Abstract

LINDEMANN, CRAIG ERIC. Evaluation of Commercially Available PFOA-Free Repellent Finishes. (Under the direction of Dr. Brent Smith and Dr. Keith Beck.)

Due to recent scrutiny of perfluorooctainoic acid (PFOA)-containing fluorochemical (FC) repellent finishes, research was conducted to evaluate the performance of commercially available alternative to such products. Such products have been shown to be environmentally persistent and potentially hazardous.

A list of available finishes was compiled and a number of products were selected for evaluation. Finishes were applied using a pad-dry-cure process, and their performance was evaluated using industry-approved, standard test methods for oil and water repellency. Finishes were applied to six fabric substrates, with end uses in the apparel, home furnishings, and automotive industries.

Results of the work indicate that none of the commercially available products tested performed at the same level as the traditional, PFOA-containing products. Novel, short-chain FCs showed the highest level of performance among the alternatives tested. These were the only products which were able to provide oil repellency. Silicone and hydrocarbon wax products were also evaluated and showed varying levels of performance.

Short-term and long-term durability to home laundering of the finishes were evaluated. Hydrocarbon wax products showed the poorest durability of the finish types tested. None of the alternative products tested were found to have the same level of durability as the traditional FC finishes. Influence of isocyanate and melamine-formaldehyde crosslinking resins was found to be fabric specific, showing significant benefit on cotton substrates.

EVALUATION OF COMMERCIALLY AVAILABLE PFOA-FREE REPELLENT FINISHES

by

CRAIG ERIC LINDEMANN

A thesis submitted to the Graduate Faculty of
North Carolina State University
In partial fulfillment of the
Requirements for the degree of
Master of Science

TEXTILE CHEMISTRY

Raleigh, North Carolina

March 21, 2007

APPROVED BY:

Dr. Keith Beck Co-chair of Advisory Committee	Dr. C. Brent Smith Co-chair of Advisory Committee
Dr. Kristin Thoney	Dr. Henry Boyter ITT Committee Member

Dedication

For my parents who keep me motivated, and the cyclists who keep me sane.

Biography

Craig Lindemann grew up in Baltimore, Maryland with his parents, Russ and Linda, and younger brother, Kevin. He graduated with honors in 2004 from Loyola College in Maryland with B.S. degree in Chemistry and a minor in Mathematics. Craig is interesting in pursuing sustainable manufacturing practices and environmentally responsible product development. After graduation, he hopes to pursue a career in materials development in the performance textiles and apparel industry.

Acknowledgements

First and foremost, I would like to acknowledge my advisory committee for their input and guidance. I would also like to thank the Institute of Textile Technology and its member companies for funding this work, as well as providing fabric samples and additional resources for the completion of my research. Many chemical suppliers offered products for testing; their participation is greatly appreciated. Keith Zimmerman at Cotton, Incorporated offered many contacts and ideas for this work, and was critical to the success of this work. Finally I would like to especially thank Jeff Krauss and Judy Elson, both of whom provided immeasurable time, resources, and guidance to my work.

Table of Contents

List of Figures

List of Tables

Chapter 1 : Introduction

Wetting properties of textiles are important considerations when developing products for a number of desired end uses including, but not limited to apparel, home furnishings, and automotive textiles. These properties are determined by the fiber type being used, the product structure, and the finish applied to the substrate. The ability to alter and control the wetting properties of textiles is an important challenge for the textile industry.

A number of repellent finishing options, which can generally be divided into three main classifications: fluorocarbons, hydrocarbons, and polysiloxanes, have been available for the past 50 years. Since the 1980s, fluorocarbons have been the most widely used of the three classes due to their superior repellency and fastness properties, as well as their additional utility in applications where oil-repellent and soil-release properties are desirable.

Recently, concern has been raised regarding the ecological persistence and toxicity of a number of chemicals including perfluorooctanoic sulfonate (PFOS), and perfluorooctanoic acid (PFOA), which are associated with fluorochemical (FC) finishing, [10]. While research to determine the long term impact of such chemicals is still ongoing, it seems prudent to begin considering the utility of various alternatives to such finishing options. In addition to environmental concerns being raised, identification of a viable alternative could provide a significant cost savings considering the relatively high cost of FC finishes.

The work compiles a list of all currently available commercial repellent finishing options. Based on product literature, screening according to safety, availability, durability, and application for selected substrates and end uses has been conducted to

develop a list of chemical alternatives for performance testing. Substrates and end uses have been chosen according to the recommendations of an industry panel. The main objective of this project is to present a comparative study of repellent finishing alternatives to traditional eight-carbon FCs, focusing on novel, short chain FCs.

Chapter 2 : Literature Review

Recent literature related to repellency and water repellent finishing of textiles has been researched. General principles of wettability and theory of repellency are briefly reviewed in this chapter. Materials used for the water repellent finishing of textile substrates are covered in this review. Background information regarding the ecological persistence and toxicology of select FCs are also discussed as justification for the proposed research.

2.1 Wettability and Repellency Theory

The purpose of this section is to establish an understanding of general theories of wettability and repellency. Areas covered include principles of wetting, equilibrium and dynamic contact angles, surface energy and surface tension, and repellency in fabrics.

2.1.1 Wetting

Though highly subjective in measure, repellency can be generally thought of as a state of limited or low wettability[1]. Stated another way, repellency is the ability of a material to resist wetting under prescribed conditions. More specific definitions of repellency are discussed later in this review.

Since the two topics are intricately related, it is critical to develop an understanding of the general principles of wetting in order to fully discuss repellency. In Gibbs' thermodynamic treatise, he described the tendency of a system to move toward the lowest free energy state. In the specific case of wetting, this free energy is related to

the sum of the interfacial energies across the solid-liquid, solid-vapor, and liquid-vapor boundaries, as described in Equation (1) [11],

$$F = A_S \gamma_{SV} + A_L \gamma_{LV} + A_{SL} \gamma_{SL} \qquad (1)$$

where F denotes the interfacial energy, A denotes area, and γ in the surface energy per unit area. The subscripts S, L, and V, denote the solid, liquid and vapor phases, respectively.

In this case, wetting is spontaneous when the change in free energies, ΔF is negative. In a three-dimensional textile substrate, wetting involves four basic processes, related to Equation (1): immersion, capillary sorption, adhesion, and spreading. These processed are illustrated in Figure 1.

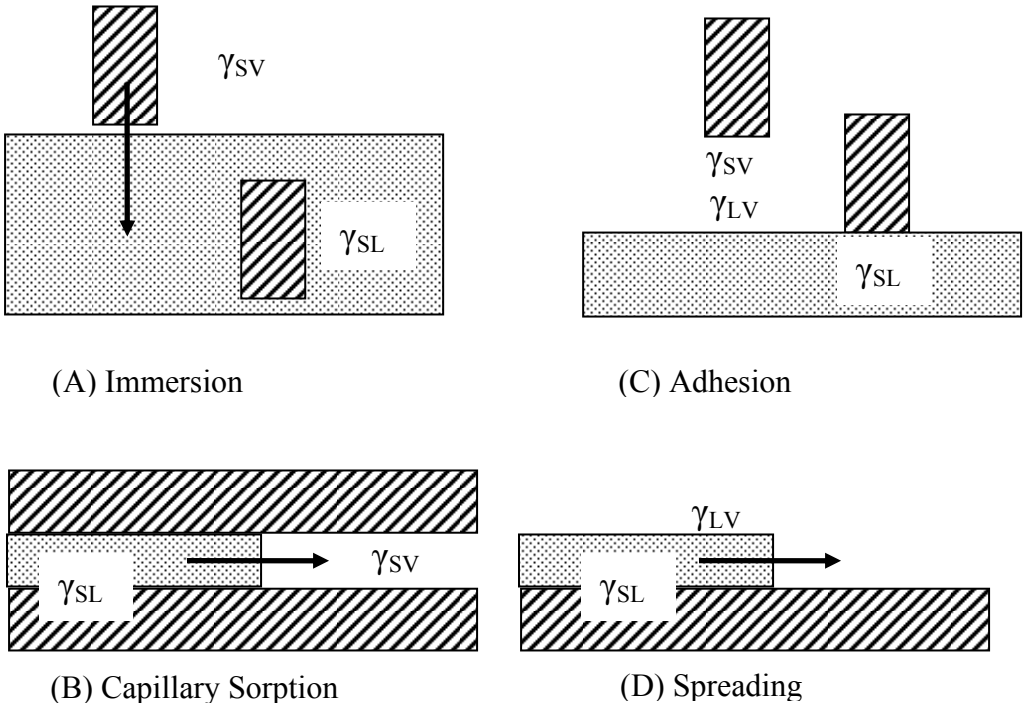


Figure 1: Illustrations of basic wetting processes, based on the work of Kissa[1].

As described by Kissa[1], immersion and capillary sorption are both instances in which a solid-vapor interface is replaced by a solid-liquid interface, as described by Equation (2), where W_I is the work of immersion and W_P is the work of capillary sorption.

$$W_I = W_P = \gamma_{SV} - \gamma_{SL} \quad (2)$$

Adhesion is the force of attraction between two contacting surfaces, in the case of wetting, a solid and a liquid. The Dupré equation describes the work of adhesion, W_A, as the difference between the surface free energies of the solid-vapor and liquid-vapor interfaces when the two surfaces are separated and the free energy of the solid-liquid interface formed between the materials in contact (Equation 3) [12].

$$W_A = \gamma_{SV} + \gamma_{LV} - \gamma_{SL} \quad (3)$$

Finally, spreading is defined as the flow of at least two molecular layers of liquid over the surface of a solid. The work of spreading, W_S, can be described mathematically by Equation (4).

$$Ws = \gamma_{SV} - \gamma_{LV} - \gamma_{SL} \quad (4)$$

When considering these equations, it is important to note that they are defined as the energy per unit area required to separate the two materials. That is, they describe the energy associated with the reversal of partial wetting. Also, it must be noted that these equations are derived based on ideal surfaces which are perfectly smooth, homogeneous, impermeable, and non-deformable[1]. With this in mind, we must note that actual wetting of textile substrates is, in practice, much more complicated.

2.1.2 Contact Angles in Real vs. Ideal Systems

When attempting to apply the equations discussed in the previous section to predict wetting activity, a complication arises. The quanitity γ_{SV} is not readily available. Therefore alternate measurements must be used to determine wetting interactions. Equation (1) can be shown mathematically to be equivalent to equation (5) [7],

$$\frac{F}{\gamma_{LV}} = A_{LV} - \iint_{A_{SL}} \cos\theta_a \, dA \tag{5}$$

where $\cos\theta_a$ is the intrinsic contact angle, defined by Young's equation (6) [13], and which is illustrated in Figure 2. As previously discussed, when a liquid is placed on a solid surface, the system tends toward the state of lowest energy. From the vector diagram in Figure 2 it can be seen that the system reaches this state when the vectors γ_{SV}, γ_{SL}, and the in-plane component of the vector γ_{LV} sum to zero[14]. This balance of forced is described by the Young equation:

$$\cos\theta_a = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} \tag{6}$$

When a sessile drop of water is placed on a surface, a contact angle >90° indicates a hydrophobic surface, while contact angles of <90° are considered hydrophilic, with wettability approaching a maximum limit as θ approaches 0[1].

While the Young equation helps to understand the equilibrium involved in the wetting properties of an ideal surface, it is still important to recall that wetting of textiles does not act ideally, and equilibrium measures can be difficult to determine. Therefore,

there is no standard textile test method that measures contact angle, and actual wetting properties are explored empirically.

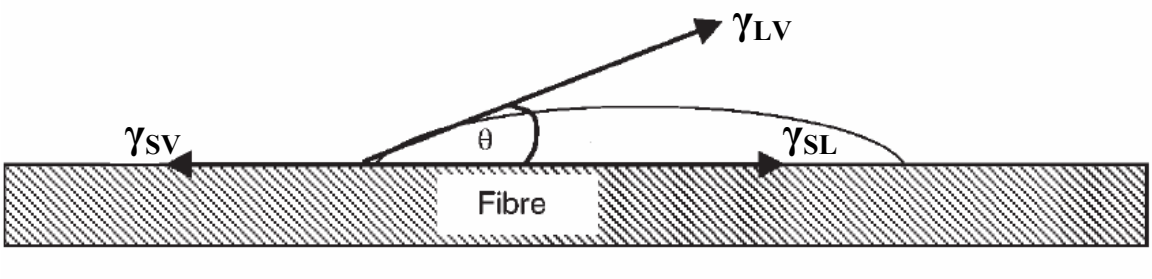


Figure 2: Illustration of the Equilibrium Contact Angle[2]

In all of the above equations, the term γ_{SV} for surface energy at partial vapor pressure, *p*, has been used instead of the alternate terms, γ_{SV° for saturated vapor, and γ_{SO} for surface energy, which can be used to provide a more comprehensive understanding of general wettability. These terms can be associated using a corrective factor, π, for the spreading pressure of the vapor adsorbed on the solid surface. However, based on empirical data, Zisman asserted that this value is negligible in instances where the contact angle is constant and does not approach 0, as is the case when examining repellent surfaces[8]. This claim was later supported by Good, who found π to be reasonably small in instances where the liquid surface tension was close to the critical surface tension of the solid, which are discussed later[15]. Therefore, the above equations are sufficiently accurate for the subject in question.

There is a great deal of disagreement regarding how to handle the concept of the equilibrium contact angle in an ideal system, since it is easily confused with apparent contact angles in nonequilibrium systems[1]. A number of notable attempts have been made to reconcile or understand this difference. Wenzel proposed a theory based on non-ideal surface rugosities, stating that the actual area of contact of a liquid on a rough

surface is greater than the contact area is a smooth surface is assumed[16], on the basis of which he proposed the notion of the roughness factor, r, given by,

$$r = \frac{A_o}{A_r} = \frac{\cos\theta'}{\cos\theta} \qquad (7)$$

where A_o is the observed area, A_r is the real contact are including roughness, θ is the intrinsic contact angle, and θ' is the observed contact angle. What this factor represents is the difference between an assumed smooth, two-dimensional surface area, and the actual surface area of a three-dimensional, rough surface (Figure 3).

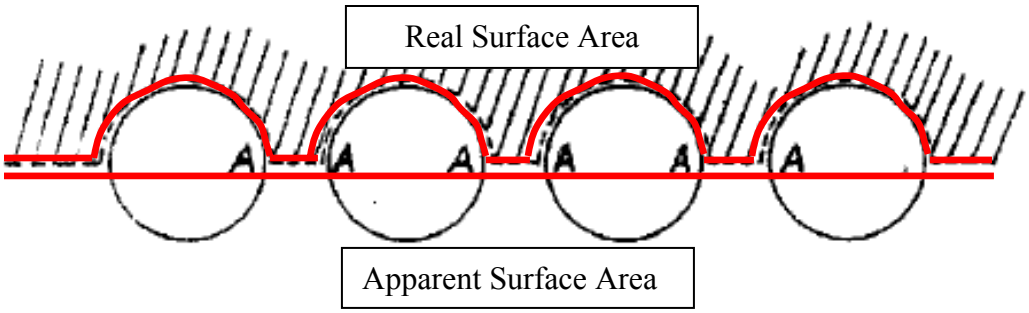


Figure 3: Illustration of apparent vs. real surface areas for wetting of a textile surface. Illustration from the work of Cassie and Baxter[3].

This three-dimensional surface similarly bears on the real angle at which the liquid contacts the surface compared to the observable contact angle which assumes a smooth surface. Cassie[17] further developed this theory, examining heterogenous compound surfaces, as is typical of a porous textile surface. Rather than considering the solid surface ridges fully wetted, as Wenzel did, Cassie and Baxter assumed that the liquid rested at the peaks of the low energy, and was bridged by a region of contact with air, which has a higher interfacial energy[3]. Cassie asserted that the energy gained by wetting is, essentially, an area weighted average of the individual energy gains of the compound materials (in the case of textiles, this compound material is a fiber/air compound

"surface"). This phenomenon is described by Equation (8), where σ_1 denotes the contact area of material 1 having relatively low surface energy, and σ_2 denotes the contact area of material 2 having a relatively high surface energy.

$$F = \sigma_1(\gamma_{S1V} - \gamma_{S1L}) + \sigma_2(\gamma_{S2V} + \gamma_{S2L}) \tag{8}$$

This equation can be directly related to the contact angle measures, for observed and ideal systems as given in Equation (9),

$$\cos\theta' = \phi_1 \cos\theta_1 + \phi_2 \cos\theta_2 \tag{9}$$

where θ_1 and θ_2 are the intrinsic contact angles associated with smooth surfaces of materials 1 and 2 respectively. Cassie and Baxter expanded on Cassie's compound surface concept by assuming that the high energy surface in question is air. A value of 1 can then be assigned for $\cos\theta_2$, since a water-air interface has the equivalent of a contact angle of zero. This value can be substituted into Equation (9) can be combined with the Young equation (6) to yield:

$$\cos\theta' = -1 + \phi_s(\cos\theta + 1) \tag{10}$$

The Wenzel and Cassie models propose two different equilibrium conditions for a drop of liquid on a solid surface. Bico has proposed the application of the Wenzel model to hydrophilic surfaces, and the Cassie model for hydrophobic interactions[5]. Figure 4 illustrates the Wenzel and Cassie models along with the combined Bico model, showing dissymmetry between hydrophilic and hydrophobic behaviors which is realized in practice.

