

**STUDYING THE INTERACTIONS OF IONIC LIQUIDS
AND BLENDED FABRICS**

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198491B

Degree of Master of Engineering in Environment Engineering and
Management

Department of Civil Engineering

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ABSTRACT

Textile waste is a huge contributor of solid wastes piling up in landfills every second around the world. It has been a challenge over decades to recycle textile waste due to reasons including high durability of the materials and difficulty in sorting.

Blended fabrics are a category that contributes for solid wastes that are difficult to be recycled due to combined properties of different fabric types used. Spandex is one such blending yarn type often combined with fabrics to enhance stretchability and elastic recoverability. It is a synthetic fiber with segmented polyurethane with superior elasticity, durability and shape retention. It has more durability and elasticity than natural rubber. Spandex is commonly used in sportswear, lingerie, swim wear and leisurewear. Spandex has an exceptional elasticity due to alternating rigid and flexible segments. Composites of polyester-spandex, nylon-spandex and cotton-spandex are commonly used in garment industry. The spandex percentage ranges from about 5% to 40%. There is a high tendency of landfilling Spandex blended fabrics because highly elastic fibers have totally different characteristics than other fibers which make them difficult to be recycled (Langley and Kim, 2006).

Even though there are few studies focused on recycling of Spandex fabric blends, hardly any study is available on recycling of spandex -polyester blends and spandex-cotton blends. This study was aimed to see the possibility of selectively dissolving spandex in an ionic liquid and recovering polyester and cotton in pure form. Recovered polyester can be used to make polyester yarns, bottles etc. Regenerated cotton can be used to produce cotton yarns. The study will initially be focused on ionic liquids as those are environmentally friendly since they can be used as solvents at room temperature.

The experiments were carried out and found out that the polyester + spandex fabric and cotton + spandex fabric does not show any significant change after dissolution.

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LIST OF ABBREVIATIONS

IL – Ionic Liquid

AMIMCl - 1-Allyl-3-methylimidazolium chloride

BMIMCl - 1-Butyl-3-methylimidazolium chloride

MMT – Million Metric Tons

1. INTRODUCTION

There's a huge contribution from textile and apparel industry to the solid waste generated every year globally. A recent report has highlighted that \$ 400 bn worth of fabric wastes are generated annually. This number reflects the impact that it can create. In other words, a garbage truck of apparels is landfilled or dumped every second (Reichart & Drew, 2019). These are the Economic, Social and Environmental Impacts of Fast Fashion. Fiber production worldwide for textile and apparel amounts to more than 110 MMT/yr. Production of textiles ranges from basic daily clothes to technical clothing (Ütebay et al., 2020).

Textiles are made up of yarns made with natural and synthetic fibers. Natural textile fibers are made from plant sources (i.e. Cellulose compounds) or animal sources (i.e. proteins). Cotton is an example for natural fibers which produces around 40% of world fabric demand. Synthetic fabrics are artificially man-made with cellulosic fibers such as Rayon, Tencel and Lyocell or synthetic fibers such as polyester & nylon that are fully chemically synthesized. Synthetic and natural fibers are combined to get better properties (Pinheiro & de Francisco, 2016). Spandex is one of those synthetic yarns made up with segmented polyurethane. It is blended with different yarn types due to its superior stretch and elastic recoverability. Therefore, they are widely used in sportswear and in cases where maintaining stable shape under a loading is required. Even though the fiber type is enhancing the properties immensely, it is challenging to be recycled (Lv et al., 2015). Natural fibers take decades to decay whereas polymers such as spandex take 200 years in a landfill to breakdown (Shirvanimoghaddam et al., 2020).

Not only the wasted textile wastes but also the product itself (i.e. apparels) ends up in contributing to the solid wastes piling up around the globe. Only a small amount of this waste is recycled whereas most of it end up in a landfill, an open dump or in an incinerator (Ütebay et al., 2020) It is expected if the current trend in generating textile waste continues further, by 2050 26% of the global carbon budget will be from the Textile and Apparel industry. Since most of the raw materials are non-renewable it is

expected that 22 MMT of micro plastics will be released to oceans by 2050 (Ellen MacArthur Foundation, 2017).

Alongside the natural increase of production of textiles due to increase in population, the trend of fast fashions also contributes to the huge overconsumption of resources. Even though the prime purposes of clothing are to protect human body from cold, heat and to be modest, now it has moved to a completely different level where it is connected to personalities, wealth and fashion (Yalcin-Enis et al., 2019). However, as a result, from the point of sourcing raw materials to delivery of final product to retail shops the industry generates huge amounts of wastes and have become a significant portion of municipal solid waste (Karaosman et al., 2016). Another factor that causes increase overconsumption and high wastage is that the idea created by the leaders of fashion that people have to go for new fashions depending on the seasons of the year. This is very prominent in the markets of the developed world (Zamani, 2014)

Solid fabric wastes can be categorized into three main categories broadly as production waste, pre-consumer waste and post-consumer waste. Further categorization is shown in Figure 1.

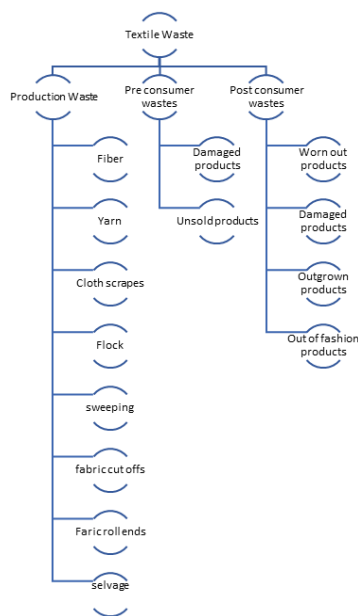


Figure 1: Textile Waste Categorization (Yalcin-Enis et al., 2019)

1.1. Management of Wastes Generated from Spandex Blended fabrics

Spandex is among those yarn types that are blended with other fibers to make fabrics with superior properties but difficult to be recycled. Managing spandex blended fabric wastes is challenging due to several reasons. Therefore, it is important to view it from different perspectives including waste management hierarchy and concepts of circular economy. The main objective is to minimize the amount of fabric that is being landfilled which is the burning issue.

1.1.1. Hierarchy of Waste Management & Circular Economy

In the hierarchy of waste management (Figure 2), prevention and reduce are the options that are most favoured. Then it is followed by reuse, recycle, energy recovery, and disposal is the least favoured option. Circular economy of textile waste management which agrees with above concept is also followed by many of the leading brands currently which has resulted favourably on 3R to get adopted and to prevent large amounts of wastes that are landfilled (Yalcin-Enis et al., 2019).

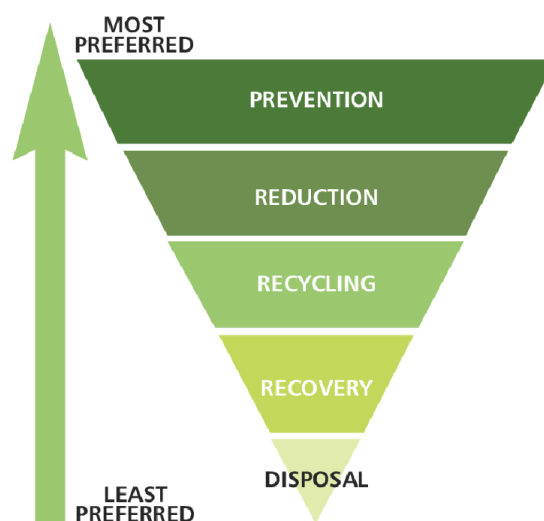


Figure 2:Textile Waste Management Hierarchy (The Waste Hierarchy as Described in the EU Waste Framework..., n.d.)

Reuse of blended fabrics can be done by transferring the products to new owners once they are used. It can be done by methods of re-selling, renting, trading or swapping. It is practiced in many of the communities and in Africa the clothing market is

predominantly handled by this type of secondhand sales (Bukhari et al., 2018). However, it cannot be considered as a solution to the global issue as the behaviour of the markets are different from place to place.

On the other hand, recycling of blended textiles is done up to some extent by converting pre- or post-consumer textile products into textile products or into non-textile products. Producing textile yarns using PET bottles can be taken as an example for this. The methods of recycling include thermal, chemical, and mechanical processes. In chemical recycling depolymerization of polymers such as polyester, and dissolution of natural fibers such as cellulose (Sandin & Peters, 2018) are done. Mechanical processes are commonly used as pre-treatment methods in most of the recycling processes. Conversion of PET flakes into fibers by melt processing can be considered as a method of thermal processing (Schmidt et al., 2016). This method is commonly used for spandex- nylon blended fabrics since they can be reprocessed to make toys, construction materials etc.

There are other forms of classification of methods of recycling including upcycling or downcycling or open loop or closed loop. Most of the fabric recycling processes result in down cycling as the strength and the length of the recycled fabric is lowered due to wearing and laundering. However, in most of the cases it does not cause the recycling to be less preferred in the hierarchy (Bukhari et al., 2018).

In closed looped systems the recycled product should have identical or similar properties to the original blended textile product. The open loop systems produce products that are different from the original product. Even though there are different classifications of recycling as described above, recycling can be considered as a valuable option if properly managed. However, recycling of blended fabrics is having complexities more than any other category of textile waste.

1.1. Challenges of Managing Spandex Blended Fabric Wastes

There are different factors that can act as barriers for recycling in different types of textile products. It can be the type of yarn or the types of dyes and chemicals that have been used in manufacturing. Irrespective of certain barriers scholars have worked on

repurposing and recycling textile wastes and have given numerous useful insights to practical recycling and repurposing interventions.

Can (2019) has conducted research on how short fiber wastes of cotton can be used as a sound absorbing material. A similar type of a study has been done by Raj et al. (2009) using Denim waste and has shown it works better than glass wool as a sound absorber. These can be considered as examples of studies on repurposing textile waste. Furthermore, many studies have been done on using textile wastes as construction materials with lower weight than traditional construction materials and with better properties including enhanced compressive and flexural strengths. (Rajput et al., 2012 and Peña-Pichardo et al., 2018). Repurposing studies have been used to identify how cotton and lyocell wastes can be used for adsorption of heavy metals in heavy metal removal (Ma et al., 2018 and Bediako et al., 2016). Another recent study has shown that different types of fabric waste such as cotton, viscose and polyester can be used as raw materials for making biochar. (Hanoğlu et al., 2019). However, these options cannot be considered as upcycling. Therefore, there is a need for exploring solutions for spandex blended fabrics.

The concept of circular economy ensures sustainability from the very first point of design. However, when it comes to elastane, starting from a petroleum based raw material making it an energy intensive product and with the microplastics released during processing it has been challenging to make it a sustainable material. Blending it with other fabrics worsens the problem as the blended fabric also has similar environmental impacts. However, since there is no substitute to the exceptional properties of this material yet, it is important to focus on green solutions to recycle spandex blended fabrics.

1.2. Problem Statement

Textile wastes including blended fabrics have created a lot of issues around the globe. Generation of CO₂ in producing textiles using raw fibrous materials is around 15kg per kilo gram of textile and the processes consume a lot of water and energy. It is not much different for both synthetic and natural yarns (Yin et al., 2013). This in turn

contributes to health hazards and a lot of harmful impacts on the environment as well. Therefore, it is essential to focus on reusing textile wastes as much as possible in order to reduce land filling and achieving sustainability through conservation of resources in a broader perspective. Conventional methods of regeneration of cotton yarns encounter numerous drawbacks in processing (De Silva et al., 2014).

The presence of spandex increases the challenges associated with blended fabrics (Wang et al., 2022). Therefore, out of the methods explained above from the perspective of circular economy and hierarchy of wastes, reusing and recycling are the main options that can be considered for spandex blended fabrics other than for both spandex-cotton blends and spandex-polyester blends which have influenced the science world to focus more on finding solutions to further reduce the amounts of wastes landfilled (Leonas, 2016).

It is commonly known in the industry that spandex can be removed from blended fabrics using solvents such as N,N-dimethylformamide. However, it is undesirable due to environmental and economic reasons (Yin et al., 2013). One of the possible options for dissolving cotton and polyester yarns is the use of ionic liquids. Furthermore, since the ionic liquids are design flexible, they can be used to convert these waste types to reusable forms. It allows textile waste to be effectively reutilized. Even though green solvents are being considered as a solution in many industries there is only a limited number of studies available in literature for textile related wastes. Therefore, it is worthwhile to explore the possibilities, especially in the context of blended textiles.

1.3. Aim and Objective of the Research

This study is mainly focused on exploring sustainable solutions for recycling of spandex blended fabrics. That being the main aim the specific objectives are,

- To explore the interactions between ionic liquids and blended fabrics
- To explore the dissolution mechanisms of polymers in Ionic liquids
- To seek solutions for spandex blended fabric wastes

2. LITERATURE REVIEW

As the textile and fashion industry grows, the demand for different types of fibers increases as the consumers are looking for new fashion and properties in fabrics. Often, different fiber types are combined in textile manufacturing to get properties that are not possible with a single type of yarn alone. Sometimes it is done due to economic reasons or due to scarcity of natural fibers. Furthermore, the blended fibers help to add missing properties to natural fibers, and it helps to give a better spinning performance for yarns. Even though, natural fibers are freely available in nature and have properties such as renewability, biodegradability, light weight and low cost, blended fabrics show better properties when performance comes into play (Bora & Padmini, 2020).

