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Durable and reusable antimicrobial textiles

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DURABLE AND REUSABLE ANTIMICROBIAL TEXTILES

A Thesis
Submitted to the Graduate Faculty of the
Louisiana State University and
Agricultural and Mechanical College
In partial fulfillment of the
Requirements for the degree of
Master of Science

in

The School of Human Ecology

by
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Authors Note

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Table of Contents

| | |
|-----------------------------------------------------------------|------|
| Acknowledgments..... | ii |
| Author's Note..... | iii |
| List of Tables..... | vi |
| List of Figures..... | vii |
| Abstract..... | viii |
| Chapter 1 Introduction..... | 1 |
| 1.1 Research Purpose and Objectives..... | 2 |
| 1.2 Hypotheses..... | 3 |
| 1.3 Definitions..... | 3 |
| Chapter 2 Review of Literature..... | 5 |
| 2.1 Why We Need Antimicrobial Textiles..... | 5 |
| 2.2 Opposition to Antimicrobial Textiles..... | 8 |
| 2.3 Types of Antimicrobial Finishes..... | 9 |
| 2.4 Why Use Cotton?..... | 12 |
| 2.5 Summary..... | 13 |
| Chapter 3 Methodology..... | 14 |
| 3.1 Materials and Equipment..... | 14 |
| 3.2 Experiment Design..... | 14 |
| 3.3 Chemical Procedure..... | 15 |
| 3.4 Fabric Characterization..... | 16 |
| 3.4.1 Mechanical Testing..... | 16 |
| 3.4.2 Physical Testing..... | 18 |
| 3.4.3 Antimicrobial Testing..... | 20 |
| 3.4.4 Durability Testing..... | 20 |
| 3.5 Data Analysis..... | 21 |
| Chapter 4 Results and Discussion..... | 22 |
| 4.1 Tensile Performance..... | 22 |
| 4.2 Bending Performance..... | 22 |
| 4.3 Shearing Performance..... | 23 |
| 4.4 Compression Performance..... | 24 |
| 4.5 Surface Friction and Roughness..... | 25 |
| 4.6 Subjective Analysis..... | 26 |
| 4.7 Durability of Antimicrobial Treatments..... | 26 |
| Chapter 5 Conclusions and Suggestions for Further Research..... | 29 |
| 5.1 Conclusions..... | 29 |
| 5.2 Suggestions for Further Work..... | 30 |

| | |
|------------------------------|----|
| References Cited..... | 32 |
| Appendix: Research Data..... | 34 |
| Vita....., | 44 |

List of Tables

| | |
|---------------------------------------------------------------------|----|
| 4-1 Average Means and Standard Deviations for Tensile..... | 22 |
| 4-2 Average Means and Standard Deviations for Bending..... | 23 |
| 4-3 Average Means and Standard Deviations for Shearing..... | 24 |
| 4-4 Average Means and Standard Deviations for Compression..... | 25 |
| 4-5 Average Means and Standard Deviations for Surface Friction..... | 26 |

List of Figures

| | |
|----------------------------------------------|----|
| 3-1 Tensile and Shearing machine..... | 18 |
| 3-2 Bending machine..... | 18 |
| 3-3 Compression machine..... | 19 |
| 3-4 Surface testing machine..... | 20 |
| 4-1 Assay using samples washed 25 times..... | 27 |
| 4-2 Assay using samples washed 10 times..... | 28 |

Abstract

Antimicrobial textiles are a large research focus in the textile industry. There is an apparent need for creating reusable and durable antimicrobial textiles. Most of the textiles with antimicrobial properties effective against *Staphylococcus aureus* are disposable.

To address the issue, two types of biopolymer solutions were created in a USDA lab. Both solutions displayed antimicrobial properties. A medium weight, plain weave cotton sheeting was used as the test fabric. Samples of the sheeting were cut, treated, and tested to determine their efficacy as antimicrobial textiles. The tests performed included tensile deformation, bending, shearing, compression, surface friction, surface roughness, and treatment durability. To compare the effect of the finish on the cotton sheeting, untreated samples were also tested.

Results showed that the antimicrobial finishes negatively affected most of the fabric properties. Tensile, bending, and shearing were greatly affected by the treatments while compression and surface friction and roughness showed only slight impairments. The samples that were washed up to 25 times could still retain certain microbial resistance. The samples washed by 50 times showed no antimicrobial properties. Overall, the treatments were not effective to be used as antimicrobial finishes on plain weave cotton fabrics.

Chapter 1 Introduction

Over the past century, much focus has been placed on the sustainability of the earth's environment. This concern, accompanied by a recent boom in the interest of healthy living, has influenced many research projects. Because textiles play such a large part in the daily lives of humans, many of these projects are based on enhancing the properties of textiles. Fabrics that are fire resistant, wrinkle resistant, and stain repellent are already on the market while other property enhancing treatments, such as UV protective and antimicrobial, are being researched.

Antimicrobial textiles have been tested for use in the medical industry for some time. Currently, the only antimicrobial textiles being used in the field of medicine are disposable and nonwoven. Some of the treatments being used are harmful to our environment not only because of the chemicals used in the treatments but also because the treated textiles are not reusable. To address the growing concerns about the environment, research should focus on the use of reusable textiles with durable finishes. By developing this type of textile, consumers are reducing the amount of chemicals and trash being disposed of in landfills, resulting in a negative effect on the environment.

With growth in world population and the spread of disease, the number of antibiotic resistant micro-organisms is rising along with the occurrence of infections from these micro-organisms. The need for antimicrobial textiles goes hand-in-hand with the rise in resistant strains of micro-organisms. Since the only antimicrobial textiles currently on the market are either disposable or used primarily for odor control, the availability of a reusable and durable antimicrobial textile effective against harmful

pathogens will not only be beneficial to both medical industry workers and patients but to the general public as well.

Just as with any other marketable product, a target market must be well defined. The group should be diverse yet aware of their need for this type of product. Antimicrobial textiles can be useful to recovering transplant patients, people with immunodeficiency viruses, and those with low immune systems such as premature babies. Garments treated with antimicrobial finishes can benefit these same customers if they are worn by those coming into close contact with them, such as roommates, home health care nurses, and parents of premature babies. Childcare workers and grade school teachers are also appropriate candidates for this target market. There is a need for this type of product.

1.1 Research Purpose and Objectives

The purpose of this research is to determine if an antimicrobial finish that is effective against *S. aureus* and will be active for up to 50 home launderings. This research will provide useful information on woven, reusable, antimicrobial fabrics that will be beneficial to the medical industry and the general public. Although research is well underway for producing reusable antimicrobial textiles in hospitals, there is a lack of research for this same type of textile in the apparel industry. Many people fit into the target market for this type of product even though hospital beds do not bind them.

