THESIS

IMPARTING PROTECTIVE PROPERTIES TO LYOCELL FABRIC VIA SINGLE AND MULTI-FUNCTIONAL FINISHING TREATMENTS

Submitted by

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In partial fulfillment of the requirements

For the degree of Master of Science

Colorado State University

Fort Collins, Colorado

Spring 2010

COLORADO STATE UNIVERSITY

March 12, 2010

WE HEREBY RECOMMEND THAT THE THESIS PREPARED UNDER OUR SUPERVISION BY GUNJAN VORA ENTITLED IMPARTING PROTECTIVE PROPERTIES TO LYOCELL FABRIC VIA SINGLE AND MULTI-FUNCTIONAL FINISHING TREATMENTS BE ACCEPTED AS FULFILLING IN PART REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE.

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ABSTRACT OF THESIS

IMPARTING PROTECTIVE PROPERTIES TO LYOCELL FABRIC VIA SINGLE AND MULTI-FUNCTIONAL FINISHING TREATMENTS

Lyocell is a fiber made from wood pulp cellulose. Like other cellulosic fibers, it is breathable, absorbent, very comfortable to wear, and biodegradable. Lyocell fiber is also eco-friendly and sustainable since the solvent used to manufacture the fiber is environmentally not hazardous and the manufacturing of lyocell is a closed-loop process. Lyocell has been treated a myriad of ways to enhance its value-added potential. However, no studies were found to have been done to impart protective properties such as resistance to UV-radiation and resistance to microbes.

In this study, untreated lyocell fabric was analyzed for its protective properties against UV radiation and disease causing microbes. Lyocell was found to afford no protection against UV radiation and also possessed no anti-microbial activity against the three microbes investigated in this study. To improve its protective properties, lyocell was finished with UV-absorbers. To enhance its antimicrobial property, lyocell was treated with an antimicrobial agent. It was experimentally determined that a UV-absorber concentration of 2% on weight of fabric was sufficient to improve the UV properties of lyocell fabric to an excellent degree. The antimicrobial concentration for excellent antimicrobial activity was found to be 0.5% on weight of fabric.

Combining several processing steps to reduce time and cost is preferred in the textile industry so the synergistic effect of UV absorber treatment and antimicrobial treatment from a multi- functional treatment bath was explored in the next phase of the study. Lyocell fabrics were treated with the optimum amount of UV-absorber and antimicrobial agent. The data showed the UV-protection of lyocell fabric was not imparted negatively when a multi-functional bath was employed. Similarly, the antimicrobial efficiency was not reduced on multi-functional finishing treatment. Further, the finishing treatments, both single and multi-functional were durable to laundering and to light exposure.

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ACKNOWLEDGEMENTS

I would like to thank God for all the happiness and success he has blessed upon me till today and for the future. I thank my family for their unconditional support, love, endless encouragement and for truly believing in my ability to succeed in any endeavor I might embark on.

I would like to express the deepest appreciation to Dr. Ajoy Sarkar, my advisor. He has been a guide to me and has always been there to correct my mistakes and direct me in the right direction. Without his rightful guidance, help and support, I would never have completed this work successfully. He took time off his busy schedule to go through my manuscript, edit any mistakes, and give his insight and suggestions without which the outlook of this thesis would have been hard to describe.

I would like to thank Dr. Mary Littrell and Dr. Jennifer Ogle for their valuable advice and motivation. I would also like to thank my friends and colleagues Ms. Deedee DeMiranda, Mr. Subhash Appidi and Ms. Larissa Gibson for their help in my journey through graduate school.

I wish to thank my committee members, Dr. Kenneth Tremblay and Dr. Dawn Mallette for their support and enthusiasm in this project. Each individual provided insights that guided and challenged my thinking, substantially improving the finished product.

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INTRODUCTION

1.1 History of lyocell

Lyocell is a fiber made from wood pulp cellulose. It was first manufactured in 1987 by Courtaulds Fibers, United Kingdom. Lyocell was designated by BISFA (Bureau International Standardisation Fibres Synthetiques) as belonging to a new generic class. Lyocell was also the first new generic fiber group to be approved by the U.S. Federal Trade Commission (FTC) in thirty years. The FTC defines Lyocell as "a cellulose fabric that is obtained by an organic solvent spinning process". The current manufacturer of lyocell is Lenzing Inc, Austria, who marketS it under the trademarked brand name Tencel® and classifies the fiber as a sub-category of rayon. Twenty-two years ago at its debut, lyocell fiber was predicted to have a big future and by all accounts is on its way to fulfilling that prediction.

1.2 Production of lyocell

The principal advantage of lyocell fiber is that it is naturally biodegradable. The forests that provide the raw material for lyocell are also always being replenished. Additionally, other materials used in the fiber production process are re-cycled with very little loss. Lyocell is produced from wood pulp via a solvent spinning process. The pulp is dissolved in amine oxide (usually N-methylmorpholine-N-oxide, Figure 1) and the resulting viscose solution is extruded into a water bath through fine jets. As the solvent is washed out, the fiber forms into fine filaments that are collected as a tow, from which the staple fiber is produced. More than 99% of the solvent is recycled within the process, making the fiber production extremely environmentally responsible (Taylor, 1998).

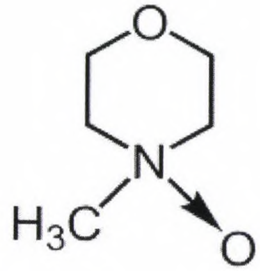


Figure 1. N-methylmorpholine-N-oxide (Hayhurst, White & Taylor, n.d.)

The standard fiber produced is 1.4 dtex, 38 mm, but it can be produced in a range of linear densities and staple lengths. The fiber has a smooth surface and a round cross section, providing high luster in the raw state. A flow-chart of the production process is depicted in Figure 2.

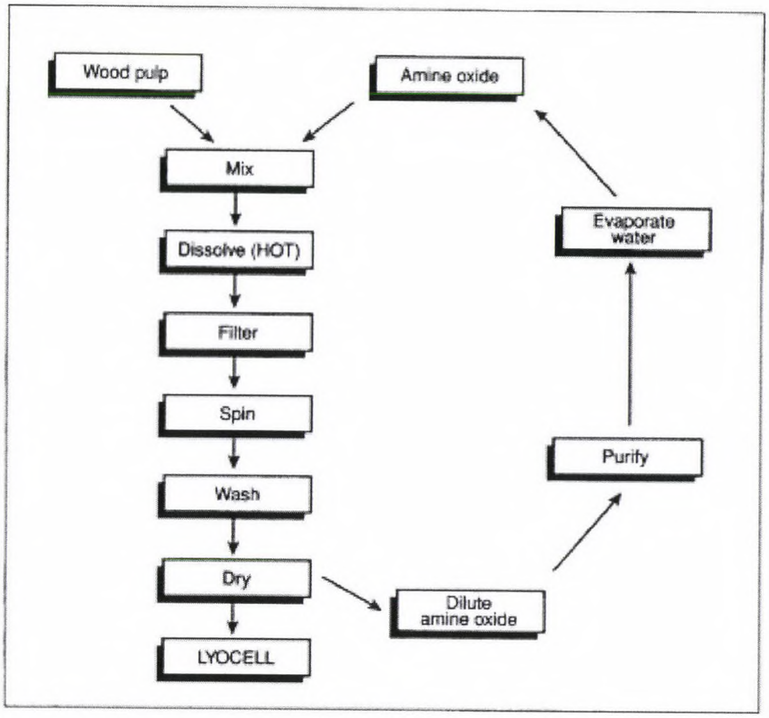


Figure 2. Production process for lyocell fiber (Boniface, 1990)

1.3 Properties of lyocell

Lyocell is a manufactured fiber, but it is not synthetic. Because it is made from a plant material, it is cellulosic and possesses many properties of other cellulose fibers, such as cotton, linen, ramie, and rayon - another manufactured but non-synthetic fiber. Compared to cotton, lyocell wrinkles less, is softer, more absorbent, and much more resistant to ripping. In material physical properties, lyocell is more like cotton than rayon. Like other cellulosic fibers, it is breathable, absorbent, and very comfortable to wear. In fact, lyocell is more absorbent than cotton or silk, but slightly less absorbent than wool, linen, or rayon. Lyocell has good resiliency: it does not wrinkle as badly as rayon, cotton, or linen, and some wrinkles will fall out if the garment is hung in a warm moist area. Lyocell is as stable a fiber as silk and better than cotton or linen. Lyocell has strength and durability. It is the strongest cellulosic fiber when dry, even stronger than cotton or linen and is stronger than cotton when wet. Lyocell is much stronger than rayon when wet. This property of high wet strength usually determines the extent to which fabrics can be machine washed successfully. Lyocell can generally be either hand washed or machine washed and tumbled dried successfully. Other desirable properties of lyocell are its luster and soft drape which makes it an aesthetically pleasing fiber. Since it is a manufactured fiber, the diameter and length of fibers can be varied. Lyocell can be made into microfibers, offering depth and body to fabrics combined with luxurious drape. Short staple length fibers give a cotton-like look to fabrics. Long filament fibers are successful in silk-like end uses. Lyocell blends well with other fibers including wool, silk, rayon, cotton, linen, nylon, and polyester. It successfully takes many finishes, both functional and those designed to achieve different surface effects and dyes easily. Overall, lyocell is a versatile fiber with many desirable properties (Finlen, 1998).

1.4 Rationale for study

A number of finishing treatments have been done on lyocell to improve its properties; examples of treatments include enzymatic hydrolysis, reactive dyeing, sulphur dyeing, low-temperature plasma treatment, treatment with chitosan, and aqueous emulsion surface finish treatment. All of these treatments are discussed in the chapter on literature review. Very few studies however, have studied the protective properties of lyocell via either a single-bath finish or multi-functional finishing. Multi-functional finishing may be defined as treating textile fabrics with two or more finishing agents in one single step. Multi-functional finishing therefore results in savings both in terms of energy usage as well as water consumption. In this study, both single-bath finishing as well as multi-functional finishing for lyocell fabric was investigated. The intent was to impart value-added protective properties to lyocell viz., resistance to ultra-violet (UV) radiation and resistance to disease-causing microbes. The specific objectives of this study were:

Objective 1: To determine the UV properties of untreated lyocell fabric.

Objective 2: To determine the antimicrobial properties of untreated lyocell fabric.

Objective 3: To enhance UV protection characteristics via a finishing treatment with UV absorbers.

Objective 4: To improve antimicrobial properties via a finishing treatment with an antimicrobial agent.

Objective 5: To study the feasibility of multi-functional UV and antimicrobial finishing treatment on protective properties of lyocell fabric.

CHAPTER 2

LITERATURE REVIEW

The review of literature is organized in three sections. The first section discusses general finishing treatments for lyocell fabrics. The second section is a specific discussion on ultraviolet (UV) finishing of fabrics and the third section is a detailed discussion on antimicrobial finishing of textiles.

