

Program of Study Committee:
Rachel Eike, Major Professor
Chunhui Xiang
Ling Zhang
Alex Braidwood

The student author, whose presentation of the scholarship herein was approved by the program of study committee, is solely responsible for the content of this thesis. The Graduate College will ensure this thesis is globally accessible and will not permit alterations after a degree is conferred.

Iowa State University
Ames, Iowa
2020

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ABSTRACT

Negative environmental impact regarding overconsumption and textile waste is a major sustainable issue facing the apparel and textiles industry. Increased consumption stemming from fast fashion has led to an increase in overall textile production and therefore, waste. Oftentimes, textile waste ends in landfills or is incinerated, therefore contributing to pollution and environmental burden. While nearly all textiles have the capacity to be repurposed or recycled, recycling at the fiber level (such as chemical recycling) is limited in industry practice.

This experimental study was conducted to explore an alternative textile recycling method for the production of single-fiber content nanofiber textiles. Chemical recycling was assessed to determine the processes and percentages needed for successful fiber blend separation in order to develop a scientific protocol. The outcome of this protocol produced a solution for electrospinning a nano-textile. Additionally, electrospinning techniques were assessed to determine the quantity of solution and spinning time required to produce a recycled nano-textile that maintained similarity in weight (mass density) to the preliminary non-recycled textile. Results from this study documented the findings for chemical processing and nano-textile production, and a protocol combining the chemical recycling processes with electrospinning of recycled fiber blends was created.

The purpose of this research addressed the concept of life cycle circularity through the recycling of textiles. This research provided valuable feedback regarding the potential to remove textile waste from traditional waste streams for circular lifecycles through chemical recycling. Chemical recycling processes incur minimal to no fiber degradation through repetitive recycling, while electrospinning provides an opportunity to minimize the use of fiber blends through maintaining enhanced fiber properties.
CHAPTER I

INTRODUCTION

Opening and Outline

Each step of a textile’s life cycle produces negative environmental impact including the production, maintenance, and disposal of the textile. Because of the significant increase in textile production, it is important to discuss the related environmental consequences as well as the potential areas for improvement. Within the introduction, environmental concerns of the apparel and textiles (AT) industry, connected to the increase of global textile consumption as a result of fast fashion markets and insatiable consumer demand, are exposed. An overview of textile recycling systems and the importance of these systems for environmental impact are brought to light. Previous work investigating the reusability and quality of recycled textiles regarding a textile’s life cycle assessment (LCA) are discussed with consideration to circular lifecycles. Finally, opportunities of nano-technology in recycled textiles are considered. Following the introduction, the purpose of this study, research questions, objectives, study assumptions and limitations, and operational definitions are presented.

Environmental Concerns of the Apparel & Textiles Industry

Overconsumption. The apparel and textiles (AT) industry is one of the most globalized industries in the world (Palamutcu, 2015). Fiber consumption has increased approximately 39%, per capita, since 2011, with expectations of continued growth due to the increasing global demand for textile products (Palamuctu, 2015; Sandin & Peters, 2018). This increase in fiber consumption is likely due to worldwide population growth, economic development, an increased market of technical textiles, and an increase in fast fashion consumption (Sandin & Peters, 2018; Palamutcu, 2015). The consumer-oriented nature of the industry, guided by the industry’s fast
fashion complex, results in the mass production and consumption of textiles and clothing. In 2003, the United States alone accounted for $180 billion in clothing and accessory retail sales (Chen & Burns, 2006). By 2018, it was reported that consumption practices had risen by approximately 400% since the 1980’s (CBC News, 2018). “Fashion fuels momentum for change, which creates demand for ongoing replacement of products with something that is new and fresh” (Hawley, 2009, p.181). Fast fashion is one of the largest contributing factors impacting the un-sustainable cycle of the AT industry (BSR, 2017). This cycle of unsustainability largely impacts social, environmental, and economic well-being (Forum for Fashion, 2007). Fierce competition in the industry commonly results in outsourced production, poor production practices, and extremely low retail prices for the consumer; leading consumers to a ‘throw-away’ mentality that ultimately impacts landfill waste (Stephens, 1985; Claudio, 2007). In one year alone, it is claimed that the AT industry contributes an estimated 25 billion pounds of solid waste to landfills in North America (CBC News, 2018). While increased production and consumption of textiles drives the industry economically, negative impacts regarding textile disposal increases as well (Hawley, 2009).

**Pollution.** The AT industry contributes to pollution during each phase of a textile’s life-cycle including the production and finishing of fibers and textiles, the use and maintenance of the product, and finally the disposal of the product (Chen & Burns, 2006). Forum for the Future (2007) argues that the AT industry requires greater resources for lower returns. The production of synthetic fibers requires high levels of energy consumption and are considered to be a “secondary carbon hot spot” in the emission of harmful greenhouse gases (BSR, 2017; Palamuctu, 2015). Approximately 63% of fibers, mainly synthetic, derive from non-renewable resources such as petrochemicals and crude oil (Sandin & Peters, 2018; Claudio, 2007). As oil-
based products take longer to break down in a landfill, they accumulate over time and negatively contribute to end-of-life pollution (Claudio, 2007). This accumulation of synthetic textiles in landfills as well as incineration of end-of-life textiles impacts water, soil, and air quality. The production of nylon, a widely manufactured synthetic fiber, has negative environmental impact at all levels: use of valuable petroleum resources, prolonged decomposition within a landfill, and harmful nitrous oxide emissions during production (Fletcher, 2014; Watson & Warnock, 2003; Rodie, 2010; Forum for the Future, 2007). In addition to the aforementioned impacts of synthetic fiber production, the increase in worldwide textile consumption has also created a steady decline in environmental status as valuable resources are being used daily that cannot be replaced. Due to limited petroleum resources, the textiles industry is finally being forced to adopt alternative resources (Chen & Burns, 2006). Textile recycling offers an alternative to the current textile production standards set forth by the industry by using pre-existing materials and reducing negative environmental impact. This study will specifically focus on the textile recycling of nylon due to current research gaps and negative environmental impacts created by nylon fiber production.

**Textile Recycling**

Recycling refers to the removal of waste from traditional waste streams in order to repurpose or re-process a product for re-use (Hawley, 2009; Merriam-Webster, 2019). Textile recycling is specific to the re-use of textile goods utilizing specific processes to break down the textiles at the fiber level for the re-creation of new yarns and textiles. Mechanical, thermal, or chemical processes are needed for the extraction and reprocessing of fibers, while upcycling and downcycling refer to repurposing items at the textile level versus fiber level. Difficulties related to the quality of recycled textiles deter the industry from implementing textile recycling on a
large scale. Such difficulties include fiber length, uniformity, imperfections, and blended fiber content in textiles (Vadicherla & Saravanan, 2017). However, chemical recycling has proven to maintain the integrity of the fiber resulting in no degradation during recycling processes (Zamani, 2011). Therefore, the chemical recycling method provides a feasible option when considering a circular life cycle.

**Life cycle assessment of recycled textiles.** Life cycle assessment (LCA) of a product is often used as a measure for both quality and sustainability within systems analysis. Waste management can be analyzed within LCA and classified for second life potential as the principal input or raw material for the creation of a new product (Amini et al, 2007; Domina & Koch, 1997). Connections between waste and new life can be described by Commoner’s Law of Ecology, which recognizes that everything is connected and must go somewhere (Domina & Koch, 1997). Whether a product is recycled or placed into a landfill, an impact is made that affects the environment as well economic infrastructure. Therefore, understanding the life cycle of a textile from production, to consumption, and then disposal is necessary for sustainable action to take place.

A conventional linear flow of waste, as shown in figure 1, utilizes new resources for production and is disposed of into a landfill or incinerated after use. However, for a sustainable shift to occur, a circular lifecycle of material flow should be considered. A circular lifecycle, also known as a cradle-to-cradle lifecycle or closed-loop system, is referred to by McDonough & Braungart (2002) as a cyclical system that nourishes continual, prolonged use of biological and technical mass through design and waste consideration. Circularity within a life cycle calls for the repurposing and recycling of materials and products within a closed-loop system.
Closed loop systems, shown in figure 2, re-purpose the current product for a new or different product of identical stature or quality level, such as re-purposing a button-up shirt into a dress. Open loop systems, on the other hand, re-purpose a product for use within a different product, which could be of the same or lesser quality (Vadicherla & Saravan, 2017; Sandin & Peters, 2018) such as re-purposing t-shirts into rags.
As textile production is projected to continue increasing (Palamutcu, 2015), focus on sustainable systems change such as repurposing and recycling will help reduce environmental impact. Chemical recycling, the main topic of this exploratory study, is one example of a systems change for positive environmental impact. This system allows for the reduction of virgin textile fiber production while maintaining quality through a circular, closed-loop life cycle.

**Advanced textile processes.** Advanced textile processes provide researchers with the ability to engineer fibers in order to achieve optimal fiber performance. Often times, the blending of
fibers within production is used for achieving desirable properties and precision. However, fiber blends lead to more complicated impacts on the environment and more challenging recycling processes. McDonough and Braungart (2010, p.99) refer to such blends as ‘monstrous hybrids’ as they severely lengthen the time of break-down in a land fill and also hinder a product’s ability to become ‘food’ in a circular economy. While textile processing has some negative environmental affects, these processes also offer great opportunity for change within the industry.

Nanotechnology is an example of a newer textile process that provides “considerable potential for the development of materials and products with completely new properties” (Petschow et.al., 2009, p.7). The term nanotechnology refers to the manipulation of technological materials that concern functionality at the molecular and atomic scale of one billionth (10\(^{-9}\)) normal size (Black, 2009). For size perspective, Black (2009) notes that a human hair is equivalent to 80,000 nanometers (nm). Manipulation of a material at the nano scale allows for extreme property changes of the material with little to no impact on the material’s general appearance. Current textile advancements in nano-technology include enhancements of the following properties: stain resistance, abrasion resistance, water repellency, oil repellency, self-cleaning attributes, antistatic properties, and antibacterial properties (Black, 2009). Currently within the industry, recycled fibers are often blended with virgin fibers to achieve higher quality end fabrics (Grasso, 1995). During the 2006 Consumer Conference for Nanotechnology, a call was made for the development and production of novel, pure nanofibers to decrease the amount of raw materials used within the industry as well as potentially eliminate the need for blended fibers due to the technological engineering capabilities at the nano-scale (Petschow, 2009). The combination of chemical recycling
processes with nanotechnology to provide an enhanced recycled textile has great potential to impact the number of textiles ending in landfills.

**Purpose & Research Questions**

The purpose of this experimental study was to explore an alternative textile recycling method utilizing chemical recycling processes for fiber separation in combination with electrospinning technology to create an enhanced recycled textile. This purpose addresses the proposed concept of recycling for life cycle circularity of textiles. Electrospinning provides the potential for minimization of fiber blends while maintaining enhanced fiber properties, while chemical processes provide minimal fiber damage through repetitive re-cycling.

The following research questions were developed to address the purpose of this research:

1) What processes and percentages are needed for successful fiber blend separation?
   a) What is the chemical to textile percentage ratio required for successful dissolution of nylon from nylon/polyester fiber blends?
   b) What is the necessary time for successful dissolution of nylon when separating nylon from nylon/polyester fiber blends?
   c) What is the necessary process for successful removal of the polyester fibers from the dissolved nylon solution?