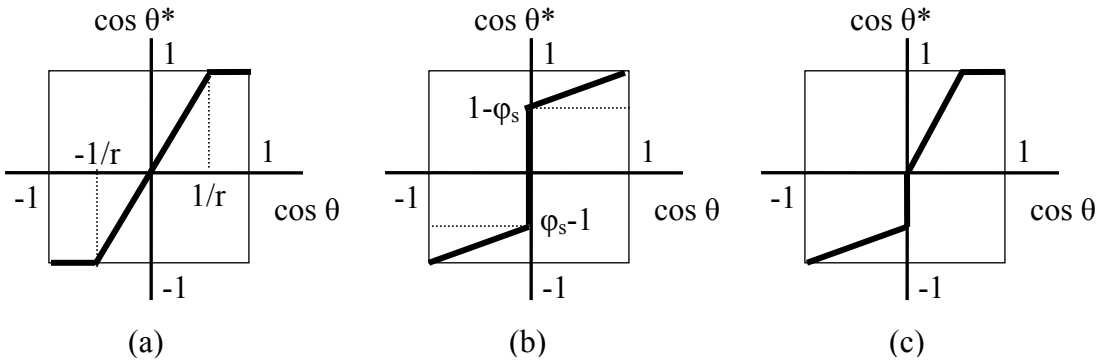


Figure 4: Ilustration of proposed wetting models for rough surfaces. (a) Wenzel's model for "wetting" contact, (b) Cassie's model for compound surfaces, and (c) Bico's combined model. Based on figures from Patankar and Bico[4, 5].

Work by Patankar suggests that, though Cassie's model generally works well to predict the behavior of hydrophobic surfaces as previously suggested, a drop can easily be altered between the proposed equilibrium states, so both Wenzel and Cassie models must be considered when examining a hydrophobic surface[4].

In further work related to Cassie's model, Johnson and Dettre[6], related the composition of the idealized compound surface to the advancing and receding contact angles observed. The hysteresis observed between advancing and receding contact angles is attributable to a variety of factors related to energy differences in the state of the solid surface in these two conditions[11]. The main findings of Johnson and Dettre can be summarized in Figure 5.

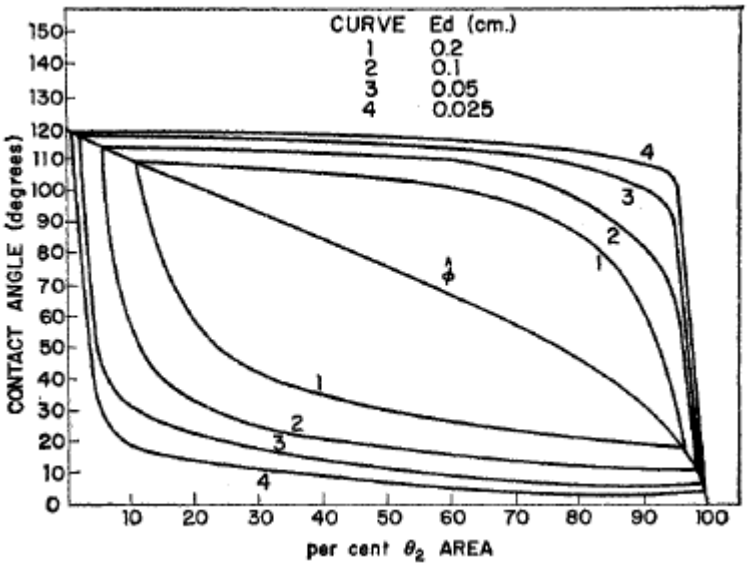


Figure 5: A Series of Advancing (top) and Receding (bottom) Contact Angles on an Idealized Heterogeneous Surface[6]

Figure 5 illustrates a series of advancing and receding contact angles for various liquids on an idealized homogeneous surface. The top set of curves relate to the advancing contact angles, while the bottom set correspond to receding contact angles. The x axis shows that the total energy of the surface increases from left to right, meaning that the surface is composed of a higher percentage of high energy material. The curve labeled θ represents the Cassie Equilibrium angle, as determined by the composition of the solid surface. From these results, we can infer that advancing contact angles tend to be associated with low wettability regions, while receding contact angles are associated with high energy, readily wetted areas[18].

Figure 6 illustrates the basic relationship between advancing and receding contact angles, and the hysteresis that results.

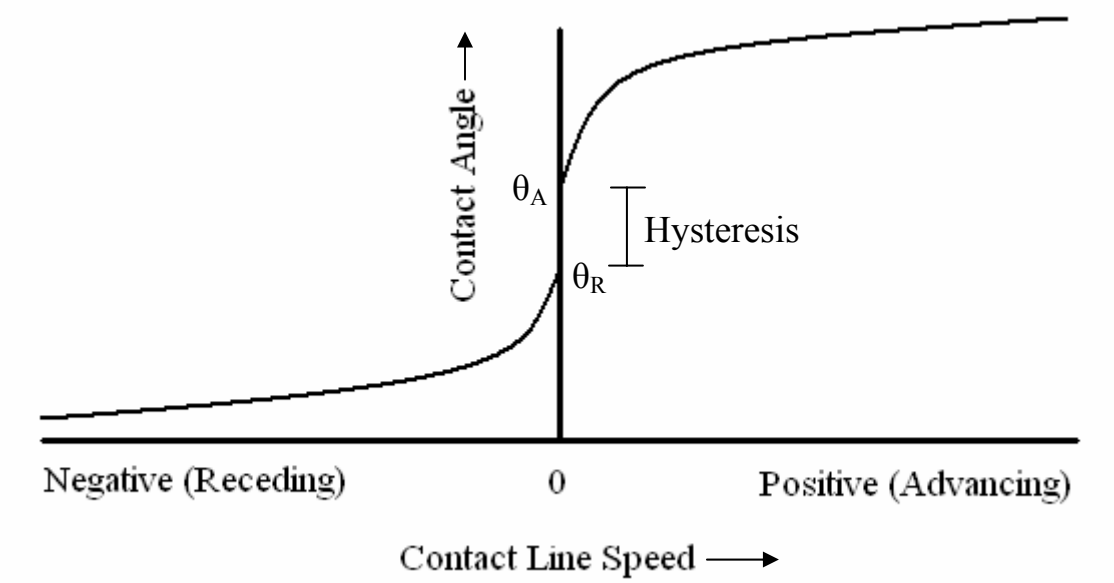


Figure 6: Illustration of Contact Angle Hysteresis as it relates to the advancing and receding contact angles in static systems (at contact line speed = 0).

In many practical applications, the interaction between a liquid and a solid surface is not static, as described in the previous section, but dynamic. The apparent contact angle is dependent on the advancing or receding speed of the contact line, as shown in Figure 6. A measure of dynamic contact angle can be obtained indirectly by measuring capillary force[14]. An in depth discussion of the theory of dynamic wetting of surfaces is beyond the scope of this review.

Recent work has contributed to further understanding of wetting behaviors of non-ideal surfaces, as Chen, et al[7] have developed a mathematical and empirical comparison of the contact angles exhibited by a liquid on an anisotropic surface, comparing the apparent contact angle along the grooves to the apparent contact angle against the grooves. The difference can be easily observed in Figure 7.

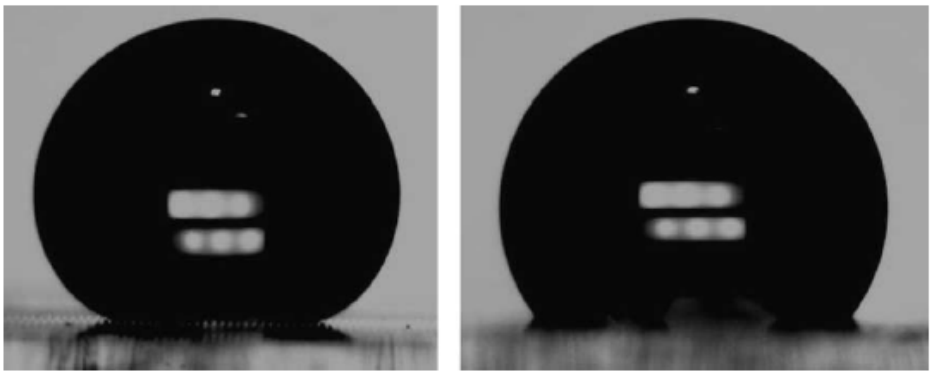


Figure 7: Front (left) and side (right) views of a sessile drop on a rough, anisotropic surface with parallel geometry. Contacts angles reported are 149.5° (front) and 126.5°[7]

While some research suggests that receding contact angles be used rather than equilibrium angles in light of the fact that, due to evaporation, the equilibrium state tends to reflect a receding angle regardless of initial condition[19], convention is to measure the advancing contact angle, as this has been shown to be more repeatable[1].

2.1.3 Surface Energies and Critical Surface Tension

The work of Zisman provided a critical breakthrough in the understanding of wetting properties of solids. This work examined the equilibrium contact angles of a series of liquids, on a low energy surface of polytetrafluoroethylene[8]. From this work, a series of graphs, plotting cos θ' against surface tension for subsets of similar liquids, such as those in Figure 8 were produced. Each subset of liquids studied produced similar linear results. The point at which cos θ = 1, indicates the surface tension below which liquids in that particular subset spreads on the surface.

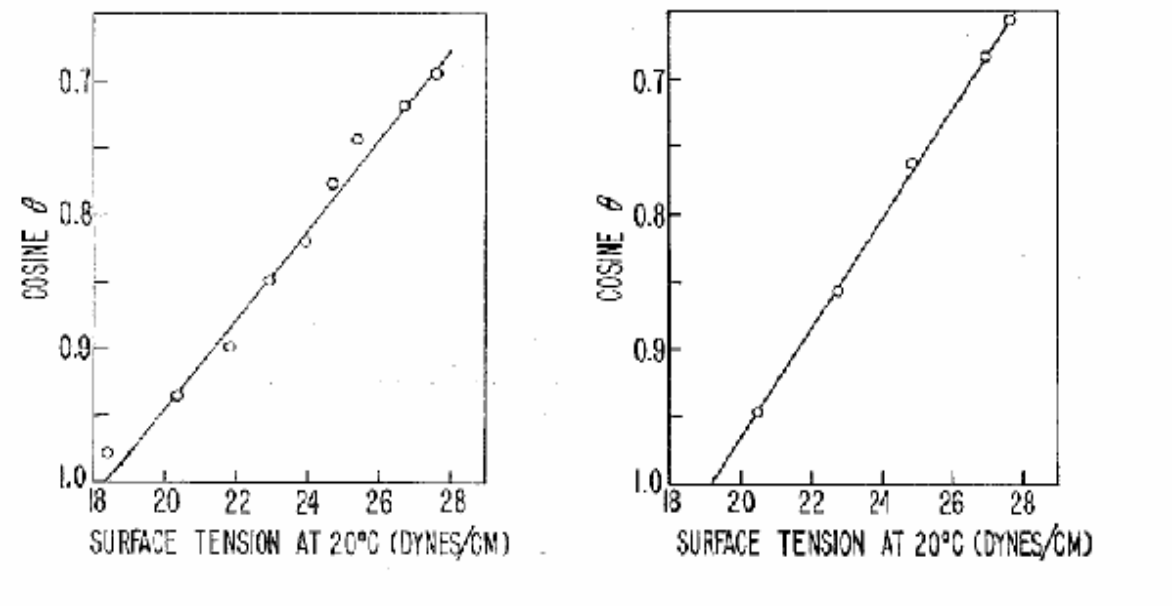


Figure 8: Zisman plots of surface tension versus cos θ for *n*-alkanes (left) and di(*n*-alkyl)ethers (right) on polytetrafluoroethylene[8].

In the study, Zisman found that there was a common range of values at which spreading began to occur, regardless of the liquid subset examined. For polytetrafluoroethylene, that value was between 17.5 and 20.5 dynes/cm. For an oleophobic coating on a platinum surface, the range was between 23.0 and 23.6 dynes/cm. From this information, Zisman proposed the concept of critical surface tension, defined as the surface tension, γ_{SV}, below which every liquid spreads on a given surface, and above which no liquid spreads[8].

Further work by Zisman analyzed the critical surface tensions for a variety of low-energy surfaces. The results of this work are summarized in Table 1 and Table 2. Table 1 shows results for a series of halogenated polyethylene derivatives, and Table 2 gives results for a series of condensed monolayers.

FC surfaces are clearly shown to have the lowest critical surface tension, and therefore presumably have the lowest surface energy. A surface comprised of closely

packed CF_3 groups was found to have the lowest critical surface tension of all surfaces tested. In Table 1 it can be seen that substitution of fluorine atoms with hydrogen atoms from a perfluorethylene chain results in an almost linear increase in surface energy.

Table 1: Critical Surface Tensions for Halogenated Polyethylenes[9]

Polymer	Critical Surface Tension (dyne/cm)
Poly(vinylidene chloride)	40
Poly(vinyl chloride)	39
Polyethylene	31
Poly(vinyl fluoride)	28
Poly(vinylidene fluoride)	25
Polytrifluoroethylene	22
Polytetrafluoroethylene	18

While the value of critical surface tension is only an empirical value for surfaces, and does not have a direct thermodynamical basis, it can be related to γ_{SV} by work related to Cassie's theory and has proven useful in the development of oil- and water-repellent finishes[1].

Table 2: Critical Surface Tensions for Low-Energy Surfaces[9]

Surface Consistution	Critical Surface Tension (dyne/cm)
Fluorocarbon Surfaces	
$-CF_3$	6
$-CF_2H$	15
$-CF_3$ and $-CF_2-$	17
$-CF_2-$	18
$-CH_2-CF_3$	20
$-CF_2-CFH-$	22
$-CF_2-CH_2-$	25
$-CFH-CH_2-$	28
Hydrocarbon Surfaces	
$-CH_3$ (crystal)	22
$-CH_3$ (monolayer)	24
$-CH_2-$	31
$-CH_2-$ and Phenyl	33
Phenyl	35

Table 2 (continued)

Chlorocarbon Surfaces	
-CClH-CH_2-	39
-CCl_2-CH_2	40
=CCl_2	43
Nitrated Hydrocarbon Surfaces	
-CH_2ONO_2 (crystal)	40
-$C(NO_2)_3$ (monolayer)	42
-CH_2NHNO_2 (crystal)	44
-CH_2ONO_2 (crystal)	45

2.1.4 Repellency in Fabrics

The preceding discussion of the principles which govern wetting and repellent behaviors of surfaces have not considered the impact of capillary spaces such as those that exist in a textile substrate. A number of factors including chemical nature, geometry, roughness, and spacing of fibers influence the capillary properties of textiles[1].

To describe the capillary properties of textiles, we can define the term ΔP, which is the hydrostatic pressure required to force a liquid through a fabric. ΔP is a function of yarn diameter (a), and spacing (b = half the distance between yarns), the surface tension of the liquid (γ_{SV}), and the contact angle between the liquid and the apparent surface (θ_A) (Equation (11))[1].

$$\Delta P = \frac{2\gamma_{LV}}{R} = \frac{2\gamma_{LV}}{a(\cos\theta_A + \sqrt{(a+b)^2/a^2 - \sin^2\theta_A}} \qquad (11)$$

Equation (11) indicates that, for yarns with a contact angle greater that 90°, high bulk densities and tight weaves require higher pressure for capillary transport, and are thus beneficial for repellent fabric structures. Accordingly, many water repellent structures

are tightly woven fabrics, made repellent through chemical modification[1]. All fabric samples tested in the present work are woven samples for this reason.

2.2 Fluorocarbon (FC) type Finishes

Repellent finishes are generally divided into three subclasses: hydrocarbon waxes, polysiloxanes, and FCs. Each of these three classes of water repellents has unique benefits and challenges associated with them, as summarized in Table 3. Paraffin waxes are the oldest used water repellent finishes and are extremely inexpensive. They provide good water repellency, but limit air permeability, low durability, and have low washfastness properties[20]. Polysiloxanes also provide good water repellency, and have the added benefits of water-vapor permeability and improved fabric hand, but do not provide oil repellency and show increased pilling[2]. FCs are the only compounds that have been found to impart both oil- and water-repellency, and thus are the most popular finishes currently for repellent finishing.

Table 3: Summary of principle classifications of water repellent finishes and general advantages and disadvantages of each

Finish Type	Advantages	Disadvantages
Paraffin Waxes	Very Inexpensive Good water repellency	Limited breathability Not oil or soil repellent Poor durability
Polysiloxanes	Good water repellency Soft hand Water-vapor permeable	Not oil or soil repellent Moderate durability
FCs	Water and oil repellent Very Durable to washing Soil Releasing	Relatively expensive Environmental concerns

2.2.1 Performance and Use of FC Finishes

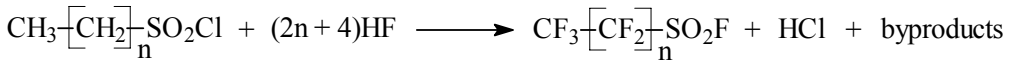
As discussed previously, surfaces consisting of $-CF_3$ or $-CF_2-$ groups have has an extremely low surface energy and are both oil and water repellent. A wide variety of FC finishes has been developed for application to textile substrates and, in the past 25 years, they have grown increasingly successful due to their superior performance characteristics and the increasing ability to develop novel structures for desired properties[2]. In the early 1990s in Europe, the apparel sector comprised about 60% of the total market for FC finishes. The remaining 40% was divided between household textiles including awnings, upholstery, drapes, and table clothes, and technical textiles for a variety of uses including automotive applications[21]. In order to encompass a wide variety of end uses, fabrics for the proposed research have been selected which have use in the apparel, home furnishings, and automotive sectors.

Much recent work on FCs has involved development of LAD, or "laundry air dry" polymers. After laundering, traditional FC finishes are only able to regain their repellent properties after heat treatment such as ironing, because laundering disorients the FC chains. LAD products utilize boosters, which are discussed later, to stabilize the FC chain during washing and reduce the level of disorientation[22].

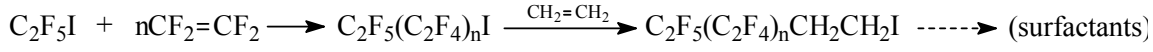
2.2.2 Methods of Fluorochemical Synthesis

Three methods are typically employed in the synthesis of FCs: electrofluorination, telomerization, and oligomerization. In electrofluorination, a hydrocarbon product is dissolved in hydrogen fluoride. Under an applied electrical current, the hydrogen atoms on the hydrocarbon are replaced by fluorine atoms. In telomerization, a fluorinated

ethylene derivative (taxogen) is reacted with an initiator for chain growth polymerization (telogen), producing the desired FC. The oligomerization process is similar to telomereization, but results in high molecular weight fluoropolymers, and thus is not generally used for production of FC finishes[23].