2.1. Finishing treatments for lyocell

2.1.1. Fibrillation and peach skin finish

Fibrillation is the peeling back or splintering of the fiber ends to form tiny "hairs" on the surface. Fibrillation is caused under aqueous conditions by the peeling away of micro-fibrils (or tiny fibers) on the surface of the fiber during mechanical action. In practice, all cellulosic fibers have a tendency to fibrillate, and it is merely the degree of fibrillation which varies from one fiber to another. Cellulosic fibers swell in presence of water, and swelling behavior of cellulosic materials is directly relevant to many stages of manufacture and use, including finishing, dyeing, wear and washing. The high degree of swelling in water is one of the reasons why wet rubbing causes the fibrils to break off and protrude from the surface. The high water absorbency of lyocell gives increased fibrillation during processing. Fibrillation is one of the main features of lyocell fibers and its understanding and control are important in developing the aesthetic appeal of fabrics based on it. Fibrillation is most commonly used in the creation of the 'mill-wash' or 'peach skin finish' fabrics (York, Donnelly, & Harnden, 2001). The process required to produce such finish involves three stages: primary fibrillation, enzyme cleaning and secondary fibrillation. The fabric is subjected to water and mechanical action in the primary fibrillation process. The hairs are most accessible to the action and therefore fibrillation occurs predominantly on these surface fibers. They can become entangled giving a characteristic pilled appearance. Cellulase enzymes remove the fibrillation fibers and pills from the fabric surface. Further wet processing causes more fibrillation also called secondary fibrillation. There were no longer any hairs so no pilling occurs, just fibrillation on the fabric highpoints. Final softening and tumbling allow the surface fibrillation to lift, giving a frosted appearance from the light scattering behavior of the fibrils and the characteristic hand (Taylor, 1998). In some applications, its fibrillation behavior is considered to be a drawback. If however, peach skin effect is not desired, the tendency of lyocell to fibrillate can be reduced or prevented by avoiding mechanical action whilst wet and which is done by various finishes on lyocell and also by crosslinking the cellulose chains immediately after spinning (Ahmed, Hawkyard, & Shamey, 2004).

2.1.2. Enzymatic treatment of lyocell fabric

Enzymatic treatment is one of the well-known developed treatments that are commonly used in the industry for improving the performance of the fabric. The benefits for treating lyocell fabric with the enzyme cellulase are softening, defuzzing, depilling and pill prevention, improved drapeability and improved surface appearance after multiple launderings. In a study by Kumar (1994), lyocell was treated with acid cellulase for up to 120 minutes in a 35 kg- lab scale rotary drum washer. Subsequently, the fabric was tested for differences in surface smoothness, handle, weight, strength and laundering durability. The effects of treating lyocell with cellulases were measured on a pilot scale and correlated well to results in commercial applications. Results of the study were reduction of loose fibers on the lyocell fabric. Tendency of pilling was also reduced and the fabric had a smooth surface appearance.

2.1.3. Caustic treatment

Treatment of a yarn or fabric with caustic soda to improve its luster, increased affinity for dyeing, strength and permanent swelling of the fiber is known as caustic treatment. The construction of lyocell fabrics has a great influence on processability and fabric properties, in dyeing, finishing and in consumer use. To reduce shortcomings in construction of the fabric caustic treatment is usually done. Since the free swelling on lyocell is up to three hundred percent, caustic treatment can open up even greater internal space within the fabric structure. Morley (2000) treated lyocell fabrics with aqueous concentrations of caustic soda between 0–7 mol dm^{-3} by a pad batch process. Samples were immersed in an aqueous caustic soda solution, passed through an Ernst Benz padding mangle applying a pressure of 10 kg cm^{-2}, and then batched for 30 min by wrapping around a glass rod and enveloping the sample in poly (ethylene) sheet. The samples were subsequently removed from the glass rod, rinsed in hot water for 5 min, and then immersed in 5% aqueous acetic acid at room temperature for 60 min. Samples were then rinsed in cold water for 5 min and dried. When dried, this increased space remains set within the fabric. As the use of caustic soda results in much greater swelling than with water, the potential for fabric shrinkage and creasing is increased during the caustic treatment process. Once treated not only the caustic treated fabric has greater bulk to cope with any subsequent wet swelling, but it also has much better set than is possible with water alone. The improved set arises from the fact that caustic soda can break hydrogen bonds within the fiber that are not accessible to water. Caustic setting also provides a lasting memory that remains undisturbed by later wetting and drying treatments. This provides lyocell fabric with an efficient and lasting set.

2.1.4. Dyeing of lyocell fabric

Lyocell is a cellulosic fiber and therefore can be dyed with any class of dyestuff suitable for other cellulosics. Willmott and Burkinshaw (1994) compared the dyeability of lyocell to that of cotton and viscose. Three types of reactive dye were applied to cotton, regular viscose and lyocell at 2% o.w.f (on weight of fabric). All dyeing were carried out in partially sealed, glass dye pots of 200 cm^3 capacities housed in a Zeltex Vistacolor laboratory-scale dyeing machine and employing a 20:1 liquor ratio. It was found that the calorimetric parameters of all the dyes on the three types of fiber reflected those of the undyed substrates and, in general, that there was relatively little difference in color between the dyeing on the three fiber types. The color strength of the dyeing was lowest on cotton, the dyeing on lyocell being of slightly lower color strength than those on viscose. From the results, it appears that lyocell exhibits similar dyeability to regular viscose with reactive dyes. In another study by Leonard and Burkinshaw (1994), four CI solubilised sulphur dyes were applied to cotton, regular viscose and lyocell at 2%, 6% and 10% o.w.f. and the color strength and colorimetric parameters of the dyeing were determined. The dyes used were Sulphosol Black SG®, Sulphosol Blue SL®, Sulphosol Yellow SR®, and Sulphosol Bordeaux SB®. Scoured and bleached cotton, lyocell and viscose were used. Again, it was found that the colorimetric parameters of all the dyes on the three types of fiber reflected those of the undyed substrates and there was relatively little difference in color between the dyeings on the three fiber types. The color strength of the dyeings was lowest on cotton, the dyeings on lyocell being of slightly lower color strength than those on viscose. From the results, it appears that lyocell exhibits similar dyeability to cotton with solubilised sulphur dyes (Leonard & Burkinshaw, 1994). Another potential use of bi-functional reactive dyes is as a visible cross linker on lyocell fabrics. This was demonstrated in a study by Burkinshaw and Leonard (1994), sole influence of CI Reactive Black 5 dye on the fibrillation tendency of lyocell single jersey knitted fabrics was investigated by dyeing with different concentrations viz. 35 g/L for medium shade and 70 g/L for dark shade (Bui, Ehrhardt, & Bechtold, 2008). The fabrics were cut into pieces and kept 24 h in standard atmosphere room (20 ± 20 C, RH 65%). The dye solutions were separately prepared with three different dyeing concentrations: blank dyeing, medium shade dyeing and dark shade dyeing. Nitrogen content was used as indication of the amount of reactive dye in fabrics during dyeing and discoloration processes. The correlation between fiber fibrillation tendency and color values of fabrics was found. Due to the fibrillation reduction, pilling formation in dyed lyocell fabrics was improved. As color values can estimate the dye fixation and reduction in fabrics, reactive dye can therefore be used as a visible marker for cross linking effect on lyocell fabrics.

2.1.5. Low-temperature plasma treatment of lyocell

Low- temperature plasma treatment is done on a fabric to modify the fabric surface area so as to increase the dye sites for dyeing and also for high quality of comfort fabrics in terms of high moisture regains and soft-hand. The effect of low-temperature plasma on the surface modification of lyocell fabric was studied using the plasma gases of oxygen and argon. The pressure of the system, the gas flow rate and plasma temperature were kept at 10 Pa, 10 cc/min and in the range of 1-5 eV, output power used was kept at 100W throughout the study and the time was varied from 5,10,20,30 and 60 min. The experimental results showed that argon plasma caused higher fabric loss. In addition there was a tremendous decrease in the whiteness of the testing specimens as a result of the long exposure of argon low plasma treatment. The results of scanning electron microscopy showed that short exposure of argon or oxygen plasma increased the surface roughness on lyocell fabric that promoted the dye uptake. The contact angle test result showed that the wettability is increased by means of short-exposure low temperature plasma treatment (Mak, Yuen, Ku, & Kan, 2005).

2.2. UV finishing of fabrics

In recent years, consumers have become increasingly aware of the need for sun protection, which is related to the incidence of sun induced skin damage and its relationship with increased exposures to ultraviolet (UV) light. UV radiation is divided into two distinct parts, viz. UVA and UVB. UVA falls into the regions of 315-400 and UVB falls in the regions of 280-315 nm, of the solar spectrum. Elevated exposure to UV radiation can result in skin damage such as sunburn, premature skin ageing, allergies, and even skin cancer. Textiles can provide effective protection against such damages. UV-resistance in fabrics can be incorporated by:

- Varying the weave structure.
- Dyeing in heavy shades.
- Applying a suitable finishing agent on the fabric such as an UV absorber.

The Ultraviolet Protection Factor (UPF) system measures the UV protection provided by fabric. It is very similar to the SPF rating system used for sunscreens, the difference being that SPF values are determined *in-vivo* whereas UPF values are based *in-vitro*. A garment with a UPF of 50 only allows 1/50th of the UV radiation falling on the surface of the garment to pass through it. In other words, it blocks 49/50ths or 98% of the UV radiation. The Table 1 below illustrates the different UPF protection ranges and the percentage UV blocked according to ASTM D 6603.

Table 1. UPF Protection Ranges

Protection Category	UPF Range	Approximate % UV Blocked
Good UV Protection	15 - 24	93.3% - 95.8%
Very Good UV Protection	25 - 39	96.0% - 97.4%
Excellent UV Protection	40 - 50+	97.5% - 98.0%

2.2.1. UV resistance as a function of weave structure

Woven fabric construction is one of the basic factors that have a direct effect on UPF value. Woven fabric construction is altered by three primary constructional parameters: yarn fineness, weave type and warp/weft density. In a study by Dubrovski and Golob (2009), forty-five 100% cotton woven fabric samples were studied. Samples varied according to weave type (plain, twill, satin) and fabric tightness (from 55% to 95%; tightness in a fabric is defined as a ratio between yarn diameter and its length in that structure). An UPF value of all the samples was measured and results indicated that none of the plain fabric showed the minimum UV protection, even if they were tightly woven. Twill fabric showed good UV protection if they were tightly woven above 70%, while satin fabrics offered good UV protection at 60% tightness. In another study by Gabrijelcic, Urbas, Sluga, and Dimitrovski (2009), cotton fabric samples were taken which were woven in eight-end sateen weave, construction of yarn used for warp and weft was identical. The construction parameters of the fabrics were considered, such as warp and weft thread density, yarn thickness and weave. UPF values of all the samples in the study were measured and the results indicated that the construction of a fabric in sateen weave provides different type of protection against UV radiation depending on surface openness/ closeness. Higher surface openness than approximately 8% does not provide sufficient protection against UV radiation. Higher actual warp/weft density means higher tightness and consequently higher UV protection, fabrics can also offer excellent UV protection at lower fabric tightness when they are woven in twill and satin weave.

2.2.2. UV resistance via dyeing

The color of clothing and fabric is known to affect UPF. Several color characteristics are there which may influence the improvement of UV protection: the shade of color, the type and chemical structure of dyestuff, and the intensity of the color. Dark colors have been reported as providing better protection than light colors because the absorption bands of those dyes can extend into UV range and thereby reduce the total UV transmission through fabric. In a study by Wang, Alfred, Kopf, Marx, Bordan, Polsky, and Bart (2000), cotton fabric was evaluated for UV protection after various dyeing treatments. Plain cotton fabric was dyed with blue and yellow dyes. Both dyes have azo and anthraquinone backbones that attach to various chromophores to give the blue and yellow colors. After dyeing treatment, the UPF of each of the fabric samples were calculated with a Labsphere® UV transmittance analyzer. Results showed that both dyes increased the UPF, however, the blue dye was much more effective than the yellow dye in reducing the UV transmission and the untreated fabric showed relatively low UPFs.