2) What quantities and times are required for successful electrospinning techniques to occur?
   a) What is the quantity of solution required to spin a specific size and thickness in order to maintain similarity to the preliminary textile?
   b) What is the time required to spin a specific size and thickness in order to maintain similarity to the preliminary textile?
Objectives

The following objectives have been identified to answer the outlined research questions:

1. Select virgin nylon/polyester blended fabrics for testing.
3. Use basic statistical central tendencies to determine the mean and standard deviation across replications for fabric weight, bow, and skew to ensure commonality amongst textile comparisons.
4. Chemically process blended fabrics to separate fiber blend contents using formic acid as suggested by the fiber identification standard AATCC 20A-2004.
5. Successfully electrospin recycled nylon samples from chemical dissolution to the standard weight and thickness comparative to that of the virgin fabric.
6. Report findings for the following:
   a) Optimal chemical percentages for fiber blend separation.
   b) Optimal time to dissolve the fiber blends.
   c) Quantity of solution required to create the appropriate weight and thickness of the electrospun material in order to maintain similarity to the initial fabrics.
   d) Time required to spin the appropriate weight and thickness of the electrospun material in order to maintain similarity to the initial fabrics.
Limitations

Limitations of this study include:

1. Testing was completed for nylon/polyester blended fiber-based textiles; other manufactured fibers should be further explored.

2. Size of nanofiber textiles developed in this study are limited to the electrospinning equipment available.

Operational Definitions

AATCC  
American Association of Textile Chemists and Colorists; Organization that provides standardized test methods for the evaluation of performance characteristics of fibers and fabrics (‘About AATCC’, 2019).

ASTM  
American Society for Testing and Materials; Organization that provides a voluntary consensus for standards for testing across a wide range of industries and materials (‘Detailed Overview’, 2019).

Circular Lifecycle  
A closed-loop or cyclical system that nourishes continual, prolonged use of biological and technical mass through design and waste consideration. This term is interchangeable with cradle-to-cradle lifecycles, closed loop systems, or circular systems (McDonough & Braungart, 2002).
<table>
<thead>
<tr>
<th>Term</th>
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<tr>
<td>Disposal</td>
<td>The act of discarding textile-based products involving “trashing, donating, swapping, recycling, repurposing, and/or re-selling” (Eike, Irick, McKinney, Zhang, &amp; Sanders, 2020).</td>
</tr>
<tr>
<td>Down-cycling</td>
<td>The re-purposing of a product to a lower value/quality product (Sandin &amp; Peters, 2018).</td>
</tr>
<tr>
<td>Electrospinning</td>
<td>A process employed for spinning fibers at the nano-scale using electrostatic forces to spin extruded polymer solution into a nonwoven textile.</td>
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<tr>
<td>Fast Fashion</td>
<td>Fashion that is produced in mass quantities with high turnover and low prices and does not follow the typical style seasons (Forum for the Future, 2007).</td>
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<tr>
<td>Fiber</td>
<td>The basic unit of fabrics and textiles which influence product aesthetics, durability, comfort, appearance retention, care, environmental impact, sustainability, and cost. Fibers can be naturally occurring or manufactured (Kadolph, 2010).</td>
</tr>
<tr>
<td>Life Cycle Assessment</td>
<td>The full life cycle and life cycle impacts of a textile from production, through consumer use, to end-of-life (Domina &amp; Koch, 1997).</td>
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<tr>
<td>Nano-fiber</td>
<td>A nano-fiber is a fiber that is one billionth (10^{-9}) scaled size (Black, 2009).</td>
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Nanotechnology is the manipulation of materials at the atomic or molecular level; used within technology (Nanotechnology, n.d.).

Pre-Consumer Waste
Waste that is retrieved during and after production that does not make it to the consumer market such as industrial factory waste (Hawley, 2009).

Post-Consumer Waste
Waste that is retrieved from the consumer once the owner no longer needs and therefore discards (Hawley, 2009).

Recycling
The removal of waste from traditional waste streams in order to repurpose or re-process the product for re-use (Hawley, 2009; Recycle, n.d.).

Sustainability
The consideration of environmental protection, economic growth, and social equity to ensure that present needs are being met without compromising future generations by permanently depleting or damaging resources (The Brundtland Report, 1987; Sustainable, n.d.).

Textile
“A flexible material that is composed of thin films of polymer or of fibers, yarns, or fabrics” (Kadolph, p.6, 2010).

Textile Recycling
The reprocessing of a textile product using chemical, mechanical, or thermal processes to return the textile back to its original fiber form (Lewis et. al., 2017).
<table>
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<th>Category</th>
<th>Definition</th>
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<tr>
<td>Textile Re-Purposing</td>
<td>The re-creation of an item for an alternate use through down-cycling, up-cycling, or re-design (Eike, Irick, McKinney, Zhang, &amp; Sanders, 2020).</td>
</tr>
<tr>
<td>Up-cycling</td>
<td>The re-purposing of a product to a higher value/quality product (Sandin &amp; Peters, 2018).</td>
</tr>
<tr>
<td>Textile Waste</td>
<td>The pre-consumer and post-consumer waste which reflects textile products that are excessive or unwanted and often donated, incinerated, or sent to a landfill (Domina &amp; Koch, 1997; Hawley, 2009).</td>
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CHAPTER II

REVIEW OF LITERATURE

In the following literature review, the lifecycle of discarded textiles is examined with consideration of textile waste as potential for a circular lifecycle using textile recycling systems. Textile recycling methods are explored in depth with emphasis placed on quality management during recycling. Limitations and difficulties of textile recycling are presented to provide insight for potential innovation and expansion of current methods. A market analysis is also presented to justify material selection for recycling testing. Finally, electrospinning technology is explored for potential impact on the quality of textile products.

Lifecycle of Discarded Textiles

Textile Waste

It has been estimated that the environmental impact per garment must be drastically reduced (30-100%) by the year 2050 for the industry to be considered sustainable (Sandin, Peters, & Svanström, 2015). Sustainability can be defined as the consideration of environmental protection, economic growth, and social equity to ensure that present needs are being met without compromising future generations by permanently depleting or damaging resources (The Brundtland Report, 1987). Throughout the last few decades, shifts in consumer demand and industry practice have contributed to the common ‘throw-away’ mentality associated with textiles and clothing, therefore increasing overall textile waste within the industry (Stephens, 1985; Claudio, 2007; CBC News, 2018). Textile waste is classified by Domina and Koch (1997) into two waste categories: pre-consumer and post-consumer waste. Pre-consumer waste consists of post-production waste by manufacturers and retailers often resulting from defects, excess, or unwanted goods. Post-consumer waste, however, is discarded by the consumer and consists
largely of garments or household articles that the owner no longer needs or wants (Hawley, 2009). These waste products are typically donated or thrown out and then sent to a landfill. While popular belief relates recycling with economic and environmental benefits, it has been noted that textile waste often does not reach the ‘recycling pipeline’ (Hawley, 2009). It has been found that approximately 75% of pre-consumer waste and 48% of post-consumer waste is re-routed from the traditional waste stream for re-use (Council for Recycling, 2003 as cited in Chen & Burns, 2006). However, only approximately 1% of clothing is actually recycled at the fiber level for the creation of a new product (CBC News, 2018). Additionally, the United States Environmental Protection Agency (2017) reported 11.2 million tons of textiles contributing to municipal landfill waste.

Waste is often referred to as anything that is left over (in excess), or unwanted (Domina & Koch, 1997). However, waste is a far more complex topic deserving consideration relating to circular systems. McDonough and Braungart (2002) equate waste to ‘food’ within a circular economy, categorizing waste as biological or technical nutrients for future life-cycles. Unwanted waste from one sector of a product’s lifecycle could be a useful raw material for another sector. Biofuels and re-usable energy are key examples of this notion. Because textiles are nearly 100% recyclable, they are able to function in a circular system and be redeveloped into new textiles and products (Hawley, 2009). Textile recycling production also has very limited contributions to new hazardous waste or harmful emissions (Hawley, 2009). Therefore, more textile recycling should be taking place within the industry to divert textile waste away from landfills and back into the production cycle.
Extending Life of Discarded Textiles

Some studies state that re-purposing and recycling within the textile industry is already well established (Sandin & Peters, 2018). However, this tends to apply to downcycled products (products of lesser value or quality from the original). Sandin & Peters (2018) note some key statistics provided by the Textile Recycling Association (2005) backing their claims of a well-established industry. These statistics state that only 15-20% of disposed textiles in Europe are distributed to landfills or incinerated; whereas, approximately 50% is downcycled and 50% is reused via exports to developing countries (Sandin & Peters, 2018). These statistics, however, are not implicative of practices within other countries. Additionally, studies by Roos et.al. (2017) and Woolridge et.al. (2006) note that many textiles are also disposed of before the end of their technical service life. Therefore, opportunities exist for the improvement and expansion of reuse and recycling systems (Sandin & Peters, 2018).

Textile repurposing and recycling. Textile repurposing utilizes different methods in order to prolong the service life of a textile (Fortuna & Diyamandoglu, 2017). This can be done through downcycling or upcycling via modifications, repairs, or giving the product to a new owner (Fortuna & Diyamandoglu, 2017; Sandin & Peters, 2018). Downcycling is the repurposing of a product to lower value/quality product, while upcycling is the repurposing of a product to a higher value/quality product (Sandin & Peters, 2018). End-of-life products can also be upcycled and incorporated into value-added products such as stuffing, insulation, carpet underlays, or automotive components. Recycling differs from repurposing as product collection and re-use occurs at the fiber level and is then re-spun into new yarns or fabrications (Hawley, 2009). Textile recycling refers to the reprocessing of a textile product by returning the textile back to its original fiber form for re-use in a future product (Lewis et. al., 2017). In order to create a
sustainable shift in material flow, circular, or closed-loop, systems should be considered in the life cycle assessment of textiles (Vadicherla & Saravanan, 2017). Life cycle assessments evaluate the environmental performance or impact of products from cradle (production) to grave (end of life). This includes the extraction of new materials for production, the actual production phase, the use phase, the waste treatment of the product, and the final disposal of the product (Zamani, 2011).

**Circular systems.** A circular system “calls for a coordinated redesign of production and consumption patterns, ensuring that cascading material and product resource use continues for as long as possible” (Boiten, Li-Chou, & Tyler, 2017, p.1). Circular systems, as shown in figure 2, shift away from the traditional linear lifecycle resulting in the disposal of products, and instead re-imagine a product’s ‘end-of-life’ for future regeneration (Ellen MacArthur Foundation, 2013). The main purpose of circularity within a system is to keep materials in productive use for as long as possible (Boiten, Li-Chou, & Tyler, 2017). The following principles are considered in a circular system: zero-waste design, product-life extension, resource recovery, repair, and remanufacture services. For a successful circular economy to occur, innovative policy changes need to be put in place and integrated at all levels of the supply chain (Boiten, Li-Chou, & Tyler, 2017). The collection of discarded textiles is also integral for the success of a circular lifecycle. General knowledge of the collection infrastructure as well as easy accessibility for the public would be necessary for textile collection to occur. Because drivers for textile circularity are connected to a triple bottom line concerning the environment, society, and economics, incentives would most likely be required at both the manufacturer and consumer level to promote use of the system and further encourage recycling efforts (Boiten, Li-Chou, & Tyler, 2017). Until recently, circular systems were relatively unheard of within textile waste collection. In order for a circular
system shift to occur within textiles, increased demand of sustainably recycled products, as well
the incorporation of a strong infrastructure for the recycling of textiles, would need to take place.