The objective of this research is to determine if an antimicrobial finished textile that is effective against *Staphylococcus aureus* can be used in the medical industry as a durable textile. The tests are designed to characterize the antimicrobial treated textiles, chitosan and chitosan/PEG, in terms of mechanical properties such as bending, compression, shearing, and tensile strain along with surface properties such as surface

friction and roughness. The sample textiles will then be laundered to determine if they can be used as durable textiles. Last, the samples will be exposed to *Staphylococcus aureus* to determine if they have antimicrobial properties.

1.2 Hypotheses

- Both the chitosan treated and the chitosan plus PEG treated textiles will be effective against microbes.
- The chitosan solution will have a higher efficacy against microbes than the chitosan and PEG solution.
- The chitosan treated and chitosan plus PEG treated textiles will withstand 50 home launderings.
- The chitosan treated and chitosan plus PEG treatments will lose efficacy with each home laundering.
- The chitosan treated and chitosan plus PEG treatments will negatively affect the mechanical properties of the woven cotton.
- The treatments will not affect the physical properties of the woven cotton samples.
- The treated samples will have a different hand than the untreated control samples.

1.3 Definitions

Acetylation – the addition of an acetyl group to an organic compound.

Antibacterial – a descriptive term used to indicate harmful effects to bacteria.

Antimicrobial – a general term that is used to indicate that a product has a negative effect on the vitality of micro-organisms [18].

Biocidal – a descriptive term used to indicate that microbes are killed by the product.

Deacetylation – the removal of an acetyl group from an organic compound.

Durable – a finish that remains active for 50 or more launderings.

Functional finish – a treatment added to a textile to increase its value and functionality for the wearer.

Gram-negative bacteria – bacteria that is not dyed purple when treated with Gram's stain [1,4].

Gram-positive bacteria – bacteria that remain purple when treated with Gram's stain [1,5,6].

Microbes – the tiniest creatures not seen by the naked eye, such as bacteria, fungi, algae, and viruses [18].

Mechanical properties – those properties that affect the performance of the fabric.

Pathogen – a highly infectious organism or agent that produces disease in humans [12].

Physical properties – those properties that affect the feel of the fabric and have no effect on the performance.

Polyethylene Glycol (PEG) – a series of ethylene glycol polymers $H(OCH_2CH_2)OH_n$; used to promote good surface properties of test samples [14].

Reusable – a textile that is used, washed, and re-used for the life of the garment.

Please note that the terms textile and fabric are used interchangeably. Also used interchangeably are the terms treatment and finish.

Chapter 2 Review of Literature

With a rising interest in personal health and hygiene and a decrease in disposable time, the market for functional textiles is steadily increasing. Antimicrobial finishes are currently being used on disposable, nonwoven textiles for the medical industry. Presently, testing is being conducted to find safe and effective antimicrobial finishes for woven fabrics. The most important question for this research is, “Is an antimicrobial finished garment practical for everyday use by the average person?” A better understanding of antimicrobial finishes and textiles will aid in answering this question. Everyone should recognize why we need this type of textile and be aware of why antimicrobial textile use is opposed by some. There are many different types of antimicrobial finishes for textiles. Each finish serves a different purpose and targets a different group of bacteria or pathogens. Each of these finishes can be useful when applied to fabrics. It is important that the right fabric is chosen for both the treatment and the desired end use. Cotton was chosen for use in this study.

2.1 Why We Need Antimicrobial Textiles

Antimicrobial textiles have been in use for many years. The concern and need for protection against micro-organisms during World War II is what began the research race to find, or make, a suitable antimicrobial finish. One of the first antimicrobial textile finishes, used during World War II, was made to prevent cotton textiles, such as tents, tarpaulins, and vehicle covers, from rotting [18, 24]. At this point in time, the main concern for scientists was to preserve the textile. It was not until environmental protection became a universal concern did researchers realize the damage of the current antimicrobial finishes on our environment. Consequently, finishes began to evolve. Synthetic fibers were also a focus for the textile industry at this time [18, 24].

Experimentation with synthetics and antimicrobial finishes opened many doors for scientists.

As knowledge of functional finishes and manmade fibers evolved, so did society's view on health and safety. With this increase in health awareness, many people focused their attention on educating and protecting themselves against harmful pathogens. It soon became more important for antimicrobially finished textiles to protect the wearer from bacteria than it was to simply protect the garment from fiber degradation [24]. The media played a large role in bringing these concerns into the spotlight. A person cannot watch a television program without being exposed to advertisements about using Clorox® or Lysol® to clean household items and clothing.

A home is full of things for micro-organisms to live on. All textiles provide a growing environment for these micro-organisms. In fact, some finishes accelerate the growth of microbes [16, 18]. Natural fibers, such as cotton and wool, are especially susceptible to microbial growth and even dust mites because they retain oxygen, water, and nutrients [11, 16, 18]. Micro-organisms can embed themselves in clothes in a closet, curtains, carpets, bed, bath, and kitchen linens, and even pillows and mattresses. Many bacteria also live on the skin while dust mites live on shed human skin cells that have been deposited on items such as sheets, towels, and clothing [11, 18].

Like a house, a hospital contains an immense amount of textiles with the added threat of high volumes of traffic. Because of the constant flow of people, especially those with infectious diseases, many researchers have focused on creating finishes specifically for hospital use. Both patients and employees are at risk for cross-transmission of diseases and other health issues. Current medical protective wear,

such as gloves, masks, and gowns are insufficient in protecting the wearer against both air-borne pathogens and blood-borne viruses, like HIV/AIDS and hepatitis B. One researcher even attributed outbreaks of severe acute respiratory syndrome (SARS) in hospitals to the inadequacy of this protective gear [19]. The majority of these micro-organisms are passed from person to person by various textiles [20]. Previous research has shown that bacteria are able to live on hospital curtains for up to ninety days. This same research study claims that the costs of hospital acquired infections can reach \$4.5 billion per year [21]. The increasing rate of drug-resistant bacteria only heightens the importance of finding safe and durable antimicrobial finishes. The medical industry is not the only industry to have to deal with these threats.

Terrorist threats have become a top priority for militaries worldwide over the past decade. More recently, threats of biological warfare, like anthrax, have increased health concerns for both militaries and citizens [3]. While developing antimicrobial finishes to protect against chemical warfare is a life saving strategy, other industries require similar finishes to simply cater to their customers. Companies that produce clothing for outdoor recreation and sports aim to make the wearer more comfortable and preserve the integrity of their active wear. Odor control is a big concern for these companies. “Micro-organisms metabolize nutrients, such as sweat and soil present in textile products, producing odor causing intermediates that cause irritation” [16]. Controlling moisture is also a major concern for many manufacturing companies because micro-organisms only attack fibers when they are damp [16]. Moisture control is linked to odor control because the sweat that produces the odors can also increase the damage done to textiles by providing a moist environment for mildew to grow. Mildew damages a fabric by staining and discoloring the textile [11, 16, 18]. Health concerns along with

customer satisfaction have made functionally finished textiles a fast-paced and fast-growing industry.