Electrofluorination of perfluoroalkylsulfonyl chlorides



Telomerization of perfluorinated surfactants

Figure 9: Electrofluorination and telomerization reactions used to produce fluorinated surfactants

2.2.3 Application

Application of FC finishes can be accomplished either by exhaust or pad-dry-cure methods. Exhaust methods are typically used for yarn packages, with padding being common for application to fabrics[24]. Since the present work focuses on application to fabrics, this review examines the padding process.

Pretreatment of the fabric is critical to effective FC application. The fabric to be treated must be cleaned and free of all residues, e.g. alkali or sizes from the preparation process. Rewetting agents must also be removed prior to application to prevent the reduction of oleophobic properties. Polysiloxanes, typically in the form of defoamers, tend to have the largest negative impact on finished properties of fabrics, reducing oil repellent properties and cause shade change. To optimize repellent properties, the above chemicals must not be present on the fabric or in the bath[24, 25].

Depending on substrate type and fabric construction, the wet pick up for the padding process can range between 20 and 100%, which generally corresponds to a fluorine add on between 0.25 and 2.0% on the weight of the fabric[24]. Drying and curing temperatures depend largely on the substrate being finished and can fall anywhere between 120° and 190° C[24, 26].

FC finishing is often carried out using a variety of additives designed to provide additional functionality or to improve the functionality offered by the FC treatments. These additives include extenders and boosters, crosslinkers, and durable press finishes, among others[25, 27].

2.2.4 Use of boosters and other additives

Due to the expense of using FC finishes, it is advantageous to use extenders or boosters to maintain repellency with lower FC levels. Hydrocarbon waxes have often been used as FC extenders[1], but isocyanate boosters have recently begun replacing waxes in this role. The hydrocarbon waxes allow a similar level of repellency from a significantly reduced level of FC add on, but have a negative impact on the durability of the product. Unlike hydrocarbons, though, isocyanates improve the functionality of the FC finishes by acting as a crosslinker. The isocyanate molecules can react either with the FC functional groups, with the fibers, or with other isocyanate molecules. This interaction increases the stability of the FC finish, and produces LAD products which were previously discussed[2].

Durable press finishes are also often added to FC finishes to provide added wrinkle and crease resistance, while serving as FC extenders. By combining finish types

in this way, a lower FC level can be obtained while maintaining repellency properties and providing the added functionality associated with a durable press finish[27].

2.3 Toxicology and Ecological Considerations Concerning PFCs

While FCs have certainly added a great deal of functionality to the area of repellent finishing, there has recently arisen concern relating to the environmental persistence and bio-accumulation of several key decomposition products, including perfluorooctanoic acid (PFOA), and perfluorooctane sulfonate (PFOS), surfactants that have been utilized in the manufacture and application of FC finishes.

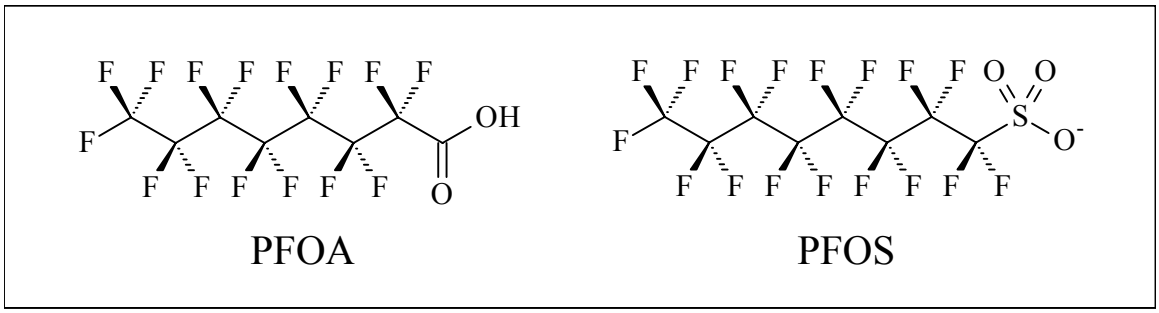


Figure 10: Chemical structures of fluorinated compounds PFOA, and PFOS.

2.3.1 Focus on Eco-Friendly textiles

The use of FCs for a number of applications, including textile finishing, has come under increasing scrutiny since 2000. Initial concern was raised regarding PFOS, a byproduct of the then common electrofluorination process for FC synthesis[23]. At this time, several companies including DuPont, 3M, Daikin and Clariant formed the Telomer Reasearch Group to work with the Environmental Protection Agency (EPA) to address concerns over this issue[28]. In response to the concerns raised, the TRP and EPA have developed the 2010/15 PFOA Stewardship Program. This program specifies that PFOA levels be

reduced 95% from a year 2000 baseline level by 2010 and eliminated completely from emissions and product by 2015[29].

2.3.2 Toxicological Concerns Associated with Fluorocarbon Finishes

It was originally thought that utilizing a telomerization synthesis would circumvent this problem by producing FCs that would not degrade to perfluorosulphonates[23]. More recently, however, additional concerns have been raised about the persistence of PFOA[10, 30-35].

The work of Kennedy[10] regarding the toxicity of PFOA indicates that potential effects of exposure include body weight loss, liver weight gain, and necrosis of the liver at high concentrations. Hinderliter[30] has also found that PFOA inhalation exposure had similar effects in male and female rats when compared with oral dosings. As with previous studies, male rats showed an accumulation of the chemical over the course of several weeks. The results of Loveless, et al[35] indicate that linear perfluorinated chains characteristic of telomerized FCs showed similar bio-persistence in rats when compared with branched ammonium perfluorooctanoate (APFO) studied previously.

Kannan[31] has reported identification of PFOS, and to a lesser extent, PFOA in the livers of birds in Japan and Korea, as well as parts of North America and Europe, indicating the risk for environmental exposure to such chemicals. Further, the work of Karrman[34], Olivero-Verbel[32], and Calafat[33] all demonstrate the presence of perfluorinated compounds in human samples from various regions of North America and Columbia.

While some work suggests that articles treated with fluorotelomers are fully degraded under typical municipal incineration conditions[36], there is still significant

evidence to support concerns regarding environmental persistence and possible toxicological effects related to exposure. With further work pending, it is vital for the textile industry to begin consideration of alternatives to FCs for use in the water repellent finishing of textiles.

2.3.3 Novel Fluorocarbon Finishes

The primary concern surrounding FC production has been the use of PFOA as a surfactant in the polymerization process. To address this issue, a number of companies, primarily those in the Telomer Research Group, have worked toward developing products that use alternative surfactants[37]. This, however, has not addressed the concern entirely, as there is still a suspicion that traditional eight-carbon based chemistries could decompose into PFOA or related products.

In order to avoid this issue entirely, several companies have recently introduced alternative FC products based on four- or six-carbon based chemistries which are not able to be converted into the chemicals of present interest[38]. There has not been a documented evaluation of these chemistries to date. The present research proposes to evaluate the performance of these novel products relative to both traditional fluorine-based chemistries, as well as other available alternatives, discussed below.

2.4 Alternatives to Fluorocarbon Type Finishes

A number of alternatives are available to traditional FC type finishes. Primary among them are hydrocarbon hydrophobes and polysiloxane based finishing options. The following section discusses these finishes as well as a number of novel approaches to

repellent finishing which are not currently used commercially, but which merit mention from a research perspective.

2.4.1 Hydrocarbon Waxes

Paraffin wax containing repellents are the oldest and least expensive water repellent finishing option for textiles. Typically, waxes are applied as Aluminum or Zirconium soaps, which have a cationic charge and interact ionically with the fiber surface to improve durability. Zirconium soaps are more commonly used in this manner since they are more hydrophobic and more resistant to alkali, which improves the washfastness properties of the finishes.

Further improvements on the performance of wax-based treatments were developed, but found little use due to significant negative aspects and environmental concerns. Chrome complexes created by DuPont provided good water repellency and soft hand, but had a blue-green color which was objectionable for many uses. Pyridinium complexes and N-methylol compounds were developed but have seen limited use due to concerns of formaldehyde release.

Current textile finishing uses waxes primarily as extenders for fluorocarbons, which helps to reduce the cost of such finishes[1].

2.4.2 Polysiloxanes

Polysiloxane based finishes are commonly used for their water repellent properties, and are generally preferred to hydrocarbon waxes since they are more durable and provide a number of additional property enhancements to the treated fibers, including imparting a soft hand, improved sewability, and improved tear strength[2]. Also, where

FCs tends to lose their repellent properties after washing, and require a re-setting step to regain repellency, polysiloxanes do not experience such a reduction, and surface properties have been shown to be less impacted by washing[39].

The hydrophobicity associated with polysiloxane finishes is related to their orientation to the fiber surface. A typical polydimethylpolysiloxane forms a three dimensional polymer matrix around the fiber upon curing, oriented with an (–O—Si—O—Si—) backbone toward the fiber surface, and the methyl groups facing outward, providing the repellency. In order to facilitate this orientation and crosslinking, a metal catalyst such as tin octoate is typically used[2].

Disadvantages associated with polysiloxane finishing involve the repellency reduction from over-application. This results in a polysiloxane double-layer with a polar exterior. Additionally, since repellency is related to the polysiloxane sheath around the fibers, repellency can be lost if the sheath is cracked, as often occurs when fibers swell during laundering. Another disadvantage that is associated with polysiloxane contamination of fabrics and equipment is uncorrectable spotting of fabrics which results in second quality product. Also, as with hydrocarbon waxes, oil repellency can not be achieved with polysiloxane finishes alone[1, 2].

Several alternative approaches which utilize polysiloxane chemistries exist. Fluorinated polysiloxanes have been shown experimentally to have excellent performance properties[40-43], and silica nano-particles have been studied extensively for application in studies mimicking the lotus effect for superhydrophobicity[5, 44, 45]. These applications are discussed later in this review.

2.4.2 Novel Approaches

A number of novel approaches to repellent finishing, several of which deserve mention, have been examined in recent years. Each of the novel approaches has shown significant benefits and could be a potentially important finishing option in the near future.

Plasma Finishing

Plasma aided textile finishing is of interest as an alternative "dry" process which could replace traditional wet finishing, eliminating demands related to waste water treatment and fabric drying processes. Fabric properties, such as breathability and hand, are also left largely unaffected by plasma treatment.

Zhang, et al. compared cotton fabrics treated with plasma finishing using a FC to samples treated with a Scotchgard™ spray-applied FC product. The plasma treatment performed as well as or better than the Scotchgard™ sample in all performance aspects measured including softness, water vapor permeability, abrasion resistance, and whiteness index of the treated samples. Contact angles measured by a goniometer were found to be 164° for the plasma treatment compared to 137° for the Scotchgard™ treated sample[46]. It should be noted that the Scotchgard™ spray treatment used in this study is not representative of a typical industrial repellent finish, but is an after-market consumer product.

In other work, Hocker successfully evaluated the utility of plasma finishing for a variety of functionalities including repellency. Plasma treatment utilizing hexamethyldisiloxane achieved a hydrophobic surface exhibiting a contact angle

approaching 130°. Additionally, treatment using hexafluoroethane plasma produced a highly hydrophobic surface, while not influencing water vapor transmission[47]. This work cites no data regarding the durability of the plasma treatment.

Work such as that done by Zhang and Hocker has indicated the potential for industrial plasma treatment of textiles in the future.

Ultra-hydrophobic surfaces

Beginning with the works of Cassie and Baxter and the development of the Wenzel equation, the importance of surface roughness on hydrophobicity has been explored[3, 16, 17]. In general, surface roughness on the microscopic level has the effect of magnifying the wetting properties of a surface due to the significantly increased surface area[5]. This is realized in nature by the lotus leaf, from which the phenomenon of ultra-hydrophobicity takes its name.

In related research, Gao and McCarthy were able to create an ultrahydrophobic surface on a microfiber polyester fabric treated with a dimethylsilicone exhibiting advancing and receding contact angles of 170° and 165°, respectively. The authors emphasize that superhydrophobicity requires both a high contact angle, as well as minimal contact angle hysteresis, which relates to the force required to move the water across the surface[44].

Li, et al. examined superhydrophobic properties of a fluoropolymeric film on a self-assembled silica colloidal template. This method utilized the hydrophobicity of the fluoropolymers combined with the nano-scale rugosities associated with the silica

particles. The result was a superhydrophobic surface having an equilibrium contact angle greater that 150° [48].

Similarly, Soeno, et al. achieved hydrophobicity with a self-assembled film of SiO_2 nano-particles. Ten bilayers of silica nano-particles of 7 nm diameter were applied on a silica wafer, and exhibited a contact angle greater than 160° [45].

Fluorinated Polysiloxanes

The final novel approach to be discussed is the development of fluorinated polysiloxanes. One instance of this type of chemistry is found in the work of Shao, et al. A multifunctional silicone derivative is described having a perfluoroalkyl chain and three epoxy groups, described as a perfluoroalkly-containing multi-epoxy compound (PFME). The described PFME was applied to cotton fabrics and a dual functionality was realized showing oil repellency, as well as moderate durable press properties resulting from the multiple crosslinking epoxy groups. This durable press effect was increased with the addition of citric acid[42]. Mahltig and his colleagues also suggest the use of a perfluoroalkyl pendant group on a polysiloxane backbone for oleophobic textile finishing[41].

Chapter 3 : Experimental Methods and Procedures

The following chapter describes the materials, application procedures and parameters, and test methods utilized in the present research. The materials section provides a discussion of the specific chemistries used, as well as the fabric substrates tested in the present research. The application procedures portion outlines the pad-dry-cure process for the treatment of all chemical and substrate combinations as tested, as well as the washing procedure used to evaluate finish durability. Finally, the test methods section specifies the tests used to evaluate the performance of the chemical finishes.

3.1 Materials

All materials used for the present research were donated by the manufacturers for use in this study. The six fabric samples were received from Institute of Textile Technology (ITT) member companies. The chemical finishes were supplied by the product manufacturers and distributors in response to sample requests made through personal correspondence.

3.1.1 Fabric Substrates

Six fabric substrates were received from Institute of Textile Technology (ITT) member companies for use in this research. These fabrics were all woven substrates designed for end uses in the apparel, automotive, or home furnishings/upholstery markets. Details on each fabric are discussed below, including fabric weight, pick counts, tear strength, and initial color values. Fabric weights are based on an 18 inch by 24 inch sample. Pick and end counts were counted using a pick glass, and verified with the use of a lunometer.

Tear strength (across fill) and L*a*b* color values are discussed in the test methods section.

Cotton Fabric

A woven one-hundred percent cotton fabric was received from an ITT member company. The fabric was intended for application in home furnishings. Specific fabric properties are listed in Table 4 below.

Table 4: Properties of 100% cotton fabric. Measured after fabric preparation, prior to finish application.

End Use	Weight (oz/yd^2)	Ends/inch	Picks/inch
Home furnishing	5.04	70	50
Tear Strength (lbf)	**L***	**a***	**b***
4.20	93.63	-0.51	3.68

Polyester/Cotton Blended Fabric

A 65% polyester, 35% cotton blended fabric was received from an ITT member company. The woven fabric is intended for an outerwear end use. Specific fabric properties are listed in Table 5 below.

Table 5: Properties of 65/35% polyester/cotton blended fabric. Properties measured after preparation, prior to finish application.

End Use	Weight (oz/yd^2)	Ends/inch	Picks/inch
Outerwear	4.11	102	48
Tear Strength (lbf)	**L***	**a***	**b***
2.93	38.58	27.14	25.6

Polyester Fabric

A woven 100% polyester fabric was donated by an ITT member company. The product is developed for use in the automotive industry. Fabric specifications are listed below in Table 6.

Table 6: Properties of 100% polyester fabric. Properties measures after preparation, prior to finish application.

End Use	Weight (oz/yd^2)	Ends/inch	Picks/inch
Automotive	9.22	60	45
Tear Strength (lbf)	**L***	**a***	**b***
18.81	26.61	-0.41	-0.11

Nylon Fabric

A woven, one-hundred percent nylon fabric was received from an ITT member company. The fabric is intended for an end use in the outerwear market. Specific fabric properties are outlined below, in Table 7.

Table 7: Properties of 100% nylon fabric. Properties measured after preparation, prior to finish application.

End Use	Weight (oz/yd^2)	Ends/inch	Picks/inch
Outerwear	2.22	114	84
Tear Strength (lbf)	**L***	**a***	**b***
2.63	57.83	0.09	6.17

Home Furnishing Acrylic Fabric

A woven, one-hundred percent acrylic fabric was received from an ITT member company. The intended end use is in the home furnishing market. Fabric properties are outlined in Table 8, below.

Table 8: Properties of 100% acrylic fabric for home furnishing applications. Properties measured after preparation, prior to finish application.

End Use	Weight (oz/yd^2)	Ends/inch	Picks/inch
Home furnishing	9.21	75	35
Tear Strength (lbf)	**L***	**a***	**b***
9.07	81.54	1.09	5.36

Automotive Acrylic Fabric

A woven, one-hundred percent acrylic fabric was provided by an ITT member company. The fabric was designed for an automotive end use. Specific physical properties of the acrylic fabric are listed in Table 9.

Table 9: Properties of 100% acrylic fabric for automotive application. Properties Measured after preparation, prior to application of finish.

End Use	Weight (oz/yd^2)	Ends/inch	Picks/inch
Home furnishing	9.21	75	35
Tear Strength (lbf)	**L***	**a***	**b***
9.07	81.54	1.09	5.36

3.1.2 Chemical Samples

An extensive list of textile chemical manufacturers and vendors was compiled at the outset of the current research. These companies were contacted to request information and literature on any available repellent finishes currently offered. The complete list of companies and product offerings developed through personal correspondence, advice from technical advisors, website research, and company literature is provided in Appendix A.