**Textile Recycling**

**Systems Analysis**

Textile recycling is one of the oldest and most established recycling industries dedicated to removing textile waste from the solid waste stream so it can be re-processed back into the market as a new product (Hawley, 2009). Over 500 businesses participate within the textile recycling industry and redirect approximately 1.25 million tons of post-consumer waste annually (Council for Textile Recycling, 1997 as cited in Hawley, 2009). Many of the textile recycling factories in the industry are small, family-owned businesses passed down among generations. However, younger generations are beginning to seek out alternative career options, leaving the future of textile recycling in jeopardy (Hawley, 2009). Within the recycling industry, there are numerous positions and steps to follow for efficient textile recycling. Hawley breaks down the textile recycling industry systems in her 2009 study,” Understanding and improving textile recycling: a systems perspective”. ‘Rag graders’ in textile sorting companies begin the recycling process by acquiring textile waste, sorting the waste into specific categories and quality levels, processing and exporting the sorted goods, and marketing the textiles for the new marketplace. Most of the acquired textiles consists of overflow of donated clothing from charitable organizations such as Goodwill or the Salvation Army. These organizations sort the clothing first and then sell the excess product to the rag graders at an average of 3-6 cents per pound. Once purchased, the textiles are then transported to the textile recycling factories. At the factory, the rag graders carefully sort the textiles into the following categories: textiles for export; textiles for conversion via recycling or redesign; wiping and polishing cloths; textiles for landfill or incineration; and
“diamonds”, which are name brand products sold at premium prices in foreign markets. Limited knowledge exists on the true end destination of the used textile goods once they reach the foreign markets (Bartlett, McGill, & Willis, 2013). It is known that secondhand Western clothing within a foreign market, such as Africa, provides cheaper clothing alternatives to poorer community members; however, consequential economic impacts are also important to consider. Decline in both indigenous dress as well as local clothing production result from secondhand clothing exports (Hawley, 2006). The textile recycling industry is well-developed and expansive providing opportunities within both repurposing, such as upcycling and downcycling, as well as fiber-to-fiber recycling. For the purpose of this study, fiber-to-fiber textile recycling was the primary focus, exploring both current limitations as well as processes and methods.

**Limitations**

**Systemic limitations.** Limitations hinder the production of textile recycling at nearly all systemic levels. One of the largest limiting factors is the lack of consumer and industrial adoption. Economic factors drive societal changes, meaning that unless there is a well-known, prescribed economic advantage given for using textile recycling or consuming recycled goods, there will be less overall adoption of the practice. Grasso’s 1995 study surveying textile manufacturers’ relationships with recycling provides valuable insight into industry limitations of using recycling practices as well as and using recycled goods. Of the seven participating manufacturers, only 42% recycled their textile waste. The main reasons provided for not utilizing textile recycling related to the following: the inability to use the waste within existing production protocol, the lack of equipment within the facility, the lack of economic feasibility, as well as pre-existing relationships with third parties already specialized in textile waste and recycling. Upon final review of the respondent answers, 82% stated that they would consider using
recycled textile materials, however the cost would need to be lower than raw virgin materials (Grasso, 1995). This lack of technological innovation for industry producers in combination with lacking benefit perception and existence of cheap product in the market limits the overall interest of textile recycling production at a large scale (Zamani, 2011). Another study by St. Vincent de Paul Society of Eugene, Oregon and Oregon State University in 1999 attempted to research the feasibility of textile recycling processes for used clothing. However, the study was suspended due to lack of financial support and industry reluctance (Chen & Burns, 2006).

**Recycling limitations.** Recycling introduces the opportunity for a circular system as it closes the cycle between industrial and environmental elements (Amini et.al., 2007). However, the recycling of textiles presents many challenges concerning quality control during recycling processes. One study stated that, “the inherent nature of the recycling process produces fibers with short lengths, non-uniformity, unopened/partially opened fibers, and more imperfections (Vadicherla & Saravanan, 2017). This would limit the quality of recycled fiber production and produce coarser, less desirable yarn counts. Blended fiber content is another common issue when considering fiber-to-fiber recycling (Vadicherla & Saravanan, 2017; Hawley, 2009). The blending of fibers is practiced throughout the textiles industry to achieve particular desirable properties (Vadicherla & Saravanan, 2017). However, blended fibers result in increased fiber strength making them difficult to separate as well as causing greater difficulty purifying the separation and sorting process of textile recycling (Hawley, 2009). Molecular integrity of the fibers can also be impaired due to general wear and laundering by consumers (Palme, Idström, Nordstierna, & Brelid, 2014). Because recycled fibers are often associated with lesser quality, continued production of virgin fibers often results (Zamani, 2011). Blending virgin fibers with
Recycled fibers is also practiced within the industry for maintaining or increasing the quality of a recycled product (Grasso, 1995; Zamani, 2011; Sandin & Peters, 2018).

**Recycling Processes**

The three main processes for the recycling of textiles are: mechanical, thermal, and chemical. Mechanical recycling uses processes such as cutting, shredding, contaminant separation, floating, washing, drying, extrusion, and pelletizing to open the structure of the textile and collect fibers for re-spinning into yarns (Vadicherla & Saravanan, 2017; Zamani, 2011). Thermal recycling utilizes heat processes or combustion to convert the existing textile into fibers via melt extrusion (Sandin & Peters, 2018). The recycling methods for mechanical and thermal often degrade the quality of the fiber while also using intensive levels of energy for recovery (McIntyre, 2005). Chemical recycling uses depolymerization processes to break down the molecular structure of the fiber and then return it back to its original raw material form through spinning processes, maintaining the fibers integrity and quality (Sanchez et al, 2015; Zamani, 2011). Depolymerization breaks down the fiber’s polymers at a molecular level before returning it to its material fiber form via repolymerization and spinning methods (Sanchez et al, 2015; Sandin & Peters, 2018; Zamani, 2011). The reprocessing of the fiber at a molecular level has been regarded for producing recycled fibers resembling the quality of virgin fibers (Sandin & Peters, 2018). Chemical recycling at the polymer level not only broadens the capability of converting textile waste for recreation into new textiles, but also reduces plastic polymers into various levels through chemical disintegration (Sandin & Peters, 2018; Vadicherla & Saravanan, 2017). One major limitation for chemical recycling includes the lack of technology for proper sorting and separation of fibers (Sandin & Peters, 2018). Chemical recycling processes are only applicable to synthetic fibers such as polyester, nylon, and polypropylene (Zamani, 2011).
**Recycling for fiber content.** Synthetic fibers are manufactured and used daily as they highly influence and “shape the world as we know it” (Kadolph, 2010, p.152). The production of virgin synthetic fibers contributes to environmental damage by utilizing nonrenewable petroleum resources during production (Chen & Burns, 2006; Lewin & Pearce, 1998). According to Lenzing, 63% of all textile fibers derive from petrochemicals. Greenhouse gas emissions such as carbon dioxide are also created during synthetic fiber production (Lenzing, 2017 as cited in Sandin & Peters, 2018). Production of nylon, a synthetic fiber, utilizes hazardous chemicals during production and emits nitrous oxide which is a potent greenhouse gas (Fletcher, 2014). In addition to production-based environmental damage, synthetic fibers are also nonbiodegradable resulting in the accumulation of textiles in landfills without the ability to break down for many years. It has been estimated that it would take approximately 30-40 years for nylon fabric to decompose, versus a time span of 1-5 months for cotton (Forum for the Future, 2007).

Among the most popular of synthetic fibers are nylon and polyester, which are widely used within the United States and both currently being researched for their recycling capabilities. Both nylon and polyester are often used within active and outdoor clothing markets due to their excellent durability, high appearance retention, high strength, light weight, and easy-care properties (Kadolph, 2010). Therefore, a market analysis of 3 active, outdoor apparel companies was performed to observe the frequency of nylon and polyester fiber content usage within products. A total of 332 women’s jacket and vest styles were assessed, with 94 styles from Patagonia, 64 styles from The North Face, and 174 styles from Columbia. Both nylon and polyester appeared frequently among the apparel products, with a total of 111 styles containing nylon fiber content and 244 styles containing polyester content. Of those styles, 28 contained a blend of both nylon and polyester. It is also important to note that only 5 of the 332 styles did not
contain nylon or polyester, accounting for less than 0.5%. The market analysis helped provide thoughtful reasoning when choosing the final fiber blend contents for this study. The results from the market analysis are displayed within Table 1.

Table 1

*Fiber Content Market Analysis*

<table>
<thead>
<tr>
<th>Fiber Content</th>
<th>Patagonia</th>
<th>The North Face</th>
<th>Columbia</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Nylon</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100% Virgin</td>
<td>4</td>
<td>12</td>
<td>34</td>
<td>50</td>
</tr>
<tr>
<td>100% Recycled</td>
<td>15</td>
<td>3</td>
<td>--</td>
<td>18</td>
</tr>
<tr>
<td>&lt;50% Recycled</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>&gt;50% Recycled</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>1</td>
</tr>
<tr>
<td>100% Polyester</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100% Virgin</td>
<td>1</td>
<td>19</td>
<td>92</td>
<td>112</td>
</tr>
<tr>
<td>100% Recycled</td>
<td>32</td>
<td>10</td>
<td>--</td>
<td>42</td>
</tr>
<tr>
<td>&lt;50% Recycled</td>
<td>7</td>
<td>2</td>
<td>--</td>
<td>9</td>
</tr>
<tr>
<td>&gt;50% Recycled</td>
<td>13</td>
<td>--</td>
<td>--</td>
<td>13</td>
</tr>
<tr>
<td>Nylon Blends</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nylon/Spandex</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>1</td>
</tr>
<tr>
<td>Nylon/Elastane</td>
<td>--</td>
<td>3</td>
<td>7</td>
<td>10</td>
</tr>
<tr>
<td>Nylon/Cotton</td>
<td>--</td>
<td>1</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Nylon/Cotton/Wool</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>1</td>
</tr>
<tr>
<td>Nylon/Polyester Blends</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nylon/Polyester</td>
<td>1</td>
<td>3</td>
<td>14</td>
<td>18</td>
</tr>
<tr>
<td>Nylon/Polyester/Elastane</td>
<td>--</td>
<td>1</td>
<td>7</td>
<td>8</td>
</tr>
<tr>
<td>Nylon/Polyester/Wool</td>
<td>2</td>
<td>--</td>
<td>--</td>
<td>2</td>
</tr>
<tr>
<td>Polyester Blends</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Polyester/Spandex</td>
<td>10</td>
<td>--</td>
<td>--</td>
<td>10</td>
</tr>
<tr>
<td>Polyester/Elastane</td>
<td>2</td>
<td>10</td>
<td>14</td>
<td>26</td>
</tr>
<tr>
<td>Polyester/Cotton</td>
<td>--</td>
<td>--</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Polyester/Cotton/Hemp</td>
<td>2</td>
<td>--</td>
<td>--</td>
<td>2</td>
</tr>
<tr>
<td>Cotton Blends</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100% Cotton</td>
<td>1</td>
<td>--</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Cotton/Spandex</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>1</td>
</tr>
<tr>
<td>Cotton/Elastane</td>
<td>--</td>
<td>--</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Cotton/Polyurethane</td>
<td>--</td>
<td>--</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

Limited petroleum reserves place restraint on new production of virgin fibers. This restraint, combined with overfilling landfills and environmental decline, is forcing researchers and
industry leaders to consider alternative resources such as recycling for textile production (Chen & Burns, 2006). Further market analysis revealed that companies are working on ways to separate the blended fibers for recycling and re-use, with great strides being made within the recycling of polyester fibers (Watson & Warnock, 2003). While many companies have begun using processes for recycling polyester, industry efforts regarding the recycling of nylon have lagged far behind (Chen & Burns, 2006). However, because nylon is a relatively inexpensive polymer, it is beginning to gain recognition for its recycling capabilities by some major companies such as Patagonia and Shaw’s Evergreen Nylon Recycling Facility (Watson & Warnock, 2003; Rodie, 2010). Chemical depolymerization has proven to be the most effective for the recycling of nylon by converting the depolymerized caprolactam into re-usable nylon fibers. One way to transform the depolymerized chemical solution into a textile is through electrospinning technology.