In a similar study by Feng, Zhang, and Chen (2005), they used natural dyes like *rheum* and *L. erythrorhizon* to investigate the ultraviolet protective properties of plain weave cotton and plain weave silk fabrics. Dyeing was performed by a general dyeing method with a material to liquor ratio of 1:10 and pH of 9-10 for *rheum* and pH 3-4 *for L. erythrorhizon* dye at a temperature of 100^0C for about 60 minutes. Results indicated that cotton and silk fabric dyed by these natural dyes absorbed about 80% of the ultraviolet rays.

In another study by Riva, Algaba, Pepio, and Prieto (2009), three azo dyes C.I Direct Yellow 98, C.I. Direct Blue 77, and C.I. Direct Red 89 were applied to cotton woven fabric according to conventional exhaustion dyeing process to study the influence of the color on the UPF of the cotton woven fabric. The results showed that the application of any one of the dyestuffs applied at the intensities investigated produced an improvement of the UPF of the cotton fabric. Thus, coloration was considered an efficient way to increase the ultraviolet protection provided by the fabric.

2.2.3. UV resistance via finishing

2.2.3.1. Improvement of UV property using UV absorbers

Ultraviolet light absorbers are molecules used in organic materials to absorb UV light in order to reduce the UV degradation (photo-oxidation) of a material. The protection provided by uncolored cellulosic fabrics such as lyocell, cotton and rayon is, in general low, but can be improved by finishing treatment with UV-absorbers. The mechanism of action of these UV-absorbers consists in the selective absorption of the damaging radiation and its dissipation as thermal energy. UV absorbers are non-colored products with elevated extinction coefficients in the spectral range between 300 and 400 nm approximately (Zweifel 1998). One commercial example of an UV absorber is Rayosan®. In a study by Abidi, Hequet, & Abdalah (2001) Rayosan® was applied by exhaust method. The concentration of Rayosan® on the weight of the fiber was varied between 1 and 10%. The bath containing fabrics and Rayosan® was circulated for 10 minutes and gradually 70 g/l of sodium sulfate was added. After 10 minutes, the temperature was raised to 194°F. Ten g/l of soda ash were added and the bath was circulated for 30 minutes at this temperature. Then the bath was cooled and the fabric was rinsed. The Ultraviolet Protection Factor was measured using an SPF-290 Analyzer. Results showed that the treated fabrics provide excellent protection from UV. The chemical cross-linking of undyed fabric with UV-absorber appeared to be the best way to increase its UPF and to provide excellent UV protection. In a different study by Akrman and Prikyrl (2007), UV absorbers for treating cotton textiles were prepared by reactions of five different amino phenyl sulfo-benzotriazoles with the condensation product of 4-aminophenyl- sulfatoethylsulfone and cyanuric chloride. The UV absorbers with two different reactive groups monochlorotriazine and aromatic vinylsulfone; capable of forming covalent bonds with hydroxyl groups of cellulose, were applied to one cellophane foil and two cotton fabrics of different porosities. This treatment increased the ultraviolet protection factor from a value of three to one hundred and above.

2.2.3.2. Sol-Gel Treatment for enhancing UV blocking

Sol-gel technology has emerged as a promising way to functionalize fabric surfaces. A UV-blocking treatment for cotton fabrics was developed using the sol-gel method. In a study by Xin, Daoud, & Kong (2004), a nanosol was prepared at room temperature by mixing titanium tetraisopropoxide with absolute ethanol at pH 1-2. The mixture was vigorously stirred for 10 minutes prior to coating. A 10 x 10 cm white cotton substrate bleached knit was dried at 100°C for 30 minutes, dipped in the nanosol for 30 seconds, and then padded with an automatic padder at a nip pressure of 2.75 kg/cm^2. The padded substrates were dried at 80°C for 10 minutes in a preheated oven to evaporate ethanol and then cured at 150°C in a preheated curing oven. Then the structure of the titania films were studied under scanning microscope. The treatment forms a thin layer of titania on the surface of cotton fibers, and the treated fabrics show much improved protection against UV radiation with a UPF factor of 50+ or excellent protection according to the Australian/New Zealand standards. The treated fabrics were also tested for washfastness. The results showed that the excellent UV protection rating of the treated fabrics were maintained even after fifty-five home launderings, indicating a high level of adhesion between the titania layer and the cotton. A bursting strength test of the treated fabrics showed no adverse effect from the treatment.

2.2.3.3. UV protecting cotton fabric with functionalized MWNT containing water vapor permeable PU coating

A study by Hu and Mondal (2007) took a novel approach of developing excellent protection from ultraviolet (UV) radiation of cotton fabrics by means of water vapor permeable (WVP) coatings containing multiwall carbon nanotube (MWNT), a stable and strongly UV absorbing species. The WVP of MWNT containing UV protective coatings of the present development were formed from solution polymer of hydrophilic polyurethane (HPU). MWNTs were dispersed in HPU solution by functionalization of MWNT. The nanotube containing HPU coating showed excellent protection against UV radiation, with only 1% by weight of MWNT (calculated based on solid content of the polymer), a UPF of 174 and with 2.5 % by weight of MWNT a UPF of 421 was obtained, which stated excellent protection (UPF $\geq$50) according to the Australian/New Zealand standards. Scanning electron micrographs of coated fabrics surface showed a film like polymer coating confirming the fabric surface was successfully coated by polyurethane. The coated fabrics maintained very good water vapor permeability, hence confirming the wearing comfort. Room temperature (20-23°C) range soft segment crystal melting of HPU enhances the permeability of coated fabrics and also enhanced the protection of cotton fabrics against UV radiation (Kathirvelu, Dhurai, & D'Souza, 2008).

2.3. Antimicrobial finishing of textiles

Antimicrobial finishing to textile fabrics imparts maximum resistance to growth of bacteria and many viruses. It helps maintain a safer, more sanitary environment. In the present day world, all of us are very conscious about our hygiene and cleanliness. Clothing and textile materials are not only the carriers of microorganisms such as pathogenic bacteria, odor generating bacteria and fungi, but also inherent properties of the textile fibers provide room for the growth of micro-organisms (Krishnaveni & Raj Kumar, n.d.). One critical factor for transmission of a microorganism from a person to the environment and then to another person is the ability of that microbe to survive on that environmental surface. A few studies have examined the survival of gram-positive bacteria on various surfaces, such as glass, aluminum foil, polyvinyl chloride, countertops, bed rails and stethoscopes. Infections, particularly those caused by antibiotic-resistant gram-positive bacteria, such as methicillin-resistant *Staphylococcus aureus* (MRSA) and vancomycin-resistant *enterococci* (VRE) are a growing concern. Though the use of antimicrobials have been known for decades, it is only in recent years that several attempts have been made towards finishing textiles with antimicrobial compounds. The requirements of a good antimicrobial agent for textiles are:

- A good antimicrobial agent should be effective against a broad spectrum of bacterial and fungal species.
- It should exhibit low toxicity, like allergy or irritation to the consumer.
- Affinity for specific fabric and fiber types and ease of application on textile substrate.
- It should be durable to laundering, dry cleaning and hot pressing.
- It should not negatively affect the quality of the fabric like physical strength, appearance, softness and handle.

Antimicrobial agents primarily work in two ways. The conventional "leaching" antimicrobials, separate from the textile upon contact, and chemically interact with the microorganism. The result of the interaction leads to the destabilization of the microorganism, eventually killing it. The "bound" antimicrobial, on the other hand, remains affixed to the textile, and, on a molecular scale, physically penetrates the bacteria membrane and complexes the biochemicals in the microorganism on contact to kill it.

Numerous commonly used antimicrobial agents for imparting antimicrobial finish and their techniques of action are summarized below. (Payne & Kudner, 1996).

2.3.1. Triclosan

One of the most durable type of antimicrobial products is based on a diphenyl ether (bis-phenyl) derivative known as either 2, 4, 4-trichloro-2 hydroxy diphenyl ether or 5-chloro-2-(2, 4-dichloro phenoxyl) phenol (Figure 3).

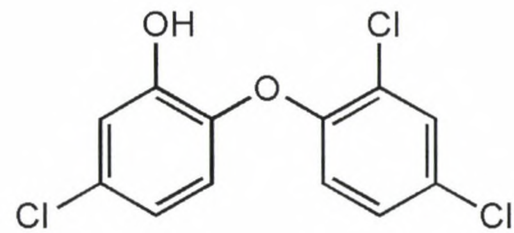


Figure 3. Structure of triclosan (Gao & Cranston, 2008)

Triclosan products have been used for more than 25 years in hospitals and personal care products such as antimicrobial soap, toothpaste and deodorants. Triclosan inhibits growth of microorganisms by using an electro chemical mode of action to penetrate and disrupt their cell walls. When the cell walls are penetrated, leakage of metabolites occurs and other cell functions are disabled, thereby preventing the organism from functioning or reproducing. Triclosan when incorporated within a polymer such as in a textile fiber migrates to the surface, where it is bound. Because it is not water-soluble, it does not leach out, and it continuously inhibits the growth of bacteria in contact with the surface using barrier or blocking action (Rajendran, Rajendrakumar, & Ramachandran, 2004). Huang and Leonas (1999) examined triclosan as an antibacterial finish along with a fluorochemical repellent finish on nonwoven fabrics. They found that a 0.25% add-on of triclosan was sufficiently high to inhibit bacterial growth of *S. aureus*.

2.3.2. Chitosan-coated Lyocell/cotton nonwoven fabric as a wound dressing

Chitosan is a bio-copolymer comprising glucosamine and *N*-acetylglucosamine, and is the alkaline deacetylated products of chitin, derived from the exoskeletons of insects and shells of crustaceans (Figure 4). An ideal wound dressing should protect the wound from bacterial infection, provide a moist and healing environment, and be bio-compatible. A study by Lin, Chao, Chun-Hsu, and Wen Lou (2008) attempted to produce such a wound dressing by coating a nonwoven lyocell/cotton fabric with Chitosan.

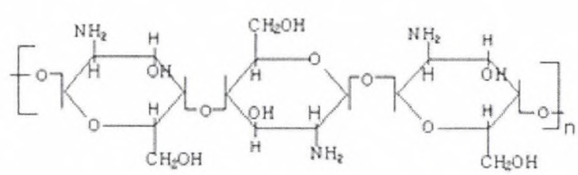


Figure 4. Structure of chitosan (Gao & Cranston, 2008)

The purpose of the study was to evaluate the double-layer structure of the chitosan-coated lyocell/cotton nonwoven fabric as a dressing for wound occlusion and accelerator in the healing process. The wound healing test was performed on Wistar rats, aged 10 weeks and weighing 300±50 g. After being anesthetized with diethyl ether and clipping dorsal hair, full-thickness square wounds (about 100 mm^2) were prepared on the upper back of each mouse using a sharp pair of scissors and a scalpel. The wound was then covered with a sample of chitosan coated lyocell/cotton nonwoven fabric. The same full-thickness square wounds without any treatment were prepared as a control. Changes in wound area were measured using a slide caliper after 2, 4, and 8 days post-surgically. The new skin and the tissues underneath with a size of $1.5\times1.5\ cm^2$ from each mouse were retrieved for histological examinations. Several properties of the chitosan-coated lyocell/cotton nonwoven fabric were studied, including the permeability to gas and the ability to control evaporative water loss. The chitosan-coated lyocell/cotton nonwoven fabrics were biocompatible causing no serious antigenicity and toxicity. The membrane had superior properties which could control the evaporative water loss and the fluid drainage properly. It could also inhibit exogenous microorganism's invasion by inherent antimicrobial property of the chitosan. Results from the wound healing studies suggested that the chitosan-coated lyocell/cotton nonwoven fabric is a viable material to be used as a wound dressing. In another study by Chung, Lee and Kim (1998), cotton fabric was treated with chitosan and citric acid to provide durable press and antimicrobial finishing to cotton fabric. The finish was applied by means of the conventional pad-dry-cure process. Citric acid was expected to react with hydroxyl groups in cellulose and chitosan or with amino groups in chitosan to form ester crosslinking or an inter-ionic attraction. Durable press appearance ratings of 3.5-4 were imparted with citric acid and chitosan treatments. The cotton fabric treated with citric acid alone showed antimicrobial properties. Durable press performance and antimicrobial properties were retained through twenty washing and tumble drying cycles. Strength retention improved more with citric acid and chitosan than with citric acid alone.