2.1. Polyester

Almost all the synthetic fibers are derived from petroleum-based polymers. Demand for synthetic fibers is increasing day by day mainly due to low-cost despite the related environmental issues. Polyester, Nylon, Polypropylene, and aramid (polyamide nylon) fibers are among the most used synthetic fibers. As polyester fibers are low cost and can be produced easily, polyester and its blends cater to a significant portion of global fiber demand. Properties including high durability, wrinkle and abrasion resistance, quick drying time have contributed polyester blends to become dominant in fiber market irrespective of the environmental impact it has created (Rashmi Nayak et al., 2022). Polymer is often blended with natural fibers such as cotton to enhance moisture management in fabrics. It is blended with Spandex to give elasticity and better stretch and recovery properties. However, these blended fabrics are responsible for a high percentage of landfilled fabric waste (De Silva et al., 2014).

2.2. Spandex

Spandex fabric has superior elastic recoverability properties and an excellent stretchability levels which helps to increase the fit and comfort qualities of a garment. 400%-800%. The other important property is that Spandex fiber returns to its original shape as soon as the deforming force is removed (Herrero et al., 2019). It is widely used in sportswear and swimwear apparel manufacturing. Dimension stability and wrinkle recovery also can be considered as special properties of Spandex fabric that make the

yarn suitable for many different applications. There are some deficient properties of spandex yarn such as low thermal stability and chemical resistance which are often managed in manufacturing by blending with other fibers like Nylon (SMM et al., 2020)

Spandex was first manufactured in 1960s. DuPont was the market leader that controlled about 80% of the market. In 2019, 7.39 billion USD has been the size of the global spandex market. Spandex is formed by reacting Polyurethane with a Di-Isocyanide (Herrero et al., 2019). It is a synthetic linear macromolecule that contains more than 85% of polyurethane that is segmented. Alternating hard and soft segments which gives a stretchability that recovers soon are linked by the Urethane bonds.

There are several different techniques used to manufacture spandex such as wet spinning, melt extrusion and dry spinning. In 2019, more than 90% of the Spandex made have been manufactured using solution dry spinning process.

In all these processes pre-polymer making by reacting the monomers is done as the first step. Then it is run through reactions to make long fibers of spandex. Figure 3 shows the chemical structure of spandex (SMM et al., 2020).

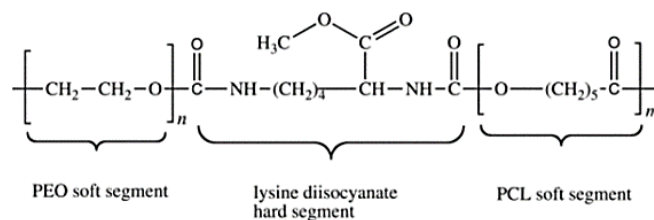


Figure 3: Chemical Structure of Spandex

Spandex fibers have a superior strength that they do not reach breaking point until it is stretched to the extreme stretch level (Arrosyad et al., 2020).

Spandex enhances stretch and recovery of fabrics in a controlled way even if it is a warp knit, weft knit, lace, mesh and any woven and non-woven fabric. The amount of spandex used is usually determined by the level of modulus required. For shape retention a small percentage of around 2% of spandex is added whereas for high performance garments such as swimwear around 30% of spandex is added (Senthilkumar & Anbumani, 2010).

2.3. Recycling of blended fabrics

In blended fabrics as the fibers are blended in a complicated manner, they cannot be separated using mechanical methods and chemical treatment is often used. There are few studies in literature focused on recycling of cotton/polyester blends. For extraction of cotton highly concentrated Sulphuric acid, phosphoric acid and Sodium Hydrochloride aqueous solutions are used. The recovered yarns are lower in tensile strength than the original ones (Yin et al., 2013). Moreover, these types of solvent usage cannot be considered as environmentally friendly. There are not many studies focused to dissolve polyester yarns as most of the substances used for dissolution of polyester are toxic. Therefore, most of the times polyester blended fabrics end up in landfills creating a huge environmental especially due to slow degradation (De Silva et al., 2014).

Nylon fiber which is having a high strength and chemical resistance but not having a good extensibility is often blended with spandex to achieve a better performance in the fabric. There are studies on recycling the blended fabric to recover the Nylon fibers which is the main component of the blended fabric (Yin et al., 2013).

When considering the stability of spandex using thermogravimetric analysis (TGA) it has been found that the chemical bonding in spandex is stable beyond 200 °C (Figure 4) and the degradation starts with thermolysis of hard segments (Boschmeier et al., 2023).

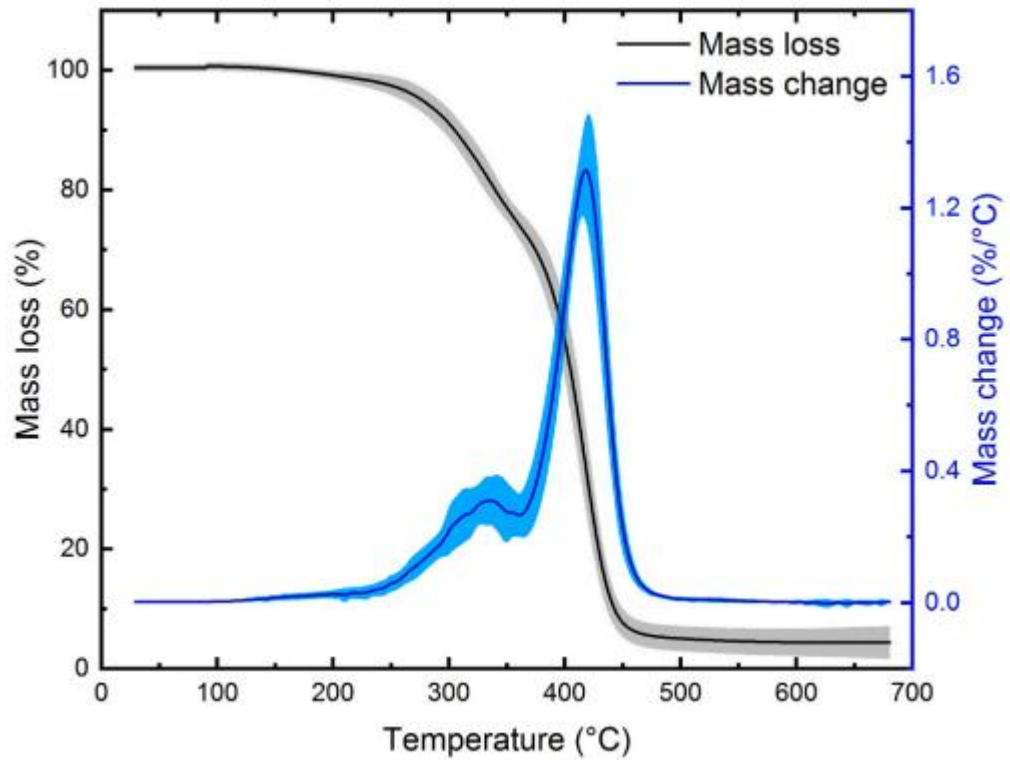


Figure 4: TGA Results of Elastane (Boschmeier et al., 2023)

2.4. Issues in recycling of blended fabrics

Attempts on recycling of spandex are limited as the recycling industries face issues when the stretchy fibers of spandex get entangled and wrapped in machinery (Langley and Kim, 2006). However, it is possible to recycle fabrics containing 10%-14% of spandex using conventional cutting and pulling machinery as the entanglement of fibers in machinery is less. However, the resultant material does not have a consistent quality as it can be a mixture of small pieces of yarn and individual fibers (De Silva et al., 2014).

There has been a study done on possibility of making flock using spandex blended fabrics. Flocking is the process of applying finely cut fibers to an adhesive coated surface. It is done by means of mechanical, electrostatic or pneumatic forces or with combinations of above forces. Flocked materials are used in sound dampening, cushioning and protection, thermal insulation and in some decorative applications (Langley and Kim, 2006).

In a study done by (Yin, 2013) possibility of dissolving spandex in a Nylon -Spandex blended fabric using an environmentally friendly method has been explored. The conventional solutions used to dissolve spandex such as using the solvent N,N-dimethylformamide are not considered as environmentally friendly and it is not economical to use them also. Selective degradation has been used to remove spandex and Nylon. It has been done as a two-step process in which firstly the blended fabric has gone through a heat treatment process which has been followed by a washing process using ethanol. They have been able to achieve an effective separation and have been able to find the optimum temperature and pressure combinations to be used (Wang et.al, 2009).

2.5. Green Solvents

Due to high volatility and large volumes used, solvents are generally considered as agents that are harmful to the environment. Therefore, globally actions are taken to reduce usage of harmful solvents, especially volatile organic compounds (VOCs) that are at highest environmental risks. It has opened a new area of study focused on environmentally friendly solvents. Mainly the solvents developed to match this criterion are using reduced amount of energy and reduces emission of VOCs. Ionic liquids (ILs) can be considered as solvents that meet these requirements (De Silva et al., 2014). ILs are organic solvents that are in liquid form at ambient temperatures. It's a class of molten salts that are completely made up of ions. Usually, they have large organic cations.

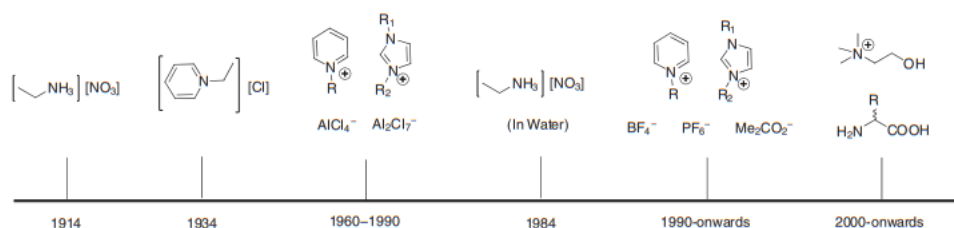


Figure 5: Important Milestones of History of ILs (Macfarlane et al., 2017)

They get paired with small anions which can be organic or inorganic. The functions of the ILS depend heavily on cations and anions combined.

There are various useful properties of ILs. Figure 6 shows the common spectrum of ILs. ILs do not produce volatile organic substances in normal pressure conditions as they are non-volatile. Furthermore, they do not tend to decompose as they are highly thermally stable so that they can be used over a wide temperature range in reactions as required. The high ionic properties of ILs enhance the reaction rates of experiments. Moreover, they can be stored over a long period of time without any deterioration (Wang et al., 2022).

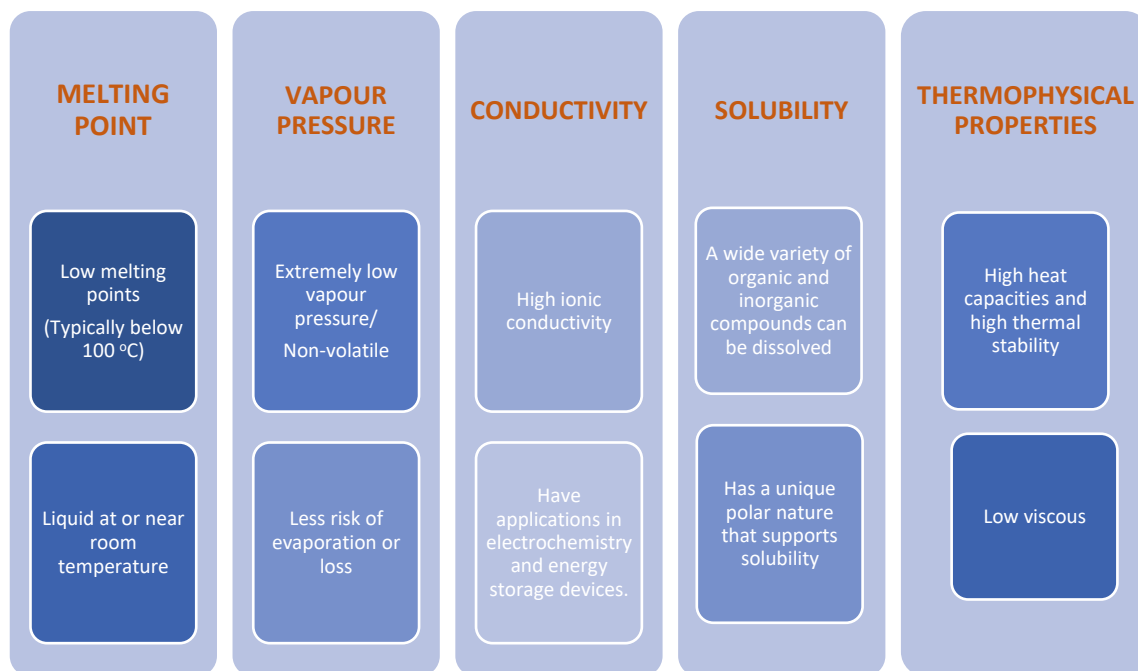


Figure 6: Spectrum of Ionic Liquids

A molten salt at room temperature has first been mentioned in a study done by Welton (1914). However, Wilks, Hussey and Seddon are considered as the pioneers of ionic liquids with their contribution to research on ILs. ILs based on Aluminum Chloride are considered as the first generation ILs.

However, they were not air stable. Eventually scholars have introduced air and water stable ILs which helped to widen the usage of ILs. Figure 5 shows the important milestones of development of ILs.