**Nanotechnology**

The study of nanotechnology within materials science is considered relatively new, having been developed only within the last thirty to forty years (Black, 2009). More specifically, the ability to manipulate matter at the nano-scale is currently being greatly considered within the focus of textiles and apparel (Black, 2009). Nanomaterials can be categorized within a 0D to 3D scope, as they can be represented in their smallest forms as nanoparticles or be manipulated into visual elements as nanotubes, nanowires, multilayered structures and thin films, or nanostructured materials (Black, 2009). The manipulation of properties and the nano-scale can inherently alter the properties of the materials.
**Textile Enhancement from Electrospinning**

Nanotechnology within textiles is currently focused in fabric coatings and finishes to enhance fibers in specialized product areas (Black, 2009). One key property change that is highly relevant within textiles is the alteration potential relating to the surface area of a textile. Alterations to the surface of the fabric can restructure and enhance surface properties altering the overall feel or performance of the textile, while keeping all manipulations invisible to the ‘naked eye’ (Black, 2009). Other properties that can be adjusted through nanotechnology include strength to weight ratio, stain and abrasion resistance, water and oil repellency, self-cleaning attributes, antistatic properties, antibacterial properties, insulative properties, as well as conductive properties (Black, 2009). Many of these properties have positive impacts on water and energy usage within a textile’s life-cycle by reducing the need for laundering and ironing. Business for Social Responsibility (BSR) notes that the use/care phase of a garment’s life cycle is the largest contributor to AT industry waste accounting for 40-80% of total life cycle green-house gas (GHG) emissions (BSR, 2009).

The improved functionality of textiles created by nanotechnology has already reached textile markets within industrial safety, sports clothing, antibacterial clothing, and ultraviolet protection. These developments provide products with increased durability and perceived higher user-value for the consumer (Petschow, Scholl, Renn, & Ulmer, 2009). It is suggested that new nano- products will eventually change the way consumers interact with, care for, and purchase textiles by shifting consumers away from a ‘throw-away’ mentality (Black, 2009). The combination of chemical recycling processes with nanotechnology to provide an enhanced recycled textile has great potential to impact and reduce the number of textiles ending in landfills.
Nano-Textile Production

Nanoparticles are integrated into a textile through the weaving of yarns or applied as a coating (finish) onto the textile surface. The application of finishes at the nano-level creates a more durable product while reducing pollution that is characteristic of traditional finishing methods (Black, 2009). Electrospinning, a process originally patented by Formhals (1934), can also be employed for spinning fibers at the nano-scale. Within this process, electrostatic forces are used to spin an extruded polymer solution into a nonwoven textile. The polymer solution gets pushed through a syringe with a capillary jet outlet entering into a strong electrostatic field, where it is spun through the field and collected onto a metal screen. This screen counters the electric charge from the solution as the two sides have opposite polarities, therefore splitting the solution into small charged fibers and evaporating the solvent upon contact with the metal screen (Chodák & Blackburn, 2009; Zheng-Ming, Zhang, Kotaki, & Ramakrishna, 2003). See figure 3 for illustration of nanofiber production via electrospinning process.

Figure 3. Electrospinning Setup by Haghi, 2012, retrieved from Apple Academic Press, Inc.
As the nanofibers collect and build on the plate during spinning, the thickness of the nonwoven/fiber-web textile is created. Many factors should be considered during electrospinning for proper fiber transformation to occur for the production of a quality nano-textile. Viscosity, elasticity, conductivity, surface tension, temperature, and humidity all provide potential risk for impact on the quality of the fiber produced (Doshi & Reneker, 1995). The collection plate should also be considered and predetermined in order to achieve optimal size, thickness, and shape (Black, 2009). Capillary thickness and diameter must be considered as well during the initial setup of the spinning equipment. Capillary diameter determines the voltage requirements for the machine, while capillary thickness determines the diameter of the nanofibers produced (Chodák & Blackburn, 2009). Finally, the distance between the tip and the collection screen also impact voltage levels, temperature, and the quality of overall fiber collection for end textile production (Ramakrishna, Fujihara, Teo, Lim, & Ma, 2005).

The integration process of nanoparticles in textiles is important to note considering sustainability impact, as nano-textiles are produced within contained systems, therefore potentially limiting environmental pollution (Petschow, 2009). The development and production of novel, pure nanofibers could also provide immense benefits regarding the decrease in raw material use for textile production as well as potentially eliminate the need for blended fibers. Fibers are commonly blended to achieve desired properties, however technological engineering capabilities at the nano-scale could provide the opportunity for enhanced single-fiber content fabrications.

**Nano-Production of Nylon**

The production of nylon nanofibers via electrospinning processes requires a similar set up as other fibers regarding the necessary electrospinning components. From the literature reviewed
for this study, components, such as voltage, distance between the needle and collection plate, and flow rate, were all examined. However, the specific protocol pertaining to requirements for optimal solution consistency as well as optimal solution amount & time for a desired mass density have been unexplored and documented. Also, overall protocol when working with solutions created from fiber blends versus 100% fiber content have not been documented.

While recycling processes are advancing, companies are still utilizing virgin fibers blended with recycled fibers to maintain textile quality. This continued practice of fiber blending suggests the need for further improvement to chemical recycling processes (Rodie, 2010) and the need to explore alternative production methods, such as electrospinning, that may maintain textile quality and performance expectations. The need for recycling improvement, the lack of literature and industry knowledge concerning nylon fiber recycling, and the negative environmental impacts from nylon were all instrumental when determining fiber selection and test methods for this study. This study aims to report all of the required components necessary for optimal spinning to occur for the production of a novel, nonwoven nylon from a chemically recycled nylon/polyester blend. Because blended fiber content is highly prevalent within the apparel and textiles market, it is important to consider the processes for fiber separation and recycling as differences in fiber content and percentages may highly impact the overall process.
CHAPTER III

METHODOLOGY

The primary purpose of this study was to explore an alternative textile recycling method utilizing chemical recycling processes with electrospinning technology. A two-step method was employed to determine the processes and percentages required for successful fiber blend separation and for successful electrospinning to occur. The following questions were addressed within this exploratory research:

1) What processes and percentages are needed for successful fiber blend separation?
   a) What is the chemical to textile percentage ratio required for successful dissolution of nylon from nylon/polyester fiber blends?
   b) What is the necessary time for successful dissolution of nylon when separating nylon from nylon/polyester fiber blends?
   c) What is the necessary process for successful removal of the polyester fibers from the dissolved nylon solution?

2) What quantities and times are required for successful electrospinning techniques to occur?
   a) What is the quantity of solution required to spin a specific size and thickness in order to maintain similarity to the preliminary textile?
   b) What is the time required to spin a specific size and thickness in order to maintain similarity to the preliminary textile?
Experimental Materials

Textiles

For this study, nylon fiber content was chosen due to environmental impact, popular market use, and gaps within research from industry and academia. Fiber content took precedence when sourcing materials for this study. However, careful considerations of the weave structure and weight were also employed throughout the sourcing of the materials. This was done as an attempt to ensure commonality and consistency of the materials for appropriate comparative analysis among the fabrications. Nylon and polyester fabrics and blends were sourced from local retailers as well as online retailers within the United States in order to ensure efficient turn-around time for testing.

Three fabrics were purchased for assessment for this study. A 50% nylon/50% polyester blend was purchased per the main purpose of this study. One fabrication of 100% nylon as well as one fabrication of 100% polyester were also purchased to provide a baseline for comparative analysis of the findings. The 100% nylon organza and the 50% nylon/50% polyester organza were both sourced from Mood Fabrics in New York City, New York. While fabric weight or thickness were not provided by the manufacturer, both fabrications were similar in appearance with a woven structure. Finally, the 100% polyester lightweight lining was purchased from a local Joann fabric store. Again, the weight was not provided, however it provided a similar hand, drape, and woven structure as the other fabrications purchased. Fabric characterization testing was employed post purchase to ensure commonality.

Solvents and Equipment

All chemical solvents were purchased through Sigma Aldrich, a science-based company that provides a multitude of lab materials such as chemicals for textile testing.
**Dissolution of nylon.** According to Table 1 in the AATCC TM20A-2004 standard method for quantitative fiber analysis, nylon is soluble in 20% hydrochloric acid, 59.5% sulfuric acid, or 90% formic acid for separation from polyester fiber content (AATCC, 2004, p.59-61). In this study, the AATCC standard for formic acid was modified and an 88% concentration was used for the dissolution of nylon. Formic acid was chosen over hydrochloric acid due to reports by Toray Industries, Inc. (AMILAN™ Nylon Resin) stating that the dissolution of nylon in concentrated hydrochloric acid could cause partial hydrolysis to occur. Hydrolysis is a decomposition reaction in which water is either reacted with or produced via a chemical reaction. This reaction results in the breakdown of chemical bonds, which could damage the molecular integrity of the nylon fiber structure (Helmenstine, 2019).

**Dissolution of polyester.** In accordance to the AATCC TM20-2004 qualitative fiber analysis standard, m-Cresol was first considered for the dissolution of polyester fiber content and adjusted slightly to a 99% concentration versus 100% concentration. Failed electrospinning attempts using the m-Cresol/polyester solution required the use of an alternative chemical compound for dissolution, therefore suggesting an adjustment in protocol from the AATCC standard test method. This adjustment involved the use of trifluoroacetic acid, TFA, (99%) in a 50/50 combination with dichloromethane, DCM, (>99%). The solvents and percentages utilized in this study were outlined by Kayaci, Aytec, and Uyar (2013).

Other required materials for use with the solvents included glass beakers, glass mason jars with lids, and two fritted glass crucibles with coarse porosity. Proper safety protocol was followed throughout the entirety of the research project. PPE (personal protective equipment) such as nitrile disposable gloves, a lab coat, and safety glasses were all used for protection of the
skin and eyes. All testing and dissolution processes took place within the vent-hood of the laboratory for safety precautions.

**Electrospinning Equipment**

A Harvard Apparatus standard infuse/withdraw programmable pump was used for the electro-spinning portion of this research. Electrospinning processes require the use of an electrically charged field for successful spinning and collection of the nano-fibers. For this study, a Gamma High Voltage box was used to produce the necessary voltage for the creation of the electrical field. Other electrospinning equipment included a metal collection plate, aluminum foil for collection plate coverage, a plastic syringe to hold the solution, and a capillary jet outlet for extruding the solution through the electrically charged field for final collection.

**Experimental Methods**

**Preparation of Textiles**

All preparation and testing of textile samples was completed in the Textile Testing Lab at the University. Prior to testing, all purchased textiles were pre-washed according to the AATCC TM135 – 2004 test method using a commercial home laundry machine to remove any potential contaminants that could occur throughout the production and shipment of the textile good. In order to ensure accuracy across temperature and water usage, a copy of the original laundry machine operating manual was obtained and followed. AATCC standard detergent was used to ensure no added fragrances, bleach, phosphates, or enzymes were used in the cleaning process. Once the fabric was weighed, careful conversion calculations were then made to ensure accuracy among the detergent and water proportions in accordance to the AATCC standard. The textiles were then hand-rinsed with distilled water to remove any mineral build that could occur through
home laundering. Finally, the textiles were carefully hung and allowed to air dry before being placed into the conditioning chamber.

In accordance to ASTM D1776/D1776M – 16, standard practice for conditioning and testing of textiles was used for all test samples. Pre-conditioning brings the textiles to a relatively low moisture content within a specified atmosphere and is important for obtaining both accurate and reproducible results. Because physical properties of textiles are influenced by relative humidity and temperature, results obtained under uncontrolled atmospheres may not allow for accurate comparative analysis. The specified atmosphere for conditioning varies among textiles and is specific to fiber content. ASTM standards note polyamide, or nylon, textiles at a standard temperature of 20 +/- 2°C [68 +/- 4°F] with a relative humidity of 65 +/- 5%. Conditioning of polyamide textiles must occur for a minimum of 16 hours for accurate results. ASTM included built-in tolerances within the standard as uncertainty of accuracy must be considered when testing. The Economy Line Humidity Chamber in the Textile Testing Lab was used for this study to bring the textile samples to standard atmosphere, therefore meeting standard requirements.