Functional textiles include everything from antimicrobial finished textiles, to durable, or permanent press finished garments, to textiles with self-cleaning properties, and also textiles with nanotechnology [15]. The global market for hi-tech textiles has grown exponentially. Over \$106.9 billion in technical textiles were sold in 2005 [15]. It was estimated that the global economy saw a \$20.4 billion increase in the sales of hi-tech textiles in 2006 alone [15]. We live in a society of ease and functionality and scientific research prospers from this trend.

2.2 Opposition to Antimicrobial Textiles

Not all people are in support of antimicrobial agents. The medical industry is seeing a rise in the number of drug-resistant pathogens. This increase is most often attributed to the number of biocidal agents being used not only in hospitals, but in homes and in the workplace. Antibacterial treatments are applied to a number of things that we use on a daily basis such as soaps, lotions, cleaning supplies, air conditioning and ventilation, materials for the food and pharmaceutical industries, and even construction materials [18]. A common idea is that the increase in the number of antimicrobial agents used will heighten a person's susceptibility to infection.

This notion is not far-fetched according to many doctors. The inaccuracy of the idea occurs because many people attribute the use of antimicrobial agents to a decrease in the efficacy of an individual's immune system. In actuality, the use of antimicrobial agents supports the growth of drug-resistant strains of bacteria. One of the best known antibiotic resistant infections is methicillin resistant *Staphylococcus aureus* (MRSA). This bacterium is known to be spread in hospitals through clothes and

by personal contact [13]. According to Professors Christopher T. Walsh and Gerry Wright [22 p.392], “Given the vast numbers of bacteria, their short generation times, and typical gene mutation frequencies of 1 in 10^7 bacteria resistance is inevitable.

Antibiotics select for those very rare bacteria in a population that are less susceptible and allow them to become dominant in the populations as susceptible bacteria die off.”

So, resistance is unavoidable. The important question is whether or not you want to take action and kill the pathogens that you come into contact with, or just allow nature to take its course and let your immune system do all the work for you.

2.3 Types of Antimicrobial Finishes

Many types of antimicrobial finishes exist. The finishes are derived from different sources; some finishes are natural and some are created in a laboratory. No matter how or where the finish originates, three common traits are necessary for any treatment. The finish must not be harmful to the environment both when the fabric is treated and during the life span of the finish. Second, the finish should be effective until the wearer is finished using the textile and if necessary, endure repeated laundering. Third, and most importantly, the finish must not be harmful to the wearer. Other desirable properties of antimicrobial textiles include, but are not limited to, “selective activity to undesirable micro-organisms,” meeting requirements of regulatory agencies, “compatibility with the chemical processes,” “easy method of application,” “no discoloration of fabric quality,” “resistant to body fluids,” and “resistant to disinfections/sterilization” [16, 18].

Categorizations of antimicrobial treatments include classifying a finish as leaching or non-leaching. A finish classified as leaching moves out from the surface of the textile to kill the micro-organism. This type of finish is not particularly durable due to

the fact that it slowly leaves the surface of the textile. A non-leaching finish remains fixed to the textile and only kills those micro-organisms that come into contact with the surface of the textile. This type of finish is durable and safe because it does not affect normal skin bacteria and it does not cause skin irritation [19]. Each antimicrobial finish displays the three common traits, safe to the environment, safe to the wearer, and antimicrobially effective, and is classified as either leaching or non-leaching.

A wide selection of antimicrobial finishing agents exists. Oxidizing agents are one type of finish. This type of finish consists of halogens, aldehydes, and peroxy compounds. Oxidizing agents affect micro-organisms by attacking the cell membrane to get into the cytoplasm and affect the organism's enzymes [18]. Halogens also fall into the group called coagulants, along with isothiazones and peroxy compounds. However, the main component of this group is alcohols. This type of finish affects micro-organisms by reacting with all organic structures in the organism [18]. Quaternary ammonium salts are classified as cationizing agents. This finish alters the permeability of the cytoplasmic membrane, affecting the vitality of the cell. Quaternary ammonium salts are effective finishes for fabrics made of natural fibers [16]. Many tests are presently being performed to add this treatment to cotton.

One of the most popular and most durable of the finishes is triclosan. This finish has been used for over twenty-seven years. Triclosan is a non-leaching finish and affects micro-organisms by penetrating their cell walls causing metabolite leakage and blocking the synthesis of lipids. Consequently, cell functions are disabled and the micro-organism cannot function or reproduce [16, 18].

At this time, much research is being performed on amines, which is part of the quaternary ammonium compound group. Other compounds in this group include

biguanides and glucoprotamine. Micro-organisms are affected by this type of finish because it binds the organism to its cell membrane which ultimately results in the breakdown of the cell [18].

Several elements and natural compounds have inherent antimicrobial properties. Heavy metals and metallic compounds hold a large portion of the market for antimicrobial textiles. Cadmium, silver, copper, and mercury are all effective antimicrobial agents. Metal based finishes are fairly durable to repeated laundering making them appropriate for use as a reusable finish. Metallics work by inhibiting the active enzyme centers in micro-organisms. Silver is most commonly known for its use as an antimicrobial treatment for drinking water [16, 18].

Several natural, non-metallic, antimicrobial finishes exist. One of these natural antimicrobial finishes, Chitosan, is the deacetylated form of Chitin which is a main component in crustacean shells. This finish is important because it does not provoke an immunological response, is biodegradable and biocompatible, and is renewable. Chitosan has been shown to be effective against both gram-positive and gram-negative bacteria. The drawback to this finish is that it has a low ability for strong chemical bonding [18, 23, 24].

Dyes are also being used as antimicrobial treatments, but this type of finish may also pose bonding problems when paired with certain types of fibers [16]. Researchers have responded to problems like this by experimenting with the current finishes available. One research paper states, “the antibacterial properties of textile materials, in general, depend on the structures and amounts of biocidal groups incorporated on their surface” [20 p.1018]. Many antimicrobial textiles are treated with combinations of finishes to enhance the antimicrobial efficacy of the finishes and counter act the

negative aspects of the treatments [16]. By combining finishes, the occurrence of drug-resistant strains forming from the finish is decreased. Another trend in experimentation with antimicrobial finishes consists of adding antimicrobial agents to synthetic fibers during the spinning process [3]. By doing this, the finish is embedded into the fiber and will last for the lifetime of the textile.

2.4 Why Use Cotton?

An antimicrobial finish can be applied to most types of textiles. A wide variety of antimicrobial finishes are currently being applied to nonwoven textiles to be used as disposable protective garments in hospitals. Antimicrobial textiles, whether woven, nonwoven, or knit, can also be made out of any type of fiber content that is suitable for garment production.