Modern day ILs have been developed as a result of experiments done with more hydrophobic anions. In modern day research is focused more on using ILs as

environmentally friendly or green solvents with the emerging environmental challenges across the globe (Wang et al., 2009).

ILs are made up of cationic and anionic structures like most of the other salts but they are less likely to crystallize than the other salts as the cationic structures are bulky and asymmetric (Senthilkumar & Anbumani, 2010). There are millions of combinations of cations and anions that can form ILs. Generally, the anion is responsible for the properties such as water solubility and stability and the cation is responsible for melting temperature and solubility. With the flexibility to tune up the ILs to meet the requirements of the applications they are known as designer solvents as well. Other physical properties such as density and viscosity also can be varied by changing the structure of the IL (Wang et al., 2022).

Another important property of ILs is their ability to recover metals. Studies have been done on possibility of using ILs to recover important metals including gold and silver from the metal matrices and also proven to be used for extraction of active metals including Na, Mg and Al. Furthermore, their ability to recover radioactive component from nuclear fuels that are spent also has been explored. There has been research done on capability of ionic liquids to remove Hydrogen Sulfide using absorption which is an important finding when it comes to oil and gas industries (Yin et al., 2013).

Studies that are focused on ionic liquids and their applications have expanded over recent years. Earlier they were considered as a solvent group with limited applications (Wang et al., 2022). Figure 6 shows the range of different applications of ionic liquids. However, now they are being used in many fields Ionic liquids that are in liquid form at room temperature have no molecules that are neutral. The main reason for possibility of expanding the usage of ionic liquids is that they can be made by combining various types of cations and anions to have different physical and chemical properties (Senthilkumar & Anbumani, 2010).

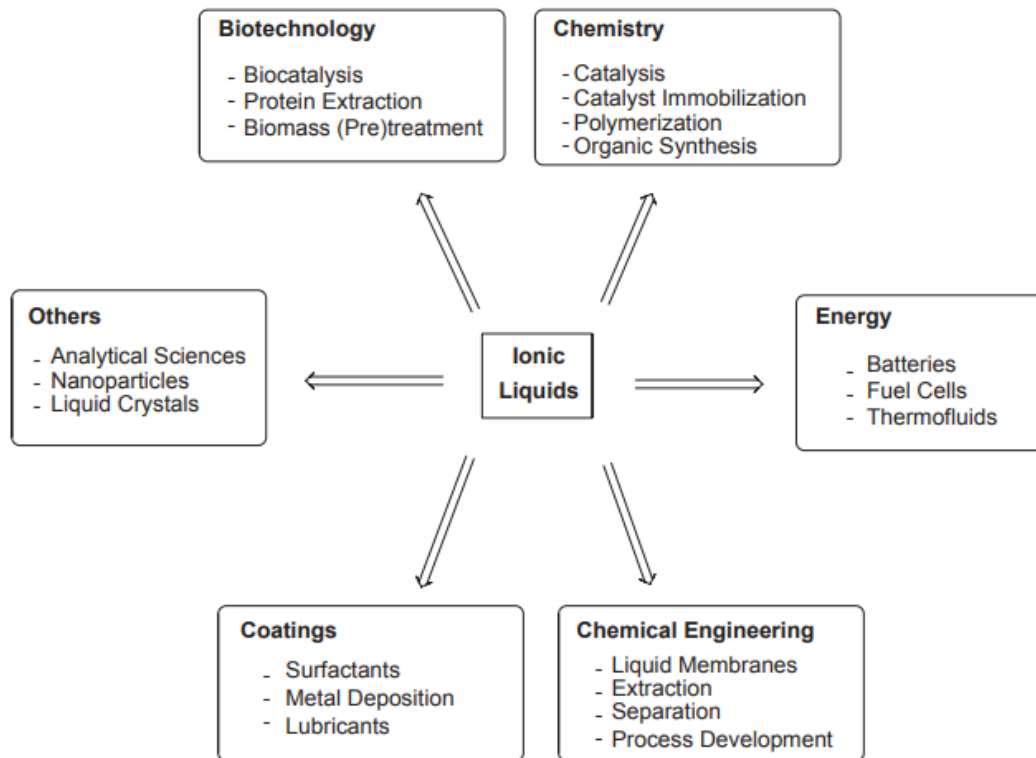


Figure 7: Range of Applications of Ionic Liquids (Macfarlane et al., 2017)

Negligible vapour pressure, chemical stability and thermal stability have been the reasons for ionic liquids to be considered as environmentally friendly. When it comes to green chemistry ionic liquids hold a significant place as they are used for formation of natural polymers. This area of research has been greatly benefited by advancements of research on ILs (De Silva et al., 2014).

Even though ionic liquids are traditionally considered to be costly solvents specially because of high costs of manufacturing the ability to regenerate almost fully helps to overcome the challenge of cost. It is a benefit that most of the other solvents do not have. There are many types of ILs that are non-toxic and comparatively less harmful to the environment than many other solvents (Shamsuri, 2011).

2.6. Dissolution Mechanisms of Polymers in Ionic Liquids

In the context of fabric fibers ILs have been used to dissolve natural fibers natural fiber dissolution mechanism has been reported in dissolution of cellulose. According to the mechanism suggested by Zhang(2009) it has been described that both ionic cation and anion get involved in the process of dissolution. Figure 4 shows the dissolution mechanism, using AMIMCl IL (Senthilkumar & Anbumani, 2010).

According to the proposed mechanism, when the liquid reaches the critical temperature at which dissolution starts, anion and the cation dissolves into AMIM⁺ and Cl⁻ ions. In the OH group of the cellulose molecule Oxygen and Hydrogen atoms start to form donor and acceptor electron complexes with positive and negative dissociated parts of the IL molecules. The Cl⁻ ion bonds with the proton of OH group and the AMIM⁺ ion bonds with oxygen in OH. It will disrupt the hydrogen bond of the cellulose. It has been found that mostly this happens C-3 OH and C-6 OH groups of nearby cellulose molecules (De Silva et al., 2014).

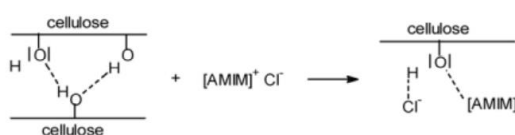


Figure 8 Dissolution Mechanism of Cellulose in AMIMCl (Zhang et al. , 1988)

2.7. Green Solvents used in Recycling of blended fabrics

There have been studies done on recycling Nylon/Cotton blended fabrics which is a commonly used fabric blend in textile industry. The volumes of this blend have significantly increased over the years and so are the wastes piling up. Although the general practice is to landfill or incinerate that are not only destroying the environment but also cause loss of valuable resources which can be improved if recovered as individual components (Wang et al., 2009). If these fabrics were in the form of single component fabrics the recycling and recovery methods to be followed will vary and will be easy to manage. That is one of the main reasons why the blended fabrics end up in landfills or incinerators without taking efforts to recover. In recent times

successful efforts have been made to explore economical and environmentally friendly methods to recycle cotton including converting into cellulose derivatives and energy recovery. Extracting microcrystalline cellulose from cotton wastes is another way of recovery. Since most of the wasted fabrics are blended fabrics this area of research has become an area of interest among many scholars focusing on textile waste management. Cotton fabrics are used as a component of reinforcement in composite materials due to the special physical and chemical properties it has (IV, 2015). When considering blends with Nylon mostly used methods are focused on dissolving or degradation. Hydrochloric acids, phenol-based solvents and aliphatic alcohol-based solvents are among main types of solvents used for dissolution. To achieve dissolution specific temperature pressure combinations, need to be used (De Silva et al., 2014). Huge volumes of solvents are consumed in these and required time is high. Since there are constraints of associated costs for pre preparation, reacting and purifying, depolymerization is least preferred in recycling of Nylon fiber. The fiber itself is expensive than polyester. Therefore, recycling methods of blends containing Nylon are focused to recover the Nylon fiber in pure form. Therefore, methods have been developed to recover Nylon. However, not most of these methods are simple and commercially viable which again pushes the industry to move ahead with landfilling as the preferred option (IV, 2015).

However, ionic liquids that are well tuned to fit the applications are used for recycling blends of biopolymers such as cellulose. In a study done by Wang et al. (2015) using AMIMCl ionic liquid separation of Nylon/cotton blend has been achieved by reacting at a temperature of 12°C. This has been followed by subsequent filtration separation. They have investigated the combinations of reacting temperatures and times in order to identify the optimum conditions in which separation can be achieved. They have confirmed that there is no effect on the physical properties of Nylon fibers and no other chemical reaction than breakage of hydrogen bonding. (IV, 2015)

2.8. Dissolution of Cellulose with Ionic Liquids

Cellulose is considered as one of the most widely used bio renewable material. Not only in the textile and clothing industry but also in other industries including paper,

polymer and paint industries. It is a well-known fact that cellulose does not dissolve in water or most of the organic liquids (Yin et al., 2013). There are different types of modified cellulose developed but the methods used for processing are not considered as green methods. Viscose Rayon yarn which is commonly used in textile industry is produced using cellulose which included processing using many chemicals and the method is not environmentally friendly. There have been studies focused on dissolving cellulose and its compounds using different types of ILs to make processes greener (De Silva et al., 2014). There are studies extended to understand methods of dissolution efficiency. A study has been done using BMIM⁺ as the cation with combinations of a variety of anions including Cl⁻, [PF₆]⁻, Br⁻, SCN⁻ and [BF₄]⁻. Cl⁻ can be considered as the smallest anion used and large non-coordinating anions including [PF₆]⁻. Butyl part of the cation has been replaced by an Octyl component as well and conducted studies (Sandin & Peters, 2018). Cellulose has been used in forms of pulps, fibers and papers. It has been identified that there is hardly any dissolution at ambient temperature, but the reaction has started when the mixture was heated up to around 110 °C. It has been summarized in the above study that dissolution happens as the intra molecular hydrogen bonding network is disrupted and broken. It has been found that when Cl⁻ is used as the cation the effectiveness of dissolution is high compared to other anions as it breaks the extensive hydrogen bonding network. This results in a reduced dissolution time than with other anions. It has been concluded that when the cation is made octyl also the dissolution efficiency is reduced as the concentration of effective chloride is reduced. Another finding that they have made is that the dissolution efficiency reduces when water is present in the IL. It has been identified that hydrogen bonding property of the solvent is an important parameter in dissolving cellulose. It has been concluded that ILs having anions that are strong hydrogen bond acceptors are more effective than the others (Mir et al., 2021).

2.9. Recycling of Polyester Blends

PET based fibers are considered as the largest single type of fiber used in the global textile manufacturing. However, the efforts taken to recycle are at a minimum and it has resulted in the amount of fabric wastes landfilled to be increased uncontrollably. The main reason for this is that PET is not a readily biodegradable type of yarn. In

many of the scenarios of textile waste recycling dyes that are present play a major role as they cause to further reduce ability to recover (Wang et al., 2009). As manmade fiber manufacturing was picking up towards the end of the decade of 2000 the larger portion which was previously dominated by cotton was converted to PET. Not only in apparel products but also in automotive and furnishing applications PET became a dominant player replacing most of the combinations of natural fibers especially due to its durability. PET is a semi crystalline thermoplastic substrate. It has a high resistance to biological and atmospheric components due to low gas permeability and high strength (Mir et al., 2021).

In 2015 the total output of PET products globally has summed up to 269 MMT which has gradually increased and in 2017 China alone has recorded 75.2 MMT of waste. Similar to many of other non-degradable solid waste the readily available solution for PET is also to end up in landfills (Yin et al., 2013).

There have been studies done on environmental impact of polyester recycling process and the findings show that the available recycling methods themselves are causing global warming. Furthermore, toxic compounds are released that are carcinogenic, harmful effects on water resources and toxic to eco systems as well (Wang et al., 2022).

A study done by (Wang et al., 2009) revealed that polyethylene terephthalate can be degraded using ionic liquids. They have been able to separate the products depending on the solubility in water. they have further investigated the impact of the time of reaction, reaction temperature, impacts of addition of catalysts to expedite the dissolution process. In another study it has been found that ionic liquids that are used for this type of applications can be used again after recycling and it has been found that solubility of PET in recycled solutions of ionic liquids is even better than in the fresh one. They have concluded that the reason for improvement of solubility could be due to availability of small amounts of water in the recycled IL. Moreover, they have suggested a degradation mechanism for PET in BMIMCl and reaction kinetics also have been investigated (Arrosyad et al., 2020).

Among the methods of recycling of PET chemical recycling is the one with the highest interest. It is achieved through depolymerization. The commonly used methods are methanolysis, glycolysis and hydrolysis through which acidic or basic conditions are promoted to achieve depolymerization. The associated disadvantages of methanolysis are involvement of methanol volatilization and associated high temperature and pressure. Purity of the recycled products of Glycolysis is less. Since there are disadvantages like these there is an opportunity for a green solvent based solution to be competitive with available methods. Cellulose has been the most commonly tested polymer with ionic liquids. It has been found that Ionic liquids with Chloroaluminate (III) could be used in degradation of PET (Sandin & Peters, 2018). The selection of ionic liquids for the study has been done based on previous studies done using PE and Cellulose. The study has concluded that PET dissolves readily in BMIMClAlCl₄, there is a solubility of 2.7% in BMIMCl at 180°C but did not dissolve in ionic liquids that contained BF₄⁻ and PF₆⁻. They have used PET in pellet form for the study and the reaction temperature have been greater than 120°C whereas in Cellulose the dissolution occurs at around 110°C. They have been able to filter the product and recycle the ionic liquid and found out that solubility is high in recycled IL than the original one (Wang et al., 2009).