**Fabric Characterization**

Fabric characterization was first conducted to assess fabric properties and determine feasibility for comparative analysis. AATCC TM20A-2004 reflects quantitative fiber analysis test standards and procedures for natural and man-made fiber blends. Quantitative testing includes chemical analysis procedures specific to varying fiber blends for the purpose of specifying exact quantities of each fiber within the textile. Because the fiber content was pre-determined with purchase, quantitative testing was done for fiber content and fiber percentage assurance. This portion of the fabric characterization is a crucial element within this study to ensure accurate fiber content as well as eliminate the potential for skewed or biased findings.
Preliminary knowledge of the intended fiber content allowed for method minimization and direction when determining the methods used.

Further characterization testing included calculations of the mass per unit area (weight) of the fabrics. Mass was calculated in accordance to ASTM D 3776/D3776M – 09a, using small swatch testing requiring a total cut area of 20 in.², 400 inches. Some samples were slightly modified in total area to accommodate the fabric size purchased. Once the samples were cut, the exact length, width, and weight were measured for each sample, and then calculated as mass per linear yard (oz/yd² = 45.73 X [Mass in grams / Area in inches²]). The mean and standard deviation were then recorded for the mass for further comparative analysis of fabric characterization.

The final step within fabric characterization included quality assessment by visual inspection and calculation of bow and skew (ASTM D 3882 – 08). Both fabrications were laid out fully under direct light and analyzed carefully by visual inspection for potential defects or limitations of the fabric quality. Three samples were then cut for assessment of bow and skew. The bow was measured by placing a straight-edge, squared ruler across the width of the sample and marking all points where the filling yarn was off balance and higher or lower than the filling yarn along the straight edge. The greatest distance between the straight edge and the variation from the straight edge along the width was recorded for the bow. The skew required removal of some of the yarns through both the warp and the filling to interpret the interlaced woven structure. A straight-edge squared ruler was again used to determine if the fabric measured at a straight 90° angle perpendicularly across the width of the specimen. If any angle was noticed, the distance from the 90° angle at the end of the width was recorded as the skew.
**Chemical Processing**

Upon completion of fabric characterization testing, chemical processing testing occurred in order to acquire findings relative to the first proposed research question regarding the processes and percentages necessary for successful fiber dissolution and fiber blend separation. This research question was divided into sub-questions, which guided the method development for this study.

a) What is the chemical to textile percentage ratio required for successful dissolution of nylon from nylon/polyester fiber blends?

b) What is the necessary time for successful dissolution of nylon when separating nylon from nylon/polyester fiber blends?

c) What is the necessary process for successful removal of the polyester fibers from the dissolved nylon solution?

To answer the first question and determine the optimal ratio for fiber blend dissolution, a series of ratios were tested starting at a 50:50 ratio of chemical solvent to fabric. From this 50:50 ratio, increments of 10 to a final ratio of 90:10 chemical solvent to fabric weight were tested. A panel of small glass mason jars were set up for a 100% nylon fabrication, a 50% nylon/50% polyester blend fabrication, and a 100% polyester fabrication. Each fabrication was carefully weighed out to 5 grams of fabric weight and placed into the jars. Calculations were then made for a 50:50, 60:40, 70:30, 80:20, and 90:10 ratio to occur for each fabrication. The solvent was also measured by grams (g) of weight for consistency of measures. All ratios and weights used for this study are listed within table 2.
Table 2

*Ratio Weights of Chemical Solvent: Fabric (g)*

<table>
<thead>
<tr>
<th></th>
<th>50:50</th>
<th>60:40</th>
<th>70:30</th>
<th>80:20</th>
<th>90:10</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nylon</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Formic Acid (88%)</td>
<td>5</td>
<td>7.5</td>
<td>11.67</td>
<td>20</td>
<td>45</td>
</tr>
<tr>
<td>Fabric</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Polyester</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trial 1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>m-Cresol (99%)</td>
<td>5</td>
<td>7.5</td>
<td>11.67</td>
<td>20</td>
<td>45</td>
</tr>
<tr>
<td>Fabric</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Trial 2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trifluoroacetic Acid (99%)</td>
<td>2.5</td>
<td>3.75</td>
<td>5.84</td>
<td>10</td>
<td>22.5</td>
</tr>
<tr>
<td>Dichloromethane (≥99.9%)</td>
<td>2.5</td>
<td>3.75</td>
<td>5.84</td>
<td>10</td>
<td>22.5</td>
</tr>
<tr>
<td>Fabric</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
</tbody>
</table>

*Note:* All measures are recorded for weight in grams (g).

Once the chemical solvents were measured by weight, they were then added to the fabrications within the mason jars one at a time to record the amount of dissolution that occurred and the time for full dissolution to occur. In order to assess commonality and variability with ease, the 100% nylon and 100% polyester fabrication were dissolved first. This provided a base for what to look for in terms of solution consistency and amount of solution that should be expected from the 50% nylon, 50% polyester blend.

Finally, the last step for chemical processing was to document the steps necessary for fiber blend dissolution and separation for the 50% nylon/50% polyester blend. This was achieved by modifying the quantitative fiber analysis standard (AATCC TM20A-2004). The 80:20 and 90:10 ratios were tested using coarse porosity fritted glass crucibles. The chemical solvent was poured over the fabric within the crucible and the contents were stirred frequently over a 12-hour period to allow for fiber blend separation and document the findings. The remaining undissolved fibers from the blend were then neutralized and rinsed to be dissolved in the secondary solvent base.

Once the optimal ratio was recorded for each fabrication, the unsuccessful ratios were modified.
either in chemical solvent weight or fabric weight and findings were documented. This was done in order to ensure that the ratio performed consistently across samples.

**Nano-Textile Production.**

The final testing for this study included documenting the procedures for recycled nano-textile production via electrospinning techniques. To answer the second proposed research question pertaining to successful electrospinning techniques, the main components needed to spin a specific weight and size similar to the preliminary fabrication were assessed. The voltage, distance, and flow rate were all assessed and recorded. However, the two main components assessed related to the quantity of solution and time necessary for spinning the specific weights of the electrospun fabrications.

The processes explained by Kayaci, Aytac, and Uyar (2013) were followed for the initial set-up of the electrospinning equipment. However, each fabrication required adjustments in voltage, distance, and flow rate in order to create a uniform nonwoven structure or fabrication. The first step within the set-up phase was to cut an aluminum piece that would cover the metal collection plate for protection. Prior to covering the metal plate, the weight of the aluminum foil piece was recorded in order to obtain mass density of the electrospun sample post spinning without having to remove it from the foil. After covering the metal plate, the plate was positioned inside the electrospinning chamber. Once the collection plate was positioned, the solution was then placed into a 10 ml plastic syringe with a capillary jet outlet for extrusion. Finally, the syringe and capillary jet outlet were positioned horizontally into the Harvard Apparatus pump and locked into place, and the metal collection plate was then repositioned to the center of capillary jet outlet for optimal collection.
After the initial set-up was complete, the machine was turned on, the flow rate was adjusted until the solution was flowing through the capillary jet outlet with ease. The voltage was then turned on at a low voltage of around 10 kilovolts and gently increased over the first 5 minutes of spinning. The electrostatic current that was produced guided the nano-fibers towards the metal plate collector, where the fibers accumulated over time producing a textile. The flow rate and voltage were continually adjusted throughout the first trial in order to obtain a uniform nonwoven textile. Once the uniform nano-fibers started to appear on the collection plate, the sample was allowed to run for 30 minutes to 2 hours to ensure that the flow rate, voltage, and distance set-up were correct. A second trial followed using the set-up determined within trial 1. This trial occurred for the purpose of documenting the amount of solution (ml) and time required to produce weight (g.). The findings were then inputted into a formula to find the amount of solution (ml) and time required to produce the same weight (mass density) as the preliminary fabrication.

**Statistical Analysis**

The dependent variables in this study consisted of the measures assessed through testing: mass density, bow, skew, ratio and time for successful dissolution of nylon and polyester, and quantity of solution and time for successful electrospinning. The independent variables were the fabric samples tested: 100% nylon, 50% nylon/50% polyester blend, and 100% polyester. Once the testing was complete, basic statistical analysis was used to determine the mean and standard deviation. Two-sample t-tests measuring t-values and confidence intervals were used to determine significant differences among the fabrications during fabric characterization testing. The t-score for the independent variables were compared with the fixed critical t-scores to draw comparisons and ensure a level of commonality was maintained. Overall, advanced statistical
analysis was not highly used in this study as the purpose of this research was exploratory in nature and aimed to formulate a replicable method for chemical recycling.
CHAPTER IV

RESULTS

This experimental study was conducted for the purpose of exploring alternative opportunities for textile recycling. The methods presented within this study explored both chemical processes and electrospinning techniques for the creation of a recycled nano-spun textile with single fiber content. The findings from this study address the following research questions and sub-questions that guided the organization of this study.

1) What processes and percentages are needed for successful fiber blend separation?
   a) What is the chemical to textile percentage ratio required for successful dissolution of nylon from nylon/polyester fiber blends?
   b) What is the necessary time for successful dissolution of nylon when separating nylon from nylon/polyester fiber blends?
   c) What is the necessary process for successful removal of the polyester fibers from the dissolved nylon solution?

2) What quantities and times are required for successful electrospinning techniques to occur?
   a) What is the quantity of solution required to spin a specific size and thickness in order to maintain similarity to the preliminary textile?
   b) What is the time required to spin a specific size and thickness in order to maintain similarity to the preliminary textile?

Fabric Characterization

Preliminary testing for fabric characterization was necessary to ensure that commonality was present among the different fabrications of 100% nylon, 50% nylon/50% polyester, and 100%
polyester. The quantitative findings for the fiber content analysis are displayed within Table 3 and verify consistent fiber content percentages with the retail reports.

Table 3

Fiber Content Analysis

<table>
<thead>
<tr>
<th>Fiber Content</th>
<th>Chemical Used</th>
<th>Preliminary Weight</th>
<th>Residue Weight</th>
<th>Solubility</th>
<th>Percent Dissolved</th>
<th>Percent Insoluble</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Nylon</td>
<td>Formic Acid (88%)</td>
<td>.5</td>
<td>0</td>
<td>S</td>
<td>100%</td>
<td>--</td>
</tr>
<tr>
<td>50% Nylon/50% Polyester</td>
<td>Formic Acid (88%)</td>
<td>.51</td>
<td>0.26</td>
<td>PS</td>
<td>50%</td>
<td>50%</td>
</tr>
<tr>
<td>100% Polyester</td>
<td>m-Cresol (99%)</td>
<td>.5</td>
<td>0</td>
<td>S</td>
<td>100%</td>
<td>--</td>
</tr>
</tbody>
</table>

Note. PS = Partially Soluble, S = Soluble.

AATCC TM20A-2004 methodology was slightly altered and used as the first test method within this study to ensure fiber content percentages were accurate. Formic acid (88%) was first used for the dissolution of the nylon fibers for the 100% nylon and the 50% nylon/50% polyester blend. As there is no method for quantitative fiber analysis for single-fiber content polyester, AATCC TM20-2004 methodology was employed. M-Cresol was heated to a temperature of 139°C (282.2°F) and used for the dissolution of the polyester fibers. It is important to note that m-Cresol can also be used for the full dissolution of nylon, in addition to acetate and vinyon. Therefore, special attention to chemical selection and proper ordering of these chemicals to achieve fiber dissolution of both nylon and polyester (separately) was needed in order to accurately carry out this study and document results.

After fiber content percentages were confirmed, the mass density, bow, and skew were recorded for each fabric. Two-sample t-tests were then calculated to compare the fabric samples. This statistical test determined the level of variance among each fabrication while measuring the probability of significant difference among results. The tests compared the following: 1) 100%
nylon fabric to 100% polyester fabric, 2) 100% nylon fabric to 50% nylon/50% polyester blend, and 3) 100% polyester fabric to 50% nylon/50% polyester blend. The mass density was recorded for the full fabrication as well as three samples (each with an area of 400 in.²), and one nano-spun sample (14.04 in.²). Tables 4 and 5 display the results and two-sample t-test statistical analyses for the mass density among the three fabrications and samples.