2.3.3. Antimicrobial activity using nano-particles of Zinc Oxide

Zinc oxide is non-toxic, and compatible with skin, making it a suitable additive for textiles and surfaces that come in contact with humans. Zinc oxide nano-particles are useful as antibacterial and antifungal agents when incorporated into materials, such as surface coatings (paints), textiles, and plastics. The bacteriostatic and fungistatic behavior of zinc oxide is well studied and utilized in personal care products A fine medium weight cotton fabric sample was used in this study. The aim was to impart anti-microbial and UV protection functions to the textile substrate and study the functional properties of coated fabrics. Zinc oxide nano-particles were prepared by wet chemical method using zinc nitrate and sodium hydroxide as precursors and solublized starch as stabilizing agent. Different concentrations of soluble starch (0.5 % & 1 %) were dissolved in 500 ml of distilled water by using microwave oven. Zinc nitrate hexahydrate, 14.874 g (0.1 mol) was added in the above starch solution. Then, the solution was kept under constant stirring using magnetic stirrer to completely dissolve the zinc nitrate. After complete dissolution, 500 ml of NaOH was added drop-wise under constant stirring, drop by drop touching the walls of the vessel. The clear aqueous solution gets turned into a milky white colloid without any precipitation. The reaction was allowed to proceed for two hours after complete addition of NaOH. These nano-particles were impregnated onto cotton fabrics by pad-dry-cure method using acrylic binder. The analysis was done by using SEM micrographs. The nano-ZnO impregnated cotton fabrics showed excellent antimicrobial activity against both gram-positive and gram-negative bacteria.

2.3.4. Silver-based antimicrobial agent

Silver has many notable contributions to human health and medicine. Silver ions are significant antimicrobials by virtue of their antiseptic properties with only few bacteria being intrinsically resistant to this metal. In early times, silver was used in the Middle East and South America for purifying drinking water, prostheses, surgical needles, catheters, dentistry, facial reconstruction and wound therapy. Halstead (1895) found that silver foil provided an effective means of dressing surgical wounds and controlling postoperative infections. Silver compounds were used to prevent infection in World War I before the advent of antibiotics. Figure 5 represents silver sulfadiazine which is most commonly used as an antibacterial agent.

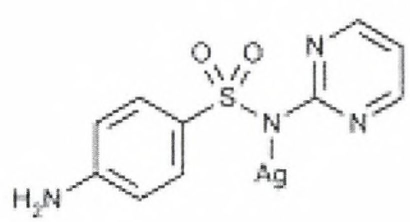


Figure 5. Silver sulfadiazine (Sun and Worley, 2005)

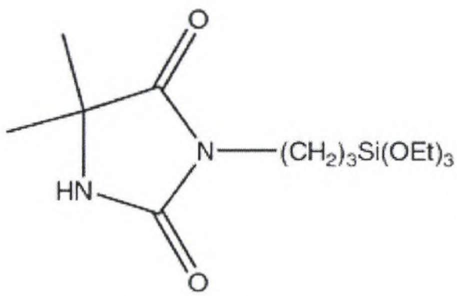
Silver compounds produce their antimicrobial effects by the time-dependent release of silver ions, and their clinical efficacy is directly related to the constant presence of free silver ions in the local microbial environment. Naturally occurring silver is composed of two stable isotopes, ^{107}Ag and ^{109}Ag, with ^{107}Ag being the most abundant. The anti-microbial properties of silver stem from the chemical properties of its ionized form, Ag^{+}. This ion forms strong molecular bonds with other substances used by bacteria to respire, such as molecules containing sulfur, nitrogen, and oxygen. When the Ag^{+} ion forms a complex with these molecules, they are rendered unusable by the bacteria, depriving them of necessary compounds and eventually leading to the bacteria's death. (Kumar and Munstedt, 2004).

In a study by Patricia, Person, Warner, Snipes, and Stevens (1986), a blend of nylon fiber and silver-coated nylon fiber was used to make the fabric bactericidal. To produce the silver coated component 12% by weight silver was added during manufacturing by means of an electrode less plating process. *Escherichia coli, Pseudomonas aeruginosa, Klebsiella pneumonia, Staphylococcus aureus* and *Streptococcus agalactiae* were the bacteria used in the study. Bacteria were exposed to Ag^{+} for 1 hour and incubation temperature was varied. Plates were incubated overnight at $37^{0}C$, and visible colonies were counted the next day to determine the number of CFU (Colony Forming Units) present. Results indicated that the rate of killing increased with increasing silver ion concentration of the fiber extract, as determined through atomic absorption spectrophotometer. The rate of killing was greater and the onset was earlier with an extract containing silver ions from fiber than with a salt solution containing the same concentration of silver ions from silver nitrate.

2.3.5. N-halamines

N-halamines are proven to be suitable biocides that could provide desired antibacterial functions without causing much environmental concerns. Recently, in a study by Ren, Kon, Liang, Worley, Tzou, and Huang (2008), N-halamine structures were incorporated into cellulose-containing fabrics by using a conventional pad-dry-cure method. The results revealed that as little as 1% weight add-on of halamine structures provided the materials with powerful biocidal properties against most common pathogens after a contact time of 2 min. Cotton swatches were soaked in solutions containing 3.0%, 5%, and 6.5% by weight of 5,5-dimethyl-3-(30 tri ethoxysilyl-propyl) hydantoin, 4-[3-triethoxysilylpropoxyl]-2,2,6,6-tetramethylpiperidine, and 3 triethoxysilylpropyl-2,2,5,5-

tetramethylimidazolidin-4-one in ethanol/water (1:1 w/w) for 15 min. These cotton swatches were cured at 95°C for 1 h, soaked in 0.5% detergent solution for 15 min, washed with water, and dried in air. Control and chlorinated fabrics were challenged with *Staphylococcus aureus* and *Escherichia coli* using a ‘‘sandwich test’’.



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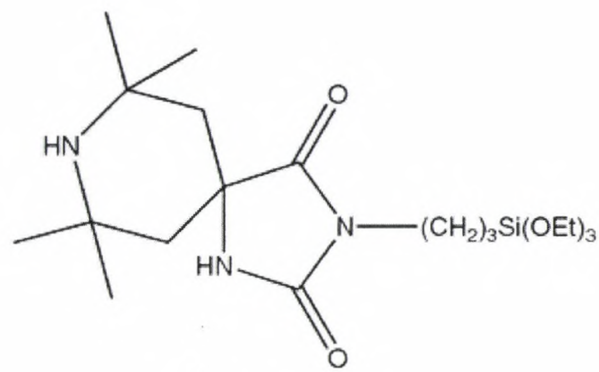

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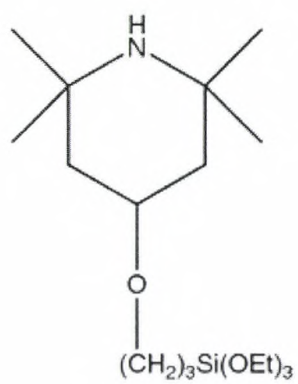

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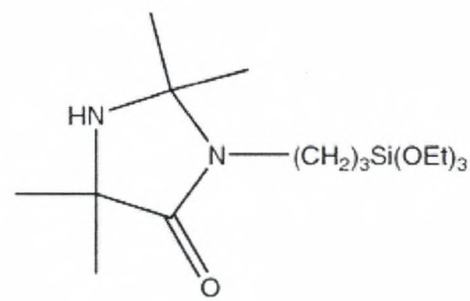

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Figure 6. Structures of the N-halamines compounds (Ren, Kon, Liang, Worley, Tzou, Huang, 2008)

Bacteria were suspended in pH 7, 100 mM phosphate buffer, and 25 μL of the bacterial suspensions were added to the middles of two pieces of one inch square cotton swatches which were held in place by sterile weights. The contact times for different samples were 1, 5, 10, and 30 min. Then the samples were quenched with 5.0 mL of sterile 0.02 N sodium thiosulfate solutions to remove all oxidative chlorine and vortexed. The plates were incubated at 37°C for 24 h, and viable bacterial colonies were recorded for biocidal efficacy analysis. Results showed that cotton fabric coated with N-halamine siloxanes with a range of chlorine loading from 0.23% to 0.25% effectively inactivated *Escherichia coli.* Another study by Sun and Worley (2005), examined the durability and stability of the antimicrobial properties on the fabric after being treated by halamines. The stability of N-Cl bonds which is formed by replacing hydrogen on the N-H moiety contributes to the stability of the antimicrobial properties on the fabric. In the study the antimicrobial properties of the finished fabrics were evaluated using gram-positive and gram-negative bacteria, funguses, yeasts, and viruses following AATCC Test Method 100. Results showed reductions of microorganisms to 90% to 99.9%.

2.3.6. Quaternary ammonium compounds

Quaternary ammonium compounds are salts of quaternary ammonium cations with an anion. They are used as disinfectants, surfactants, fabric softeners, and as antistatic agents. Certain long alkyl chain quaternary ammonium compounds are used as antimicrobials and disinfectants. Examples are benzalkonium chloride, benzethonium chloride, methylbenzethonium chloride, cetalkonium chloride, cetylpyridinium chloride, cetrimonium, cetrimide, dofanium chloride, tetraethylammonium bromide, didecyldimethylammonium chloride and domiphen bromide. Quaternary ammonium compounds are good against fungi, amoeba, and enveloped viruses, and act by disrupting the cell membrane and proteins.

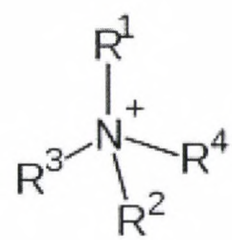

Figure 7. Quaternary ammonium cation. Any or all of the R groups may be the same or different alkyl groups. Also, any of the R groups may be connected.

In a study by Son, Lee, Ravikumar, and Kim (2006), two quaternary ammonium salts (QAS) were investigated namely, Cetylpyridinum chloride (CPC) and Benzyldimethylhexadecyl ammonium chloride (BDHAC) which are widely accepted as strong antimicrobial agents. The antimicrobial properties were quantitatively evaluated using *Staphylococcus aureus*, according to AATCC test method 100-1999. The antimicrobial activity of the reactive anionic agent modified cotton substrates treated with CPC and BDHAC was evaluated. The colonies of the bacterium on the agar plate were counted and the reduction in numbers of the bacterium was calculated. Results obtained show that the treated fabric shows higher antimicrobial activity compared with the untreated fabric.

2.4. Conclusion

The literature review indicated that various finishing products and applications were available to improve the protective properties of fabrics and they could potentially impart protective property to lyocell fabric as well. The present study was an attempt to investigate the potential of a few of these agents to impart UV and antimicrobial resistance to lyocell fabric via single and multi-functional finishing processes.