2.10. Summary

Blended fabrics are widely used in the textile and apparel industry to enhance properties of fabrics. When fabrics are blended with spandex, they get superior performance. However, recycling of blended fabrics is a challenge. There are methods available but most of them are not cost effective and sustainable.

There is a requirement to find green solutions for recycling of blended fabrics. Green solvents can be considered as a solution to issues with recycling of spandex blended fabrics as the dissolution happens at lower temperatures, solvents are non-toxic and can be regenerated.

Since polyester is among the yarn types that do not get dissolved in many of the substances and since there are successful dissolution experiments of PET being

dissolved in green solvents, it is highly likely that similar types of ionic liquids can have positive results on spandex-polyester blends.

Even though there is hardly any study focused on cotton-spandex blended fabric dissolution using ionic liquids it can be predicted that ionic liquids that work for cellulose to work to dissolve cotton in polyester- spandex blends as potentially possible.

3. METHODS AND MATERIALS

3.1 Selection of Green Solvents

Based on the available literature following liquids were shortlisted as potential solvents for experiments. The candidate solvents are third generation ionic liquids.

BMIMCl- 1-Butyl-3-methylimidazolium chloride

BMIMA₄Cl₄ - 1-Butyl-3-Methylimidazolium Tetra chloroaluminate

AMIMCl - 1-Allyl-3-methylimidazolium chloride

BMIMBr - 1-Butyl-3-methylimidazolium bromide

BMIMCH₃COO - 1-butyl-3-methylimidazolium

EMIMBr - 1-Ethyl-3-methylimidazolium bromide

BMIMCF₃COO - N-Methylaniline trifluoroacetate

AMIMCl and BMIMCl were selected as the ionic liquids to be used for the experiments depending on literature and due to practicality as the other shortlisted chemicals were not available to an acceptable price and delivery times did not match with availability of other resources for the experiments. Both the ILs were purchased from Zichang New Materials (Shandong) Co., Ltd. China. Purchasing details and MSDS of both the materials are included in Appendix 1.

Figure 9 shows the chemical structures of the two ionic liquids.

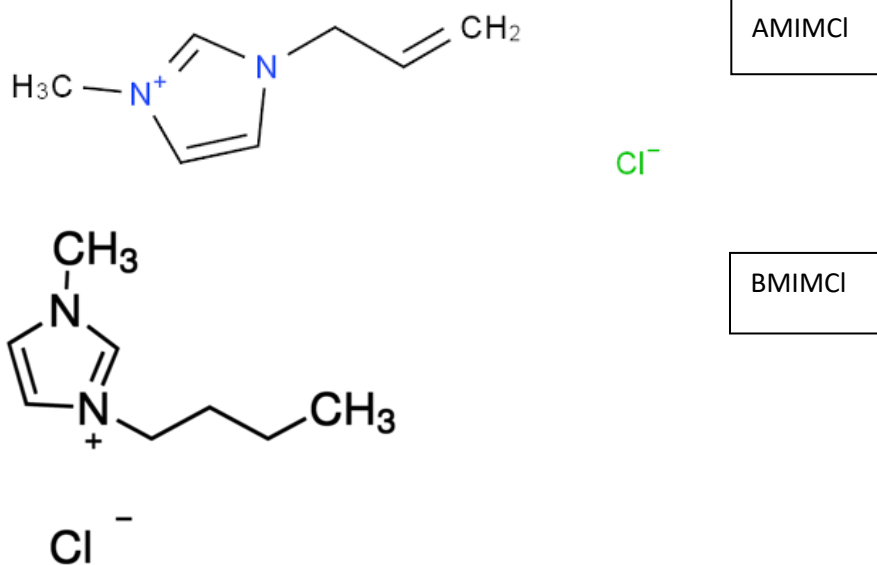


Figure 9: Chemical Structures of AMIMCl and BMIMCl

3.2 Material Types for Experiments

Table 1: Material Details for Testing

Materials in pure yarn form	Yarn 1 (Y1)	100% Polyester yarn
	Yarn 2 (Y2)	100% Cotton yarn
	Yarn 3 (Y3)	100% Spandex yarn
Materials in pure fabric form	Fabric 1 (F1)	100% Polyester fabric
	Fabric 2 (F2)	100% Cotton fabric
Materials in blended fabric form	Fabric 3 (F3)	91% cotton + 9% spandex fabric
	Fabric 4 (F4)	70% polyester + 30% spandex fabric

Table 1 summarizes the fabric details. All the fabric qualities were received from Hayleys Fabric Plc (Sri Lanka). Spandex, cotton and polyester yarns were also obtained from the same company to pre-test the ionic liquids.

Spandex alone is difficult to be knitted to a fabric structure. Therefore, it was used only in yarn form. Spandex yarns were treated in higher temperature conditions to make sure that the ionic liquids dissolve cotton and polyester selectively.

3.3 Experimental Process



3.3 Sample Combinations that need to be tested with Temperatures.

Based on the literature, temperature ranges were selected for the experiments. Temperature range for cotton blended fabrics will be 80°C – 120°C and for the polyester blends 120°C- 180°C range was selected. Spandex will be tested in yarn form in 120°C-180°C temperature range as a control sample to evaluate if any chemical/ physical change occurs in spandex.

Table 2: Sample Combinations to be Tested

Temperature	Fabric	Ionic Liquid
80°C	F2	IL1
	F3	IL1
	F3	IL1
80°C	F2	IL2
	Y3	IL2
	F	IL2
120°C	F2	IL1
	Y3	IL1

	F3	IL1
120°C	F2	IL2
	Y3	IL2
	F3	IL2
160°C	F1	IL1
	Y3	IL1
	F4	IL1
160°C	F1	IL2
	Y3	IL2
	F4	IL2
180°C	F1	IL1
	Y3	IL1
	F4	IL1
180°C	F1	IL2
	Y3	IL2
	F4	IL2

3.4 Experimental Apparatus

Dissolution was done under atmospheric pressure using a silicone bath with a magnetic stirrer. It uses a stainless-steel bath for silicone. The ionic liquid and fabric pieces are in a round bottom flask of 200 ml.



Figure 10: Experimental Apparatus

3.5 SEM Images

The morphology of samples was observed using Zeiss Supra 55VP scanning electron microscope (SEM) at an accelerating voltage of 5.00 kV.

3.6 Fourier transform infrared spectroscopy (FTIR)

FTIR spectra of the regenerated films were measured on a Bruker LUMOS FTIR microscope in ATR mode. Firstly, samples were scanned in the frequency range of 600 to 4000 cm^{-1} at the scan resolution of 4 cm^{-1} .

4. RESULTS

Table 3 summarizes the sample combinations. As the first step to get an understanding on the melting temperatures and functioning of solvents following tests were done and the results were obtained. The tests were done keeping the RPM of the magnetic stirrer a constant value of 400 RPM. Yarn pieces of the same size were taken and shredded. Mixing time was kept a constant at 4 hours. Each testing was done using 50 ml of Ionic liquid. The melting temperatures of the solvents matched with material data sheets.

Table 3: Observations of Experiments

Yarn type	Type of IL	Temperature (°C)	Observation
Pure cotton	AMIMCl	80	almost fully dissolved
Pure Polyester	AMIMCl	160	No change
Pure spandex	AMIMCl	180<	No change
Pure cotton	BMIMCl	80	almost fully dissolved
Pure Polyester	BMIMCl	160	No change
Pure spandex	BMIMCl	180<	No change

After that, dissolving testes were carried out for fabric types under the same conditions as above. 10 cm x 10 cm fabric swatches were taken and shredded. The results and observations are listed in Table 3. The fabric swatches were oven dried at a temperature of 120 (°C) for 24 hours to make sure the swatches are free from moisture. After that they were kept inside a desiccator.

The initial weights of the panels were taken using a digital scale. Dissolving was carried out using a silicone bath kept on a magnetic stirrer as per figure 10. Temperature was maintained constant as per Table 4 and testing was done under

atmospheric pressure. Mixing was done with a RPM of 400 and stirring was continued for 4 hours for each of the samples.

Then the residuals were separated using a strainer and washed with excess water. Panels were kept for drying. After that, the weight was measured.

Table 4: Observations in Detail

Fabric Type	Type of IL	Temperature (°C)	Initial Weight	Final Weight	Observations
100% Cotton	AMIMCl	80	2g	2.4g	Fabric pieces were hardened
100% Cotton	AMIMCl	120	2g	3g	Fabric pieces were hardened
100% Cotton	BMIMCl	80	2g	2.5g	Fabric pieces were hardened
100% Cotton	BMIMCl	120	2g	3g	Fabric pieces were hardened
100% Polyester	AMIMCl	160	1.6g	1.6g	No visible change
100% Polyester	AMIMCl	180	1.6g	1.6g	No visible change
100% Polyester	BMIMCl	160	1.5g	1.6g	No visible change
100% Polyester	BMIMCl	180	1.6g	1.6g	No visible change
91% cotton + 9% spandex	AMIMCl	80	1.8g	2.1g	Fabric pieces were hardened
91% cotton + 9% spandex	AMIMCl	120	1.6g	1.8g	Fabric pieces were hardened
91% cotton + 9% spandex	BMIMCl	80	1.6g	1.9g	Fabric pieces were hardened

91% cotton + 9% spandex	BMIMCl	120	1.8g	2.1g	Fabric pieces were hardened
70% polyester + 30% spandex	AMIMCl	80	1.8g	2.1g	No visible change
70% polyester + 30% spandex	AMIMCl	120	1.6g	1.8g	No visible change
70% polyester + 30% spandex	BMIMCl	80	1.6g	1.9g	No visible change
70% polyester + 30% spandex	BMIMCl	120	1.8g	2.1g	No visible change

Additional Results

- Experiments were carried out with increased temperature and increased stirring time for polyester as no change was observed. There was no change in weight.
- An experiment was carried out by increasing the pressure for polyester samples and no change was observed.
- The testing apparatus was placed in an ultrasound environment and tested. There was no change observed.

1.1 Fourier Transform Infrared Spectroscopic (FTIR) Analysis

FTIR is a widely used technique to analyze the structural composition and the functional groups in samples. In the current study FTIR was used as a mean to evaluate the results of the original polyester fabric (Figure 11) and the ILs treated blended fabrics with different temperatures (Figures 12 - 15). All the IR spectra appeared in the range of 500 - 4000 cm^{-1} .

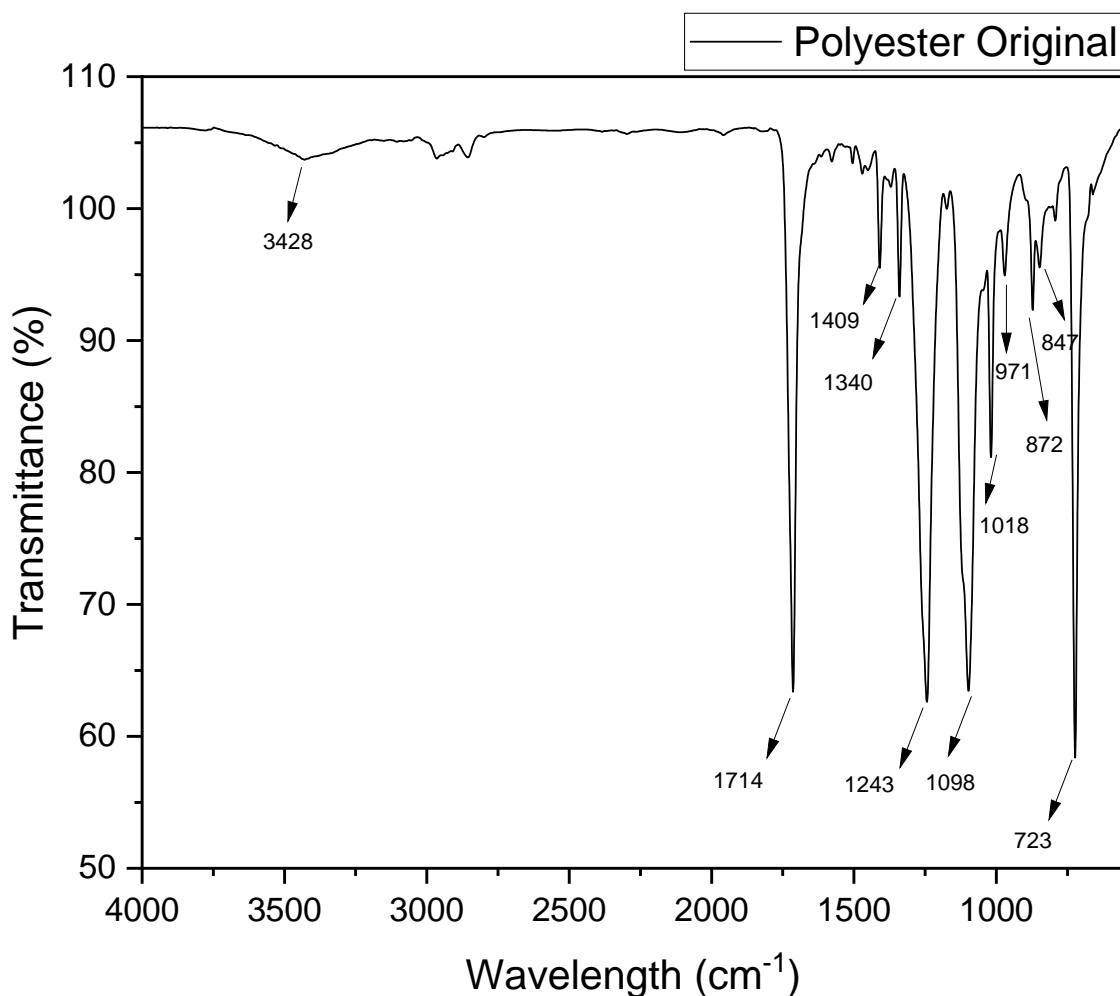


Figure 11: FTIR Spectrum of Original Polyester

The FT-IR spectrum of the original polyester fabric showed characteristic absorption peaks in 1714, 1409, 1340, 1018, 971, 872 cm^{-1} , which were compatible with the literature (Portella et al., 2016). The strong peak appeared at 1714 cm^{-1} represents the C=O vibration, assigned to the ester group. The peak at 1409 cm^{-1} , which is a characteristic absorption peak of polyester, indicates the presence of aromatic ring which is a stable group. The peaks at 1340 and 1018 cm^{-1} shows the carboxylic ester or anhydride groups whereas the 1018 cm^{-1} specifies the presence of C-O-C=O or secondary alcohol. The peak at 971 cm^{-1} is of C=C stretching amidst the five substituted H in benzene is denoted by the peak at 872 cm^{-1} .