Table 4

Fabric Characteristics – Mass Density (oz/yd²)

<table>
<thead>
<tr>
<th>Fiber Content</th>
<th>R1</th>
<th>R2</th>
<th>R3</th>
<th>Full Fabric</th>
<th>Spin Size</th>
<th>M</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Nylon</td>
<td>0.9740</td>
<td>0.9649</td>
<td>0.9786</td>
<td>0.9786</td>
<td>1.0106</td>
<td>0.9813</td>
<td>0.0361</td>
</tr>
<tr>
<td>50% Nylon/50% Polyester</td>
<td>1.0106</td>
<td>1.0244</td>
<td>1.0244</td>
<td>1.0244</td>
<td>1.0427</td>
<td>1.0253</td>
<td>0.01</td>
</tr>
<tr>
<td>100% Polyester</td>
<td>1.5777</td>
<td>1.5868</td>
<td>1.5548</td>
<td>1.5594</td>
<td>1.5960</td>
<td>1.5749</td>
<td>0.0141</td>
</tr>
</tbody>
</table>

Note. R = Replication, M = Mean, SD = Standard Deviation.

Table 5

Two Sample T-test Analysis – Mass Density (oz/yd²)

<table>
<thead>
<tr>
<th>Sample Comparisons</th>
<th>(M)</th>
<th>(SD)</th>
<th>(t)</th>
<th>(p)</th>
<th>(df)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% N to 100% P</td>
<td>0.9813</td>
<td>0.0361</td>
<td>6.009*</td>
<td>2.132</td>
<td>4</td>
</tr>
<tr>
<td>100% N to 50%/50% NP</td>
<td>1.0253</td>
<td>0.01</td>
<td>0.4588</td>
<td>2.132</td>
<td>4</td>
</tr>
<tr>
<td>100% P to 50%/50% NP</td>
<td>1.5749</td>
<td>0.0141</td>
<td>7.9307*</td>
<td>2.132</td>
<td>4</td>
</tr>
</tbody>
</table>

Note. N = Nylon, P = Polyester, (n) = sample size, (M) = mean, (SD) = standard deviation, (t) = t-value, (p) = probability, and (df) = degree of freedom. * = significant at <0.05. The degree of freedom, significance level, and p-value remained consistent for each fabrication.

Overall, the samples ranged from 0.9649 oz/yd² to 1.5960 oz/yd², which indicates a total variance of 0.6311 oz/yd² across the three fabrications. When comparing the 100% nylon to the 50/50% blend, the t-value did not exceed the critical value. However, when comparing the 100% polyester to the 50/50% blend as well as the 100% nylon, the t-values did exceed the critical
number. This indicated that there was a difference for mass density for the 100% polyester fabrication. The 90% confidence interval for the comparative analysis of 100% polyester indicated that the 100% polyester had between 0.4019 less and 0.6973 more mass density per sample than the 50% nylon/50% polyester blend and between 0.3877 less and 0.8141 more mass density per sample than the 100% nylon fabric. While there is variation among the fabrications, the variation should not impact the majority of findings for dissolution and overall electrospinning requirements. However, the time necessary to spin a sample of equivalent mass density would be impacted.

Bow and skew percentages were also assessed to determine commonality among the fabrications to ensure accuracy of the stated results. However, as nano-textiles have nonwoven/fiber-web structures, the bow and skew would not impact the overall results. This test was done primarily as an affirmative measure for representation of common fabric characteristics. Tables 6 and 7 display the findings for bow and skew and statistical analyses of the two-sample t tests.

Table 6

*Fabric Characteristics – Bow and Skew (%)*

<table>
<thead>
<tr>
<th>Fiber Content</th>
<th>Bow</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>R1</td>
<td>R2</td>
<td>R3</td>
<td>M</td>
<td>SD</td>
<td>R1</td>
<td>R2</td>
<td>R3</td>
<td>M</td>
<td>SD</td>
<td>R1</td>
<td>R2</td>
<td>R3</td>
<td>M</td>
<td>SD</td>
<td></td>
</tr>
<tr>
<td>100 % Nylon</td>
<td>0.6</td>
<td>0.3</td>
<td>0.3</td>
<td>0.4</td>
<td>0.141</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>R1</td>
<td>R2</td>
<td>R3</td>
<td>M</td>
<td>SD</td>
<td></td>
</tr>
<tr>
<td>50% Nylon/50% Polyester</td>
<td>0.9</td>
<td>3.1</td>
<td>0.9</td>
<td>1.6</td>
<td>1.038</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>R1</td>
<td>R2</td>
<td>R3</td>
<td>M</td>
<td>SD</td>
<td>0.014</td>
</tr>
<tr>
<td>100% Polyester</td>
<td>0.7</td>
<td>0.5</td>
<td>0</td>
<td>0.4</td>
<td>0.294</td>
<td>2.8</td>
<td>0.6</td>
<td>7.1</td>
<td>3.5</td>
<td>2.699</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Note.* R = Replication, M = Mean, SD = Standard Deviation.
Table 7

Two Sample T-test Analysis – Bow and Skew (%)

<table>
<thead>
<tr>
<th>Sample Comparisons</th>
<th>Bow</th>
<th>Skew</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(M)</td>
<td>(SD)</td>
</tr>
<tr>
<td>100% N to 100% P</td>
<td>0.4</td>
<td>0.141</td>
</tr>
<tr>
<td>100% N to 50%/50% NP</td>
<td>1.6</td>
<td>1.038</td>
</tr>
<tr>
<td>100% P to 50%/50% NP</td>
<td>0.4</td>
<td>0.294</td>
</tr>
</tbody>
</table>

Note. N = Nylon, P = Polyester, (n) = sample size, (M) = mean, (SD) = standard deviation, (t) = t-value, (p) = probability, and (df) = degree of freedom, * = significant at <0.05. The degree of freedom, significance level, and p-value remained consistent for each fabrication.

Assessment of the bow among the samples determined commonality between the 3 samples as the t-values for each test did not exceed the critical value. When assessing the skew percentages among the samples, the t-test comparing the 100% nylon fabric to the 50/50% blend was the only comparison that displayed commonality of the means. The t-value for the comparison of the 100% nylon fabric to the 100% polyester fabric exceeded the critical value with a 3.688% skew difference. The 90% confidence interval indicated that the 100% polyester fabric had between 0.729% and 6.271% more skew per sample than the 100% nylon fabric. The 100% polyester fabric comparison to the 50/50% also exceeded the critical value with a 3.67% skew difference. The 90% confidence interval indicated that the 100% polyester fabric had between 0.713% less and 6.267% more skew per sample than the 50/50% blend.

The overall assessment of fabric characteristics through mass density, bow percentage, and skew percentage testing indicated some significant differences in the 100% polyester fabrication when compared to the 100% nylon fabrication and the 50/50% blend. Mass density could impact the test results for time. Fiber content percentages took precedent when selecting fabrics in this study as the mass density, bow, and skew would not highly impact the results.
Chemical Processing

Chemical processing was employed to determine the overall processes and percentages needed for successful dissolution and fiber blend separation. Referencing the proposed method, a panel of small glass mason jars were organized to test chemical ratios for each fabrication. The jars were labeled for the appropriate ratio trial to each fabrication type. The fabrics along with their corresponding chemical solvent were then carefully weighed in grams and placed into the jars. Each ratio was worked through individually before moving to the next to ensure accurate documentation of results. As a reminder, the ratios used for testing within this study were 50:50, 60:40, 70:30, 80:20, and 90:10 for chemical solvent to fabric. Formic acid (88%) was used for the dissolution of nylon fiber content. Polyester fiber content was dissolved via two different methods and solvents. The ratios were first tested using m-Cresol (99%) for the chemical solvent. However, this solution did not work during the electrospinning process, and a change in solvent was made. In accordance to literature by Kayaci, Aytec, and Uyar (2013), a 50/50 mixture of trifluoroacetic acid (99%) combined with dichloromethane (>99%) was used as a secondary option for the dissolution and re-spinning of the polyester fibers. The solubility and time findings for each chemical solvent at each ratio for the three fabrics are listed in Table 8.
### Table 8

**Chemical Processing Observations**

<table>
<thead>
<tr>
<th>Fiber Content &amp; Chemical Solvent</th>
<th>Ratios</th>
<th>50/50</th>
<th>60/40</th>
<th>70/30</th>
<th>80/20</th>
<th>90/10</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Nylon Formic Acid (88%)</td>
<td>Solubility</td>
<td>IS</td>
<td>IS</td>
<td>PS</td>
<td>S</td>
<td>S</td>
</tr>
<tr>
<td></td>
<td>Time</td>
<td>--</td>
<td>--</td>
<td>3-5 m.</td>
<td>3-5 m.</td>
<td>3-5 m.</td>
</tr>
<tr>
<td>50% Nylon/50% Polyester Formic Acid (88%)</td>
<td>Solubility</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>PS</td>
<td>PS</td>
</tr>
<tr>
<td>m-Cresol (99%)</td>
<td>Time</td>
<td>--</td>
<td>--</td>
<td>12 h.</td>
<td>12 h.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Solubility</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>S</td>
<td>S</td>
</tr>
<tr>
<td></td>
<td>Time</td>
<td>--</td>
<td>--</td>
<td>20 m.</td>
<td>15 m.</td>
<td></td>
</tr>
<tr>
<td>50% TFA (99%), 50% DCM (&gt;99%)</td>
<td>Solubility</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>PS</td>
<td>PS</td>
</tr>
<tr>
<td></td>
<td>Time</td>
<td>--</td>
<td>--</td>
<td>5-7 m.</td>
<td>5-7 m.</td>
<td></td>
</tr>
<tr>
<td>100% Polyester m-Cresol (99%)</td>
<td>Solubility</td>
<td>PS</td>
<td>PS</td>
<td>PS</td>
<td>S</td>
<td>S</td>
</tr>
<tr>
<td></td>
<td>Time</td>
<td>20 m.</td>
<td>20 m.</td>
<td>20 m.</td>
<td>20 m.</td>
<td>15 m.</td>
</tr>
<tr>
<td>50% TFA (99%), 50% DCM (&gt;99%)</td>
<td>Solubility</td>
<td>IS</td>
<td>IS</td>
<td>S</td>
<td>S</td>
<td>S</td>
</tr>
<tr>
<td></td>
<td>Time</td>
<td>--</td>
<td>--</td>
<td>5-7 m.</td>
<td>5-7 m.</td>
<td>3-5 m.</td>
</tr>
</tbody>
</table>

*Note:* IS = In-Soluble, PS, Partially Soluble, S = Soluble, m = minutes, h = hours. TFA represents the chemical solvent Trifluoroacetic Acid and DCM represents the chemical solvent Dichloromethane.

### Solubility Ratios

**Single-fiber solubility ratios.** According to the findings, fiber solubility using formic acid (88%) or the mixture of TFA (99%) with DCM (>99%) did not occur for the 50:50 or 60:40 ratios. When the solvent was added to the fabric, the solvent absorbed into the fabric due to the volume of fabric present. This caused some breakdown to occur, however no solution was produced. The use of m-Cresol (99%) allowed for partial solubility for the 50:50 and 60:40
ratios, likely due to the high concentration level and chemical composition of the solvent. When testing the 70:30 ratio for all chemical solvents (formic acid, TFA/DCM, and m-Cresol), solubility was partially to mostly achieved, however, full fiber dissolution did not occur at this ratio. Full dissolution did occur for each solvent at both the 80:20 and 90:10 ratios, however the 90:10 ratio provided a solution with too much liquidity to carry out the electrospinning process. Overall, the optimal ratio of solvent to fabric weight among the majority of fabrications was the 80:20 ratio, as it provided the best consistency for electrospinning. Once the 80:20 ratio was chosen as the optimal configuration for dissolution of fibers for electrospinning, chemical weight or fabric weight was added to the other ratios to achieve an 80:20 ratio. This was done to ensure consistency of the findings as well as produce adequate solution to execute the electrospinning processes.