The fiber content of an antimicrobial textile must be chosen carefully. Synthetic fabrics may not be appropriate for some end uses due to the fact that most synthetic fibers are hydrophobic. This means that fabrics made of synthetic fibers hold a larger amount of perspiration wetness in their weave structures than do natural fibers. This property can cause an increased chance of irritation and odor due to microbial growth on the body [16].

The use of natural fibers is encouraged because end-use products from natural fibers are biobased, not petrobased. Natural fibers are also good sources for textiles because they are renewable resources and their export can be good for many economies. Cotton is abundant and its mechanical properties are well suited for garment production. It is easy to care for and takes well to bleaching. How a fiber reacts to bleaching is important when dealing with antimicrobial finishes because many of these finishes require that the textile be bleached to regenerate its antimicrobial

properties. Both chlorine and oxygen bleach are adequate in renewing a textiles antimicrobial finish as long as the appropriate type of bleach is used for the regeneration.

2.5 Summary

Antimicrobial textiles are easily finding a place in the global textile market. Their end uses can be tailored to fit the needs of many different people and their professions. Most antimicrobial experimentation is being performed for the medical industry. The apparel industry can definitely benefit from this experimentation because the products made for the two professions are closely related. The number of safe and durable antimicrobial finishes is steadily growing. An emphasis is being put on the use of fabrics made of natural fibers because the global economy is trying to reduce the overall use and production of petroleum-based products (synthetic fibers). The global trend for a safer environment is apparent all around us.

Chapter 3 Methodology

Antimicrobial textiles can be beneficial to a wide variety of people. It is very important that the particular type of finish used is appropriate for its intended consumer. Chitosan is a good choice when the intended consumer does not fit into a specific category. Since the target audience for this research is the general public, chitosan is an appropriate agent. The PEG is added to enhance the aesthetic and physical properties of the textiles.

3.1 Materials and Equipment

The textile treated with the antimicrobial finishes and used in the mechanical and physical testing process was a desized, bleached, 100% cotton sheeting bought by the LSU Textile Science program from Test Fabric Inc. USDA SRRC provided the chitosan and PEG formulations and the *Staphylococcus aureus*. The padder and dryer used in treating the fabric samples was a Birch Bros. and Mathis Labdryer oven. The micrometer used in measuring the thickness of the fabric samples was a Model 553 from Testing Machines Inc., Meneola, NY. The testers used in measuring the mechanical and surface properties of the test fabrics were the Kawabata KES-FB instruments.

3.2 Experiment Design

The cotton sheeting was cut into 40 rectangles measuring 14x20 cm. Twenty of the rectangles were treated with a chitosan solution and the remaining 20 were treated with a chitosan and PEG solution. After treatment, the rectangles were cut into 10x10 cm squares for testing. Each test was performed 3 times on 3 randomly chosen samples treated with chitosan and 3 times on 3 randomly chosen samples treated with chitosan and PEG. Twenty samples were previously cut measuring 10x10 cm and left

untreated to function as control samples. Each test was also performed 3 times on 3 randomly chosen control samples.

After tensile, bending, shearing, compression, and surface tests were concluded, three sections were drawn onto each 10x10 cm sample. These sections were labeled for the purpose of allowing the testers to know exactly which section the 5/8 in. circular swatch used for antimicrobial testing was cut from on the larger piece of fabric. The samples were first numbered one through 20 in each of the treatment groups. Then, the samples treated with chitosan only were given a capital letter “A,” the chitosan and PEG samples were given a capital letter “B,” and the control samples received a capital letter “C.” Next, each section drawn on the samples was labeled with a lower case “a,” “b,” or “c” depending on the location of the section on the sample. Lastly, three samples from each of the treatment groups were numbered 5, 10, 25, or 50 depending on how many times the samples were washed. For example, a swatch labeled 1Ac(5) was the first sample numbered in the chitosan only group with a swatch that was cut from the bottom right side of the larger sample and washed 5 times.

3.3 Chemical Procedure

A pad-dry-cure method was used to treat fabric samples with both the chitosan and chitosan/PEG formulations. This method is a conventional process. First the fabric samples measuring 14x20 cm were immersed in the pad bath containing the designated solution and were then padded through squeeze rolls at a specified pressure to give a wet pick-up of 100%. Next, the fabric samples were mounted on pin frames, dried and cured at a specified temperature in the oven. The specific pressure and oven temperature used in the chemical procedure is considered confidential until the USDA SRRC gives further notice.

3.4 Fabric Characterization

Tests were performed to evaluate mechanical and physical properties of textiles as well as durability and effectiveness of the antimicrobial agents used. A fabric's mechanical properties determine its performance in regards to movement and perceived comfort by a wearer. The physical properties determine how a fabric looks and feels to the consumer. All tests results were evaluated and when compared their results were used to determine if the fabric was right for its proposed end use.

3.4.1 Mechanical Testing

Mechanical properties tested included tensile deformation, pure bending, shearing, and compression. Mechanical properties were tested and interpreted according to the method developed by S. Kawabata [7, 8, 9, 10]. Fabric samples were tested using the KES-FB instruments (Kawabata's evaluation system for fabrics). This system consists of four testers: KES-FB-1, KES-FB-2, KES-FB-3, and KES-FB-4. Each of these testers is connected to a main amplifier and a computer. Each tester has a corresponding computer program to accurately record and calculate the data received from the KES-FB instrument.

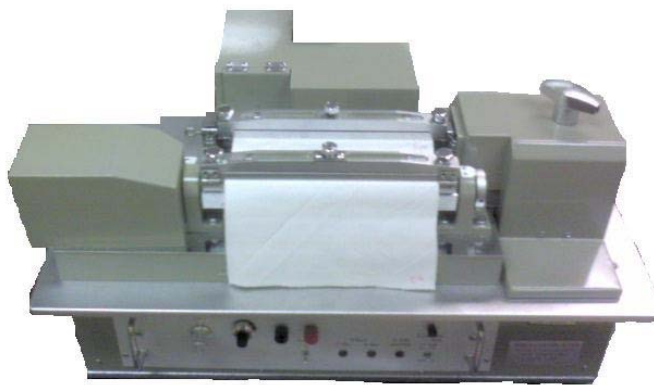
The tensile deformation was measured using the KES-FB-1 tester (figure 3-1). The characteristic value measurements taken from each tensile test were linearity (LT), tensile energy per unit area (WT), resilience (RT), and tensile strain (EMT) [7, 8, 9, 10]. Testing began by placing the sample of fabric in two clamps, or chucks, that were 5 cm. apart. The back chuck then moves away from the front chuck while the computer reads the amount of strain being put on the sample. The output from the computer was the values for LT, WT measured in $\text{gf} \times \text{cm}/\text{cm}^2$, RT measured in %, and EMT measured in %.