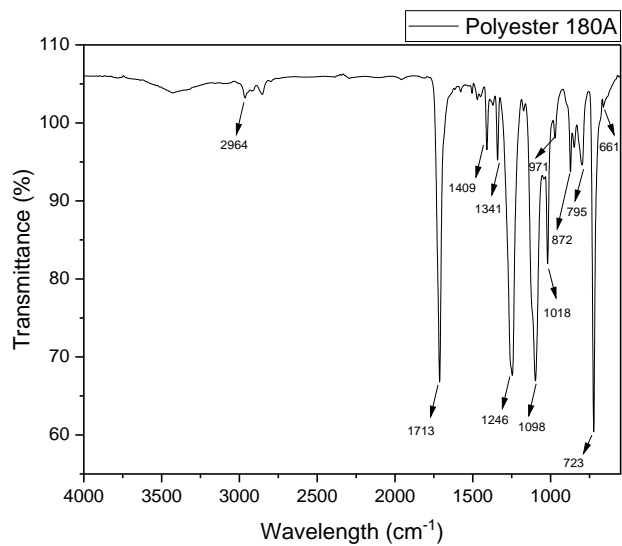


Figure 12: FTIR Spectrum of AMIMCl Treated Polyester at 180 °C

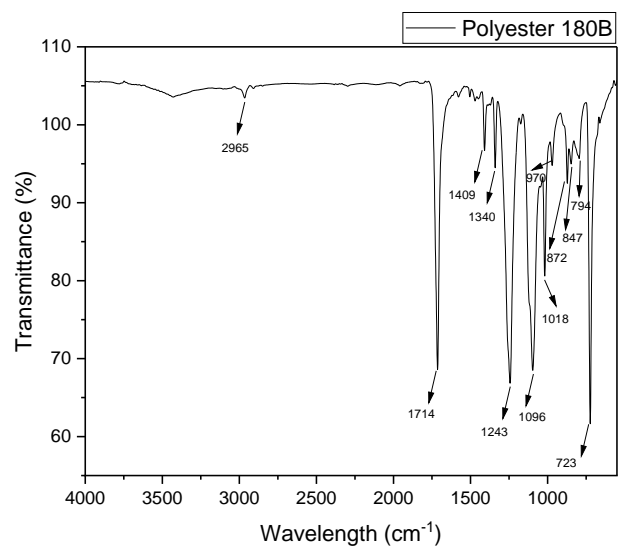


Figure 13: FTIR Spectrum of BMIMCl Treated Polyester at 180 °C

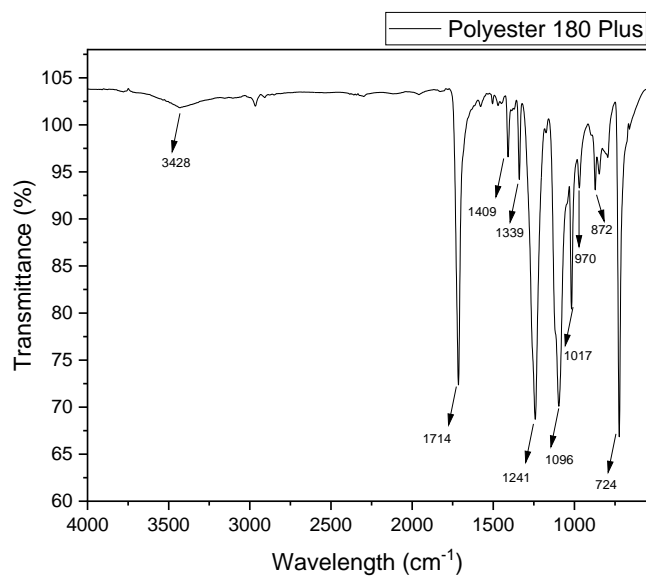


Figure 14: FTIR Spectrum of AMIMCl Treated Polyester above 180 °C

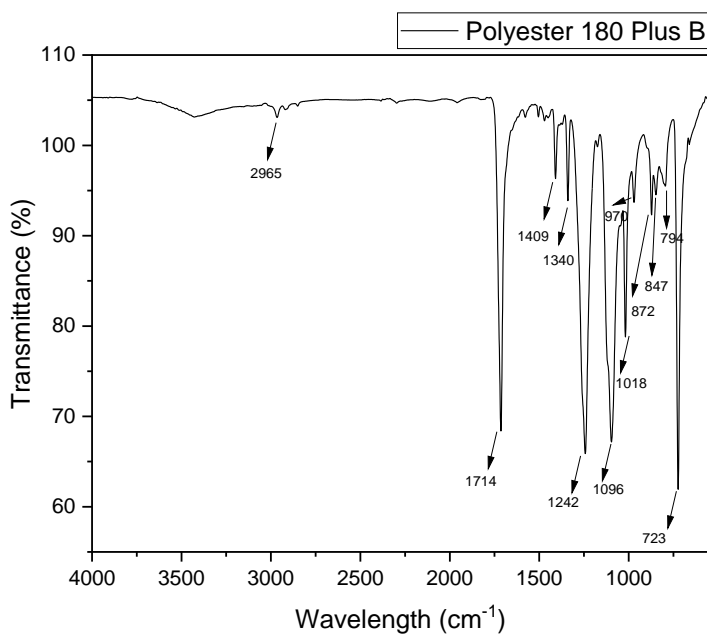


Figure 15: FTIR Spectrum of BMIMCl Treated Polyester above 180 °C

When considering the FT-IR spectra of AMIMCl and BMIMCl treated polyester fabrics at 180 °C temperatures (Figures 12-15), they all resemble a common spectrum in appearance with almost same peak positions and intensities.

All the above spectra of IL treated polyester fabrics revealed the presence of absorption peaks at specific wavenumbers: 1714, 1409, 1340, 1018, 971, and 872 cm^{-1} , which are compatible with the original non-treated polyester. Accordingly, the prominent peak around 1714 cm^{-1} corresponds to the vibration of the C=O bond, indicating the presence of the ester group. At 1409 cm^{-1} , the appearance of the characteristic absorption peak for polyester, confirms the presence of an aromatic ring, which is the stable functional group. The peaks around 1340 and 1018 cm^{-1} indicate the presence of carboxylic ester or anhydride groups, while the 1018 cm^{-1} peak specifically signifies the existence of C-O-C=O or secondary alcohol. Moreover, the peak at 971 cm^{-1} corresponds to the stretching of the C=C bond. Furthermore, the five substituted hydrogen atoms in benzene are represented by the peak at 872 cm^{-1} .

Since the FT-IR spectra of the original and the IL treated polyester fabrics are practically identical, it can be assumed that no changes have occurred in the functional groups and in the chemical structure of the fabric samples treated.

These results suggest that the IL treatment on polyester at higher temperatures has not caused a significant impact on the polyester fabric. Finally, it can be assumed that the absence of new peaks and the non-disappearance of existing peaks at IR spectra indicate the polyester dissolution in ILs is a kind of physical dissolution process (Tomczak et al., 2007).

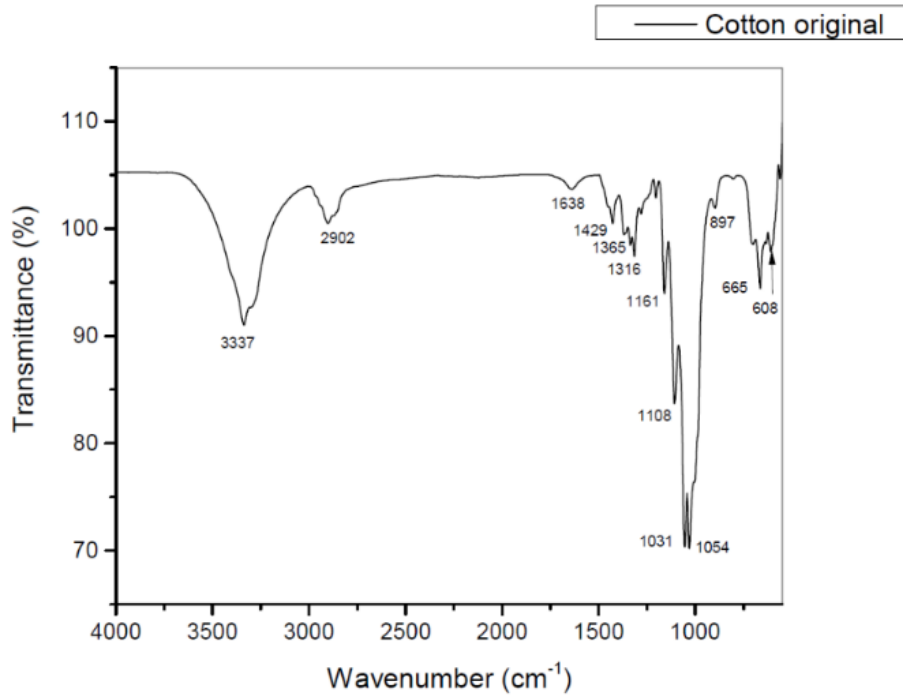


Figure 16: FTIR Spectrum of Original Cotton

The FT-IR spectrum of the original polyester fabric is shown in Figure 11. There are characteristic absorption peaks in 3330, 2896, 1622, 1428, 1360, 1315, 1032, and 894 cm^{-1} , which were compatible with the literature. The strong peak observed at 3330 cm^{-1} is characteristic of the hydroxyl (OH) groups of cellulose, lignin, and water (Tomczak et al., 2007). The peak at 2902 cm^{-1} can be the characteristic peak of the stretching vibration of C-H present in cellulose and hemicellulose (Senthilkumar & Anbumani, 2010). The band at 1638 cm^{-1} may be related to the presence of water in the fibers (De Rosa et al., 2010). The absence of a peak at 1730 cm^{-1} which is a characteristic of the carboxyl group of hemicellulose, shows the low content of hemicellulose in cotton. The absorption band at 1429 cm^{-1} is associated with the CH_2 symmetric bending of the cellulose. The absorption bands at 1365 and 1316 cm^{-1} are relative to bending vibrations of the C-H and C-O groups, respectively, of the aromatic rings in cellulose polysaccharides. Intense peak vibrations observed at 1031 cm^{-1} are related to the (CO) and (OH).

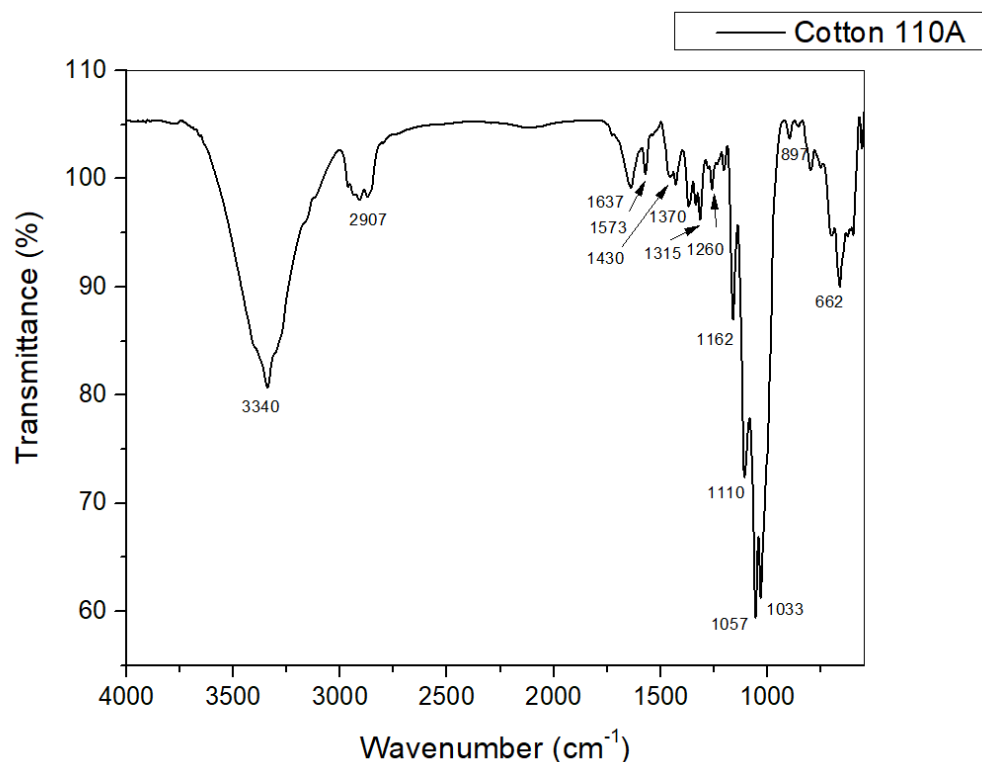


Figure 17: FTIR Spectrum of AMIMCl treated Cotton at 110°C

stretching vibrations of the polysaccharide in cellulose. The presence of β -glycosidic linkages between monosaccharides is indicated by the peak at 897 cm^{-1} (Romanzini et al., 2012).