From this list, several products were selected for testing. Selection was made based on availability and ensures that a range of available chemistries was evaluated. The initial chemical selection identified two eight-carbon based FCs, two novel short-chain FCs, two polysiloxane based finishes, and one fluorinated polysiloxane product.

After initial testing, which is discussed in detail in the Results section, several of these products were eliminated from further testing. Eliminated were one of the traditional eight-carbon FC products as well as the fluorinated polysiloxane product. For

the final application, three hydrocarbon waxes were added to the selected finishes. Additionally, several melamine and isocyanate resins were used in conjunction with the remaining FC finishes to evaluate their impact on both the performance and durability of such finishes.

Table 10: Listing of all chemicals used for initial screening and continuous application process, including product name and supplier.

Product	Supplier	Initial Screening	Continuous Application	Product Type
Aerotex® M3	Emerald Materials		X	Melamine Resin
Apexosist® 186	Apexical		X	Isocyanate crosslinker
AsahiGuard™ E-061	MIC Specialty Chemicals	X	X	Short-chain FC
Baygard® RT	Lanxess	X		Fluorinated polysiloxane
Fluorochem A1*	N/A	X	X	Short-chain FC
Freepel® CCS	Emerald Materials		X	Zirconium Wax
Impregnole® FH	Omnova		X	Zirconium Wax
Norane® 100	Omnova		X	Reactive Wax
Norane® Sil (w/Catalyst Sil)	Omnova	X	X	Polysiloxane
Phobotone® WS Conc (w/Phobotone® BC New)	Huntsman	X	X	Polysiloxane
TMI®[META]	Cytec		X	Isocyanate crosslinker
Waterproofon™ 246	Apexical	X	X	C-8 FC
Zonyl® 7040	Huntsman	X		C-8 FC

* The supplier of this product requested that results for the product tested be published anonymously.

Chemical Samples for Initial Screening

The initial screening process consisted of seven chemicals, including four FCs and three polysiloxane based products. Traditional FCs tested were WATERPROOFON™ 246,

obtained from Apexical in Spartanburg, South Carolina, and Ciba® ZONYL® 7040 obtained from Huntsman, Inc. in Salt Lake City, Utah. Both of these products are marketed as being free of PFOA at a manufacturing level. They were chosen to represent the state of the art in traditional eight-carbon fluorochemistry.

Two short-chain FCs were used in the initial screening: AsahiGuard™ E-061, from MIC Specialty Chemicals, Inc. in Iselin, NJ, and another proprietary technology, which is referred to as Fluorochem-A1 in this document at the request of the supplier.

Polysiloxane chemistries used were NORANE® Silicone with CATALYST SIL from Omnova Solutions, Inc. in Fairlawn, OH, and PHOBOTONE® WS CONC with required catalyst, PHOBOTONE® BC New, from Huntsman, Inc. A fluorinated polysiloxane product, BAYGARD® RT, from LANXESS Corporation in Pittsburgh, PA, intended for room temperature, post-manufacture application for carpet finishing was used as a benchmark for fluorinated silicone chemistry. This product was used in lieu of BAYGARD® SFA-02, an industrial finish, which Lanxess has recently taken off the market for unspecified reasons.

Chemical Samples for Continuous Application and Evaluation

After the initial screening process using the chemicals listed above, the ZONYL® 7040 and BAYGARD® RT samples were eliminated from consideration, as is discussed in the results section. Several hydrocarbon wax based chemistries were added to the study, to evaluate their potential as extenders for the FCs tested. These additional products were: IMPREGNOLE® FH, and NORANE® 100, a zirconium and reactive wax, respectively, from Omnova Solutions, Inc., as well as FREEPEL® CCS, a zirconium wax from Emerald Performance Materials, in Charlotte, NC.

To further examine the potential application of novel FCs, AEROTEX® M-3 melamine resin from Emerald Materials, TMI®[META] unsaturated isocyanate crosslinker from Cytec Industries, Inc., in Charlotte, NC, and APEXOSIST® 186 isocyanate crosslinker from Apexical were all obtained for experimental application.

For later determination of dry chemical add-on each of the chemicals used in the continuous pad application process was dehydrated to determine approximate dry chemical concentration. For this evaluation, approximately 2.5g samples of each chemical was weighed in fresh Petri dishes using a Mettler AG-260 analytical balance. These samples were then dried in an oven at 105ºC overnight, and placed in a desicator to cool for 4 hours. After cooling, the Petri dishes with chemicals were reweighed and the dry mass of the solids was determined.

These data are used to determine the approximate percent solids of the products being studied. Percent solids was calculated using the following formula:

$$PercentSolids = \frac{ChemicalDryWeight}{Initial\ \mathrm{Pr}oductWeight} \times 100\% \quad (12)$$

Data for the TMI®[META] product, show close to zero dry solids, indicating the chemical as well as the solvent may have volatilized at the experimental temperatures. The remaining results all fall within expected levels. Results are summarized in Table 11.

Table 11: Summary of Percent Dry Solids, calculated as a function of chemical dry weight/chemical liquid weight. Published values are also shown, where provided by the manufacturer.

Sample	Initial Product Weight (g)	Chemical Dry Weight (g)	Calculated Percent Dry Solids (%)
Freepel® CCS	2.5343	0.6184	24%
Impregnole® FH	2.5676	0.4451	17%
Norane® 100	2.6437	0.5497	21%
Catalyst Sil	2.5424	0.2369	9%
Phobotone® BC New	2.0621	0.5763	28%
Norane® SIL	2.5778	0.6144	24%
Phobotone® WS Conc	2.0503	0.9886	48%
AsahiGuard™ E-061	2.3890	0.3584	15%
Waterproofon™ 246	2.5809	0.6949	27%
Fluorochem A1	2.5506	0.6148	24%
Aerotex® M3	2.1005	1.5534	74%
Apexosist™ 186	2.1100	0.3334	16%
TMI®[Meta]	2.3967	0.0114	0.5%

3.2 Experimental Application Procedures

The following section discusses the procedures used to prepare and treat the fabric samples for both the initial screening process and the continuous application and performance evaluation. Equipment used for application and testing is listed in Table 12.

Table 12: Listing of equipment used for application and testing, including in text references used in procedure details.

Machine Manufacturer	Product Name	In Text Reference
Rodney Hunt Machine Co	Tru-Shade Winch Beck	Winch Beck
Theis	Mini-Soft Jet	Theis Sample Jet
W Mathis AG	THN Oven	THN Tenter Oven
W Mathis AG	HVF Padder	Pilot Scale Padder
Marshall & William Co	S.O.No.4451 2 Zone Tenter	Full-width Tenter
Sew-Eurodrive	S92	Full-width Padder
MTS	Q-Test/5 with Elite Controller	Q-Test
X-Rite	SP64 Handheld Unit	Portable Spectrophotometer
Despatch	LAC1-38A	Despatch Oven

3.2.1 Fabric Preparation

Each of the six fabric substrates was prepared prior to application of any chemical finishes. Preparation was completed to ensure that any lubricants or surfactants used in the weaving or dyeing processes were completely removed from the fabric prior to finish application as any contamination of the fabric surface can significantly hinder the performance of the repellent finishes. Preparation was verified using AATCC Test Method 81-2001, pH of the Water-Extract from Wet Processed Textiles, and AATCC Test Method 97-1999, Extractable Content of Greige and/or Prepared Textiles, which is discussed in depth in the test methods section. The preparation procedure for each substrate is discussed individually, except where two fabrics were prepared in tandem. After preparation, each full width fabric sample was cut into 18 inch widths for application on the pilot plant padder and THN tenter oven, except the two acrylic samples which were cut to width prior to preparation.

Preparation of Cotton

Cotton fabric was prepared in the pilot plant winch beck. The beck was filled with approximately ninety gallons (341 L) of hot water. Half of the fabric to be treated was loaded, and the scouring chemicals were added: Keiralon NF (600 g) non-ionic surfactant and soda ash (1,500 g) (see Table 13). The remaining fabric was then loaded into the beck and the front door was closed.

Table 13: Summary of chemical baths used for preparation of 100% cotton fabric.

Chemical Bath Ingredients	Bath Concentration (g/L)	Amount added to bath (g)
Scouring Chemical Bath (90 gallons total)		
Keiralon NF Surfactant	1.8 g/L	600 g
Soda Ash (Na_2CO_3)	4.4 g/L	1,500 g
Bleaching Chemical Bath (90 gallons total)		
Keiralon NF Surfactant	1.1 g/L	380 g
50% Caustic (NaOH)	0.8 g/L NaOH	550 g
35% Hydrogen Peroxide (H_2O_2)	1.1 g/L H_2O_2	1,100 g

The reel was run at 35 rpm for the duration of the scouring process. Bath temperature was raised to 180°F and held for 30 minutes. Temperature was then decreased to 150°F, and the bath was dropped and refilled with hot water to rinse. This rinse cycle was completed twice, and the bath was refilled to 90 gallons for the bleaching process.

With the bath refilled, bleaching chemicals were added, as shown in Table 13. The beck was again closed, and the bath was heated and maintained at a boil for 30 minutes. The bath temperature was lowered to 150°F, the bath was dropped, and the hot rinse cycle was repeated three times as previously described. During the third rinse, glacial acetic acid (210 g) was added in order to neutralize the alkaline bath. Neutralization was verified using pH paper, which recorded a value of 7. After a final cold rinse was completed, the fabric was unloaded from the winch beck and plaited into a transport bin.

The fabric was then dried on the full width tenter frame. Fabrics were squeezed at 4.0 bar on the full width padder to remove excess moisture from the fabric prior to entering the tenter. Heating zones one and two were both set to 300°F, and run speed

was adjusted between four and eight meters per minute as needed to obtain dryness. The exit end rail width was set to 100 percent of the incoming wet fabric width.

Preparation of Poly/Cotton Fabric and Nylon Fabric

The poly/cotton and nylon fabrics were prepared together in the winch beck in a process similar to that used for the cotton fabric. The poly/cotton and nylon fabrics were sewn end to end in a continuous loop for the preparation. The cotton process for scouring was followed through the hot rinse steps that preceded bleaching. Bath volume was 100 gallons (379 L) since two full fabrics were being treated in tandem. The adjusted chemical concentrations are reported in Table 14. After the second hot rinse, the bath was drained and refilled with cold water, and the pH was tested with pH paper. With a pH reading of 8.5, glacial acetic acid (250 g) was added to the bath to neutralize, and the pH was rechecked after 10 minutes of cycling in the cold bath. The pH after neutralization was 7.0, so the bath was dropped and one final cold rinse was performed.

Table 14: Chemical bath formulation for scouring of poly/cotton and nylon fabric samples.

Chemical Bath Ingredients	Bath Concentration (g/L)	Amount added to bath (g)
Scouring Chemical Bath (100 gallons total)		
Keiralon NF Surfactant	2.1 g/L	800 g
Soda Ash (Na_2CO_3)	5.3 g/L	2,000 g

After the cold rinse, the fabric was unloaded from the bath and plaited into a transport bin. Both substrates were dried on the full width tenter. Fabric was squeezed at 4.0 bar through padder rolls to remove excess moisture prior to drying. Exit end rail width was set to 100% of the wet fabric width. Zone one temperature was set to 320°F

and zone two was set to 300°F. Fabric run speed was adjusted between four and nine m/min as needed to obtain dryness.

Preparation of Polyester

Preparation of the 100% polyester fabric was completed in the pilot plant Theis Jet, according to procedure 1021 as outlined in the operating software package. According to this procedure, the jet was filled to the high level mark with warm water, and the fabric was loaded and sewn together into a continuous loop. With the fabric loaded, the bath was drained and refilled to a low level of approximately 100 L, and the scouring chemicals were added through the mix tank at the side of the jet. The chemical recipe followed the same recipe as used for the poly/cotton and nylon scouring procedure discussed above and outlined in Table 15. With chemicals added, the temperature was raised to 180°F and maintained for 30 minutes. The temperature was lowered to 150°F, before two full hot rinse and drain cycles were completed at approximately 100°F. During the second hot rinse, a bath sample was taken from the mix tank and pH was measured using pH paper. A pH value of 7.5 indicated that fabric was sufficiently neutral, and acetic acid addition was not needed, so steps 15 through 17 in the outlined procedure were replaced with a cool water rinse and the fabric was unloaded into a transport bin.

Table 15: Chemical recipe for scouring of polyester fabric in Theis jet.

Chemical Bath Ingredients	Approximate Formulation (g/L)	Amount added to bath (g)
Scouring Chemical Bath (100 liters total)		
Keiralon NF Surfactant	2.0 g/L	200 g
Soda Ash (Na_2CO_3)	5.0 g/L	500 g

The polyester fabric was dried on the full width tenter frame following the same procedures and machine settings discussed in the poly/cotton and nylon preparation section.

Preparation of Acrylic Samples

The acrylic fabric samples were received after the preparation of the other fabric samples had been completed. Due to the delayed arrival of these samples, the Extractables Content Analysis and pH of Hot Water Extract, which is outlined in the Test Methods section, were completed prior to fabric preparation. These tests revealed that a hot water rinse would be sufficient to remove sizing from the acrylic fabrics, and the preparation process was adjusted accordingly.

The acrylic fabrics were cut into three 18 inch wide rolls prior to preparation in the winch beck. The six full length sections were prepared in two batches according to the following procedure. The fabrics were loaded into a 100-gallon hot water bath which was heated to 180°F and maintained for 30 minutes, with the winch beck running at 35 rpm. After 30 minutes, the bath was allowed to cool to 150°F before draining and two hot rinse cycles were completed. A third cold rinse was used to reduce the fabric temperature and ensure that all extractable materials were removed. The fabric was then squeezed through the full width padder at 4.0 bar to remove excess moisture, plaited out, and placed in a standing oven overnight at 105°C to dry.

3.2.2 Initial Screening of Available Treatments

After verifying that all six fabric substrates had been well prepared by the use of the previously indicated test methods, an initial screening of the seven finishing options

previously discussed was performed. The primary objective of this screening was to select an appropriate FC control, and to eliminate any finishes which were significantly poorer performing using the available application equipment. The screening was conducted using 18-by-24 inch fabric samples, applying finishes in the standard pad-dry-cure method in the pilot plant facility at North Carolina State University. Samples were treated according to manufacturers' recommendations.

Wet Pick up of Fabric Samples

To ensure appropriate add-on, padder pressure settings were selected in order to achieve approximately 100% wet pick up (wpu) for each fabric, where possible. For each finish, several chemical baths were used in order to obtain desired levels of chemical add-on for various substrates, as recommended. The pad pressures listed in Table 16 were used as benchmarks for the application process. In several cases, the pad pressure for nylon and poly/cotton samples was reduced to 0.4 bar from the listed 0.6 value to obtain a wpu value closer to 100%. The values for wet pick up at this pressure setting are also given in Table 16.

Table 16: Pad pressures and wet pick up values used for initial product screening. Secondary pad pressures used for nylon and poly/cotton samples are listed in parentheses.

Fabric Sample	Pad Pressure (bar)	Dry Weight (g)	Wet Weight (g)	Wet Pick Up (%)
Cotton	1.4	2.7	5.6	107%
Poly/Cotton	0.6 [0.4]	2.8 [2.8]	5.1 [5.3]	82% [89%]
Polyester	2.4	4.1	8.7	112%
Nylon	0.6 [0.4]	1.2 [1.2]	2.2 [2.4]	83% [100%]
HF Acrylic	0.8	4.9	9.9	102%
AU Acrylic	0.8	4.1	8.1	98%

Wet pick up was determined by weighing a dry 4-by-6 inch fabric swatch. This swatch was then passed through a pad containing a water bath at various pressures. The padded sample was then reweighed, and the wet pick up determined by the following equation:

$$wpu(\%) = \frac{weight_{wet} - weight_{dry}}{weight_{dry}} \times 100\% \qquad (13)$$

Chemical Bath Formulations

A series of chemical baths was prepared using each of the selected finishes in order to meet the manufacturers' recommendations for chemical add-on as closely as possible. The formulations used for each application is outlined in this section.

AsahiGuard E-061

For the application of AsahiGuard E-061, two chemical baths were used. Based on manufacturer recommendations, the target value for the E-061 product was 2.0% based on the weight of the dry fabric (owf). To accomplish this, the first bath, which was used to treat the cotton, polyester, and both acrylic fabrics, was formulated using AsahiGuard E-061 (10.0g) in water (500mL), for a bath concentration of 20 g/L. The second bath, used in the treatment of poly/cotton and nylon samples was applied at 25 g/L concentration by adding E-061 (5.0g) to water (200mL). The chemical concentrations, pad pressures, and calculated add-on are outlined in Table 17.

Table 17: Summary of padder settings and finishing bath concentrations used for the initial screening application of AsahiGuard E-061.

Fabric Sample	Pad Pressure (bar)	Bath Concentration (g/L)	WPU (owf)	Theoretical Dry Add-on (% owf) (Target: 2%)
Cotton	1.4	20	107%	2.14%
Poly/cotton	0.6	25	82%	2.05%
Polyester	2.4	20	112%	2.24%
Nylon	0.6	25	83%	2.08%
HF Acrylic	0.8	20	102%	2.04%
AU Acrylic	0.8	20	98%	1.96%

Fluorochem-A1

Treatment of fabrics using finish Fluorochem-A1 was done using three bath formulations in order to reach recommended target add-on values. The first bath, used to treat the cotton and poly/cotton fabrics, had a chemical concentration of 30g/L obtained by adding Fluorochem-A1 (7.5g) to water (245mL). The second bath had a chemical concentration of 10g/L (Fluorochem-A1 (7.5g) in water (750mL)) and was used to treat the polyester, and both acrylic substrates. The final bath, used for nylon finishing, consisted of Fluorochem-A1 (3.5g) in water (250mL), for a concentration of 14 g/L. The padder settings, chemical baths, and calculated add-on values are summarized in Table 18.