**Fiber blend solubility ratios.** The 50:50, 60:40, and 70:30 ratios displayed good to poor dissolution for formic acid and TFA/DCM findings and were eliminated from the dissolution trials of the 50% nylon/50% polyester fiber blend. The nylon component of the blend required the use of an 80:20 ratio for dissolution, while the polyester component required the use of a 90:10 ratio for a proper spinning consistency. The increased amount of chemicals necessary for full dissolution of the polyester was caused by the structure of the polyester post separation and neutralization. After neutralization occurred, the remaining polyester fibers hardened into a plastic mass in areas, which was more difficult to break down for spinning. Additionally, the TFA/DCM chemical combination evaporates quickly when combined so a higher initial amount was necessary to break down the hardened mass of fibers.
Dissolution Processes

Single-fiber dissolution process. The time and processes for dissolution of single-fiber content were recorded within this study. The time required for successful dissolution varied based upon the solvent: fabric ratio used, the chemical solvent used, and if fiber separation needed to occur. For a single fiber content, the time to complete dissolution was much less than the time for dissolution of the blended fiber. In the 100% nylon fabric, formic acid (88%) instantly began the dissolution of nylon when combined with the fabric. For partial and full dissolution to occur, the fibers needed to be mixed with the chemical solvent using a stirrer stick. This process required a 3-5-minute total time for full dissolution from the initial addition of the chemical solvent to the fabric. In the 100% polyester fabric, m-Cresol (99%) was initially tested for dissolution and spinning. Because dissolution with m-Cresol (99%) requires heating the solution to 139°C (282.2°F), the time for dissolution increased exponentially. Partial to full dissolution required 15-20 minutes after placing the solution on the pre-heated hot plate. As the chemical ratio increased, the time for dissolution decreased.

Fiber blend dissolution process. Fiber blend dissolution and separation processes were the final chemical processing components assessed and documented within this study. Because polyester solvents such as m-Cresol can also dissolve nylon fibers, formic acid was used to dissolve the nylon fiber content first. When performing the dissolution process on the fiber blend, formic acid (99%) needed to be mixed with the fiber content and filtered through a coarse fritted glass crucible while being constantly stirred. After the first hour, the solution needed to be stirred approximately every 3-5 hours. While the initial dissolution occurred quickly, it took 12 full hours in order to filter and produce the solution for spinning. The dried polyester fibers remaining were then rinsed out in a beaker with distilled water to remove any residual formic
acid from the fibers. The fibers were then re-dried via air-drying inside of the vent hood, which required another 3-5 hours of time. Finally, the polyester fiber content was weighed, and tested for dissolution by TFA/DCM. Performing similarly to the formic acid solvent, the TFA/DCM solvent combination reacted instantly when combined with the fabrication. This solvent also required stirring and anywhere from 3 to 7 minutes for partial and full dissolution to occur. As the chemical solvent increased, the time for dissolution decreased.

**Nano-Textile Production**

Nano-textile production was the second process explored within this study in order to determine the solution quantities and spinning times that would be required for spinning a full sample with similar dimensions as the preliminary textile. After dissolution occurred and the initial arrangement of the electrospinning equipment was complete, each fabrication went through a trial to test equipment settings. Within the trial, multiple flow rates, voltage rates, and distances were tested in order to determine the appropriate settings of electrospinning at the nano-scale for each fabrication. The trial process was very fluid and required constant changes to be made based upon visual inspection of the metal collection plate as well as the flow of the nano-fibers through the electrostatic field. The preliminary trial required anywhere from 30 minutes to 2 hours per fabrication in order to determine the final settings for each fabrication.

The final electrospinning process utilized settings for flow rate, voltage, and distance determined from trials. The following items were then recorded: flow rate, solution used, voltage, distance, time of spin, weight, and mass density. These measures can be found in Table 9. Photo representation of the electrospun fibers is displayed consecutively in Figures 3-6.
Table 9

*Electrospinning Observations*

<table>
<thead>
<tr>
<th>Fiber Content</th>
<th>Rate (ml/hr.)</th>
<th>Solution (ml.)</th>
<th>Voltage (kv)</th>
<th>Distance (in.)</th>
<th>Time (hr.)</th>
<th>Weight (g.)</th>
<th>Mass (oz/yd²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Nylon</td>
<td>2</td>
<td>8 ml</td>
<td>20</td>
<td>5</td>
<td>4:52:49</td>
<td>0.20</td>
<td>0.5716</td>
</tr>
<tr>
<td>50% Nylon from Blend</td>
<td>0.005</td>
<td>2 ml</td>
<td>17</td>
<td>5</td>
<td>3:36:05</td>
<td>0.04</td>
<td>0.1280</td>
</tr>
<tr>
<td>50% Polyester from Blend</td>
<td>.00125</td>
<td>.25 ml</td>
<td>20</td>
<td>5</td>
<td>1:17:10</td>
<td>0.03</td>
<td>0.0960</td>
</tr>
<tr>
<td>100% Polyester</td>
<td>5</td>
<td>3 ml</td>
<td>15</td>
<td>5</td>
<td>0:40:29</td>
<td>0.35</td>
<td>1.1387</td>
</tr>
</tbody>
</table>

*Note:* (ml) = milliliters, (hr) = hour, (kv) = kilovolts, (in) = inches, (g) = grams, (oz) = ounces, and (yd) = yard.

*Figure 4.* Recycled nylon nano-fibers from 100% nylon fabrication.  
*Figure 5.* Recycled nylon nano-fibers from 50/50% blend.  
*Figure 6.* Recycled polyester nano-fibers from 100% polyester fabrication.  
*Figure 7.* Recycled polyester nano-fibers from 50/50% blend.

Findings from electrospinning research resulted in extreme variations for the necessary flow rate for each fabrication. When visually comparing the electrospinning of the nylon recycled
from the 50/50% blend to the 100% nylon fabrication, the results differed drastically. Because
the process for fiber blend separation and dissolution is quite different from the process for single
fiber content dissolution, the resulting 80:20 solution developed had a higher liquidity. This in
turn created variances in the voltage, the flow rate, and the time required for production.

Similar results occurred within the 50% nylon/50% polyester fiber content. Once a fiber
content is separated via chemical processes, the residual fiber content must be cleaned and dried
before a different solution can be used to dissolve the remaining fiber content. However, when
the fabric is rinsed and dried, it hardens to a solid plastic mass. In order to dissolve the hardened
fibers, a higher chemical solution was required. The 90:10 ratio was used for the dissolution of
the remaining polyester blend resulting in a higher liquidity as well. Therefore, the flow rates and
voltage rates had to be heavily decreased for safety purposes. When the voltage is increased to
correspond to the flow rate, the process would yield a dangerous spark.

These findings were then used for a final calculation of the requirements needed to create an
electrospun textile with similar properties to the preliminary textile. In order to do this step, the
formula for mass density was calculated to solve the problem for grams versus oz/yd² using the
mass density of the preliminary textile. The next step was to assess the amount of solution (ml)
used to produce the weight of the nano-textile. The weight in grams per 1 milliliter of solution
used during the spinning trial was calculated in order to determine the total solution required for
the desired total weight. Finally, the spin time was assessed according to solution (ml) produced
per 1 minute, and the total time required for milliliters of solution per desired weight was
calculated. Table 10 displays the calculations and formulas for each step of this process.
Table 10

Electrospinning Requirements for Mass-Density of Preliminary Textile

<table>
<thead>
<tr>
<th>Fiber Content</th>
<th>Weight (g.)</th>
<th>Solution (ml.)</th>
<th>Time (hr.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% Nylon</td>
<td>0.3019</td>
<td>12.076</td>
<td>6:06:34</td>
</tr>
<tr>
<td>50% Nylon from Blend</td>
<td>0.3145</td>
<td>15.725</td>
<td>29:07:20</td>
</tr>
<tr>
<td>50% Polyester from Blend</td>
<td>0.3145</td>
<td>2.621</td>
<td>13:39:00</td>
</tr>
<tr>
<td>100% Polyester</td>
<td>0.4830</td>
<td>4.128</td>
<td>0:55:42</td>
</tr>
</tbody>
</table>

*Note:* Formula for required weight \[\text{g.} = 45.73(x/14.04)\] = insert prelim. fabric mass in oz/yd.\(^2\)
Formula for required solution \([\text{trial weight (g)}/\text{solution (ml) used}] / [\text{required weight}]\). Formula for required time \([\text{solution (ml) used/time (min) used}] / [\text{required solution}]\) and then divided again by 60 to convert back to hours.

The calculations created for the required weight, solution, and time for the production of a similar textile indicate a much higher time requirement for the solution created from the fiber blend versus the solution created from the single-fiber content. This result reflects the observations made within Table 9. It is also important to note that the polyester fiber content requires much less solution for the production of the nano-textile structure. These findings contribute to the current knowledge and literature regarding both chemical textile recycling and the electrospinning of nano-fibers. Additionally, this research provides a detailed methodology for the alternative textile recycling explored combining chemical processes with nano fiber electro-spinning techniques. These components greatly varied based upon the solution consistency produced during the dissolution phase. Therefore, the overall time to spin and amount of solution (ml) necessary for successful weight and size replication of the preliminary textile also varied for each fabrication.
CHAPTER V

DISCUSSION

Overview of Chemical Recycling Processes

Chemical processing was chosen for this study as previous literature has stated that chemical recycling processes maintain the integrity and quality of the fiber (Sanchez et al, 2015; Zamani, 2011). Other recycling processes, such as mechanical and thermal, often degrade the quality of the fiber and require intensive energy usage for fiber recovery (McIntyre, 2005). Chemical recycling utilizes chemical solvents for depolymerization, otherwise known as the breakdown of the fiber’s molecular structure (Sanchez et al, 2015; Zamani, 2011). The fiber can then be reclaimed or re-spun through processes, such as the electrospinning techniques that were explored in this study.

According to previous literature, one of the biggest limitations for chemical recycling includes the lack of technology, standardized procedures, and difficulty for the separation of fiber blends (Sandin & Peters, 2018). Fiber blends saturate today’s market for apparel and textiles and require the use of valuable, nonrenewable petroleum resources (Chen & Burns, 2006; Lewin & Pearce, 1998). These petroleum-based products are also non-biodegradable, meaning that it would take many years for break down to occur when placed into a landfill (Forum for the Future, 2007). The purpose of this thesis was to explore an alternative method for textile recycling through the combination of chemical processes and electrospinning at the nano-level. Within this research, the necessary chemical ratios and corresponding times for successful dissolution of single fiber content as well as blended fiber content were documented and used for comparative analysis. Also documented was the process of fiber blend separation for re-collection and re-spinning into a new, single fiber content fabrication. Overall, dissolution and
fiber separation were successful, indicating that chemical processing can be accomplished for the separation of fiber blends for single-fiber content recycling.

**Benefits & Risks of Chemical Processing**

**Recycling Textile Waste**

Chemical recycling processes offer an alternative solution to traditional textile waste platforms such as landfill release and incineration. With the increased market adoption of fast fashion styles and rampant consumerism at a global scale, textile production and therefore textile waste disposal has skyrocketed. In a CBC News report (2018), consumption practices had been reported to have risen by approximately 400% over the last 30-40 years. Additionally, the EPA (2017) reported 11.2 million tons of textile landfill waste in the year 2017 alone. Currently, only 1% of clothing is estimated to be recycled at the fiber level. However, textiles are nearly 100% recyclable meaning that when recycling efforts are used, the textiles can function in a circular economy (Hawley 2009). Fiber-to-fiber recycling through chemical processes offers numerous benefits for the environment, such as the aforementioned diversion of textile waste, the potential for fiber blend separation, and the maintenance of quality throughout numerous lifecycles.