When considering the FTIR spectrum of AMIMCl-treated cotton fabrics at $110\text{ }^{\circ}\text{C}$ temperatures (Figure 17), it resembles a common spectrum in appearance with almost the same peak positions and intensities (Portella et al., 2016). The strong peak observed at 3340 cm^{-1} is characteristic of the hydroxyl (OH) groups of cellulose, lignin, and water (Rashmi Nayak et al., 2022). The characteristic peak of the stretching vibration of C-H present in cellulose and hemicellulose can be observed at 2907 cm^{-1} (Senthilkumar & Anbumani, 2010). The band at 1637 cm^{-1} may indicate the presence of water in the fibers (Senthilkumar & Anbumani, 2010). The absence of a peak at 1730 cm^{-1} indicates the low content of hemicellulose in cotton. The CH_2 symmetric bending of cellulose is associated with the absorption band at 1430 cm^{-1} . Bending vibrations of the C-H and C-O groups, respectively, of the aromatic rings in cellulose polysaccharides are relative to the absorption bands at 1370 and 1315 cm^{-1} . The intense peak vibrations at 1033 cm^{-1} are related to the (CO) and (OH) stretching vibrations of

the polysaccharide in cellulose. A peak at 897 cm^{-1} suggests the existence of β -glycosidic linkages between monosaccharides (Tomczak et al., 2007).

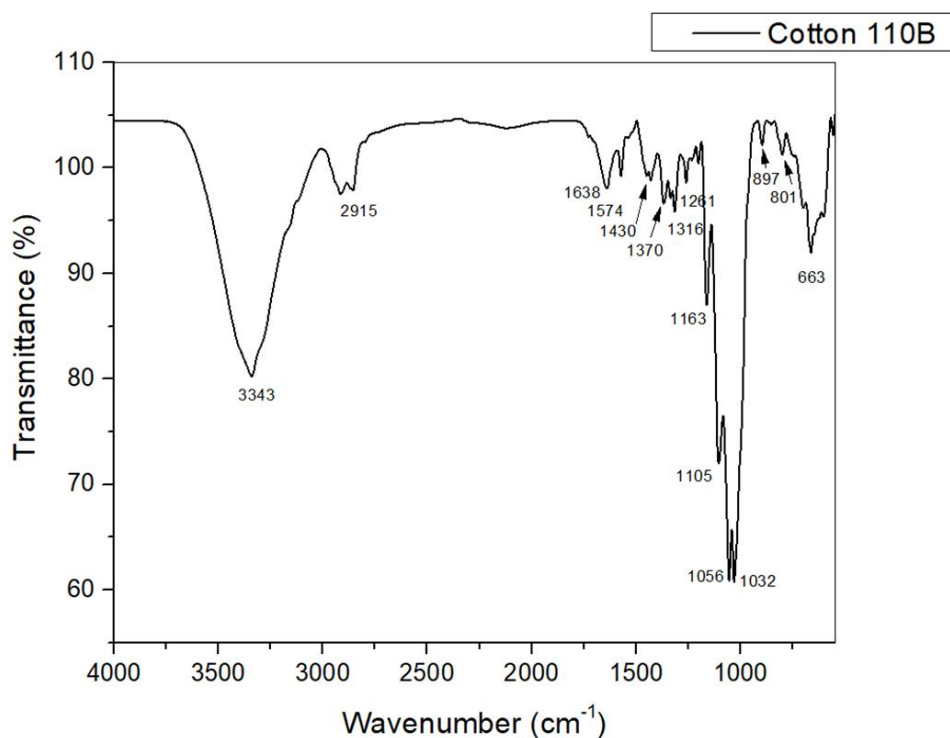


Figure 18: FTIR Spectrum of BMIMCI treated Cotton at $110\text{ }^{\circ}\text{C}$

When considering the FTIR spectrum of BMIMCI-treated cotton fabrics at $110\text{ }^{\circ}\text{C}$ temperatures (Figure 18), it resembles a common spectrum in appearance with almost the same peak positions and intensities (Portella et al., 2016). The strong peak observed at 3343 cm^{-1} is characteristic of the hydroxyl (OH) groups of cellulose, lignin, and water. At 2915 cm^{-1} , it can be observed the characteristic peak of the stretching vibration of C-H present in cellulose and hemicellulose.³ The presence of water in the fibers may be related to the band at 1638 cm^{-1} (Rashmi Nayak et al., 2022). A peak at 1730 cm^{-1} is absent, which is characteristic of the carboxyl group of hemicellulose in cotton. The absorption band at 1430 cm^{-1} is linked to the CH_2 symmetric bending of cellulose. The absorption bands at 1370 and 1316 cm^{-1} correspond to bending

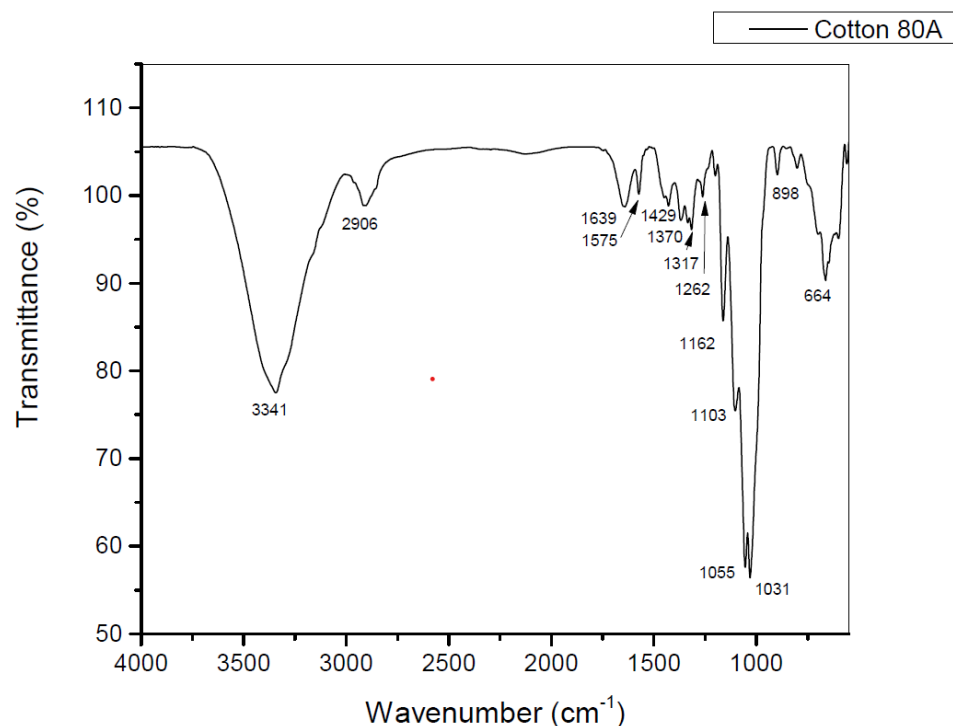


Figure 19: FTIR Spectrum of AMIMCI treated Cotton at 80 °C

vibrations of the C-H and C-O groups, respectively, of the aromatic rings in cellulose polysaccharides. Intense peak vibrations at 1032 cm^{-1} are associated with (CO) and (OH) stretching vibrations of cellulose polysaccharides. The existence of β -glycosidic linkages between monosaccharides is denoted by a peak at 897 cm^{-1} (Tomczak et al., 2007).

When considering the FTIR spectrum of AMIMCI-treated cotton fabrics at 80 °C temperatures (Figure 19), it resembles a common spectrum in appearance with almost the same peak positions and intensities. The strong peak observed at 3341 cm^{-1} is characteristic of the hydroxyl (OH) groups of cellulose, lignin, and water (Portella et al., 2016). The stretching vibration of C-H present in cellulose and hemicellulose has a characteristic peak at 2906 cm^{-1} (Romanzini et al., 2012). The band at 1639 cm^{-1} could be an indication of water present in the fibers (Senthilkumar & Anbumani, 2010). A peak is not present at 1730 cm^{-1} , which is a characteristic feature of the carboxyl group of hemicellulose in cotton. The CH_2 symmetric bending of cellulose corresponds to the absorption band at 1429 cm^{-1} . Bending vibrations of C-H and C-O groups in aromatic rings of cellulose polysaccharides are relative to absorption bands

at 1370 and 1317 cm^{-1} , respectively. Intense peak vibrations at 1031 cm^{-1} are due to

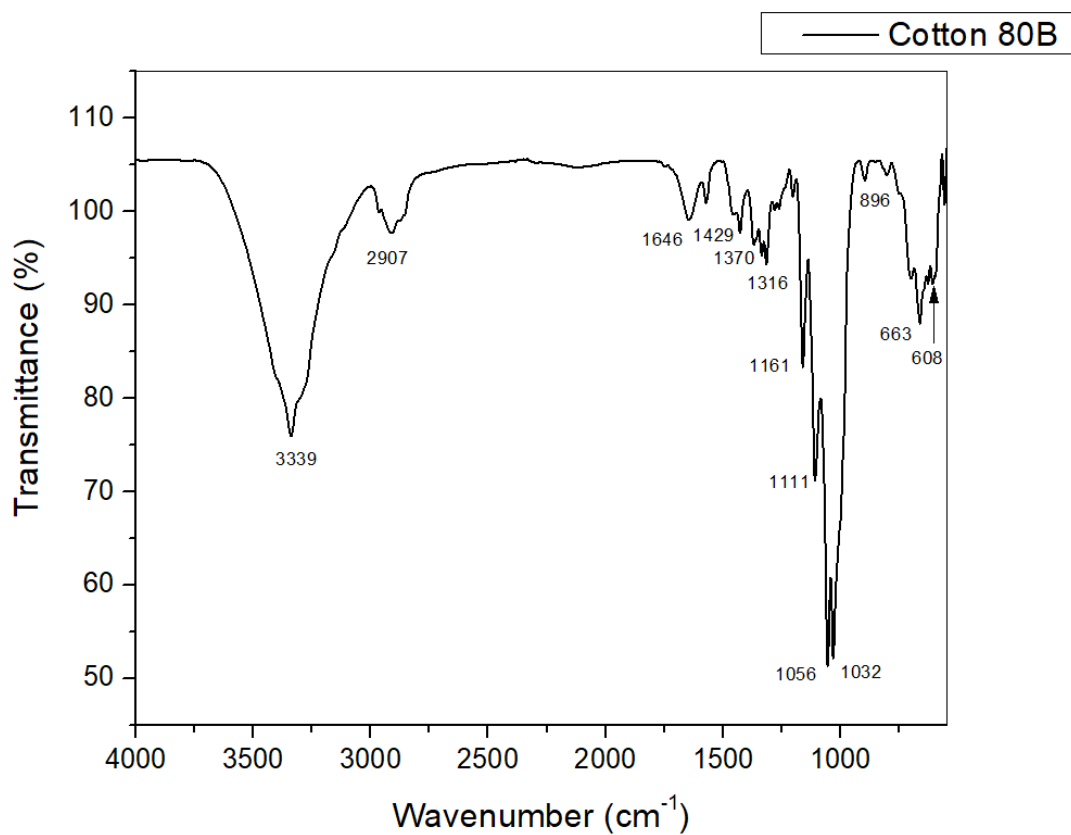


Figure 20: FTIR Spectrum of Cotton 80 °C BMIMCI

(CO) and (OH) stretching vibrations in cellulose polysaccharides. β -glycosidic linkages between monosaccharides can be inferred from the peak at 898 cm^{-1} (Tomczak et al., 2007).

When considering the FTIR spectrum of BMIMCI-treated cotton fabrics at 80 °C temperatures (Figure 20), it resembles a common spectrum in appearance with almost the same peak positions and intensities. The strong peak observed at 3341 cm^{-1} is characteristic of the hydroxyl (OH) groups of cellulose, lignin, and water. A characteristic peak can be observed at 2907 cm^{-1} for the stretching vibration of C-H present in cellulose and hemicellulose (Senthilkumar & Anbumani, 2010). Water present in fibers may be indicated by a band at 1646 cm^{-1} . The absence of the peak at 1730 cm^{-1} shows the low content of hemicellulose in cotton. The absorption band at 1429 cm^{-1} corresponds to CH₂ symmetric bending in cellulose. Absorption bands at

1370 and 1316 cm^{-1} are relative to bending vibrations for C-H and C-O groups, respectively, within aromatic rings found in cellulose polysaccharides. Intense peak vibrations observed at 1032 cm^{-1} correspond to (CO) and (OH) stretching vibrations within cellulose polysaccharides. The peak at 896 cm^{-1} is a sign of the presence of β -glycosidic linkages between monosaccharides (Tomczak et al., 2007).