Table 18: Summary of padder settings and chemical bath concentrations used for the initial screening application of Fluorochem-A1.

Fabric Sample	Pad Pressure (bar)	Bath Concentration (g/L)	WPU (owf)	Target Add-on (% owf)	Theoretical Dry Add-on (% owf)
Cotton	1.4	30	107%	3.0%	3.21%
Poly/cotton	0.6	30	82%	2.0%	2.46%
Polyester	2.4	10	112%	1.0%	1.12%
Nylon	0.6	14	83%	1.0%	1.16%
HF Acrylic	0.8	10	102%	1.0%	1.02%
AU Acrylic	0.8	10	98%	1.0%	.98%

Waterproofon 246

Waterproofon 246 was applied to fabrics in order to obtain 2% add-on to cotton and poly/cotton fabrics, and 1% add-on to the remaining synthetic substrates. To obtain these levels, a bath of Waterproofon 246 (5.0g) in water (250mL) (20g/L) was used to treat the cotton and poly/cotton substrates. For the synthetic fabrics, a 10g/L bath was made using Waterproofon 246 (6.0g) in water (600mL). The padder settings and calculated add-on values are reported in Table 19.

Table 19: Summary of padder settings and chemical bath concentrations used in initial screening application of Waterproofon 246.

Fabric Sample	Pad Pressure (bar)	Bath Concentration (g/L)	WPU (owf)	Target Add-on (% owf)	Theoretical Dry Add-on (% owf)
Cotton	1.4	20	107%	2.0%	2.14%
Poly/cotton	0.6	20	82%	2.0%	1.64%
Polyester	2.4	10	112%	1.0%	1.12%
Nylon	0.4	10	100%	1.0%	1.00%
HF Acrylic	0.8	10	102%	1.0%	1.02%
AU Acrylic	0.8	10	98%	1.0%	1.02%

Zonyl 7040

Zonyl 7040 was applied to all six fabrics using a single bath formulation, consisting of Zonyl 7040 (20.0g) in water (1.0L). The target add-on value for this treatment was 2%, so each fabric was run with the pad pressure set to achieve approximately 100% wet pick up. The pad settings and finish bath concentrations are summarized in Table 20.

Table 20: Summary of padder settings and chemical bath concentrations used in the initial screening application of Zonyl 7040 and Baygard RT.

Fabric Sample	Pad Pressure (bar)	Bath Concentration (g/L)	WPU (owf)	Target Add-on (% owf)	Theoretical Dry Add-on (% owf)
Cotton	1.4	20	107%	2.0%	2.14%
Poly/cotton	0.4	20	89%	2.0%	1.78%
Polyester	2.4	20	112%	2.0%	2.24%
Nylon	0.4	20	100%	2.0%	2.00%
HF Acrylic	0.8	20	102%	2.0%	2.04%
AU Acrylic	0.8	20	98%	2.0%	1.96%

Baygard RT

The initial screening application of Baygard RT followed the same bath concentrations and padder settings as used for the Zonyl 7040 treatment described above. Refer to Table 20 for pad pressures and bath concentrations used in this application.

Phobotone WS CONC with Phobotone BC Catalyst

A one liter bath was made for application of Phobotone WS CONC to all six substrates. A catalyst, Phobotone BC New, was added at a 1:1 ratio with Phobotone WS CONC as recommended by the manufacturer. The bath consisted of Phobotone WS CONC (25g) and Phobotone BC New (25g), in water (950mL), for a 25g/L concentration of each chemical in the total bath. Padder settings and chemical bath concentrations are listed in Table 21.

Table 21: Summary of padder pressure and chemical bath concentration for Initial Screening application of Phobotone WS CONC.

Fabric Sample	Pad Pressure (bar)	Bath Concentration (g/L)	WPU (owf)	Target Add-on (% owf)	Theoretical Dry Add-on (% owf)
Cotton	1.4	25 25*	107%	2.5% 2.5%*	2.68% 2.68%*
Poly/cotton	0.4	25 25*	89%	2.5% 2.5%*	2.23% 2.23%*
Polyester	2.4	25 25*	112%	2.5% 2.5%*	2.80% 2.80%*
Nylon	0.4	25 25*	100%	2.5% 2.5%*	2.50% 2.50%*
HF Acrylic	0.8	25 25*	102%	2.5% 2.5%*	2.55% 2.55%*
AU Acrylic	0.8	25 25*	98%	2.5% 2.5%*	2.45% 2.45%*

*Catalyst values

Norane Sil with Catalyst Sil

Norane Sil was applied to all six fabrics using a single finishing bath. The finish was applied with the required catalyst, Catalyst Sil, at a level of 60% on the weight of the Norane Sil finish. The one-liter bath was made with Norane Sil (50g), and Catalyst Sil (30g) in water (920mL). As with the Phobotone application described above, padder pressure was set to obtain approximately 100% wet pick up from all six substrates. Table 22 summarizes the padder settings and chemical bath concentrations used in this application process.

Table 22: Summary of padder pressure and chemical bath concentration for initial screening application of Norane Sil.

Fabric Sample	Pad Pressure (bar)	Bath Concentration (g/L)	WPU (owf)	Target Add-on (% owf)	Calculated Add-on (% owf)
Cotton	1.4	50 30*	107%	5.0% 3.0%*	5.35% 3.21%*
Poly/cotton	0.4	50 30*	89%	5.0% 3.0%*	4.45% 2.67%*
Polyester	2.4	50 30*	112%	5.0% 3.0%*	5.60% 3.36%*
Nylon	0.4	50 30*	100%	5.0% 3.0%*	5.00% 3.00%*
HF Acrylic	0.8	50 30*	102%	5.0% 3.0%*	5.10% 3.06%*
AU Acrylic	0.8	50 30*	98%	5.0% 3.0%*	4.90% 2.94%*

*Catalyst values

THN Oven Settings-Drying and Curing

Treated samples, with the exception of the Baygard RT finish, were dried and cured using a pilot scale THN Oven at North Carolina State University. Drying was completed at a temperature set point of 120°C. The actual run temperature was 127°C. The treated fabrics were mounted on sample racks, and passed through the oven at a speed of 0.70-0.92 m/min to obtain a dwell time of 1:30 to 2:00 as needed to achieve dryness.

Table 23: Drying conditions used for Initial Screening application, completed on the pilot scale THN oven.

Drying Set Temp (°C)	Drying Actual Temp (°C)	Drying Run Speed (m/min)	Drying Dwell Time (min)
120	127	0.70-0.92	1:30-2:00

Samples were then cured according to manufacturers' recommendations for the individual finishes. Fabrics treated with AsahiGuard E-061 and Fluorochem-A1, the two short-chain FC finishes, were cured at a temperature setting of 160°C at a run speed of

0.70 m/min to obtain a two minute dwell time. Fabrics treated with Zonyl 7040 finish were cured at 1.4 m/min with a temperature setting of 170°C, allowing for a one minute curing dwell time. Samples finished using Waterproofon 246, Phobotone WS CONC, and Norane Sil finishes were all cured at 1.4 m/min, to obtain a one minute dwell time, with a temperature setting of 180°C. Recorded oven temperature for all curing processes was 6-7 degrees higher than the set point, as reported in Table 24. This discrepancy is not believed to have any significant effect on the performance of the finishes. The nylon samples exhibited brown streaks, indicating potential scorching, after the curing procedure for all finishes tested.

Table 24: Curing parameters used for the initial screening application on the pilot scale THN Oven. Baygard RT samples were not dried and cured, as the product is designed to air dry.

Finishing Treatment	Curing Set Temp (°C)	Curing Actual Temp (°C)	Curing Run Speed (m/min)	Curing Dwell Time (min:sec)
AsahiGuard E-061	160	166	0.7	2:00
Fluorochem-A1	160	167	0.7	2:00
Zonyl 7040	170	176	1.4	1:00
Waterproofon 246	180	186	1.4	1:00
Phobotone WS CONC	180	187	1.4	1:00
Norane Sil	180	187	1.4	1:00

The Baygard RT finish was dried separately, on a vertical hanger in a Despatch oven. The recirculating fan was run, but the oven was not heated, and the fabric samples were dried overnight.

3.2.3 Continuous Application of Finishes Durability Evaluation

Following the initial screening process, and elimination of several finishes, a continuous pad application was designed to evaluate the durability properties of the finishes, and to examinine the influence of isocyanate and melamine resins on the performance and wash durability of the fabric samples. As previously discussed, three hydrocarbon waxes were also added to the finish selection for continuous application. In all, fifteen treatment combinations were applied and evaluated. In addition to an evaluation of repellency, physical properties including tear strength, stiffness, and color change were evaluated after the finishes were applied. Finish durability was studied by reevaluating the repellency performance after five and twenty-five wash cycles as is described in the durability section.

To perform this evaluation, five-yard sections of each of the six fabrics were sewn end-to-end to create a fabric "set." The sets were sewn together, separated by eight yards of leader fabric to allow for the pad to be cleaned between finish treatments. This arrangement allowed the fabrics to be treated continuously, with each set of six fabrics being treated from a common, 4-L chemical bath, as described. This continuous application process is detailed in the following sections.

Wet Pick up of Fabric Samples and Padder Settings

Due to the continuous treatment of fabric samples, it was not possible to individually select padded settings for individual fabrics as done in the initial screening. For this reason, a moderate padder pressure of 1.5 bar was selected, and the wet pick up for each

of the six substrates was calculated as described previously. The wet pick up values calculated are shown in Table 25.

Table 25: Calculated wet pick up values for continuous application of finishes. Padder pressure was set to 1.5 bar.

Fabric Sample	Dry Fabric Weight (g)	Wet Fabric Weight (g)	Calculated Wet Pick Up (% owf)
Cotton	2.8	5.7	104%
Poly/Cotton	2.6	4.3	65%
Polyester	6.1	14.4	136%
Nylon	1.5	2.4	60%
HF Acrylic	5.6	11.2	100%
AU Acrylic	4.9	9.7	98%

During the continuous treatment, the THN oven speed was set at 0.91 m/min, as is discussed later. The padder speed was set to 0.7-0.8 m/min due to a difference in calibration between the padder and THN oven speeds. The padder speed was adjusted as needed to match the speed of the THN oven.

After each treatment set was complete, the leader fabric was allowed to run through the padding and drying inline system consisting of the pilot scale HVP padder and THN oven. Once the treated fabric set was completely through the THN oven, the fabric was stopped and cut out from the padder. The pad bath was then drained and the entire padder was cleaned well with hot water. The pad trough was then dried and the drain was closed. After cleaning, the leader fabric was rethreaded through the padder and resewn in continuous fashion, and the subsequent finishing bath was added. This process was completed after each of the fifteen finishing treatments.

Chemical Bath Formulations

This section discusses the finishing bath formulations evaluated in the continuous application. Fifteen treatments were applied to each of the six fabrics, as outlined below. All baths discussed were 4kg total weight. A complete outline of chemical bath formulations, along with THN oven parameters for the continuous application procedure can be found in Appendix B. Table 26 outlines the fifteen treatments and the type of chemistries used in each.

Table 26: Overview of finishing treatments used for primary evaluation of finish performance.

Treatment	Primary Chemical	Secondary Chemical	Finish Type
SI-1	Norane Sil	Catalyst Sil	Polysiloxane
SI-2	Phobotone WS CONC	Phobotone BC New	Polysiloxane
HR-3	Norane 100	--	Reactive Wax
HZ-4	Impregnole FH	--	Zirconium Wax
HZ-5	Freepell CCS	--	Zirconium Wax
FC-6	Waterproofon 246	--	C-8 FC
FC-7	AsahiGuard E-061	--	Short Chain FC
FC-8	Fluorochem A1	--	Short Chain FC
FM-9	Waterproofon 246	Aerotex M3	C-8 FC with Melamine Resin
FM-10	AsahiGuard E-061	Aerotex M3	Short Chain FC with Melamine Resin
FM-11	Fluorochem A1	Aerotex M3	Short Chain FC with Melamine Resin
FI-12	Waterproofon 246	TMI[META]	C-8 FC with Isocyanate Crosslinker
FI-13	Waterproofon 246	Apexosist 186	C-8 FC with Isocyanate Crosslinker
FI-14	AsahiGuard E-061	Apexosist 186	Short Chain FC with Isocyanate Crosslinker
FI-15	Fluorochem A1	Apexosist 186	Short Chain FC with Isocyanate Crosslinker

Treatment SI-1: Norane Sil with Catalyst Sil

The chemical bath applied in Treatment SI-1 mirrored the chemical concentrations used for the initial screening. Norane Sil (200g) and Catalyst Sil (120g) were added to water (approximately 2kg) in a two-gallon steel beaker. Water was added to obtain a total bath of 4.0kg. Chemical concentrations for the treatment are outlined in Table 27.

Table 27: Finish formulation for Treatment SI-1 in the continuous application procedure. Chemicals are listed in their order of addition to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
Norane Sil	200 g	50 g/L
Catalyst Sil	120 g	30 g/L

Treatment SI-2: Phobotone WS CONC with Phobotone BC New

For the continuous application, the concentrations of Phobotone WS CONC and Phobotone BC New were increased from the levels used in the initial screening. Phobotone WS CONC (120g) was diluted with an equal volume of water. Phobotone BC New (160g) was also diluted 1:1 with water. Glacial Acetic Acid (5g) was added to water (approximately 2 kg) to help maintain a slightly acidic bath, before the diluted finish and catalyst were added to the bath and mixed. Water was then added to bring the total bath to the desired 4.0 kg. Table 28 outlines the chemical formulation for this treatment.

Table 28: Finish formulation for Treatment SI-2 in the continuous application procedure. Chemicals are listed in their order of addition to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
Acetic Acid (glacial)	5 g	1.25 g/L
Phobotone WS CONC	120 g	30 g/L
Phobotone BC New	160 g	40 g/L

Treatment HR-3: Norane 100

The first hydrocarbon wax tested was Norane 100, which is a reactive wax-based finish. This product was applied at 120 g/L, which consisted of Norane 100 (480g) and water (3.52 kg) for a 4-kg total bath. As with prior treatments, the finish was mixed into

approximately half the total bath mixture. The bath was then brought to the desired 4.0 kg weight by adding water. This treatment is shown in Table 29.

Treatment HZ-4: Impregnole FH

Treatment H4 contained Impregnole FH (400g), a zirconium wax-based finish. The specified amount of finish was added to approximately half the total bath and mixed, before water was added to achieve the desired weight. This treatment is shown in Table 29.

Treatment HZ-5: Freepell CCS

The second zirconium wax finish tested was Freepel CCS, which was added at 7% on the weight of the bath. Freepel CCS (280g) was added to approximately half of the total bath volume. Water was added to bring the bath to the desired 4.0 kg total weight. Table 29, below, shows this bath.

Table 29: Finish formulations for Treatments HR-3, HZ-4, and HZ-5 in the continuous application procedure. Total bath weight was 4.0 kg for each specified treatment.

Treatment	Chemical	Chemical Weight (g)	Concentration (g/L)
HR-3	Norane 100	480 g	120 g/L
HZ-4	Impregnole FH	400 g	100 g/L
HZ-5	Freepel CCS	280 g	70 g/L

Treatment FC-6: Waterproofon 246

Treatments 6-9 consisted of FC products tested without the addition of crosslinking resins. For treatment FC-6, Waterproofon 246 (100g) was added to water (approximately 2 L). Additional water was added to achieve the target weight (4.0kg). This treatment is shown in Table 30, below.

Treatment FC-7: AsahiGuard E-061

Treatment FC-7 contained AsahiGuard E-061 at 2.5% owb. Finish (100g) was mixed into water (2 L), approximately half the total bath volume. Water was then added to obtain the total bath weight of 4.0 kg. This treatment is shown in Table 30.

Treatment FC-8: Fluorochem-A1

Fluorochem-A1 was applied in a finishing bath at 2.5% owb. Finish (100g) was added to half the bath volume of water (approximately 2 L). Additional water was added to achieve the total bath weight of 4.0 kg. This treatment is shown in Table 30.

Table 30: Finish formulations for Treatments FC-6, FC-7, and FC-8 in the continuous application procedure. Total bath weight was 4.0 kg for each treatment specified.

Treatment	Chemical	Chemical Weight (g)	Concentration (g/L)
FC-6	Waterproofon 246	100 g	25 g/L
FC-7	AsahiGuard E-061	100 g	25 g/L
FC-8	Fluorochem-A1	100 g	25 g/L

Treatment FM-9: Waterproofon 246 with Aerotex M3 Resin

Treatments FM-9 through FM-11 consisted of FC finishes combined with Aerotex M3 melamine resin to evaluate the durability and performance effects of the resin. For Treatment FM-9, Waterproofon 246 (100 g) and Aerotex M3 (30 g) resin were added separately to approximately two liters of water. Additional water was added to achieve the total bath weight of 4.0 kg. This finish formulation is outlined in Table 31.

Table 31: Finish formulation for Treatment FM-9 in the continuous application procedure. Chemicals are listed in the order they were added to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
Waterproofon 246	100 g	25 g/L
Aerotex M3	30 g	7.5 g/L

Treatment FM-10: AsahiGuard E-061 with Aerotex M3 Resin

The finishing bath for Treatment FM-10 was created by adding AsahiGuard E-061 (100 g) and Aerotex M3 (30 g) resin to approximately two liters of water. The target weight of 4.0 kg was achieved by adding water to weight. This formulation is outlined below, in Table 32.

Table 32: Finish formulation for Treatment FM-10 in the continuous application procedure. Chemicals are listed in the order they were added to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
AsahiGuard E-061	100 g	25 g/L
Aerotex M3	30 g	7.5 g/L

Treatment FM-11: Fluorochem-A1 with Aerotex M3 Resin

Treatment FM-11 consisted of Fluorochem-A1 (100 g) and Areotex M3 (30 g) in a 4.0 kg total bath. The finish and resin were added individually to approximately 2.0L water. The total bath weight was achieved by the addition of water to the target weight. Table 33 outlines this formulation.