Pure bending was tested using the KES-FB-2 tester (figure 3-2). The bending curvature (K) range for the data recorded was between $K = -3.0$ and 3.0 , but the pure bending rigidity was only measured accurately at $K = -2.5$ and 2.5 . A constant rate of $0.50 \text{ (cm}^{-1}\text{)/sec.}$ was maintained during the bending process. The radius of the circular range was 0.73 cm . The characteristic values measured were bending rigidity per unit length (B) and moment of hysteresis per unit length (2HB). The units for bending rigidity are $\text{gf} \times \text{cm}^2/\text{cm}$ and the units for hysteresis of bending moment are $\text{gf} \times \text{cm}/\text{cm}$ [7, 8, 9, 10]. Each value had four measurements that could be taken: warp face, warp back, weft face, and weft back. Subscripts were used to identify the measurement. These included f for face, b for back, 1 for warp, and 2 for weft. Therefore, a value marked with subscript f1 was the measurement for face warp. Positive and negative curvature was used to identify face and back values. Positive curvature was used for face bending and negative curvature was used for back bending. Because the bending range went from a -2.5 to a 2.5 , both face and back measurements were recorded in the same sample test. This allows for two sets of data, warp and weft, to be recorded instead of four.

Shearing properties were measured using the KES-FB-1 machine, the same system used for measuring tensile deformation (figure 3-1). The characteristic values measured for shearing were shear stiffness (G), hysteresis at shear angle $\emptyset = 0.5$ degree (2HG), and hysteresis at $\emptyset = 5$ degree (2HG5). The velocity of the shearing was a constant 25 mm/min . When taking shearing measurements, only the face was measured along with the fabric warp and the weft directions. A measurement was not taken from the sample back. The units for shearing properties consist of shear stiffness ($\text{gf}/\text{cm} \times \text{deg}$) and shear hysteresis (gf/cm) [7, 8, 9, 10]. For the shearing test, the

sample was prepared in the same way as for the tensile deformation test. For the shearing test, instead of the back chuck moving backwards, the back chuck moved sideways to measure the shear angle of the fabric sample.

Compression properties were measured using the KES-FB-3 tester (figure 3-3). The samples were compressed between two steel plates with areas of 2cm^2 . The velocity of the compression was a constant 20 micron/sec . The recovery process was measured by the same velocity once the pressure attained 50 g/cm^2 . Characteristic values measured for compression were linearity (LC) with no unit, energy required for the compression (WC) with a unit of $\text{gf} \times \text{cm/cm}^2$, and resilience (RC) with a unit of % [7, 8, 9, 10].



3-1 Tensile and Shearing machine



3-2 Bending machine



3-3 Compression machine

3.4.2 Physical Testing

Physical tests included surface friction and roughness and sample thickness. Surface friction and roughness were measured also using the Kawabata instruments. These measurements were taken using the KES-FB4 tester (figure 3-4). This tester used a steel piano wire with a diameter of 0.5 mm for performing roughness measurements. The wire was bent and used under the contact force, given by a spring, of 10g. The first test performed was roughness. While the contactor was kept stationary, the sample was moved back and forth in 2 cm intervals at a constant velocity of 0.1 cm/sec. The frequency of the system from the up and down displacement of the piano wire was measured once the wire was out of contact with the sample. The characteristic value taken for roughness was mean deviation of surface roughness (SMD) with a unit of microns [7, 8, 9, 10]. The second test was for friction. This test used the same apparatus but a different detector. The characteristic values measured for friction were the mean value of the coefficient of friction (MIU) and the mean deviation of coefficient of friction (MMD) [7, 8, 9, 10]. These two values had no units to

measure them by. The values of surface friction and roughness were also defined for face, back, warp, and weft using the same subscripts as for the pure bending. Sample thickness was measured in thousandths of inches using a dead weight micrometer.



3-4 Surface testing machine [25]

3.4.3 Antimicrobial Testing

Antimicrobial tests were performed on the fabric samples using the AATCC Standard Test Method 100 [2]. A 1:10 dilution of *Staphylococcus aureus* (*S. aureus*) was used to measure the antimicrobial properties of the test samples. The samples were sterilized under a UV light before they were exposed to the pathogen.

3.4.4 Durability Testing

To test the durability of the antimicrobial finish, treated samples were examined for antimicrobial efficacy after 5, 10, 25 and 50 home launderings. The procedure used

for home laundering was the AATCC Test Method 61 [2]. The detergent used for this test was the AATCC standard reference detergent [2].

3.5 Data Analysis

For statistical analysis, averages taken for the samples in each of the three treatment groups were used in all evaluations. Analysis of variance (ANOVA) was used to test the hypotheses. A significance level of .01 was used to gauge the data results. The statistical analysis was executed using the software SAS 9.1.

A subjective analysis was performed to assess the difference in the sample groups in a practical manner. There is no standard method for fabric subjective evaluation. The results obtained in this study were based on the perception of the tester. The purpose of the subjective test was to give realistic meaning to the instrumental data obtained by the Kawabata tests. This test should help the reader to understand the effect of the chitosan and the chitosan and PEG finishes on the test fabric.

Chapter 4 Results and Discussion

4.1 Tensile Performance

The first mechanical property tested was tensile deformation. As shown in table 4-1, for tensile linearity (LT), the means of the three groups significantly differed from each other. The treatments affected how the fabrics performed. The direction of the fabric samples did not affect the means in this case. The treatments negatively affected the fabric samples with the Chitosan and PEG sample being the worst for retaining original fabric non-elastic property. The results for tensile energy (WT) showed no significant differences in the means of the samples. Tensile resiliency of the fabric (RT) also was not affected by the treatment or the direction of the sample. For tensile strain (EMT), all variables were shown to cause significant difference in the means. Each of the treatments greatly affected the means as did the directionality of the sample. For measuring tensile strain on fabrics with an end use of apparel, higher means gave desired results. In this case, the two treatments negatively affected the samples with the chitosan and PEG samples being the worst.

4-1 Average Means and Standard Deviations for Tensile

| Group | LT | | WT | | RT | | EMT | |
|--------------|------------|--------|-------------|--------|-------------|--------|-------------|--------|
| | Mean* | Std | Mean* | Std | Mean* | Std | Mean* | Std |
| Chitosan+PEG | 1.0706(A) | 0.0766 | 9.3950 (A) | 3.3732 | 49.5870 (A) | 5.7005 | 3.4712 (B) | 1.0961 |
| Chitosan | 0.9643 (B) | 0.0713 | 10.7530 (A) | 3.8616 | 51.4250 (A) | 5.4928 | 4.4556 (AB) | 1.6284 |
| Control | 0.8651 (C) | 0.0348 | 12.1100 (A) | 2.9555 | 50.5980 (A) | 7.1795 | 5.6282 (A) | 1.4841 |

*Means with the same letter in the same column are not significantly different at the 95% confidence level.