CHAPTER 3

MATERIALS AND METHODS

The purpose of this study was to investigate antimicrobial and UV properties of untreated lyocell fabric; to improve the antimicrobial properties via finishing treatment with an antimicrobial agent; to enhance the UV protection characteristics via a finishing treatment with UV absorbers; and to study the feasibility of a multi-functional UV and antimicrobial finishing treatment. Additionally, other properties of treated lyocell were also studied such as the durability of multi-functional UV treatment to washing and light exposure.

The materials and methods chapter is presented in two parts. The first section on materials includes a description of the materials used in this study. Section two discusses the methods used to evaluate the lyocell fabric for antimicrobial and UV properties.

3.1. Materials

3.1.1. Substrate

The fabric used in this study was lyocell woven fabric. Fabric characteristics are shown in Table 2. The woven lyocell fabric was generously provided by Lenzing AG (Lenzing, Austria).

Table 2. Fabric Characteristics

Characteristic	Description
Weave	Plain
Processing	Singed and Desized
Fiber count	1.3 dtex
Warp / inch	13
Filling/inch	11
Spinning type	Ring Spinning

3.1.2. UV Absorbers

Rayosan® (Clariant Corporation, Charlotte, NC) and UV-Sun Cell® (Huntsman Textile Effects, Charlotte, NC) were the two UV absorbers used to increase the ultraviolet protection factor of fabrics.

3.1.3. Antimicrobial Agent

The antimicrobial agent used was Tinosan® (Ciba Specialty Chemicals, High Point, NC).

3.1.4. Test Organisms

Staphylococcus aureus (ATCC® 6538, MicroBioLogics), *Enterococcus faecalis* (ATCC® 51299, MicroBioLogics) and *Escherichia coli* (ATCC® 25922, PML Microbiologicals) were the three microorganisms that were used in this study. They are among the three most common bacterial isolates found in medical environments and have been identified as the major causes of cross infections. *S. aureus* and *E. faecalis* are gram-positive bacteria and *E. coli* is a gram-negative rod-shaped bacteria.

3.2. Methods

3.2.1. UPF measurement

Ultraviolet Protection Factor (UPF) represents the amount of Ultra Violet (UV) protection provided by a textile substrate. UPF values are similar to SPF values provided by sunscreens, the difference being the SPF values are determined *in-vivo* whereas UPF values are based *in-vitro*. The UPF evaluates the reduction in the amount of the UV radiation that passes through the fabric to the skin. For example, when a fabric has an UPF of 20, only 1/20th of UV radiation reaches the skin. Higher values are better because it prolongs the time before the skin beneath the sample turns red and this in turn means a person can stay in the sun for a longer time. An effective UVR dose (ED) for unprotected skin is calculated by rotating the incident solar spectral power distribution with the relative spectral effectiveness function and summing over the wavelength range 290-400 nm. The UPF was measured using a labsphere® UV-100F Ultraviolet Transmission Analyzer and calculated using Equation 1

$$\text{UPF=ED}\frac{ED}{EDm} = \frac{\sum_{400}^{290} E_\lambda S_\lambda \Delta\lambda}{\sum_{400}^{290} E_\lambda S_\lambda T_\lambda \Delta\lambda} \qquad \text{Equation 1}$$

In equation 1, E_λ corresponds to the erythema sensitivity of the average human skin, S_λ is the spectral irradiance of terrestrial sunlight under controlled conditions, T_λ is the transmission spectrum of the garment, λ denotes the wavelength of the UV radiation (between 290 and 400). The numerator of the above equation describes the quantity of the UV radiation, which reaches the skin if unprotected. The denominator describes the quantity of the UV radiation reaching the skin protected by a fabric (Shuierer, 1997).

3.2.2. Treatment with UV absorber

The UV absorbers were applied by the exhaust method. Concentration of UV absorber was varied from 1 to 4% (on weight of fabric). The fabric samples were introduced in the bath containing the UV absorber and circulated for 10 minutes. Glauber's salt (60 g/L) was gradually added and the temperature of the bath was raised to 70°C. Four milliliters per liter caustic soda was then added and application continued further. Total treatment time was 60 minutes. Finally, the bath was cooled and the fabric samples were rinsed and air dried.

3.2.3. Treatment with antimicrobial agents

Samples (15 cm x 15 cm) from the three substrates were treated with the antimicrobial agent. Tinosan® was applied to the samples using an Ahiba Nuance ECO-B infrared machine. Samples were introduced in the treatment beakers at room temperature at a material to liquor ratio of 1:50. Temperature was raised to 130^{0}C and treatment continued for 90 minutes. The treated samples were then rinsed in deionized water and air dried.

3.2.4. Qualitative Evaluation

Qualitative evaluation of treated fabrics was done by AATCC Test Method 147: Antibacterial Activity Assessment of Textile Materials: Parallel Streak Method. In this method five streaks of the microorganisms are inoculated on the nutrient agar plates. Fabric samples of size 25mm x 50mm are placed in intimate contact with the bacteria inoculated agar. The plates are placed in incubators at 37C for 24 hours and then after 24 hours the plates were observed for presence of clear area of interrupted growth underneath and adjacent to the test fabric which gives an indication of the antibacterial activity of the fabric. The zone of inhibition for the samples was calculated using equation 2.

$$W = \frac{(T - D)}{2} \qquad \text{Equation 2}$$

Where:

W= width of clear zone of inhibition, mm

T= total diameter of test specimen and clear zone, mm

D= diameter of the test specimen, mm

3.2.5. Quantitative Evaluation

Quantitative evaluation was done by enumerating the colony forming units (CFU) using a Reichert Darkfield Quebec Colony Counter and calculating the percent reduction in bacteria using equation 3.

$$R\% = \frac{(B - A)}{B} \times 100 \qquad \text{Equation 3}$$

Where:

R= percent reduction in bacteria

A= CFU for treated fabric

B= CFU for untreated (control) fabric

3.2.6. Durability of treated fabrics to laundering

Treated fabrics were subjected to laundering according to AATCC Test Method 61. The fabrics were laundered at 40 °C for 45 minutes in an Atlas Launder-Ometer with an M:L ratio of 1:50. The antibacterial properties and UV properties of the laundered fabrics were then quantitatively evaluated.

3.2.7. Durability of treated fabrics to light

Treated fabrics were subjected to light fastness by exposing the samples in an Atlas Sun Test XLS+ Weatherometer chamber with the following parameters: Black Standard Temperature (BST) of 63 °C, phase time of 300 minutes, irradiance (E) of 500 W/m^2, and final dosage of 9000 kJ/m^2. The samples were exposed front and back in the chamber on successive days. The antibacterial and UV properties of the light exposed samples was then quantitatively evaluated.

CHAPTER 4

RESULTS AND DISCUSSION

4.1. UV property of untreated lyocell fabric

The first part of the study was to determine the UV property of untreated lyocell fabric. Lyocell fabric as supplied by the manufacturer had a UPF value of 0.6. Based on Table 1 (page 12), a UPF value below 15 indicates the fabric offers no protection against UV radiation.

4.2. Antimicrobial property of untreated lyocell fabric

Qualitative and quantitative evaluation was done for analyzing the antimicrobial property of untreated lyocell fabric. Qualitative evaluation showed the mean zone of inhibition for untreated lyocell fabric to be zero millimeters for all microorganisms which mean there was microbe growth adjacent to and underneath the fabric. Representative pictures are shown in Figures 8, 9, and 10 for *S. aureus, E. faecalis* and *E. coli* respectively.

Quantitative evaluation was measured by enumerating the number of colony forming units (CFU). The mean number of colony forming units (CFU) was 829 for *S. aureus* and 2434 for *E. faecalis*. The extremely high values for CFU indicates that untreated lyocell fabric has no antimicrobial activity against *S. aureus, E. faecalis* and *E.coli*.