Table 33: Finish formulation for Treatment FM-11 in the continuous application procedure. Chemicals are listed in the order they were added to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
Fluorochem-A1	100 g	25 g/L
Aerotex M3	30 g	7.5 g/L

Treatment FI-12: Waterproofon 246 with TMI[META]

The finishing bath for Treatment FI-12 consisted of Waterproofon 246 (100 g) and TMI[META] (40 g), isocyanate crosslinker. The chemicals were added individually to approximately two liters of water. The total bath weight was achieved by adding water to a total of 4.0 kg. This was the only treatment which utilized the TMI[META] crosslinker as the product was immiscible in the water bath and produced an extremely irregular finish and showed significant residue build up on the padder rolls. This formulation is outlined in Table 34.

Table 34: Finish formulation for Treatment FI-12 in the continuous application procedure. The chemicals are listed in the order in which they were added to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
Waterproofon 246	100 g	25 g/L
TMI[META]	40 g	10 g/L

Treatment FI-13: Waterproofon 246 with Apexosist 186

Treatments FI-13 through FI-15 consisted of FC finishes in conjunction with Apexosist 186, an isocyanate crosslinking agent. These treatments were used to evaluate the effect of the crosslinker on the durability and performance of the finishes. Treatment FI-13 consisted of Waterproofon 246 (100 g) with Apexosist 186 (40 g). The chemicals were added to approximately two liters of water individually. The total bath weight was

brought to the desire 4.0 kg with additional water. This treatment is outlined below in Table 35.

Table 35: Finish formulation for Treatment FI-13 in the continuous application procedure. The chemicals are listed in the order in which they were added to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
Waterproofon 246	100 g	25 g/L
Apexosist 186	40 g	10 g/L

Treatment FI-14: AsahiGuard E-061 with Apexosist 186

The finishing bath for Treatment FI-14 contained AsahiGuard E-061 (100 g) and Apexosist 186 (40 g), isocyanate crosslinker. The chemicals were added individually to approximately two liters of water. Additional water was added to obtain the total bath weight of 4.0 kg. This formulation is shown in Table 36.

Table 36: Finish formulation for Treatment FI-14 in the continuous application procedure. The chemicals are listed in the order in which they were added to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
AsahiGuard E-061	100 g	25 g/L
Apexosist 186	40 g	10 g/L

Treatment FI-15: Fluorochem-A1 with Apexosist 186

The final treatment in the continuous application process utilized Fluorochem A1 (100 g) and Apexosist 186 (40 g). The chemicals were individually added to approximately two liters of water. Water was added to obtain the total bath weight of 4.0 kg. This formulation is shown in Table 37.

Table 37: Finish formulation for Treatment FI-15 in the continuous application procedure. The chemicals are listed in the order in which they were added to the bath.

Chemical	Chemical Weight (g)	Concentration (g/L)
Fluorochem A1	100 g	25 g/L
Apexosist 186	40 g	10 g/L

THN Oven Settings-Drying and Curing

As previously discussed in the Padder Settings section, the continuous nature of treatment for this phase of the experimentation did not allow for multiple machine settings for each fabric and finish combination.

The drying process occurred inline with the pad application of finishing chemicals. The oven speed was set to 0.91 m/min to obtain a two minute dwell time, which was maintained throughout the drying process for all treatments. The collection roll at the exit end of the THN oven was adjusted to maintain a minimum amount of tension on the dried fabric without introducing overstretching the fabric or allowing it to become loose. The collection roll was able to hold four sets of fabric before becoming full. At this point the collection roll was doffed and replaced. The dried fabric was then backwound onto a cardboard core for the curing process.

The target drying temperature was 135°C. The initial temperature set point was 135°C, which was adjusted to 132°C so that the maximum drying temperature of 140°C was not exceeded. Drying set points and actual run temperatures are shown in Table 38. A complete review of finish formulations, oven set points and actual temperature ranges for drying and curing of the continuous application procedure is available in Appendix B.

Table 38:THN Oven set points and observed temperature ranges for each of the 15 treatments applied in the continuous application procedure.

Treatment	Drying Set Point (°C)	Observed Min. Temp (°C)	Observed Max Temp. (°C)
SI-1	135	136	139
SI-2	135	136	139
HR-3	135	138	138
HZ-4	135	134	140
HZ-5	132	135	137
FC-6	132	134	135
FC-7	132	134	135
FC-8	132	135	137
FM-9	132	135	136
FM-10	132	133	136
FM-11	132	136	137
FI-12	132	136	136
FI-13	132	135	136
FI-14	132	134	137
FI-15	132	135	137

After drying, all fifteen fabric sets were cured continuously on the THN oven. The set temperature on the oven was 180°C and the observed run temperatures were 184-185°C. The oven was set to 1.8 m/min run speed to obtain a one-minute dwell time.

After curing, each fabric sample was cut into three equal pieces for washing and durability testing. Test methods for unwashed fabric samples and durability studies are described below.

3.2.4 Durability

Durability analysis was done at short and long term levels for the fabrics treated in the continuous application process. Repellency performance was measured after five and

twenty-five washes for short and long term durability evaluation, as is described in the following sections.

Short Term Durability

Short term durability was evaluated for fabrics after five standard wash cycles. The fifteen treatment sets were divided into two loads for washing. One load contained all the cotton, poly/cotton, and polyester fabric samples. The second load contained the nylon and acrylic fabric samples. Each load contained approximately six pounds of fabric.

The five wash cycles were completed on a Whirlpool Design 2000 machine. The Regular ten minute wash cycle was selected, with a warm wash temperature, and cold rinse temperature. A large load size setting was selected. Each load was washed using AATCC Standard Detergent without optical brighteners (110 g).

Each wash cycle was followed by a 30-minute drying cycle in a Kenmore 700 series dryer at Medium/High temperature setting. Following the five washing and drying cycles, a sixth cycle was completed following the same procedure as the first 5, without the use of detergent in order to ensure that all detergent was effectively removed from the fabric prior to testing.

Short term durability was evaluated using the Spray Test, Hydrocarbon Resistance Test, and Water/Alcohol Solution Resistance Test, which are discussed in the Test Methods section.

Long Term Durability

Long term durability was evaluated for fabric samples from treatments SI-2, HR-3, FC-6, FC-7, FI-13, and FI-14. Twenty-five washing and drying cycles were completed for all six fabric samples for each of these treatment sets.

Cycles 1-5, 12-15, and 22-25 were completed on a Whirlpool Design 2000 machine, as described in the Short Term Durability section. These wash cycles were followed by drying in the Kenmore 700 series dryer as described above.

The remaining cycles, 6-11, and 16-21 were completed on a Whirlpool WTW5100S machine. The Regular ten-minute wash cycle, which has a warm wash cycle and cold water rinse cycle, was chosen. Each load was washed with AATCC Standard Detergent without optical brighteners (110 g). These wash cycles were followed by 30-minute drying cycles completed in a Whirlpool WED5300S Dryer on the Normal drying setting with a high temperature setting.

Long term durability of the finish treatments was evaluated using the Spray Test, Hydrocarbon Resistance Test, and Water/Alcohol Solution Resistance Test, which are discussed in the Test Methods section.

3.3 Test Methods

The following sections outline the test methods used in the evaluation of the fabrics at various stages of the experimental process, from fabric preparation through durability evaluation. Any necessary modifications that were made to the test methods are noted.

3.3.1 pH of the Water-Extract – AATCC TM 81-2001

AATCC Test Method 81 was used to verify the preparation of the fabrics prior to finish applications. The test required a 10±1 g sample of each fabric. Six 250-mL volumes of distilled water were boiled in 400 mL beakers for ten minutes on a hotplate. The fabric samples were immersed in the water, and the beaker was covered with a watch glass and boiled for an additional ten minutes. The beakers were then removed from the hotplate, uncovered and allowed to cool to room temperature. The fabric specimens were removed from the beakers and the pH of the excess liquid was measured with a handheld pH meter. The pH meter was calibrated with 4.0, 7.0, and 10.0 pH buffer solutions prior to testing. The target pH range for the water extract was 6-9[49].

3.3.2 Extractable Content – AATCC TM 97-1999

AATCC Test Method 97 outlines the procedure to quantify the amount of extractable materials in prepared textiles. The test method was modified and only water and 1,1,1-trichloroethane extractions were analyzed.

Samples of each fabric weighing approximately ten grams were cut and put in a glass weighing jar, in the Despatch oven set at 105±3°C for four hours. Samples were

removed from the over and placed in a desiccator overnight. The initial sample mass was then measured on a Mettler AG260 analytical balance.

The samples were then placed in beakers containing water (200 mL), and maintained at a temperature of 180±5°C for two hours. After two hours, the water and specimen were poured into a Buchner funnel, and then fabric was rinsed with two 25-mL aliquots of distilled water. The fabric samples were then returned to the weighing jars and placed in the oven at 105±3°C for four hours. The fabric specimens were removed from the oven and placed in a desiccator overnight to cool. The fabrics were reweighed on the Mettler balance, and the water extractable content was determined as the percent change in fabric weight.

The 1,1,1-trichloroethane extractable materials such as oils, fats, and waxes were quantified using a Soxhlet extractor. The fabric samples were placed in cellulose thimbles and extracted for 2.5 hours to complete 12-16 extraction cycles with 1,1,1-trichloroethane (approximately 150 mL). The thimbles were removed from the Soxhlet apparatus, and evaporated to dryness under a fume hood overnight. The specimens were removed from the thimbles and returned to the weighing bottles, and placed in an oven at 105±3°C for four hours. The fabric samples were then removed from the bottles, and cooled in a desiccator overnight. The samples were reweighed on the Mettler balance to determine the extractable content. Several samples showed higher weights than previously recorded, so the samples were returned to the oven for four hours, and placed in the desiccator overnight. Again, the samples were weighed on the Mettler analytical balance.

For both water and 1,1,1-trichloroethane extractable content, a value of less than 0.4% was desired to verify the fabric preparation, prior to finishing[50].

3.3.3 Water/Alcohol Solution Resistance Test – AATCC TM 193-2004

The Aqueous Liquid Repellency Test outlined in AATCC Test Method 193 is useful in the evaluation of the repellent properties of textile surfaces. The test utilizes a series of standard solutions of distilled water and isopropyl alcohol, having decreasing surface tensions. Standard solutions and the respective surface tensions are listed in Table 39. Standard conditions were not achievable in the testing environment, so samples were conditioned to 70±5°F and 35±3% relative humidity.

Table 39: Standard Test Solutions and surface tensions for Aqueous Liquid Repellency, from AATCC Test Method 193-2004.

Liquid Composition Water:Isopropyl Alcohol (v:v)	Aqueous Liquid Repellency Grade Number	Liquid Surface Tension (dynes/cm at 25°C)
None (Fails 98% Water)	0	--
98:2	1	59.0
95:5	2	50.0
90:10	3	42.0
80:20	4	33.0
70:30	5	27.5
60:40	6	25.4
50:50	7	24.5
40:60	8	24.0

To begin the test, five drops of the first standard solution are placed on a 8x8 inch sample of the fabric across the filling direction, as described in the test method. If, after ten seconds no wetting occurs, proceed to the next standard solution and repeat the drops.

A failed test occurs when three of the five drops in a series exhibit complete wetting or wicking as shown in drops C and D in Figure 11. A pass occurs when three of

the five drops show no wetting or darkening of the fabric surface, as seen in drop A, below. Drop B illustrates a borderline pass in which three of the five drops maintain a rounded contact angle with slight darkening of the fabric surface. In the case of a borderline pass, the reported value is 0.5 lower than the borderline pass liquid rating.

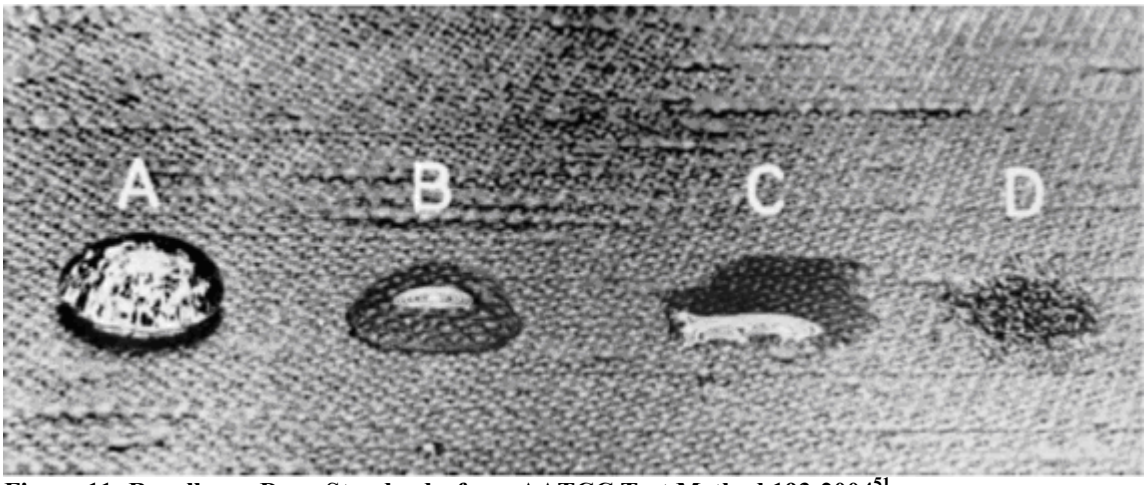


Figure 11: Repellency Drop Standards, from AATCC Test Method 193-2004[51].

The test is repeated on a second fabric sample. If the two tests agree, the value is reported and no further testing is needed. If the first two tests result in different repellency ratings, a third test must be completed. The reported value is then shared value of two of the three tests, or the median value if none of the three agree[51].

3.3.4 Hydrocarbon Resistance Test – AATCC TM 118-2002

The Oil Repellency Test described in AATCC Test Method 118 follows a similar procedure to that outlined about for Test Method 193. The test utilizes a series of standard hydrocarbon solutions with decreasing surface tensions to evaluate the repellent properties of a textile surface.

Table 40: Standard Test Solutions and surface tensions for Oil Repellency Test, from AATCC Test Method 118-2002

Liquid Composition	AATCC Oil Repellency Grade Number	Liquid Surface Tension (dyne/cm at 25°C)
None (Fails Kaydol Test)	0	--
Kaydol	1	31.2
65:35 Kaydol: n-hexadecane by volume	2	28.7
n-hexadecane	3	27.1
n-tetradecane	4	26.1
n-dodecane	5	25.1
n-decane	6	23.5
n-octane	7	21.3
n-heptane	8	19.8

As with the Aqueous Liquid Repellency Test, standard conditions were not achievable in the testing environment, so samples were conditioned to 70±5°F and 35±3% relative humidity. The Oil Repellency Test requires a 30-second test period, while the Aqueous Repellency Test only requires a ten-second test period. Other than this exception, the Oil Repellency Test was performed according to the sample procedures and standards at Test Method 193, described above[52].

3.3.5 Water Repellency: Spray Test – AATCC TM 22-2001

The spray test is the most commonly used test method to simulate exposure to rain, and is useful to evaluate the performance of a repellent finish applied to a textile substrate. For the test, the fabric was mounted in a 6" embroidery hoop. The hoop is mounted on a stand at a 45° angle, with the fabric warp direction oriented vertically. Distilled water (250 mL) was sprayed on the fabric from a height of 6 inches. The wetting pattern of the fabric is compared to standards provided in the test method, and the repellency is rated

with a grade of 0, 50, 70, 80, 90, or 100. Samples scoring above 50 are able to be given intermediate values. For each fabric and finish combination to be evaluated, three replicates were performed, as recommended[53].

3.3.6 Fluorine Content Analysis by ISE

Fluorine content analysis for four treatment sets was conducted by Galbraith Laboratories, Inc. in Knoxville, TN. Samples (two-inch by four-inch) of untreated fabrics, and fabrics from treatments FC-6, FC-7, and FC-8 were sent to Galbraith Labs for analysis. Fabric samples were digested and Fluorine content was determined at a ppm level through the use of an Ion Specific Electrode.

3.3.7 Evaluation of Color Change

Fabric color change after finishing was evaluated using an X-Rite SP64 handheld spectrophotometer with the Color iControl software package by Gretag-MacBeth. Fabric swatches (8x4 inch) used for stiffness evaluation were used for color testing. An untreated sample of each fabric was used as a standard, and two samples of fabric with each finishing treatment were evaluated. Each measurement consisted of four readings take from the sample, with the fabric repositioned after each reading. Values were reported for ΔL^*, Δa^*, Δb^*, and $\Delta Ecmc$. A $\Delta Ecmc$ value of 1.0 was used to determine a pass/fail rating for each sample tested.

3.3.8 Tear Strength Evaluation - ASTM-D2261

Tear strength evaluation using the tongue tear method on the MTS Q-Test tensile tester was performed on the continuous application treatments prior to washing. The Q-test

was set using the 250-lb load cell with a jaw pressure of 80 psi, and was calibrated prior to testing. For this test, three samples of each fabric and finish combination were tested. Samples were conditioned overnight to standard conditions of 70±2°F and 65±2% relative humidity. Samples were eight inches by three inches, with a three inch cut, as described in ASTM-D2261. All samples were torn in the warp direction, tearing the fill yarns. Reported values are the average of the five highest peaks as the tear propagated from 30 to 110 mm of jaw separation from the starting point.

Polyester samples for finishes SI-1, SI-2, HR-3, HZ-4, and HZ-5 required the use of rubber grips between the Q-Test clamps in order to prevent fabric slippage during testing[54].

3.3.9 Stiffness Evaluation – ASTM-D4032

Fabric stiffness was evaluated for the continuous application treatments prior to washing to determine the effect of the finishes. The fabrics were tested by the circular bend method as outlined in ASTM-D4032. The stiffness tester used had a digital gauge with a pneumatic actuator. The gauge was set to display the max force in pounds recorded as the fabric sample was engaged.