4.2 Bending Performance

The second mechanical property tested was pure bending. As indicated in table 4-2, for the parameter bending rigidity (B), the means significantly differed from each

other. The cause of the differences originated from the treatments used and not from the directionality of the samples tested. Lower means of bending rigidity were more desirable for apparel applications. The treatments negatively affected the samples with the chitosan and PEG samples being the worst. Histerasis of bending (2HB) showed much smaller differences in the means of the samples. Direction of the samples was not shown to have affected the means. With histerasis of bending, lower means are also desirable. The treatment using chitosan only did not have a significant negative impact on the samples as did the chitosan and PEG treatment. Overall, both treatments negatively affected the samples in regards to an end use of apparel.

4-2 Average Means and Standard Deviations for Bending

| Group | B | | HB | |
|----------------|------------|--------|------------|--------|
| | Mean* | Std | Mean* | Std |
| Chitosan + PEG | 0.4337 (A) | 0.1327 | 0.2590 (A) | 0.0656 |
| Chitosan | 0.2057 (B) | 0.0783 | 0.1438 (B) | 0.0286 |
| Control | 0.0851 (C) | 0.0151 | 0.1083 (B) | 0.0158 |

*Means with the same letter in the same column are not significantly different at the 95% confidence level.

4.3 Shearing Performance

The third mechanical property tested was shearing. As listed in table 4-3, all three parameters, G, 2HG, and 2HG5, showed significant differences among the sample means. The means for shear stiffness (G) differed greatly among the treatment groups with the direction of the samples having no affect on the means. The parameter G determines fabric stiffness and drape and lower sample means were more desirable. Both of the treatments negatively affected the samples with the chitosan and PEG treatment being the worst. Histeresis at $\varnothing = 0.5$ degree (2HG) also showed significant differences among the means. Here, the directionality of the samples also did not affect

the means. While the chitosan and PEG samples did not greatly differ from the control samples, the chitosan only treatment showed a significant negative impact on the samples. Histeresis at $\theta = 5$ degree (2HG5) had a similar outcome to G. The means of the three groups differed greatly with the direction of the samples playing no significant part in their differing means. Both the chitosan and the chitosan and PEG treatments negatively affected the samples with the chitosan and PEG treated samples being the worst affected. Overall, the treatments had a negative impact on the shearing properties of the fabric samples.

4-3 Average Means and Standard Deviations for Shearing

| Group | G | | 2HG | | 2HG5 | |
|----------------|------------|--------|------------|--------|------------|--------|
| | Mean* | Std | Mean* | Std | Mean* | Std |
| Chitosan + PEG | 8.4224 (A) | 1.4598 | 5.5767 (A) | 1.7274 | 18.759 (A) | 3.3072 |
| Chitosan | 4.9174 (B) | 0.6706 | 4.0909 (B) | 0.2381 | 11.713 (B) | 1.4651 |
| Control | 2.5903 (C) | 0.2070 | 5.4985 (A) | 0.4705 | 8.325 (C) | 0.2150 |

*Means with the same letter in the same column are not significantly different at the 95% confidence level.

4.4 Compression Performance

The last mechanical test performed was compression which determines fabric bulkiness and softness. The only parameter slightly affected by the treatments was the compressive linearity (LC). The chitosan only samples negatively affected linearity while the chitosan and PEG samples positively affected linearity. This result is to be expected since the purpose of adding the PEG to the treatment was to improve the hand, or feel, of the treated samples. All other parameters, energy required for compression (WC), resilience (RC), thickness (TO), and compression rate (EMC), showed no significant differences between the control samples and the treated

samples. Overall, the chitosan and PEG treated samples had the best compressive results for an end use of apparel.

In terms of sample thickness, the treated samples showed no difference in thickness from the control samples. It can be determined that the chitosan and chitosan and PEG treatments had no affect on the thickness of the test fabric.

4-4 Average Means and Standard Deviations for Compression

| Group | LC | | WC | | RC | | TO | | EMC | |
|--------------|-------------|--------|------------|--------|------------|--------|------------|--------|------------|--------|
| | Mean* | Std | Mean* | Std | Mean* | Std | Mean* | Std | Mean* | Std |
| Chitosan+PEG | 0.2397 (B) | 0.0176 | 0.1490 (A) | 0.0226 | 50.643 (A) | 4.7830 | 0.5353 (A) | 0.0575 | 48.090 (A) | 5.3769 |
| Chitosan | 0.3070 (A) | 0.0377 | 0.1893 (A) | 0.0720 | 45.016 (A) | 2.6872 | 0.5360 (A) | 0.0859 | 44.392 (A) | 6.8438 |
| Control | 0.2817 (AB) | 0.0169 | 0.1687 (A) | 0.0166 | 47.108 (A) | 5.7982 | 0.5127 (A) | 0.0116 | 47.565 (A) | 2.3618 |

*Means with the same letter in the same column are not significantly different at the 95% confidence level.

4.5 Surface Friction and Roughness

Surface friction and roughness were measured together since they were tested using the same Kawabata tester. As indicated in table 4-5, the mean frictional coefficient (MIU) showed only a slight significant difference in the means resulting from the treatments used and not the directionality of the samples. A lower mean was more desirable. Both treatments negatively affected the samples with the chitosan and PEG treatment being the worst. Both the mean deviation of coefficient of friction (MMD) and the mean value of the coefficient of friction (SMD) showed no significant differences in the means of the samples. It was expected that the treatment would not have affected the parameter for roughness, SMD, since the test results are directly related to the yarns and the weave of the sample fabric and not to the treatment used on the fabric. Overall, antimicrobial treatments showed little influence on the apparel fabric. This means the treated fabric can still retain good surface friction and roughness.

4-5 Average Means and Standard Deviations for Surface Friction and Roughness

| Group | MIU | | MMD | | SMD | |
|----------------|-------------|--------|------------|--------|------------|--------|
| | Mean* | Std | Mean* | Std | Mean* | Std |
| Chitosan + PEG | 0.2005 (A) | 0.0092 | 0.0453 (A) | 0.0104 | 3.6999 (A) | 0.4007 |
| Chitosan | 0.1894 (AB) | 0.0227 | 0.0462 (A) | 0.0098 | 4.1657 (A) | 0.3158 |
| Control | 0.1749 (B) | 0.0092 | 0.0403 (A) | 0.0074 | 4.0248 (A) | 0.4433 |

*Means with the same letter in the same column are not significantly different at the 95% confidence level.