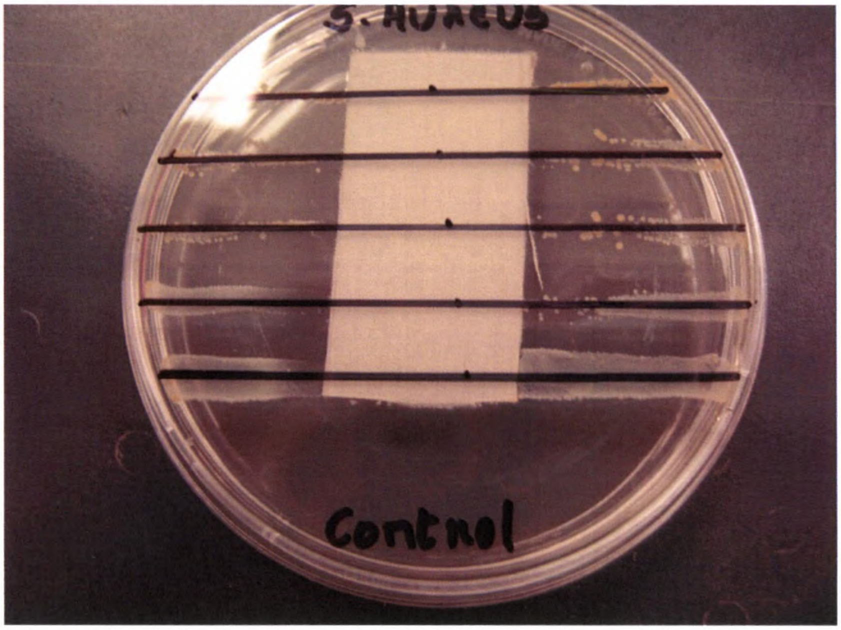

Figure 8. Zone of inhibition of untreated lyocell fabric against *S. aureus*

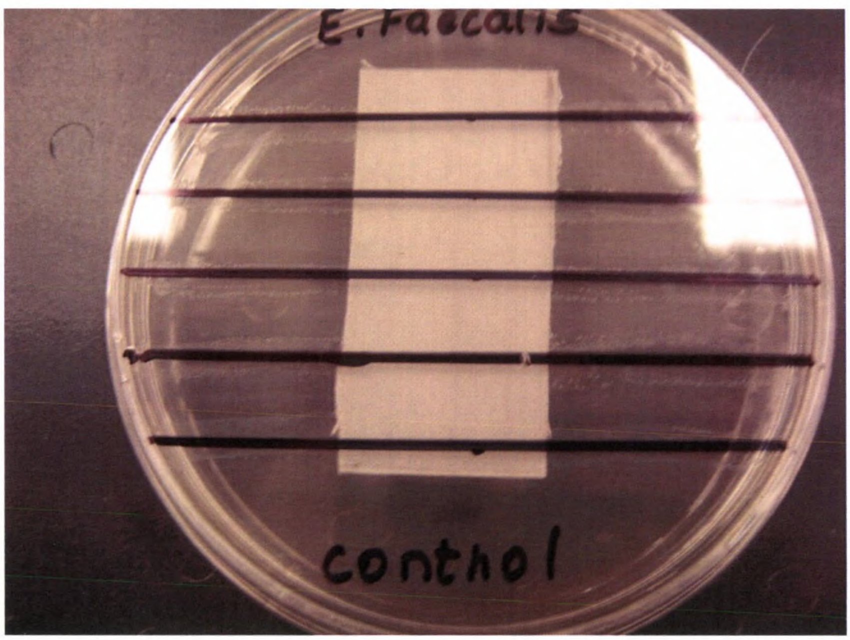

Figure 9. Zone of inhibition of untreated lyocell fabric against E. *faecalis*

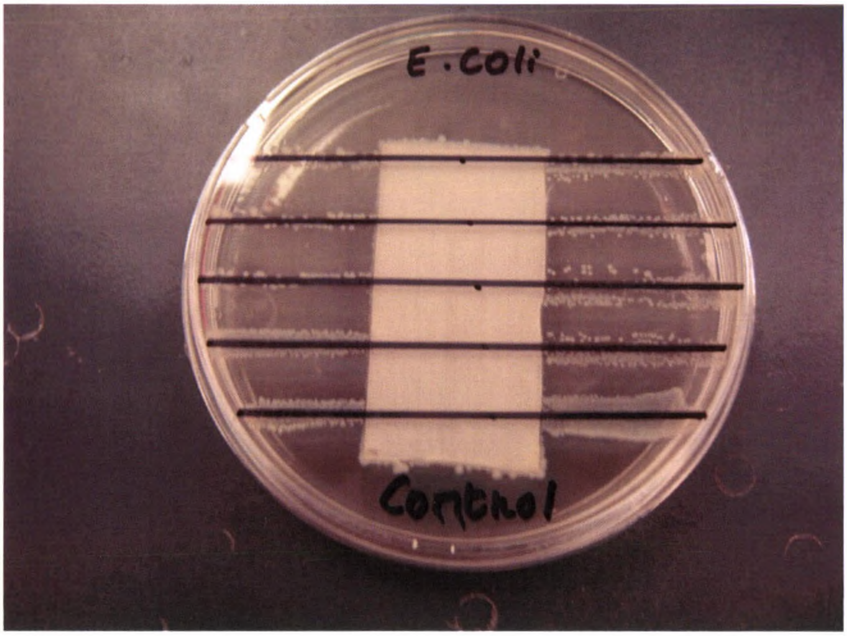

Figure 10. Zone of inhibition of untreated lyocell fabric against *E. coli*

4.3. UV protective characteristics of lyocell after finishing with UV absorbers

Since untreated lyocell fabric had no UV protective properties, lyocell was treated with two commercial UV-absorbers viz. Rayosan® and UV-Sun Cell®. Results obtained are shown in Table 3.

Table 3. UPF values of lyocell fabric after finishing with UV- absorbers

UV Absorber	Concentration (%)	Rep I	Rep II	Rep III	Mean UPF	UVA transmission (%)	UVB transmission (%)
Rayosan®	0% owf	0.6	0.6	0.6	0.6	225.2	151.2
	1% owf	16.9	14.9	14.5	15.4	28.6	2.8
	2% owf	15.1	15.7	15.8	15.5	28.2	2.8
	3% owf	16.9	18.2	16.9	17.4	23.3	2.7
	4% owf	18.2	17.5	17.0	17.6	26.6	2.5
UV-Sun Cell®	0% owf	0.6	0.6	0.9	0.7	212.9	136.5
	1% owf	46.4	46.7	44.5	45.9	4.6	1.9
	2% owf	**52.9**	**52.6**	**50.4**	**51.9**	**3.4**	**1.8**
	3% owf	55.7	54.8	55.4	55.3	2.9	1.7
	4% owf	80.4	81.8	81.8	81.8	1.8	1.2

An effective UPF value for fabrics should be 50 or more. Rayosan® was marginally effective in that UPF values on treating with 1%, 2%, 3% and 4% owf varied between 15.4 to 17.6 which falls in good UV protection based on Table 1 (page 12). As opposed to Rayosan® treatment, a 2% owf treatment with UV-Sun Cell® gave a mean UPF value of 51.9 which falls in the category of "Excellent UV protection". A 2% treatment with an UV-absorber such as UV-Sun Cell® can therefore be taken as an optimum concentration to be applied on lyocell fabric for high UPF value and hence excellent protection against

UV-radiation. The relationship between UV absorber concentration and ultraviolet protection factor for lyocell fabric treated with UV-Sun Cell® at 1%, 2% and 3% is illustrated by a graph as depicted in figure 11.

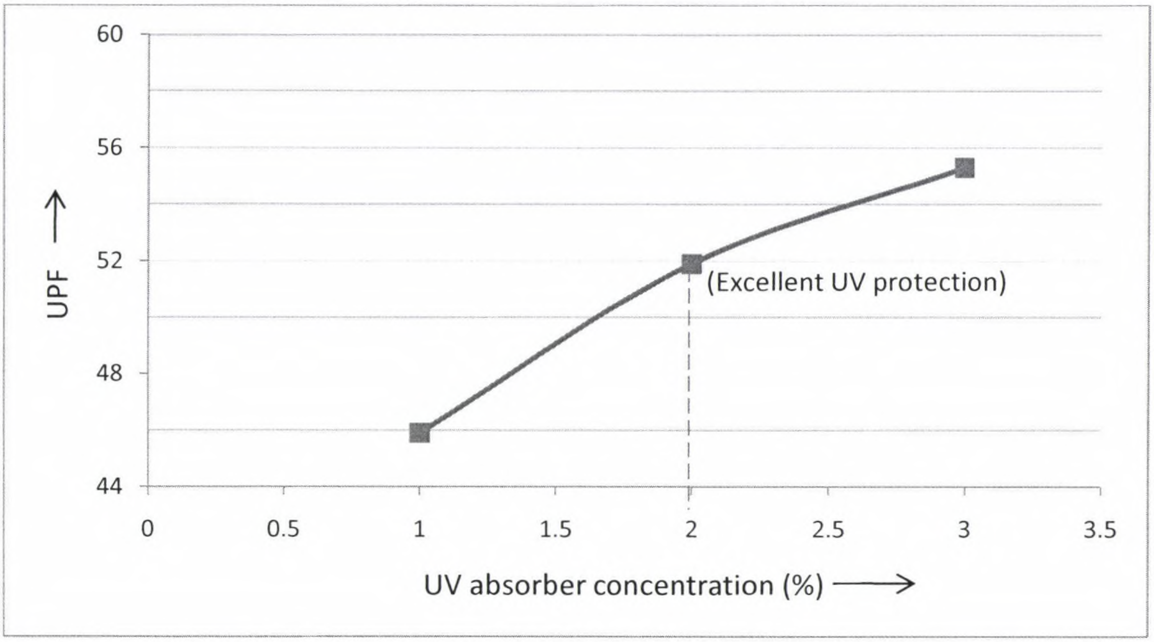

Figure 11. Relationship between UV absorber concentration and UPF for lyocell fabric treated with UV-Sun Cell®

The data in Figure 11 were also used to derive an equation (Equation 4) via least square regression for potentially predicting UV protection factor without the need for instrumental UPF measurement.

UPF=4.27 (UV Absorber concentration) +0.4280 Equation 4

Equation 4 was tested for its robustness in predicting UPF and the results are included in Table 4.

Table 4. Predicting UPF values via a regression equation

UV-absorber concentration (% owf)	Experimental UPF	UPF (Predicted)
1%	45.9	47.1
2%	51.9	51.3
3%	55.3	55.2
5%	64.1	64.1
7.5%	72.1	74.9

The results show that a regression equation of the form shown in Equation 4 can be used fairly well in predicting UPF, since both the experimental and predicted values fall in the same broad range of UPF protection categories.

Since 2% UV-Sun Cell® was the optimum concentration of UV-absorber for excellent UPF values, fabrics treated with this concentration were studied for their durability against laundering and light exposure. The results are shown in Table 5.

Table 5. UPF values of 2% owf UV-Sun Cell® treated lyocell fabric after laundering and exposure to light

	UPF values before laundering and light exposure	**UPF (after laundering)**	**UPF (after light exposure)**
Replication I	52.9	51.5	52.9
Replication II	52.6	50.6	50.3
Replication III	50.4	51.5	51.6
Mean	**51.9**	**51.2**	**51.6**
UVA transmission (%)	3.6	4.5	3.5
UVB transmission (%)	1.8	1.7	1.8

Laundering and exposure to light does not diminish the UV protection of the lyocell substrates. Two percent UV-Sun Cell® is therefore proven to be the optimum concentration of UV –absorber for lyocell fabric; both in terms of protection against UV-radiation and durability to laundering and light exposure.

4.4. Antimicrobial properties of lyocell after treatment with an antimicrobial agent

Untreated lyocell fabric had no protection against microorganisms as reported in section 4.2. Lyocell was therefore treated with Tinosan® a commercially available antimicrobial agent.

4.4.1. Qualitative determination of antimicrobial activity of treated fabrics

Qualitative evaluation of the anti-microbial treated samples was done by AATCC Test Method 147. The mean zones of inhibition of lyocell treated with Tinosan® against three bacteria at various concentrations studied are shown in Table 6. A treated sample exhibiting a zone of inhibition greater than 2 mm was considered as having good antibacterial property.

Table 6. Mean zones of inhibition of untreated fabric and Tinosan® treated lyocell substrates against *S. aureus* , *E. faecalis* and *E.coli*

Antimicrobial Concentration (%)	**Mean zone of inhibition (mm)**		
	S. Aureus	***E. faecalis***	***E. coli***
0.0%	0	0	0
0.5%	**10**	**3**	**6**
1%	10	6	7
1.5%	10	8	10
2%	10	10	10
2.5%	10	10	10

As is clearly evident from Table 6, the mean zone of inhibition for lyocell fabric against all the three bacteria are greater than 2mm clearly indicating that Tinosan® treated substrates possess excellent antibacterial activity. When the antibacterial effect of the substrate is considered in terms of concentration, the lowest concentration of 0.5% can be considered sufficiently effective as the zone of inhibition is higher than the required minimum of 2mm. Photographs illustrating the effectiveness of Tinosan® as an antibacterial agent against the three bacterial are shown in Figures 12, 13, and 14 for *S. aureus, E. faecalis* and *E. coli* respectively. Compared to Figures 8, 9 and 10 it is seen that there is no growth of bacteria adjacent to and beneath the fabrics.