As outlined in the test method, two 8x4 inch samples of each fabric and finish combination were tested. The fabric samples were conditioned to standard conditions of 70±2°F and 65±2% relative humidity prior to testing. The samples were folded in half and placed on the 4x4 inch platform on the tester. The plunger was then actuated, and the max value was recorded for each fabric sample tested[55].

Chapter 4 : Results and Discussion

The results of the above described experimental procedure are discussed below. The initial screening and selection process is discussed, and the results of the continuous application experimentation are presented.

4.1 Fabric Preparation

After fabrics were prepared using the procedures described, the preparation was verified using the previously specified test methods. Results of these tests are presented in this section.

4.1.1 pH of the Water-Extract

The pH of Water-Extract test was performed as described. The target for this test was pH values between six and nine. Results are shown in Table 41.

Table 41: pH of Water-Extract. Cotton fabric tested by Marc C. Mathews

Fabric	pH of the Water-Extract
Cotton	8.26
Poly/Cotton	8.26
Polyester	7.79
Nylon	8.62
HF Acrylic	7.54
AU Acrylic	7.74

4.1.2 Extractable Content

The modified Extractable Content of Greige and/or Prepared Fabrics test was performed as detailed in Section 3.3.2. Extractable content for both hot water and 1,1,1-trichloroethane should be not greater than 0.4% for well-prepared textiles. Results of this

test are shown in Table 42. For acrylic fabrics, testing was performed prior to preparation. Initial results showed high water extractable content for AU Acrylic (0.9%) and HF Acrylic (0.8%). Water extractable content was retested after preparation and these results are shown in Table 42. 1,1,1-trichloroethane extractable content was acceptable for both fabrics prior to preparation, so retesting was not required.

Table 42: Extractable Content results.

Fabric	Hot Water Extractable Content	TCE Extractable Content
Cotton	<0.2%	<0.2%
Poly/Cotton	<0.2%	<0.2%
Polyester	<0.2%	0.40%
Nylon	0.35%	0.20%
AU Acrylic	0.37%	<0.2%*
HF Acrylic	<0.2%	<0.2%*

*Tests performed prior to preparation.

4.2 Initial Screening and Chemical Selection

The initial screening process was used to eliminate any products that failed to achieve repellency after application and to select a product to use as a traditional FC standard for comparison. The results of this screening process are outlined below.

4.2.1 Water/Alcohol Repellency Test

The Aqueous Liquid Repellency Test was performed as described in Section 3.2.3. Results from this test are summarized in Table 43. These results were considered in selecting chemicals to be used for the continuous application process.

Table 43: Results of Aqueous Liquid Repellency Test for initial screening process

Treatment / Fabric	AsahiGuard E-061	Fluorochem A1	Waterproofon 246	Zonyl 7040	Baygard RT	Phobotone WS	Norane Silicone	Untreated
AU Acrylic	6.5	3.0	8.0	8.0	5.5	3.0	3.0	0
HF Acrylic	5.0	3.5	8.0	8.0	5.5	3.0	4.0	0
Cotton	5.0	3.5	7.5	5.0	4.0	3.5	3.5	0
Polyester	8.0	4.0	8.0	8.0	4.0	3.0	4.0	0
Nylon	6.0	3.5	8.0	4.5	3.5	3.0	3.5	0
Poly/Cotton	5.5	3.5	8.0	4.5	4.5	3.5	3.5	0

4.2.2 Oleophobicity Test

The Hydrocarbon Resistance Test was performed according to the method described in Section 3.2.4. Results from this test are summarized in Table 44.

Table 44: Results of Hydrocarbon Resistance Test for initial screening process

Treatment / Fabric	AsahiGuard E-061	Fluorochem A1	Waterproofon 246	Zonyl 7040	Baygard RT	Phobotone WS	Norane Silicone	Untreated
AU Acrylic	5.5	4.0	6.5	6.0	4.0	0	0	0
HF Acrylic	1.5*	6.0	6.5	6.0	5.0	0	0	0
Cotton	3.0	4.5	6.5	5.0	2.5	0	0	0
Polyester	6.0	5.0	7.0	6.5	4.5	0	0	0
Nylon	6.5	3.0	6.5	0.5	2.5	0	0	0
Poly/Cotton	4.5	6.0	6.5	0.5	2.5	0	0	0

The AsahiGuard E-061 finish applied to HF Acrylic substrate resulted in an uneven finish, as noted by large discrepancies in the repellent properties across a small localized region of fabric. This value is not considered to accurately represent the performance of the chemical finish. The remaining data were considered in selecting chemicals to be used in the continuous application process.

4.2.3 Chemical Selection

Based on the test results given above, Zonyl 7040 was eliminated from consideration, and Waterproofon 246 was selected as the standard for tradition FC chemistries. This selection was made due to the poor performance of Zonyl 7040 on nylon and poly/cotton substrates. A comparison of these two products is shown in Figure 12.

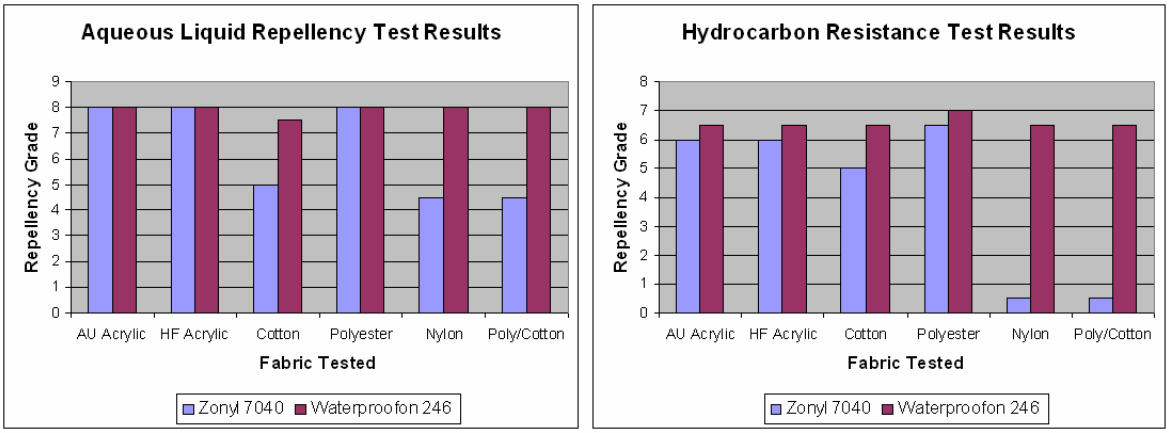


Figure 12: Test results for initial screening of Waterproofon 246 and Zonyl 7040 FC finishes.

Additionally, Baygard RT was eliminated from consideration, as the product was removed from the market by the manufacturer and was no longer available. All remaining treatment options were selected for use in the continuous application process. The two polysiloxane products performed comparably to each other. The novel FC finishing options showed some marked differences. Specifically, AsahiGuard E-061 performed significantly better on the Aqueous Repellency Test compared to the performance of Fluorochem A1. However, results from the hydrocarbon repellency test showed this product performing comparably to AsahiGuard E-061. In fact, Fluorochem A1 produced higher repellency grades for Hydrocarbon Repellency than for Aqueous Liquid Repellency for all substrates, with the exception of nylon (Figure 13).

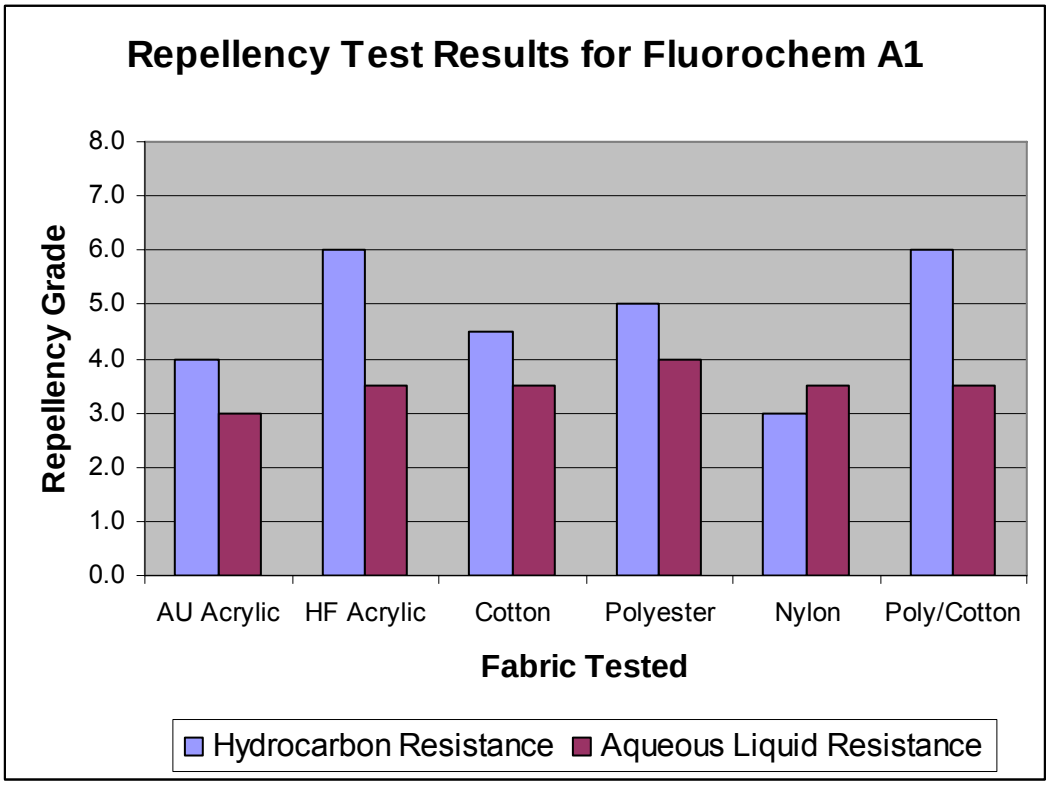


Figure 13: Repellency test results for initial screening of fabrics treated with Fluorochem A1.

4.3 Continuous Application

The test results from the continuous application process are given in the following section. The interpretation and discussion section analyzes the data on a fabric-specific level.

4.3.1 Test Results

Outlined below are the test results for the continuous application process. All tests were performed according to the methods described in the Experimental chapter.

Chemical Add-On

Theoretical chemical add-on was calculated using wet pick up values listed in Table 25 and dehydrated chemical weights (Table 11). The calculated add-on values are given in Table 45.

Table 45: Calculated chemical add-on levels for all treatment combinations as a function of bath formulation, dehydrated chemical concentration, and fabric wet pick up.

Calculated Total Dry Chemical Add-on (% owf)						
Finish Bath	**Cotton**	**Poly/Cotton**	**Polyester**	**Nylon**	**Auto Acrylic**	**Home Acrylic**
SI-1	1.53	0.96	2.00	0.88	1.44	1.47
SI-2	2.88	1.80	3.76	1.66	2.71	2.77
HR-3	2.59	1.62	3.39	1.50	2.45	2.50
HZ-4	1.80	1.13	2.36	1.04	1.70	1.73
HZ-5	1.78	1.11	2.32	1.02	1.67	1.71
FC-6	0.70	0.44	0.92	0.40	0.66	0.67
FC-7	0.39	0.24	0.51	0.23	0.37	0.38
FC-8	0.63	0.39	0.82	0.36	0.59	0.60
FM-9	1.28	0.80	1.67	0.74	1.20	1.23
FM-10	0.97	0.60	1.26	0.56	0.91	0.93
FM-11	1.20	0.75	1.57	0.69	1.13	1.16
FI-12	0.70	0.44	0.92	0.41	0.66	0.68
FI-13	0.86	0.54	1.13	0.50	0.81	0.83
FI-14	0.55	0.35	0.72	0.32	0.52	0.53
FI-15	0.79	0.49	1.03	0.46	0.75	0.76

Spray Test

Spray test results for unwashed fabric samples are listed below. Average values for three replicates are displayed in Figure 14. For a complete review of all test results, refer to Appendix C. A similar trend can be seen for the six fabrics with respect to the relative performance of the fifteen finish combinations.

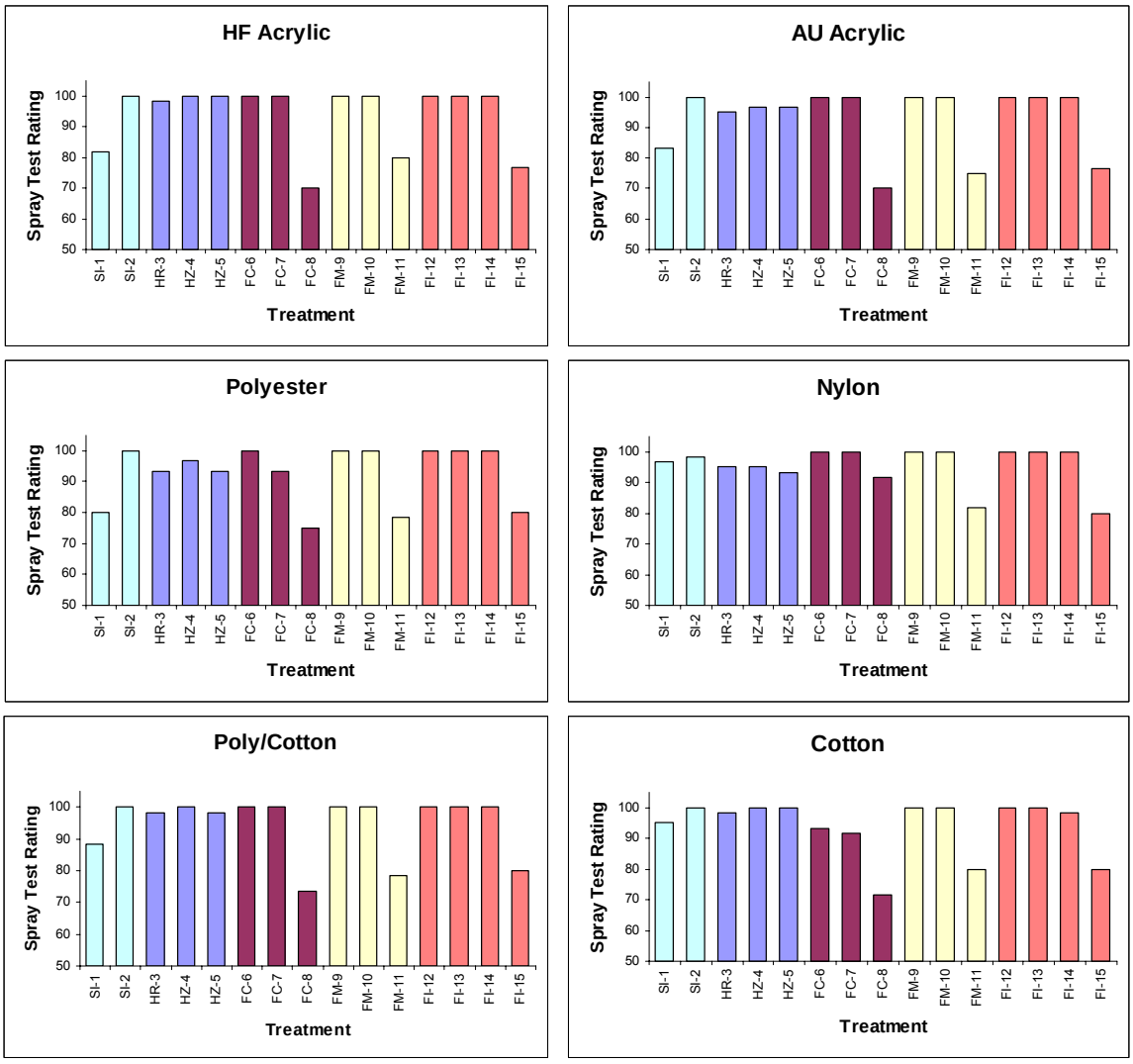


Figure 14: Spray Test results for unwashed continuous application samples. Results reported by fabric type and colored according to type of finishing treatment.

Aqueous Liquid Repellency

The Aqueous Liquid Repellency Test was completed as described in Section 3.3.3. Shown in Figure 15 are the reported values from the samples tested. Data for individual fabric types are presented. It can bee seen from the graphs that, in general, FC finishes performed better than the polysiloxane or hydrocarbon products for this test method. The AsahiGuard E-061 treated samples (FC-7, FM-10, and FI-14) performed comparably to

traditional FC finishes (FC-6, FM-9, FI-12, and FI-13) for all fabrics, except those containing cotton. Complete test results can be found in Appendix C.