**Potential for Closed-Loop Textile Systems**

As previously stated, chemical processing provides a recycling opportunity that incurs no degradation of the fiber and maintains the fiber quality. Therefore, this process of recycling allows the textile to enter into a circular economy for numerous lifecycles. The purpose of this study was to explore the combination of chemical processing and nano-fiber electrospinning for the creation of an enhanced recycled textile with single-fiber content that can perform similarly to a fiber blend. The concept of blend separation aligns with circular economy principles as
unwanted textiles (waste) may be recycled and remade into a new textile that can then be used for a multitude of products.

Findings of this study indicate that fiber blend separation is more difficult to achieve and requires larger quantities of chemical usage compared to the dissolution process for single-fiber textiles. Therefore, when blended fibers are separated during textile recycling, it would be more efficient to keep the fibers separated in single-fiber form for systematic ease in the circular system. The industry could utilize electrospinning technology to achieve the desired fiber characteristics and performance presently found in blended textiles. With textile advancements offered through nanotechnology, the challenging and time-consuming process of separating fiber blends would only need be performed a single time during the initial recycling phase. Using electrospinning technology to produce a high-performance single-fiber textile may also result in longer product use (and re-use/re-purposing) and deter routing as waste into landfills. According to Boiten, Li-Chou, & Tyler (2017), innovative thinking and policy needs to be implemented for a successful circular economy to occur. The ideas presented within this exploratory study offer an innovative approach that combine the studies of science with technology that may be further researched and adopted by apparel and textile industries.

Risks

While Hawley (2009) reported that textile recycling has limited new hazardous waste or harmful emissions, findings from this study show a high use of dangerous and toxic chemicals connected to the chemical dissolution process. The findings within this study reported that in order for full dissolution to occur, the ratio of chemicals used to fabric weight were very high with an average ratio of 80:20. The chemicals, when not used safely, could also be dangerous to the environment or the health of the worker. Within this study, formic acid (99%), m-Cresol
(99%), trifluoroacetic acid (99%), and dichloromethane (>99%) were all used. There are many safety risks regarding the use of these chemicals as they can be toxic, corrosive, and flammable/combustible. Additionally, large chemical quantities were required to dissolve relatively small fabric quantities. For perspective, 500 ml of formic acid was used within this study for testing the dissolution of less than two yards of nylon and nylon/polyester textiles.

Considering the high rate of apparel production, consumption, and disposal, the chemicals needed to chemically recycle nylon and polyester would be exponentially increased to align with the scale of the apparel and textile industries. More research is needed to more accurately forecast the environmental impacts and safety risks of scaling use of chemicals needed for chemical textile recycling.

**Overview of Nano-Textile Production**

The production of a nano-textile utilizes electrospinning processes that produce fibers within an electrostatic field at the nano scale. This process was originally patented in 1934 by Formhals and has since been used for the development of innovative and enhanced textiles. Aside from textile production, other processes can also be used to integrate nanoparticles into an existing textile through weave structure or surface finishes (Black, 2009). When researching electrospinning literature, there was an apparent gap of documented specifications to create electrospun textiles from chemical solutions. For example, limited information was found regarding the requirements for nano-textile creation by fabric characteristics such as size and mass density. Therefore, nano-textile production within this study was explored for recycled single fiber content as well as a recycled blend. The components assessed were voltage, flow rate, and distance between the needle and collection plate. These components are necessary for all electrospinning processes regardless of fiber content. However, the requirements for each
component change depending on fiber content and solution consistency. Reports from this exploratory study found large variances in these components based on fiber dissolution processes and fiber content of nylon versus polyester. The documentation of this work will allow researchers to build upon knowledge learned and processes employed to assist in streamlining future electrospinning efforts. As electrospinning a nano-textile is time-intensive, the reports for time requirements will allow researchers to more accurately allocate time needed to execute studies. Additionally, because blended fiber content is commonly used within the apparel and textile industries, it was important to document the electrospinning processes and findings post-chemical recycling to disseminate for future research and/or industry application.

**Benefits & Risks of Electrospinning**

Research on nanotechnology within textiles is limited, however suggestions for perceived risks and benefits exist. Potential benefits are discussed at an environmental and societal level and include opportunities for sustainable energy as well as extended product life through effective nano-scale finishes (Black, 2009). The development and production of both recycled and novel, pure nanofibers could provide benefits resulting in decreased raw material use for textile products. With continued advancements in electrospinning fiber technologies, the need and production of blended fibers may decrease over time as advanced technological engineering is capable at the nano-scale, yielding enhanced single-fiber textiles.

One study, conducted for the Federal Institute for Risk Assessment (BfR), analyzed risk assessment concerning nanotechnologies by setting up the first consumer conference on nanotechnologies in the spring of 2006 (Petschow et.al., 2009). Results of the conference showed clear evidence that the advantages of nanotechnologies within textiles far outweighs the risks (Petschow et.al., 2009). Within terms of production safety, nanoparticles for textiles
should be produced within enclosed and contained ventilation systems as these systems would ventilate the potential release of nanoparticles into the general environment (Petschow et al., 2009). Regulations concerning critical ranges and labelling of nano-finished textiles were not required at the time of the conference study; therefore, such regulations were suggested as necessary moving forward (Petschow et al., 2009). Other suggestions regarding improving regulations of nanotechnology moving forwarded included: uniform scientific definitions surrounding “nano”, implementation of methods measuring risk assessment, and shared information of nanotechnologies to the general public (Petschow et al., 2009). The research completed within this exploratory study allows for the dissemination of study findings regarding processes and time requirements of electrospinning.

Within this exploratory study, benefits of nano-textile production for closed loop systems when combined with chemical processing are also presented. The opportunity to successfully separate fiber blends and re-spin into a single fiber content via electrospinning were achieved. Further testing, however, is needed to assess qualitative attributes of the textile. When using the electrospinning method for this method, extreme caution was used when handling the solution, cleaning materials (acetone), and voltage. There were associated risks concerning the chemicals present that occurred throughout all stages of the study, include the electrospinning of the nano-textile. While some of the potential risks and benefits have been discovered and discussed, a full life-cycle analysis and potential ecotoxicology of nanotechnology in food, cosmetics, and textiles is yet to be explored (Petschow et al., 2009). Further research also needs to be done regarding the disposal and recycling of nanotechnologies within textiles.
Feasibility for Large-Scale Application

When considering feasibility of method application at a large-scale, it is important to consider the time and financial constraints presented by the findings of this study. In order for successful fiber dissolution and blend separation to occur, high volumes of chemical solvents were required in the depolymerization process. These chemicals are often expensive and difficult to obtain, particularly in the quantities needed to execute the dissolution processes. Throughout this study, the researcher was subjected to chemicals throughout the testing phase. It is important to consider safety protocols and associated costs that would need to be put in place for chemical processing to occur at a large-scale. Additionally, the risks associated to workers when subjected to chemicals at a large-scale would need to be considered and further assessed, potentially exploring long-term handling and exposure.

The time for dissolution for the fiber blend also presents a challenge in a time-oriented industry. The total time for dissolution of the fiber blend in the fritted glass crucible took half of a day (12 hours) to accomplish. Most companies simply will not allocate such lengthy time and financial resources when they can purchase virgin fiber for a much more affordable price in a timely manner. While the dissolution time for recycling fiber blends is longer compared with 100% single-fiber content, the dissolution using acids occurs quickly (with tendency for evaporation and unusable solution if not contained). Additionally, when a solution is left sitting for too long, the solution may re-harden into a plastic-like mass, depending upon the fiber content and solvent used. This increases the difficulty for re-dissolution of the fibers.

Finally, the costs and time associated with nano-textile production via electro-spinning process, are important to consider. For a small fabrication to be produced, it could take anywhere from 4-30 hours, based on findings from this study. In order to make a profit by producing nano-
spun textiles a company would need to set a high wholesale price (which also impacts the retail price) to be able to allocate time, materials, and equipment costs. High purchase prices would most likely deflect the opportunity to reach a large (mass-market) consumer base.
CHAPTER VI
CONCLUSION

Significance

In this study, chemical recycling was combined with the process of electrospinning for the production of single-fiber nano-textiles. This research was exploratory in structure with the intent of creating a protocol of the processes for chemical dissolution and electrospinning among single fiber and blended fiber nylon and polyester fabrics. For chemical processing, ratios, times, and processes were assessed for successful dissolution of fiber content and fiber blend separation. For nano-textile production via electrospinning, the parameters for voltage; flow rate; and distance were assessed and used for calculation of the time and solution necessary for specific textile production based on mass density and size. Based on the data collected for chemical dissolution, one ratio (80:20) was selected as optimal for dissolution among the samples, however time for dissolution displayed variance among the results. Based on the data collected for nano-textile production, large variances were present among both similar fiber contents and differing fiber contents as the fiber recovered from the blend displayed higher solution liquidity. This knowledge provides valuable information for researchers and industry professionals.

The apparel and textiles industry is both consumer driven and ever expanding in size ranking as one of the most globalized industries in the world (Palamutcu, 2015). With the prevalence of fast fashion markets promoting the shift to consumerism and mass consumption within apparel and textiles, it is imperative that alternative methods are assessed regarding textile waste. For perspective, consider that the per capita fiber consumption of textile products has increased by 39% with no expectations of slowing down. With the shift to mass consumption also comes mass disposal of unwanted or damaged textile goods. According to CBC News (2018), North America
contributes an estimate 25 billion pounds of textile waste to landfills each year. The combination of chemical recycling and electrospinning assessed and discussed within this research promotes the potential for recycling for single-fiber content while properties and qualities have the potential to remain enhanced or superior. This also provides the opportunity for minimized overall use of fiber blends and the circular or closed-loop textile lifecycle.

**Implications**

One of the implications of this research is the guidance provided to other researchers for the processes of chemical dissolution, fiber blend separation, and electrospinning. Not only does this study demonstrate that fiber blend separation is possible for recycling and re-use, but it also provides precise chemical to fabric ratio percentages and weights required for successful dissolution of both nylon and polyester. Processes and formulas for determining exact levels of solution and time needed for electrospinning also provide valuable information which will allow for efficient project management. The most beneficial finding from this study was the successful use of chemical recycling combined with electrospinning to produce a functional textile. This finding alone indicates promise for long-term positive environmental change for apparel and textiles by returning the unwanted textile into a circular recycling system for reuse; diverting this waste from landfills.

**Future Research**

Future research considerations to build upon the work and findings outlined in this study include conducting multiple trials for electrospinning to ensure the formulas produced provide the expected results. Further trials should also be conducted on dissolution testing for the ratios not tested in this study, such as 75:25 or 85:15. Testing should also be done across more variations of nylon and polyester fiber blend percentages to determine similar or different
findings. Details documented in future textile chemical recycling and electrospinning work should be consistent with the documented findings of this study including: the blend separation process, the ratio required for the collection of usable solution, and electrospinning requirements for voltage, flow rate, and distance.

The exploration of other fiber contents may also benefit future research. The protocol and processes outlined in this study may be replicated for fibers such as spandex and acetate as market analysis showed a prevalent use of these two additional synthetic fibers. Fiber blend separation documenting the removal of a synthetic fiber from a natural fiber would also yield beneficial findings for the area of textile recycling via chemical processes. Procedures used in this study may also be replicated for fiber blends with cotton or wool (natural fiber) content.

Finally, a full qualitative analysis comparing performance characteristics of electrospun textiles to virgin and industry recycled textiles is recommended for future research. Qualitative analysis procedures, such as launderability, strength, abrasion resistance, wrinkling, etc. would evaluate the concept of enhanced property attributes for specific fiber content through electrospun textile production. Assessing the qualitative properties would provide essential feedback regarding the proposed notion set forth within this study. This notion indicated the concept of enhanced property potential of the recycled single-fiber nano-textile, therefore eliminating the need for blended fiber content.
REFERENCES