4.6 Subjective Analysis

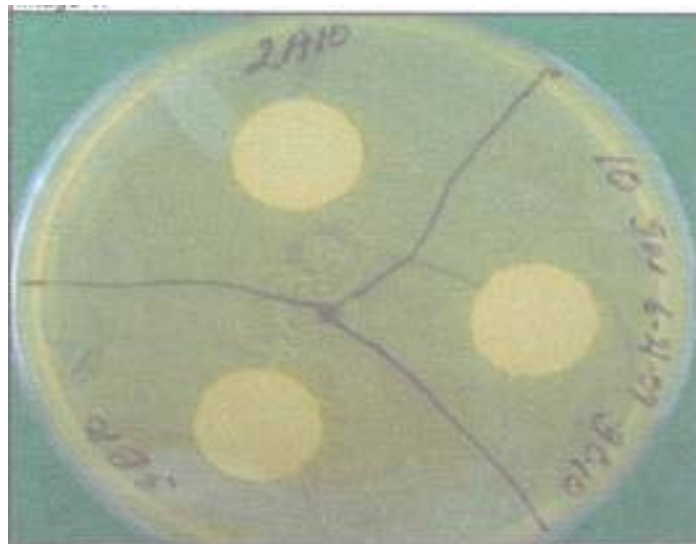
A subjective analysis was performed to compare the differences in the hand of the samples. All samples felt the same when rubbed with the fingertips. The differences in hand occurred when the samples were squeezed and crumpled. The control samples were soft when squeezed and crumpled easily. The samples treated with chitosan were slightly stiffer when squeezed and did not crumple as easily. Also, wrinkle recovery was not as noticeable on the samples treated with chitosan as they were on the control samples. The samples treated with chitosan and PEG were even stiffer and crumpled less easily than either the control samples or the samples treated with chitosan. Wrinkle recovery was less noticeable with the chitosan and PEG treated samples than with control samples or the chitosan only treated samples. The subjective evaluation in this section supports the numerical data presented in the previous sections.

4.7 Durability of Antimicrobial Treatments

Notable clearing of about 1mm existed with swatches 2A10, 3B10, 3C10. These swatches had been washed 10 times and consisted of a chitosan and PEG sample, a chitosan only sample, and a control sample. While clearing was expected for the 2A10 sample and the 3B10 sample, it was not expected for the 3C10, or control, sample. The 3C10 sample had been washed but not treated and should not have shown any

antimicrobial properties. A minute clearing was shown for samples 2B25, 3A25, and 2C25. The same can be expected for these samples as was for the previous samples. It is possible that the control samples were contaminated by the treated samples during testing.

All other samples showed no resistance to the *S. aureus* used in the assays. Decreased areas of clearing of the *S. aureus* were expected around the samples as washing frequencies increased. Samples that had only been washed 5 times were expected to have the largest area of clearing, however these samples were noted to have no affect on the *S. aureus*. The samples that underwent 50 washings were expected to have little to no affect on the *S. aureus* and this same result was noted.



4-1 Assay using samples washed 10 times



4-2 Assay using samples washed 25 times

Chapter 5 Conclusions and Suggestions for Further Research

5.1 Conclusions

In response to the international need for durable, antimicrobial resistant clothing, samples of 100% cotton sheeting were treated with two different finishing formulations. The finished fabrics were tested along with untreated control fabric samples to compare the mechanical and physical properties of the samples. The treated samples were also washed and tested to determine the antimicrobial efficacy and the durability of the biopolymer finishes.

After evaluating the quantitative results for the tensile, bending, shearing, and compression tests, it can be concluded that both the chitosan only and the chitosan/PEG finishes negatively affected the cotton textile. The treatments stiffened the structure of the plain weave cotton sheeting which resulted in the degradation of these properties. Surface friction and roughness, however, were not negatively affected by the antimicrobial treatments. This outcome was expected with the chitosan and PEG treated samples. One of the benefits of adding PEG to the chitosan is increased surface smoothness. The subjective analysis performed by the author revealed that the three treatment groups had a different hand from one another. This concludes that the instrumental assessment and the subjective assessment for the antimicrobial treatments were consistent.

Two groups of washed samples showed minimal clearing in the *Staphylococcus aureus* assays. The samples washed 10 and 25 times displayed signs of antimicrobial resistance with a clearing of 1mm. It was expected that the samples washed 5, 10, and 25 times would show this resistance. The samples washed 50 times showed no antimicrobial resistance as did the samples washed only 5 times. If the samples

washed 10 and 25 times showed antimicrobial resistance, then the samples washed only 5 times should have shown this resistance also. It is possible that the samples were either mixed up or contaminated before the antimicrobial testing began. Cross contamination may explain why the samples that were effective against the *S. aureus* came from each of the three treatment groups, chitosan only, chitosan and PEG, and control untreated. Retesting of the antimicrobial assays would have occurred if the treatments had not negatively affected the mechanical properties of the fabric.

It can be concluded that the two antimicrobial treatments in their current formulations, chitosan and chitosan/PEG, were not effective as reusable and durable antimicrobial treatments. The scientific community in finding the right durable and reusable antimicrobial treatment is making progress. Testing should continue until the right treatment can be marketed to the medical and apparel industries.

5.2 Suggestions for Further Work

The chitosan and chitosan and PEG finishes showed a small amount of resistance against the *S. aureus*. However, the finishes applied were inappropriate for use as garments. Most of the mechanical properties were negatively affected and therefore outweigh the few positive results caused by the treatments.

Suggestions for continued work include continued biological testing on the chitosan and chitosan and PEG treated and washed samples. Retesting should also be conducted with decreased concentrations of *S. aureus*. Another option would include adding a fabric softening agent to the current chitosan and chitosan and PEG finishes to improve properties such as bending, shearing, and surface roughness and friction. Fabrics with low bending rigidity and shear rigidity should be selected when applying the

chitosan and PEG treatment. Lastly, a completely new antimicrobial finish should be formulated to test on the same 100% cotton sheeting.

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Appendix: Research Data

Table I

Results from Tensile test

| Sample | Direction | LT | WT | RT | EMT |
|--------|-----------|--------|---------|---------|--------|
| 2Ab | warp | 0.9676 | 10.5884 | 46.7084 | 4.3773 |
| | fill | 0.9371 | 17.6115 | 43.8915 | 7.5176 |
| 8Aa | warp | 1.0307 | 8.6234 | 50.1858 | 3.3467 |
| | fill | 1.0365 | 11.1081 | 53.3812 | 4.2868 |
| 15Aa | warp | 0.8418 | 5.9770 | 57.2580 | 2.8401 |
| | fill | 0.9723 | 10.6102 | 57.1230 | 4.3648 |
| 1Ba | warp | 0.9793 | 6.1760 | 55.7124 | 2.5225 |
| | fill | 1.0386 | 11.6441 | 50.0716 | 4.4847 |
| 7Ba | warp | 1.0301 | 6.4572 | 53.4451 | 2.5075 |
| | fill | 1.1361 | 14.0071 | 40.5766 | 4.9318 |
| 14Bb | warp | 1.0516 | 6.6625 | 52.5739 | 2.5344 |
| | fill | 1.1877 | 11.4209 | 45.1452 | 3.8463 |
| C1 | warp | 0.8263 | 12.6813 | 40.4680 | 6.1385 |
| | fill | 0.8471 | 16.6982 | 42.3616 | 7.8850 |
| C2 | warp | 0.8842 | 8.9299 | 55.8696 | 4.0396 |
| | fill | 0.8326 | 12.8842 | 53.8225 | 6.1900 |
| C3 | warp | 0.8870 | 8.7596 | 55.9652 | 3.9502 |
| | fill | 0.9134 | 12.7095 | 55.1008 | 5.5658 |