Since the FTIR spectra of the original and the IL-treated cotton fabrics are practically identical, it can be assumed that no changes have occurred in the functional groups and in the chemical structure of the fabric samples treated.

MICROSCOPIC IMAGES

Images were taken for polyester-spandex sample to further understand on any visible change in the structure. There was no significant change observed.

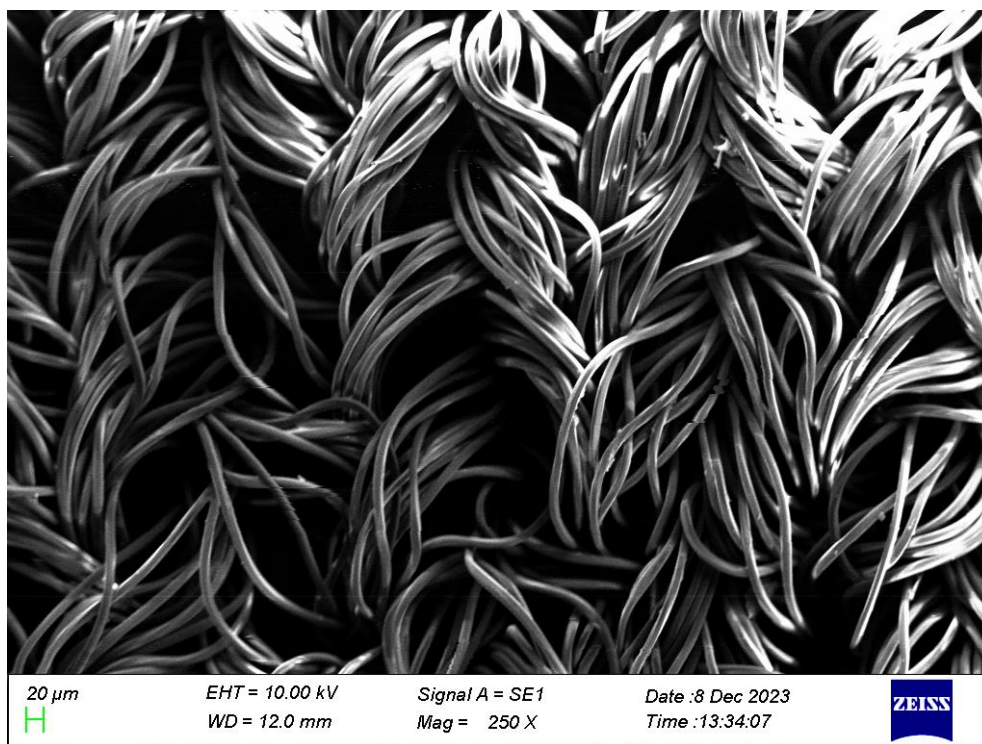


Figure 21: Surface Structure of the Treated Polyester-Spandex Sample

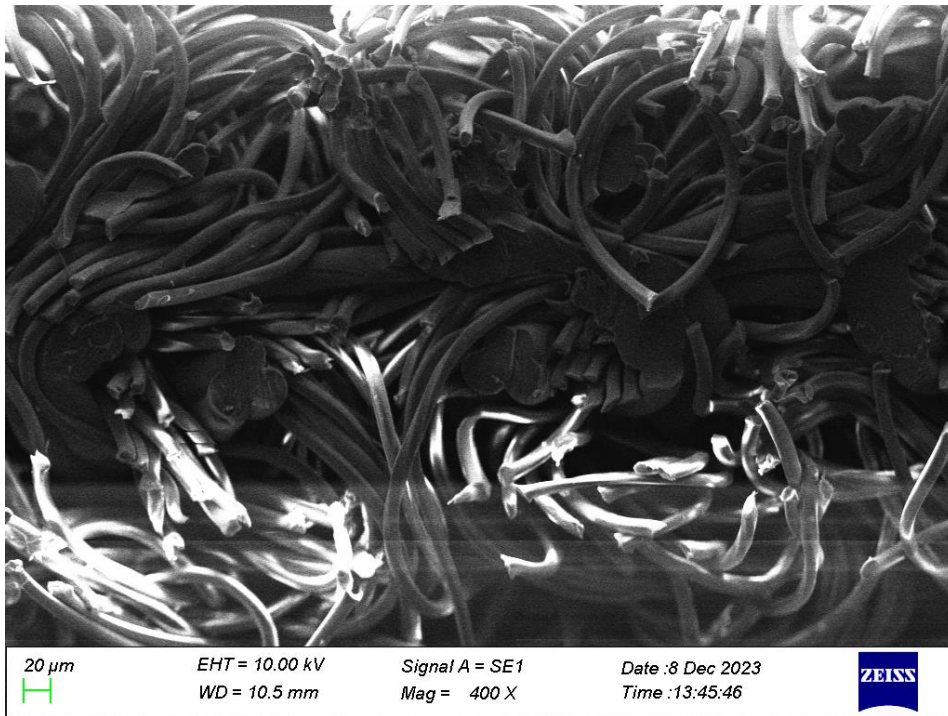


Figure 22: Cross Section of the Treated Polyester Sample

5. DISCUSSION

As discussed at the introduction the problem of blended textiles-especially spandex based blended textiles- is an alarming issue in both global and Sri Lankan contexts. Since the organizations struggle with the issue of finding a balance between profitability and sustainability in their operations they choose to move ahead with incineration or landfilling. There was a need for a solution as the problem remained. As discussed in earlier chapters there is very limited number of literature available which were focused on finding a solution through chemical recycling. This research looked at an innovative approach by using ionic liquids to enable chemical recycling of the waste blended textiles.

The study and exploration of available dissolution mechanisms led to the shortlisting of seven ionic liquids. Within Sri Lanka there was no availability of any of these chemicals even as imported chemicals. This led to scoping of suppliers, and which eventually led to the narrowing down to a supplier from Zichang New Materials (shandong) Co., Ltd. China. which was selected based primarily on cost but also on the ability to supply within a reasonable time frame. The commercial grade chemical selection was based on the fact that, for any large-scale implementation the use of analytical grade would never be practical. Hence from the outset a practical route was experimented upon.

A limited number of experimental iterations were possible with the chemicals received and the initial experiments immediately demonstrated though selected based on the limited knowledge of dissolution capacity that the chemicals were not able to deliver on supporting the chemical recycling approach. As there was an inability to compare ionic liquids from a number of suppliers and having to rely on the specifications indicated by the supplier there still may be some more experiments to be carried out in really excluding the potential of these two ionic liquids.

Wang (2009) indicated PET dissolution using the BMIMCl ionic liquid. The temperature range used in that study had been 160°C – 200°C. The experimental methodology in the current study also followed the same temperature settings with the variation. The blended matrix nor the pure fabric did not yield the intended result. The observation was completely opposite to what was expected. This indicates that there is the inapplicability of the same dissolution mechanism provided the chemical purity is valid and meets the stated specifications.

Usage of AMIMCl and BMIMCl for cellulose has been done in a study by De. Silva (2014). Dissolution has been achieved in between 80°C-120°C which was followed by the current study. Cotton yarn got dissolved giving similar results. A dissolution did not occur in fabric form. Fabric pieces got hardened once they were washed with excess water. Lindman et al. (2010) has explained that solubility of cellulose is complex to be explained. Cellulose do not get dissolved in many solvents due to strong intermolecular hydrogen bonding between cellulose molecules. They only get dissolved in solvents with intermediate properties, and ILs are one of them. In their explanation they explain about different states of cellulose. Some of them are amorphous and some of the states are crystalline. The hardening of the cotton fabric might be due to formation of one of these states. It needs to be further studied to find the actual reason.

Regeneration of Ionic Liquids

When considering both the environmental aspects and economic aspects it is important to recycle the ionic liquids used. According to Wang (2009) in case of PET pellets regeneration has been achieved by treating with excess water and filtration. It has been found that the solubility of PET in the regenerated solvent is better than the original solvent.

In the study of De Silva (2014) cellulose that has been dissolved using same ILs as the present study, has been separated using water as a coagulant. The ionic liquids have been recovered and tested for repeatability of the experiments.

Above methods indicate that if the blended fabrics can be dissolved using the ionic liquids there is a high possibility of regenerating the ionic liquids which makes usage of ionic solvents in recycling of blended fabrics an economical one.

Recommendations

A summary of the results is included in Table 5 to reflect on the results to make further recommendations.

Table 5: Summary of Results

Material Type	Solvents Used	Temperature Range Tested (°C)	Observations
Pure spandex yarn	AMIMCl/	120-180	No visible change
Pure cotton yarn	BMIMCl	80-120	Fully dissolved
Pure Polyester yarn		120- (180<)	No visible change
Pure cotton fabric		80-120	
Pure Polyester fabric		120- (180<)	No visible change
Cotton-spandex blended fabric		80-120	Hardened-Weight slightly increased
Polyester-spandex blended fabric		120- (180<)	No visible change

The current study has SEM observations for treated Polyester only due to facility constraints. It is recommended to carry out SEM on treated samples of cotton-spandex blended fabric to further observe any change in the structure which could give a direction for further treatments or change in conditions. In addition, observing any change in the structure of the treated materials through a polarized microscope can also be considered to investigate microscopic level changes.

Out of the ionic liquids shortlisted, current study only considered two types depending on pricing and import lead times. It is recommended to continue experiments using the other five shortlisted solvents.

Thermogravimetric analysis (TGA) is a powerful tool to measure thermal stability of polymers which has been successfully used by similar studies in literature. It is recommended that the current study can be further extended to perform a TGA to understand the level of solubility when temperature is increased.

Further, the current study can be extended to ultrasound coupled study to see the possibility of facilitating the reactions.

The study was exploratory in nature considering the challenging objective along with the primary goal. The final results points for more recommendations for future work than immediate results that one can deploy in order to address the problem.

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APPENDIX 1

AMIMCl and BMIMCl were received in solid state packed in 2 layers of polythene covers. Figure 23 shows the details of purchasing.

The screenshot displays a purchasing interface with two contact profiles and a product table. The first profile, 'ZICHANG CHEMICAL', lists contact name 'sweety yin', registered company address in Jinan, China, and contact details. The second profile, 'Silu', lists contact name 'Niroshinie Chathurika' and contact details in LK. The product table below lists two items, both with a quantity of 1.00 kg and a total price of USD 170.00. The first item is 1-Ethyl-3-methylimidazolium chloride (CAS 65039-09-0) and the second is 1-Butyl-3-methylimidazolium chloride (CAS 79917-90-1). Both items have a product description of '-'. At the bottom right, the total product quantity is 2.00 kg and the total price is USD 340.00. A messenger icon is visible in the bottom right corner.

Contact name: sweety yin
Registered company address: CN,Shandong,Jinan,Room A2-907, Xinyuanxin Center, Huaxin Road
Company tel: 086-0531-87565558
Company email: sweety@sdzcchemical.com#{251799927}#

Contact name: Niroshinie Chathurika
Registered company address: LK,
Company tel: -7417785
Company email: niroshinie@gmail.com

Product details

No.	Product name	Product image	Spec/Specs	Quantity	Unit	Unit price	Total
1	factory 1-Ethyl-3-methylimidazolium chloride CAS 65039-09-0		-	1.00	Kilograms	USD 170.0000	USD 170.00
Product Description: -							
2	factory supply 1-Butyl-3-methylimidazolium chloride CAS 79917-90-1		-	1.00	Kilograms	USD 170.0000	USD 170.00
Product Description: -							

Product Quantity 2.00 Total Price USD 340.00

Messenger

Figure 23: Details of Solvent Purchasing

Safety Data Sheet

1-Butyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 2/26/2017

1 IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND THE COMPANY/UNDERTAKING

Product name	1-Butyl-3-methylimidazolium chloride
Product code	IL-0014
CAS	79917-90-1
REACH No.	A registration number is not available for this substance as the substance or its uses are exempted from registration, the annual tonnage does not require a registration or the registration is envisaged for a later registration deadline.
Identified uses	Laboratory chemicals, Manufacture of substances
Supplier	IoLiTec Ionic Liquids Technologies GmbH Salzstrasse 184 D – 74076 Heilbronn Germany
Telephone	+49 (0)7131-89839-0
Fax	+49 (0)7131-89839-109
Emergency telephone	+49 (0)179-5322578
Email	msds@iolitec.de

2 HAZARDS IDENTIFICATION

2.1 Classification of the substance or mixture

Classification (REGULATION (EC) No 1272/2008)

Caution! Substance not yet fully tested. Risks cannot be excluded if the product is handled inappropriately. For laboratory use only!

Safety Data Sheet

1-Butyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 2/26/2017

Classification (67/548/EEC or 1999/45/EC)

Caution! Substance not yet fully tested. Risks cannot be excluded if the product is handled inappropriately. For laboratory use only!

2.2 Label elements

Labelling (REGULATION (EC) No 1272/2008)

Substance not yet fully tested.

Hazard statements

H-phrases

Substance not yet fully tested.