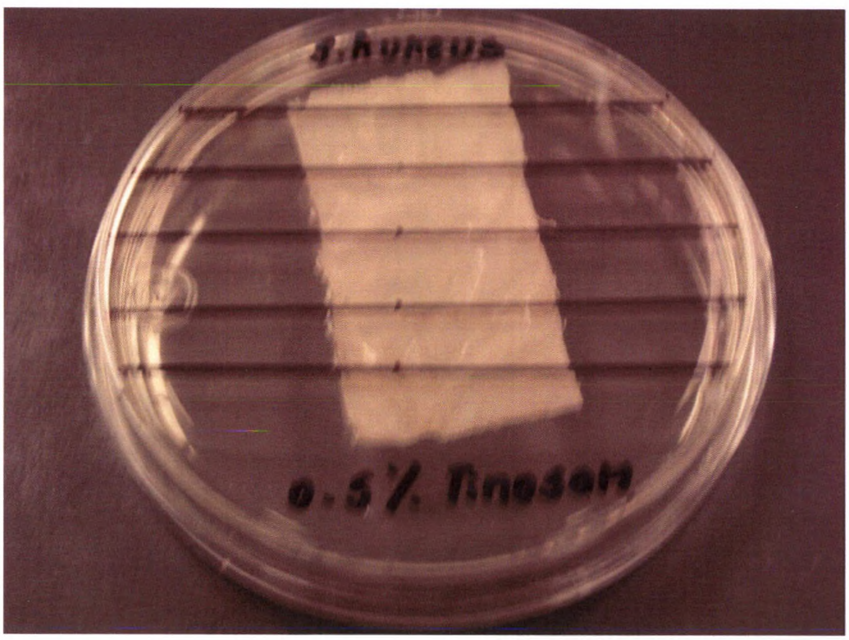

Figure 12. Zone of inhibiton of Tinosan® treated lyocell (0.5%) against *S. aureus*

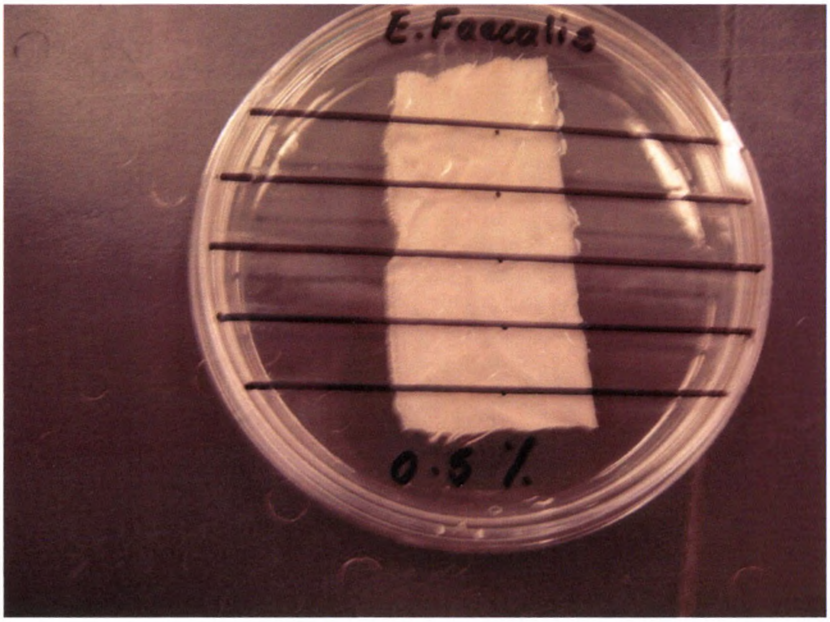

Figure 13. Zone of inhibiton of Tinosan® treated lyocell (0.5%) against *E. faecalis*

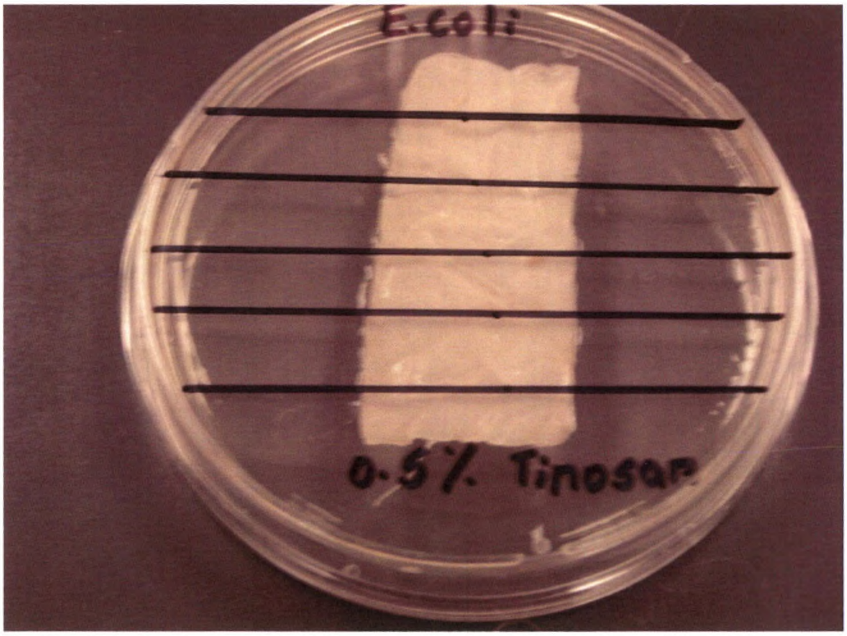

Figure 14. Zone of inhibiton of Tinosan® treated lyocell (0.5%) against *E. coli*

4.4.2. Qualitative evaluation of Tinosan® treated lyocell fabric after laundering and light-exposure

To test the durability of the antimicrobial treatment, the treated samples were laundered and exposed to light under conditions described previously. Qualitative evaluation of the laundered and light exposed samples was repeated. Zones of inhibition of the laundered and light exposed fabric samples against *S. aureus* and *E. faecalis* are illustrated in Figure 15 through Figure 18.

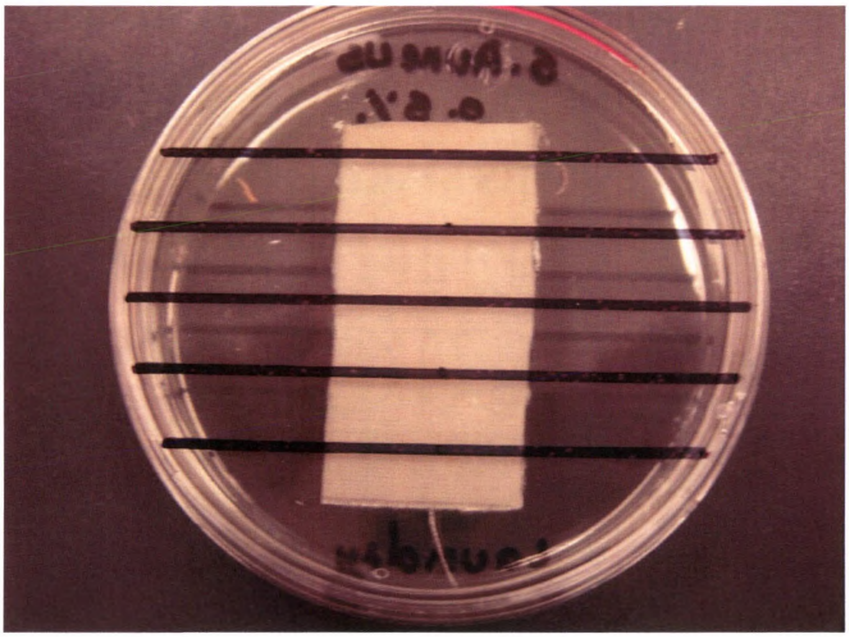

Figure 15. Zone of inhibition of Tinosan® treated (0.5%) lyocell fabric after laundering against *S. aureus*

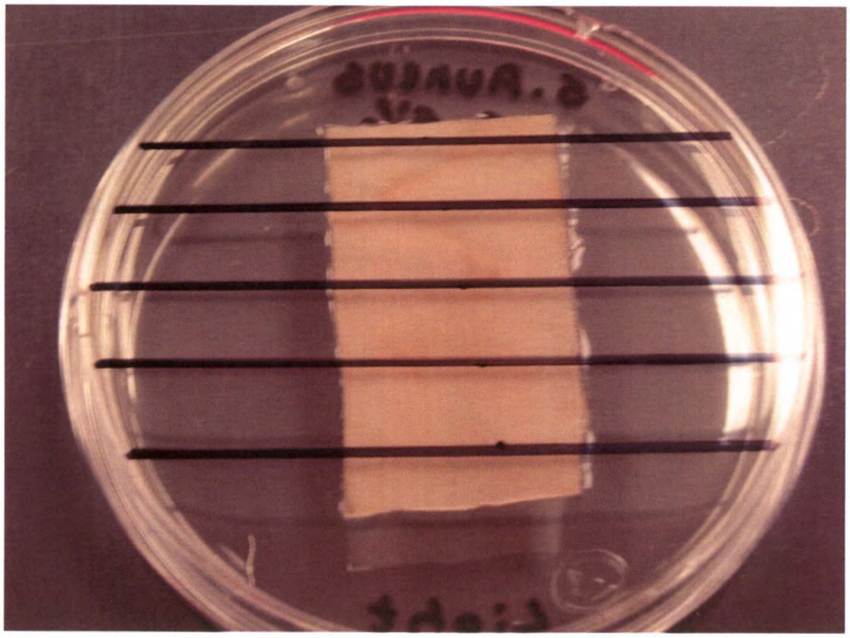

Figure 16. Zone of inhibition of Tinosan® treated (0.5%) lyocell fabric after light exposure against *S. aureus*

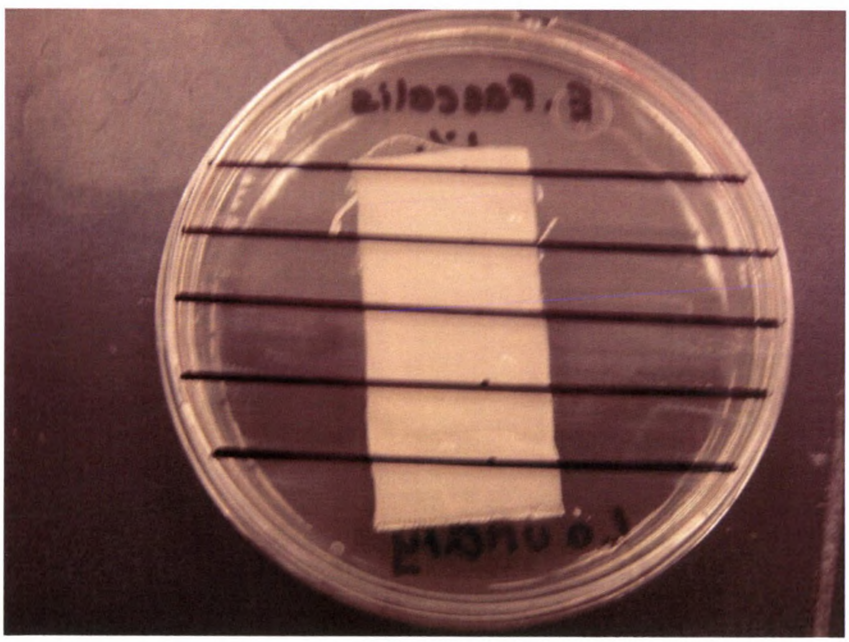

Figure 17. Zone of inhibition of Tinosan® treated (1%) lyocell fabric after laundering against *E. faecalis*

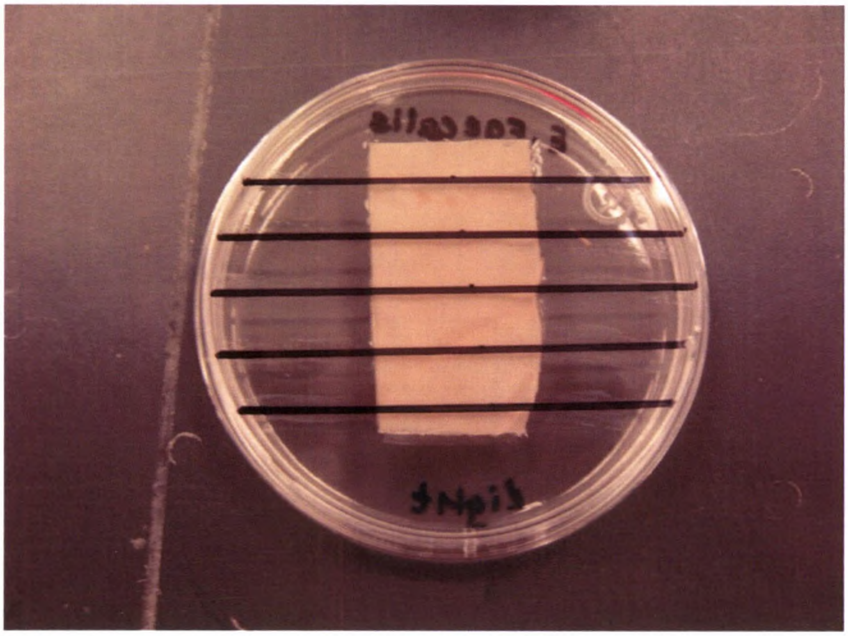

Figure 18. Zone of inhibition of Tinosan® treated (1%) lyocell fabric after light exposure against *E. faecalis*

The zones of inhibition for the laundered and light exposed samples clearly show that post-laundering and post-light exposure, lyocell retained its antimicrobial activity against *S. aureus* and *E. faecalis* indicating sufficient durability to care procedures and weathering conditions. However, lyocell did not retain good antimicrobial activity after laundering and exposure to light to *E.coli*. A possible explanation for the apparent low antimicrobial activity against *E.coli* is that, being a gram-negative bacterium, *E. coli* has a stronger outer wall which possibly restricts the antimicrobial agent from penetrating and killing the bacteria efficiently.