Table II

Results from Bending test

| Sample | Direction | B | 2HB | Sensitivity |
|--------|-----------|----------|----------|-------------|
| 1Aa | warp | 0.116353 | 0.110332 | 2x1 |
| | fill | 0.107713 | 0.110801 | 2x1 |
| 7Aa | warp | 0.300138 | 0.174912 | 2x1 |
| | fill | 0.264133 | 0.146140 | 5x1 |
| 14Ab | warp | 0.214023 | 0.173734 | 5x1 |
| | fill | 0.231859 | 0.146834 | 5x1 |
| 2Ba | warp | 0.273120 | 0.188843 | 5x1 |
| | fill | 0.283856 | 0.171776 | 5x1 |
| 7Bb | warp | 0.431624 | 0.298440 | 5x1 |
| | fill | 0.469874 | 0.255310 | 5x1 |
| 16Bb | warp | 0.557777 | 0.320440 | 5x1 |
| | fill | 0.585740 | 0.319366 | 5x1 |
| C1 | warp | 0.067218 | 0.113632 | 2x1 |
| | fill | 0.070234 | 0.090491 | 2x1 |
| C2 | warp | 0.103837 | 0.129216 | 2x1 |
| | fill | 0.088103 | 0.096538 | 2x1 |
| C3 | warp | 0.099968 | 0.122535 | 2x1 |
| | fill | 0.081353 | 0.097511 | 2x1 |

Table III

Results from Shearing test

| Sample | Direction | G | 2HG | 2HG5 |
|--------|-----------|---------|--------|---------|
| 1Ab | warp | 3.7318 | 3.6840 | 9.3485 |
| | fill | 4.7879 | 4.1903 | 11.3014 |
| 10Aa | warp | 5.0662 | 4.3616 | 11.5241 |
| | fill | 5.7827 | 4.0862 | 13.8546 |
| 13Aa | warp | 4.9973 | 4.2414 | 12.1898 |
| | fill | 5.1385 | 3.9816 | 12.0625 |
| 1Bb | warp | 8.1893 | 6.2871 | 16.5842 |
| | fill | 8.4899 | 5.5340 | 17.6649 |
| 6Bb | warp | 6.8204 | 5.7365 | 15.5721 |
| | fill | 6.8541 | 2.2806 | 17.6649 |
| 16Bb | warp | 10.0362 | 7.2985 | 20.4274 |
| | fill | 10.1446 | 6.3236 | 24.6432 |
| C1 | warp | 2.6535 | 5.7484 | 8.3136 |
| | fill | 2.3124 | 4.8279 | 8.1696 |
| C2 | warp | 2.7724 | 5.7505 | 8.3277 |
| | fill | 2.3440 | 4.9697 | 8.1577 |
| C3 | warp | 2.7160 | 5.9117 | 8.2404 |
| | fill | 2.7433 | 5.7829 | 8.7394 |

Table IV

Results from Compression test

| Sample | LC | WC | RC | TO | EMC |
|--------|-------|-------|--------|-------|--------|
| 2Aa | 0.318 | 0.248 | 46.093 | 0.614 | 50.668 |
| 9Aa | 0.338 | 0.211 | 46.997 | 0.550 | 45.412 |
| 5Ab | 0.265 | 0.109 | 41.957 | 0.444 | 37.095 |
| 4Ba | 0.229 | 0.143 | 47.657 | 0.535 | 51.194 |
| 9bb | 0.230 | 0.174 | 56.160 | 0.593 | 51.194 |
| 14Ba | 0.260 | 0.130 | 48.113 | 0.478 | 41.881 |
| 7Ca | 0.274 | 0.167 | 53.648 | 0.511 | 47.495 |
| 6Ca | 0.301 | 0.186 | 42.597 | 0.525 | 49.961 |
| 1Ca | 0.270 | 0.153 | 45.079 | 0.502 | 45.239 |

* The gap for this test was set at 1.110738

Table V

Results from Surface Friction test

| Sample | Direction | MIU | MMD |
|--------|-----------|----------|----------|
| 3Ab | warp | 0.190299 | 0.055618 |
| | fill | 0.162214 | 0.032763 |
| 10Ab | warp | 0.210478 | 0.050958 |
| | fill | 0.218622 | 0.053191 |
| 14Aa | warp | 0.166422 | 0.034938 |
| | fill | 0.188272 | 0.049634 |
| 3Ba | warp | 0.190910 | 0.032953 |
| | fill | 0.195646 | 0.052363 |
| 5Bb | warp | 0.192605 | 0.032949 |
| | fill | 0.207241 | 0.050354 |
| 13Bb | warp | 0.214412 | 0.045336 |
| | fill | 0.202215 | 0.057790 |
| 3Ca | warp | 0.171767 | 0.041722 |
| | fill | 0.166642 | 0.031583 |
| 5Ca | warp | 0.166446 | 0.031399 |
| | fill | 0.190742 | 0.048334 |
| 9Ca | warp | 0.174265 | 0.041128 |
| | fill | 0.179248 | 0.047582 |

Table VI

Results from Surface Roughness test

| Sample | Direction | SMD |
|--------|-----------|----------|
| 4Ab | warp | 4.01142 |
| | fill | 4.119759 |
| 7Ab | warp | 4.092705 |
| | fill | 4.186994 |
| 11Aa | warp | 4.758096 |
| | fill | 3.825162 |
| 6Bb | warp | 3.796877 |
| | fill | 3.377999 |
| 10Bb | warp | 3.373 |
| | fill | 4.370031 |
| 15Bb | warp | 3.892582 |
| | fill | 3.388819 |
| 2Ca | warp | 4.353929 |
| | fill | 3.328269 |
| 4Ca | warp | 4.162774 |
| | fill | 3.642022 |
| 8Ca | warp | 4.465197 |
| | fill | 4.196323 |

Table VII

Results from Sample Thickness measurements

| Sample | Thickness in 1/1000 in. |
|---------------|----------------------------------------|
| 1Ac | 6.8 |
| 7Ac | 7.0 |
| 15Ac | 7.3 |
| 3Bc | 7.0 |
| 9Bc | 7.0 |
| 13Bc | 6.5 |
| C1 | 7.0 |
| C2 | 6.7 |
| C3 | 7.1 |

Vita

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