Precautionary statements

P phrases

P262:	Do not get in eyes, on skin, or on clothing
P280:	Wear protective gloves/ protective clothing/ eye protection/ face protection
P305 + P351 + P338:	IF IN EYES: Rinse continuously with water for several minutes. Remove contact lenses if present and easy to do – continue rinsing.
P313:	Get medical advice/attention

Labelling (67/548/EEC or 1999/45/EC)

Substance not yet fully tested.

Risk phrases

Not fully tested substance.



Safety Data Sheet

1-Butyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 2/26/2017

Safety phrases

S: 24/25 Avoid contact with skin and eyes.
S: 26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S: 36/37/39 Wear suitable protective clothing, gloves and eye/face protection.
S:28,2 After contact with skin, wash immediately with plenty of water and liquid soap.

3 COMPOSITION/INFORMATION ON INGREDIENTS

Ingredient name	Contents	Health(Class)	Risk(R/No.)
1-Butyl-3-methylimidazolium chloride	>99%		Substance not yet fully tested!

4 FIRST AID MEASURES

General Contaminated clothing should be removed and washed before being reused.

Inhalation Move the exposed person to fresh air at once. If respiratory problems, provide artificial respiration/oxygen.

Ingestion Immediately rinse mouth and provide fresh air. Do not induce vomiting. Get medical attention immediately.

Skin Wash the skin immediately with soap and water.

Eyes Promptly wash eyes with plenty of water while lifting the eye lids. Continue to rinse for at least 15 minutes. Get medical attention immediately. Continue to rinse.

Safety Data Sheet

1-Butyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 2/26/2017

5 FIRE FIGHTING MEASURES

Extinguishing media	Use: Water spray, fog or mist. Carbon dioxides (CO ₂). Dry chemicals, sand, dolomite etc.
Special fire fighting procedures	Avoid water in straight hose stream, will scatter and spread fire. Keep run-off water out of sewers and water sources. Dike for water control.
Unusual fire & explosion hazards	Fire causes formation of toxic gases.
Protective measures in fire	Wear self-contained breathing apparatus as combustion may produce hazardous fumes.

6 ACCIDENTAL RELEASE MEASURES

Personal precautions during spill	Wear protective clothing and avoid inhalation of vapor, skin or eye contact.
Precautions to protect environment	Avoid washing into water courses. Avoid contaminating public drains or water supply.
Spill cleanup methods	Avoid contact with skin or inhalation of spillage, dust or vapor, Avoid dust formation. Collect and reclaim or dispose in sealed containers in license waste. Extinguish all ignition sources. Avoid sparks, flames, heat and smoking. Ventilate.

7 HANDLING AND STORAGE

Usage precautions	Keep away from heat, sparks and open flame. Do not use in confined spaces without adequate ventilation and/or respirator.
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Safety Data Sheet

1-Butyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 2/26/2017

Storage precautions	Store at moderate temperatures in dry, well ventilated area.
Storage criteria	Chemical storage.

8 EXPOSURE CONTROLS AND PERSONAL PROTECTION

Personal protective equipment

Eye/face protection

Safety glasses with side-shields conforming to EN166 Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

The selected protective gloves have to satisfy the specifications of EU Directive 89/686/EEC and the standard EN 374 derived from it.

Body Protection

Impervious clothing. The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection

For nuisance exposures use type P95 (US) or type P1 (EU EN 143) particle respirator. For higher level protection use type OV/AG/P99 (US) or type ABEK-P2 (EU EN 143) respirator cartridges. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

9 PHYSICAL AND CHEMICAL PROPERTIES

Appearance	Solid.
Color	Colorless to slightly yellow.
Odor/taste	No characteristic odor.

IL-0014

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Safety Data Sheet

1-Butyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

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10 STABILITY AND REACTIVITY

Stability	No particular stability concerns.
Conditions to avoid	Avoid contact to strong oxidizers and bases.
Hazardous	
Decomposition Products	High temperatures generate: Corrosive gases/vapor/fumes of: Carbon dioxide (CO ₂). Carbon monoxide (CO). Nitrous gases (NO _x). Hydrochloric acid (HCl).

11 TOXICOLOGICAL INFORMATION

Ingestion	not available.
Skin	not available.
Eyes	not available

Data on the toxicity of this product are not available. Hazardous properties cannot be excluded.

12 ECOLOGICAL INFORMATION

Environmental hazards	Avoid washing into water courses. Avoid contaminating public drains or water supply.
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13 DISPOSAL CONSIDERATIONS

Disposal method	Contact specialist disposal companies. Dispose of in accordance with Local Authority requirements. Recover and reclaim or recycle, if practical.
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Safety Data Sheet

1-Butyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 2/26/2017

14 TRANSPORT INFORMATION

General	Not classified as dangerous for transport purposes.
UN No.	0
Road transport notes	Not classified as dangerous for road transport.
Rail transport notes	Not classified as dangerous for rail transport.
Sea transport notes	Not classified as dangerous for sea transport.
Air transport notes	Not classified as dangerous for air transport.

15 REGULATORY INFORMATION

Label for supply	Substance not yet fully tested.
P phrases	
P262:	Do not get in eyes, on skin, or on clothing
P280:	Wear protective gloves/ protective clothing/ eye protection/ face protection
P305 + P351 + P338:	IF IN EYES: Rinse continuously with water for several minutes. Remove contact lenses if present and easy to do – continue rinsing.
P313:	Get medical advice/attention
Safety phrases	
S: 24/25	Avoid contact with skin and eyes.
S: 26	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S: 36/37/39	Wear suitable protective clothing, gloves and eye/face protection.



Safety Data Sheet

1-Butyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 2/26/2017

S:28,2

After contact with skin, wash immediately with plenty of water and liquid soap.

Country specific information

Germany

WGK: 3 (Self-Classification)

16 OTHER INFORMATION

DISCLAIMER

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPOSED TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. IOLITEC SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT. THIS INFORMATION RELATES ONLY TO THE SPECIFIC MATERIAL DESIGNATED AND MAY NOT BE VALID FOR SUCH MATERIAL USED IN COMBINATION WITH ANY OTHER MATERIALS OR ANY PROCESS. IT IS THE USER'S RESPONSIBILITY TO SATISFY HIMSELF AS TO THE SUITABILITY OF SUCH INFORMATION FOR HIS OWN PARTICULAR USE.

IN NO WAY SHALL IOLITEC BE LIABLE FOR ANY CLAIMS, LOSSES OR DAMAGES OF ANY THIRD PARTY OR FOR THE LOST PROFITS OR ANY SPECIAL, INDIRECT, INCIDENTAL, CONSEQUENTIAL OR EXEMPLARY DAMAGES, HOWSOEVER ARISING, EVEN IF THE COMPANY HAS BEEN ADVISED OF THE POSSIBILITY OF SUCH DAMAGES.

Safety Data Sheet

1-Allyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 12/8/2017

1 IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND THE COMPANY/UNDERTAKING

Product name	1-Allyl-3-methylimidazolium chloride
Product code	IL-0022
CAS	65039-10-3
REACH No.	A registration number is not available for this substance as the substance or its uses are exempted from registration, the annual tonnage does not require a registration or the registration is envisaged for a later registration deadline.
Identified uses	Laboratory chemicals, Manufacture of substances
Supplier	IoLiTec Ionic Liquids Technologies GmbH Salzstrasse 184 D – 74076 Heilbronn Germany
Telephone	+49 (0)7131-89839-0
Fax	+49 (0)7131-89839-109
Emergency telephone	+49 (0)176-84850874
Email	msds@iolitec.de

2 HAZARDS IDENTIFICATION

2.1 Classification of the substance or mixture

Classification (REGULATION (EC) No 1272/2008)

Skin irritation (Category 2)

Eye irritation (Category 2)

Specific target organ toxicity -single exposure (Category 3)

Safety Data Sheet

1-Allyl-3-methylimidazolium chloride

Revision Date: 12/01/2015

Date Issued: 12/8/2017

Classification (67/548/EEC or 1999/45/EC)

Irritating to eyes, respiratory system and skin.

2.2 Label elements

Labelling (REGULATION (EC) No 1272/2008)

Pictogram



Signal word

Warning

Hazard statement(s)

H315

Causes skin irritation.

H319

Causes serious eye irritation.

H335

May cause respiratory irritation.

Precautionary statement(s)

P261

Avoid breathing dust/ fume/ gas/ mist/ vapours/ spray.

P305 + P351 + P338

IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

Supplemental Hazard Statements none

Labelling (67/548/EEC or 1999/45/EC)

Hazard symbol(s)



R-phrases)

R36/37/38

Irritating to eyes, respiratory system and skin.

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S-phrase(s)

S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

Caution - substance not yet tested completely.

3 COMPOSITION/INFORMATION ON INGREDIENTS

Ingredient name	Contents	Health(Class)	Risk(R/No.)
1-Allyl-3-methylimidazolium chloride	>98%		Substance not yet fully tested!

4 FIRST AID MEASURES

General	Contaminated clothing should be removed and washed before being reused.
Inhalation	Move the exposed person to fresh air at once. If respiratory problems, provide artificial respiration/oxygen.
Ingestion	Immediately rinse mouth and provide fresh air. Do not induce vomiting. Get medical attention immediately.
Skin	Wash the skin immediately with soap and water.
Eyes	Promptly wash eyes with plenty of water while lifting the eye lids. Continue to rinse for at least 15 minutes. Get medical attention immediately. Continue to rinse.

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5 FIRE FIGHTING MEASURES

Extinguishing media	Use: Water spray, fog or mist. Carbon dioxides (CO ₂). Dry chemicals, sand, dolomite etc.
Special fire fighting procedures	Avoid water in straight hose stream, will scatter and spread fire. Keep run-off water out of sewers and water sources. Dike for water control.
Unusual fire & explosion hazards	Fire causes formation of toxic gases.
Protective measures in fire	Wear self-contained breathing apparatus as combustion may produce hazardous fumes.

6 ACCIDENTAL RELEASE MEASURES

Personal precautions during spill	Wear protective clothing and avoid inhalation of vapor, skin or eye contact.
Precautions to protect environment	Avoid washing into water courses. Avoid contaminating public drains or water supply.
Spill cleanup methods	Avoid contact with skin or inhalation of spillage, dust or vapor, Avoid dust formation. Collect and reclaim or dispose in sealed containers in license waste. Extinguish all ignition sources. Avoid sparks, flames, heat and smoking. Ventilate.

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7 HANDLING AND STORAGE

Usage precautions	Keep away from heat, sparks and open flame. Do not use in confined spaces without adequate ventilation and/or respirator.
Storage precautions	Store at moderate temperatures in dry, well ventilated area.
Storage criteria	Chemical storage.

8 EXPOSURE CONTROLS AND PERSONAL PROTECTION

Personal protective equipment

Eye/face protection

Safety glasses with side-shields conforming to EN166 Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

The selected protective gloves have to satisfy the specifications of EU Directive 89/686/EEC and the standard EN 374 derived from it.

Body Protection

Impervious clothing. The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection

For nuisance exposures use type P95 (US) or type P1 (EU EN 143) particle respirator. For higher level protection use type OV/AG/P99 (US) or type ABEK-P2 (EU EN 143) respirator cartridges. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).



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9 PHYSICAL AND CHEMICAL PROPERTIES

Appearance	Solid.
Color	Colorless to yellow.
Odor/taste	No characteristic odor.

10 STABILITY AND REACTIVITY

Stability	No particular stability concerns.
Conditions to avoid	Avoid contact to strong oxidizers and bases.
Hazardous	
Decomposition Products	High temperatures generate: Corrosive gases/vapor/fumes of: Carbon dioxide (CO ₂). Carbon monoxide (CO). Nitrous gases (NO _x). Hydrochloric acid (HCl).

11 TOXICOLOGICAL INFORMATION

Acute toxicity

no data available

Skin corrosion/irritation

no data available

Serious eye damage/eye irritation

no data available

Respiratory or skin sensitization

no data available

Germ cell mutagenicity

no data available

Carcinogenicity

IARC: No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.



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Reproductive toxicity

no data available

Specific target organ toxicity - single exposure

Inhalation - May cause respiratory irritation.

Specific target organ toxicity - repeated exposure

no data available

Aspiration hazard

no data available

Potential health effects

Inhalation May be harmful if inhaled. Causes respiratory tract irritation.

Ingestion May be harmful if swallowed.

Skin May be harmful if absorbed through skin. Causes skin irritation.

Eyes Causes serious eye irritation.

Signs and Symptoms of Exposure

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Additional Information

RTECS: Not available

12 ECOLOGICAL INFORMATION

Environmental hazards

Avoid washing into water courses. Avoid contaminating public drains or water supply.

13 DISPOSAL CONSIDERATIONS

Disposal method

Contact specialist disposal companies. Dispose of in accordance with Local Authority requirements. Recover and reclaim or recycle, if practical.

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14 TRANSPORT INFORMATION

General	Not classified as dangerous for transport purposes.
UN No.	0
Road transport notes	Not classified as dangerous for road transport.
Rail transport notes	Not classified as dangerous for rail transport.
Sea transport notes	Not classified as dangerous for sea transport.
Air transport notes	Not classified as dangerous for air transport.

15 REGULATORY INFORMATION

This safety datasheet complies with the requirements of Regulation (EC) No. 1907/2006.

Safety, health and environmental regulations/legislation specific for the substance or mixture

no data available

Chemical Safety Assessment

no data available

Country specific information

Germany WGK: 3 (Self-Classification)



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16 OTHER INFORMATION

DISCLAIMER

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