4.4.3. Quantitative evaluation of antimicrobial activity of treated fabrics

Accurate quantitative evaluation of antibacterial activity was done to determine the percentage reduction in bacterial population for Tinosan® treated samples as compared to an untreated control. Quantitative evaluation was repeated after laundering and after exposure to light. Antibacterial activity of Tinosan® treated fabric and control fabric after treatment, after laundering and on exposure to light are shown in Tables 7 and 8 against *S. aureus* and *E. faecalis* respectively.

Table 7. Quantitative evaluation of Tinosan® treated lyocell against *S. aureus*

Fabric Samples	**Tinosan® Treated**		**Laundered**		**Light exposed**	
	Number of Colony forming units (CFU)	Reduction in bacteria (%)	Number of Colony forming units (CFU)	Reduction in bacteria (%)	Number of Colony forming units (CFU)	Reduction in bacteria (%)
Untreated control(1)	942	-	1129	-	1059	-
Tinosan (0.5%)	0	100	0	100	0	100
Untreated fabric(2)	840	-	1824	-	1533	-
Tinosan (0.5%)	0	100	0	100	0	100
Untreated fabric(3)	704	-	1533	-	1824	-
Tinosan (0.5%)	0	100	0	100	1	99.9

Table 8. Quantitative evaluation of Tinosan® treated lyocell fabric against *E. faecalis*

Fabric Samples	Tinosan® Treated		Laundered		Light exposed	
	Number of Colony Forming Units (CFU)	Reduction in bacteria (%)	Number of Colony Forming Units (CFU)	Reduction in bacteria (%)	Number of Colony Forming Units (CFU)	Reduction in bacteria (%)
Untreated control(1)	2548	-	1442	-	1442	-
Tinosan® (1%)	48	98.1	0	100	7	99.5
Untreated fabric(2)	2258	-	1749	-	1749	-
Tinosan® (1%)	10	99.5	48	97.2	19	98.9
Untreated fabric(3)	2496	-	1120	-	1120	-
Tinosan® (1%)	8	99.6	25	97.7	16	98.5

As Table 7 and Table 8 clearly indicate, the percentage reduction in bacteria is 98% to 100% for all fabrics treated with Tinosan® at a concentration of 0.5% on weight of the fabric. Tinosan® is therefore, a highly effective antimicrobial agent for lyocell fabric.

4.5. Multi-Functional Finishing

Combining several processing steps to reduce time and cost is preferred in the textile industry so the synergistic effect of UV absorber treatment and antimicrobial treatment from a multi- functional treatment bath was explored. Lyocell fabrics were treated with the optimum amount of UV-absorber and antimicrobial determined from previous experiments. (2% owf UV-Sun Cell® and 0.5% owf Tinosan®)

Table 9 lists the ultraviolet protection factor values of lyocell fabric after multi-functional finishing, post laundering and after light exposure.

Table 9. UPF values of lyocell fabric after multi-functional finishing, post laundering and after light exposure

Replications	UPF	UPF(after Laundering)	UPF(after light exposure)
I	52.3	55.6	53.1
II	54.5	54.9	53.9
III	51.9	53.2	58.3
Mean	**52.9**	**54.6**	**55.1**
Mean UVA transmission(%)	4.2	4.4	2.8
Mean UVB transmission(%)	1.8	1.7	1.7

For purposes of comparison, Figure 19 compares the UPF values of lyocell fabric when the UV-absorber was applied from a single bath to the UPF values when the UV-absorber was applied from a multi-functional bath. As the figure shows, UPF values are not adversely impacted when a multi-functional bath is employed. Hence, UV-absorbers can safely be used in a multi-functional bath with no decrease in protection from UV-radiation.

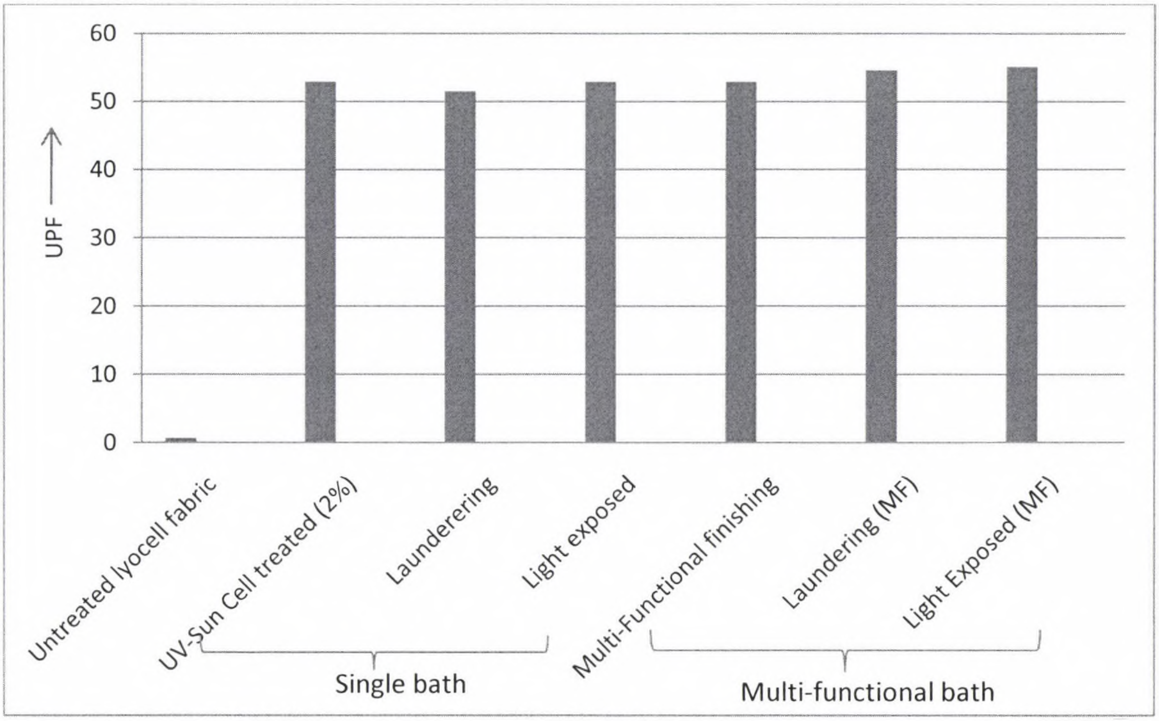

Figure 19. UPF values (Single bath versus multi-functional bath)

Table 10 and Table 11 show the antibacterial activity of lyocell fabric after multi-functional finishing, post laundering and on exposure to light against *S. aureus* and *E. faecalis* respectively.

Table 10. Antibacterial activity of multi- functional treated lyocell fabric against *S. aureus*

Fabric ID	Multi-functional finishing		Antimicrobial activity after laundering		Antimicrobial activity after Light exposure	
	Number of CFU	Reduction in bacteria (%)	Number of CFU	Reduction in bacteria (%)	Number of CFU	Reduction in bacteria (%)
Untreated control(1)	1952	-	2582	-	1850	-
Multi-functional	0	100	5	99.8	0	100
Untreated fabric(2)	2012	-	2150	-	2582	-
Multi-functional	0	100	11	99.5	10	99.6
Untreated fabric(3)	1945	-	1850	-	2150	-
Multi-functional	0	100	0	100	8	99.6

Table 11. Antibacterial activity of multi- functional treated lyocell fabric against *E. faecalis*

Fabric ID	Multi-functional finishing		Antimicrobial activity after laundering		Antimicrobial activity after light exposure	
	Number of CFU	Reduction in bacteria (%)	Number of CFU	Reduction in bacteria (%)	Number of CFU	Reduction in bacteria (%)
Untreated control(1)	2480	-	2944	-	2894	-
Multi-functional	49	98.0	10	99.6	130	95.5
Untreated fabric(2)	2480	-	2448	-	2448	-
Multi-functional	102	95.8	80	96.7	10	99.6
Untreated fabric(3)	2480	-	2894	-	2944	-
Multi-functional	74	97.7	40	98.6	27	99.1

The data presented in Tables 10 and 11 show that the percentage reduction in bacteria is not impacted on application and treatment from a multi-functional bath. A reduction in bacterial populations greater than 95% is an indication of excellent antimicrobial activity and against both *S. aureus* and *E. faecalis* the antimicrobial activity was retained when antimicrobial finishing treatment was combined with an UV-resistant treatment.

Chapter 5

CONCLUSIONS AND RECOMMENDATIONS

Untreated lyocell fabric as supplied by the manufacturer, had no protection against UV radiation. However, UV-protection was easily imparted by finishing with a UV-absorber. When treated with the UV-absorber, UV-Sun Cell®, lyocell showed excellent UV protective properties. At a concentration of 2% (owf) UV-Sun Cell®, lyocell had an UPF value of 51.9, which falls in the excellent UV protection category as clothing items with a UPF value above 50 can be labeled UPF 50+ (Federal Trade Commission) (Morley, 2000). Additionally, UV-absorber treated lyocell was found to be durable to laundering and light exposure.

Untreated lyocell fabric had no antimicrobial activity against the three bacteria used in the study. Lyocell on treatment with the antimicrobial agent Tinosan®, showed a 98-100% reduction in microbes. Tinosan® is, therefore, a highly effective antimicrobial agent for lyocell fabric against *Staphylococcus aureus* and *Enterococcus faecalis*. Qualitative results showed that a concentration of 0.5% (owf) was sufficient to impart antimicrobial properties to lyocell. In agreement with the qualitative results, quantitative studies confirmed the effectiveness of Tinosan® as an excellent antimicrobial agent for lyocell fabric. The antimicrobial efficacy of Tinosan® on lyocell fabric was also found to be durable to laundering and to light exposure.

Multi-functional finishing was done to reduce cost and time by treating lyocell fabric with an UV absorber (UV-Sun Cell®) and an antimicrobial agent (Tinosan®) from one bath. From the results of this study, a bath containing 2% UV-Sun Cell® and 0.5

Tinosan® on weight of the fabric was the optimum combination for multi-functional finishing. The data showed no adverse effect on either the UV-protective property or the antimicrobial protective property of lyocell fabric after multi-functional treatment. The protective properties were also retained after laundering and exposure to light. This is the first study to demonstrate the effectiveness of finishing treatments to enhance protective properties of lyocell fabric.

Recommendations for future study:

- Investigate additional UV absorbers and antimicrobial agents with different chemistries on lyocell fabric.
- Study the effectiveness of Tinosan® treated lyocell fabric against other bacteria and fungi such as *Vibrio cholera*, *Klebsiella pneumonia, Aspergillus fumigates* and *Candida albicans.*
- Study the feasibility of multi-functional finishing treatments on other textile substrates.

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