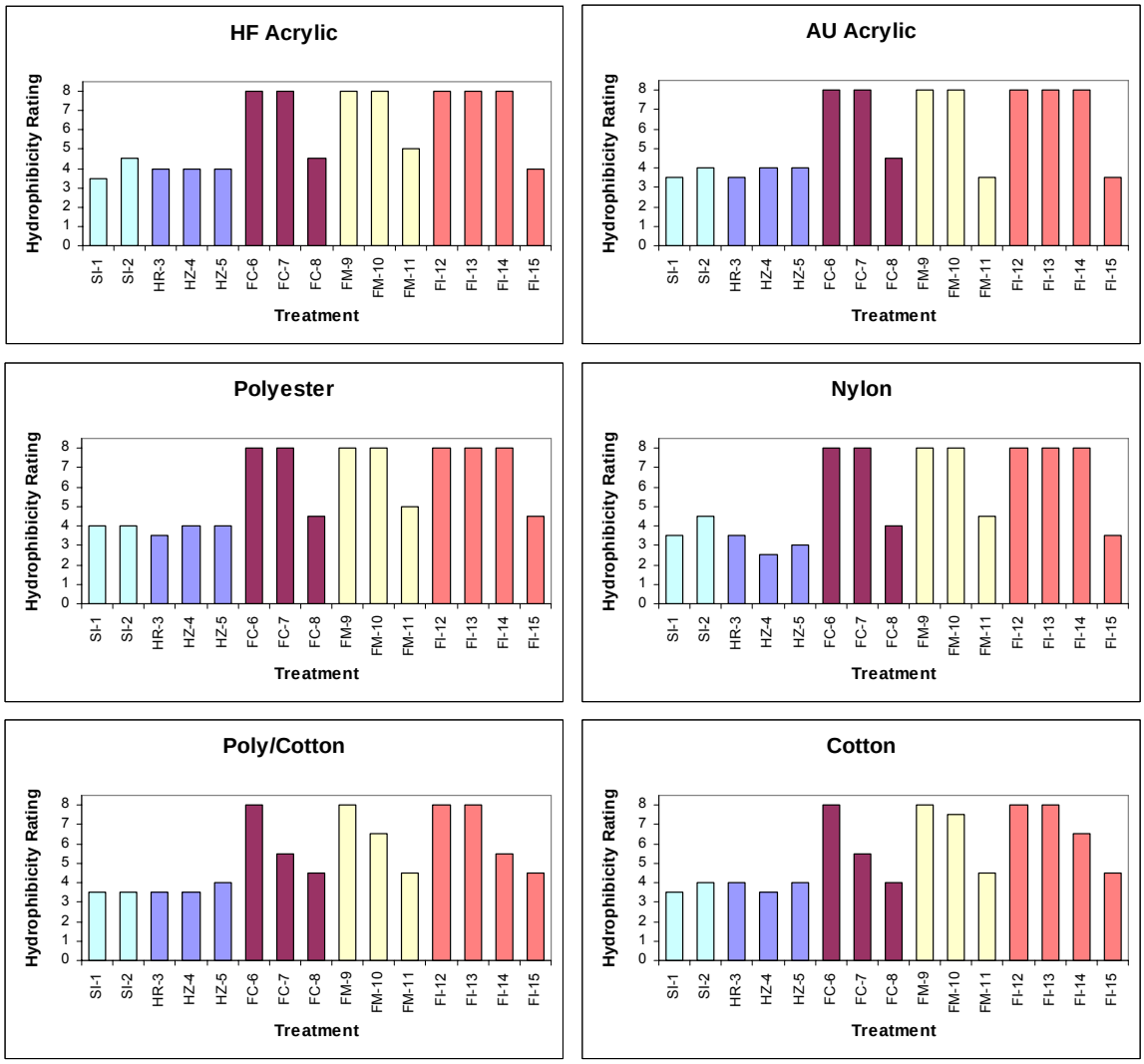


Figure 15: Aqueous liquid repellency data for unwashed fabric samples from continuous application process. Colored bars signify type of finish treatments used.

Oil Repellency

The Hydrocarbon Resistance Test was performed according to the method described in Section 3.3.4. Figure 16 displays the Oil Repellency Rating obtained for each of the various finishes applied to each fabric substrate. Values are not shown for untreated

samples, or samples finished with polysiloxane- or hydrocarbon wax-based finishes. All these samples failed at the initial Oil Repellency level, and received a rating of zero. Complete results from the Oil Repellency Test can be found in Appendix C.

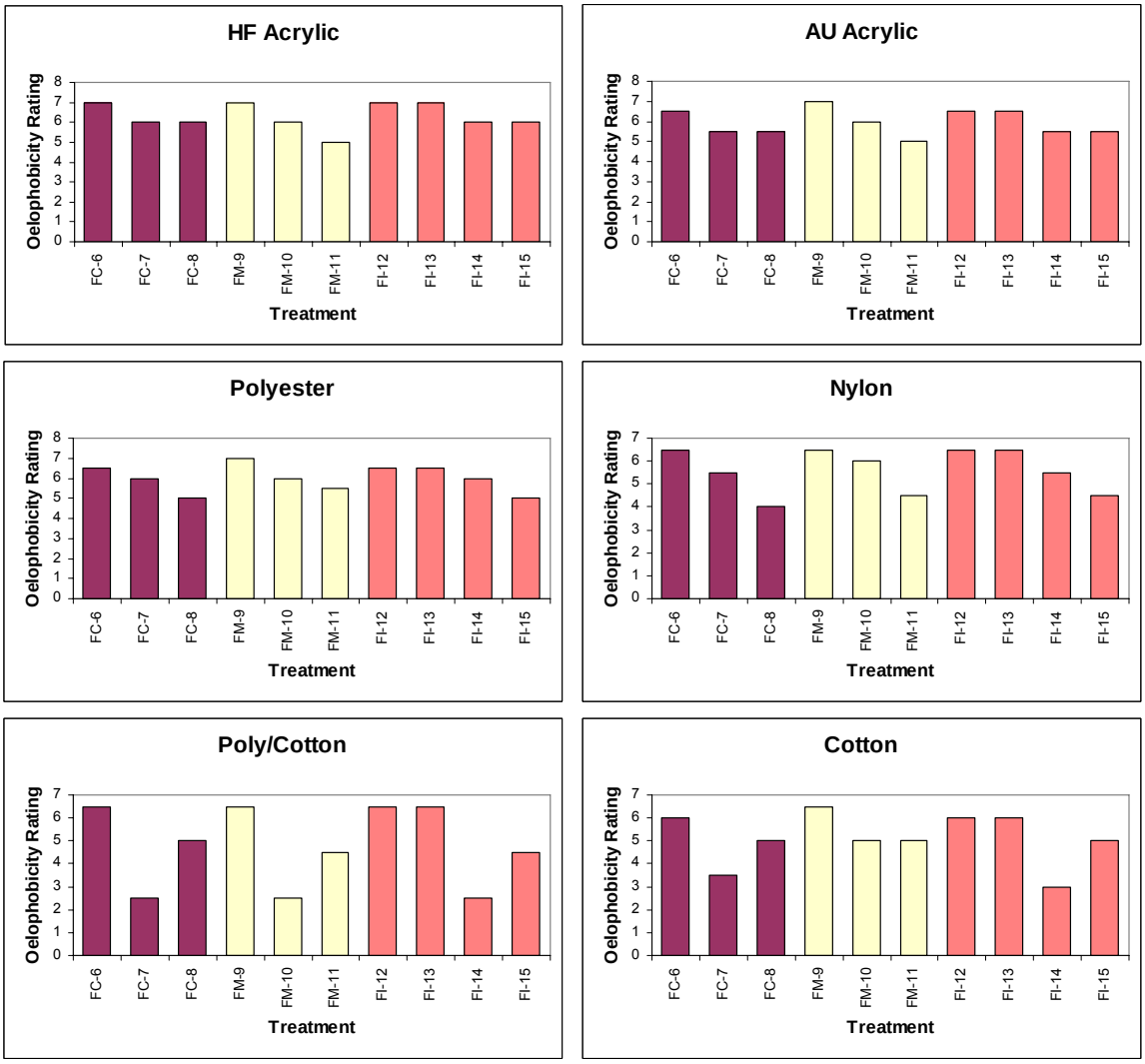


Figure 16: Oil repellency rating for unwashed samples from the continuous application process. Only FC-based finishes are displayed. Bars are colored according to the type of finishing combination utilized in treatment.

Treatments with AsahiGuard E-061 show lower performance characteristics on cotton-contianing substrates than on synthetic fabrics. Similar trends are seen among the synthetic fabrics, with Waterproofon 246 showing the best performance, followed by

AsahiGuard E-061. Melamine and Isocyanate products show no significant impact on the performance of the products prior to washing.

Fluorine Content Analysis

Total fluorine content of samples treated using FC finishes without additional crosslinkers were analyzed at Galbraith Laboratories in Knoxville, TN according to the method outlined in Section 3.3.6. Results of this analysis are shown in Figure 17, below. Complete results for this analysis can be found in Appendix D. For each fabric, the sample treated with AsahiGuard E-061 showed the lowest fluorine levels, while Fluorochem A1 tended to show levels more comparable to those obtained with Waterproofon 246. Discrepancies between oil and aqueous liquid repellency for samples treated with Fluorochem A1, which is explored more fully in the Discussion of Sample Selection section, could be partially explained by the high fluorine content of these samples.

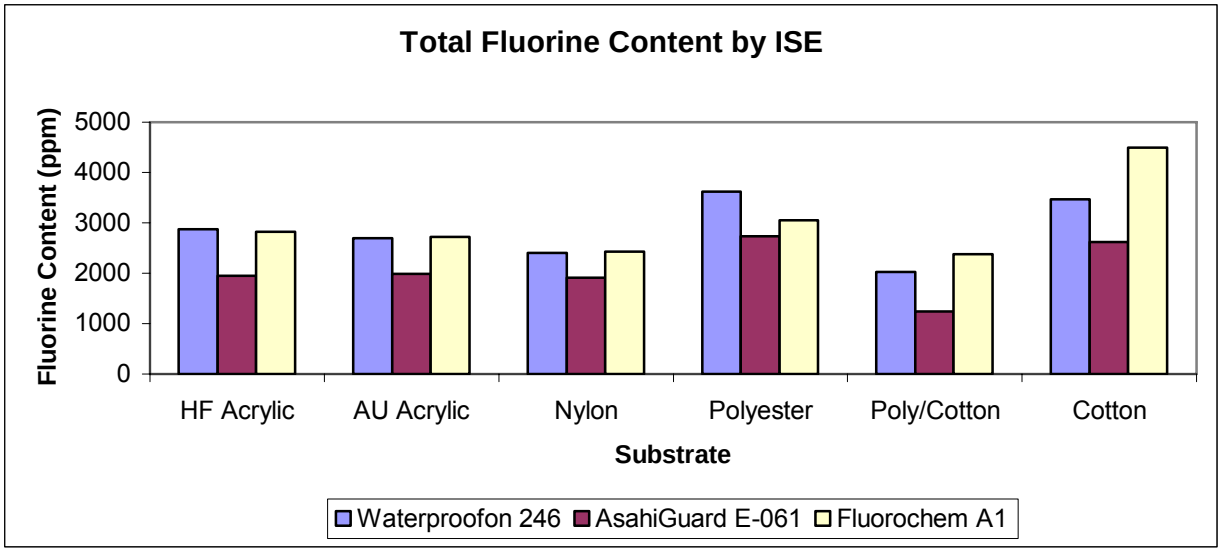


Figure 17: Total fluorine content of samples treated with finishing baths FC-6, 7, and 8. Samples tested at Galbraith Laboratories.

Evaluation of Color Change

Color change for treated fabrics was evaluated as described in Section 3.3.7. An untreated fabric sample was used as a standard for testing. Complete L*, a*, b* values for all samples are listed in Appendix E.

Table 46 shows the average DEcmc values for two replicates of each fabric and finish combination. In the table, values above 1.00 are shown in bold text, as this is a common pass/fail point in textile applications. This value roughly corresponded with visual assessment of the acceptability of treated samples, compared to the untreated standard.

Table 46: DEcmc values for unwashed samples from the continuous application process. Untreated fabric samples were used as the standard. Values represent the average of two specimens.

Treatment	Cotton	Poly/Cotton	Nylon	Polyester	AU Acrylic	HF Acrylic
SI-1	**1.63**	0.85	0.42	**1.11**	0.60	**3.71**
SI-2	**3.22**	0.97	0.86	0.86	**1.17**	**3.00**
HR-3	**2.23**	**1.18**	0.31	0.60	0.55	**2.42**
HZ-4	**2.40**	0.82	0.27	**2.38**	0.33	**2.02**
HZ-5	**1.97**	0.68	0.39	0.93	0.33	**1.94**
FC-6	**1.44**	0.78	0.46	**1.26**	0.63	**1.43**
FC-7	**1.26**	0.53	0.83	0.91	0.63	**1.77**
FC-8	**1.26**	0.56	0.45	0.86	0.56	**1.78**
FM-9	0.93	**1.17**	**3.39**	**1.68**	0.62	**2.14**
FM-10	**1.34**	0.71	**3.64**	0.81	0.69	**2.80**
FM-11	**1.07**	0.48	**3.37**	0.45	0.52	**2.57**
FI-12	**1.38**	0.87	**1.09**	0.82	0.60	**1.47**
FI-13	**1.33**	0.70	0.79	0.99	0.63	**1.81**
FI-14	**1.86**	0.65	0.19	0.92	0.71	**2.48**
FI-15	**1.47**	0.59	0.34	0.78	0.57	**1.99**

Cotton and HF Acrylic samples showed failing values for nearly all finishes applied. This may be related to the initial color of the samples. The cotton fabric is a white, bleached specimen, and the HF Acrylic fabric is an off-white, tan color. Significant yellowing was visible in the treated samples of each of these fabrics, and

spectrophotometric results showed a color shift in the yellow direction along the b* axis for the treated samples.

Also of note is the significant color change in the nylon samples treated with an FC finish and melamine resin. These samples were noticeably changed from their untreated color, shifting significantly in the green direction.

Tear Strength

Tear strength of treated fabric was tested according to the method described in Section 3.3.8. Complete results can be found in Appendix F. Figure 18 shows the mean values of three replicates for each treatment combination, with each replicate being an average of the five highest peaks during the propagation of the tear. Samples are graphed by fabric type, with the horizontal line depicting the tear strength of the untreated control.

In general, treated samples show greater tear strength than the untreated controls, though, in many cases the change is insignificant. Of the finishes, polysiloxane and hydrocarbon wax finishes tended to produce the largest increases in tear strength. This is likely related to the lubricating properties of these finishes. There is no significant trend among samples treated with FC finishes, and no apparent impact of melamine or isocyanate resin.

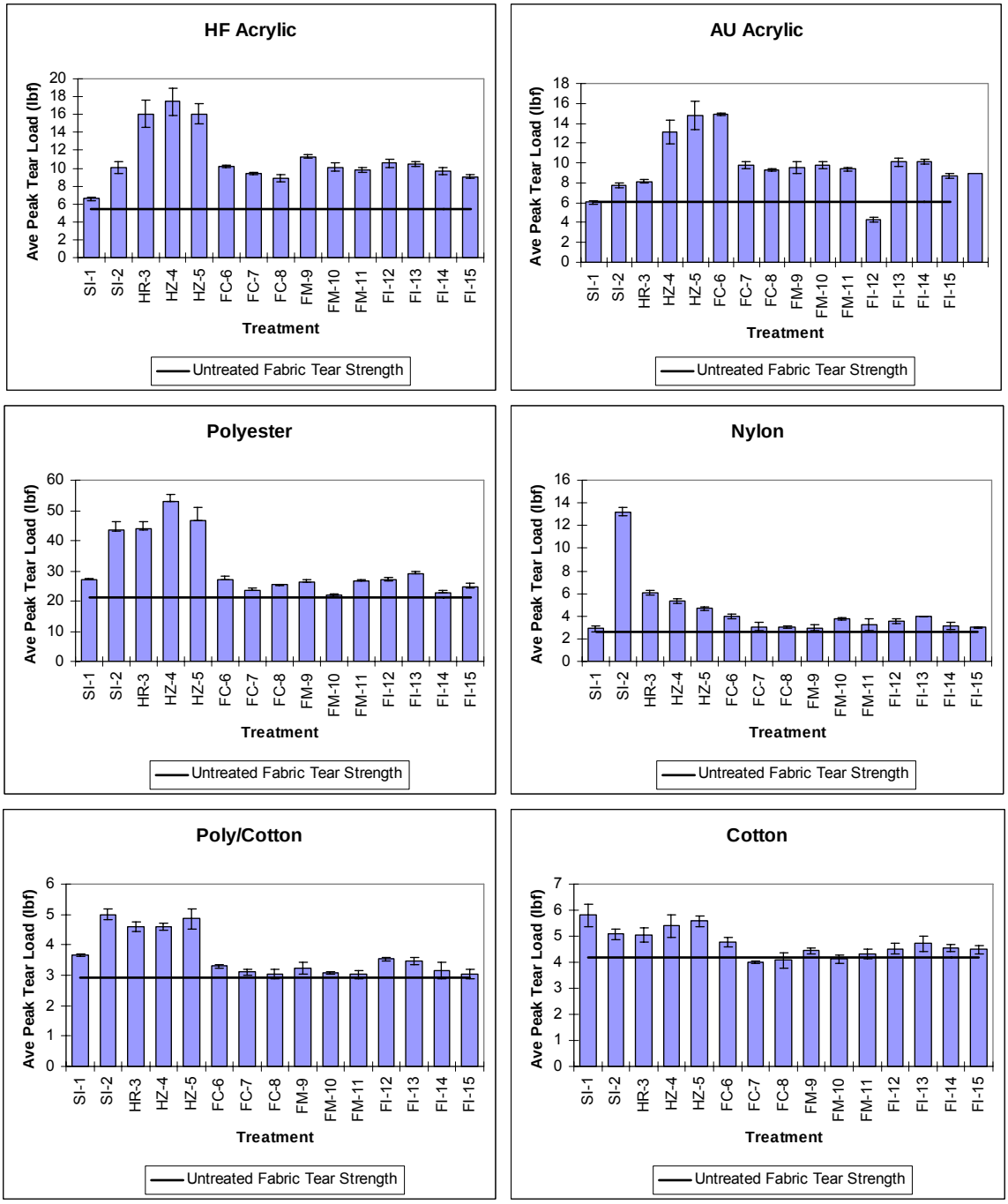


Figure 18: Tear strength data for unwashed samples from the continuous application process. Shown are averages of three replicates, calculating the five highest force peaks during tear propagation. The horizontal bar represents the tear strength of the untreated fabric.

Stiffness Evaluation

Stiffness of treated fabrics was evaluated using the circular bend method described in Section 3.3.9. The average value for two replicates of each treatment combination is shown in Figure 19, below, and complete results can be found in Appendix F. There are no universal trends, so these data are discussed in the interpretation section at a fabric specific level.

Durability – Short Term

Short term durability to laundering was determined after five complete wash cycles as described in Section 3.2.4. Repellency testing was repeated and is presented in the following sections, along with the results reported above for unwashed samples for comparison. Complete results for these tests are provided in Appendix C.

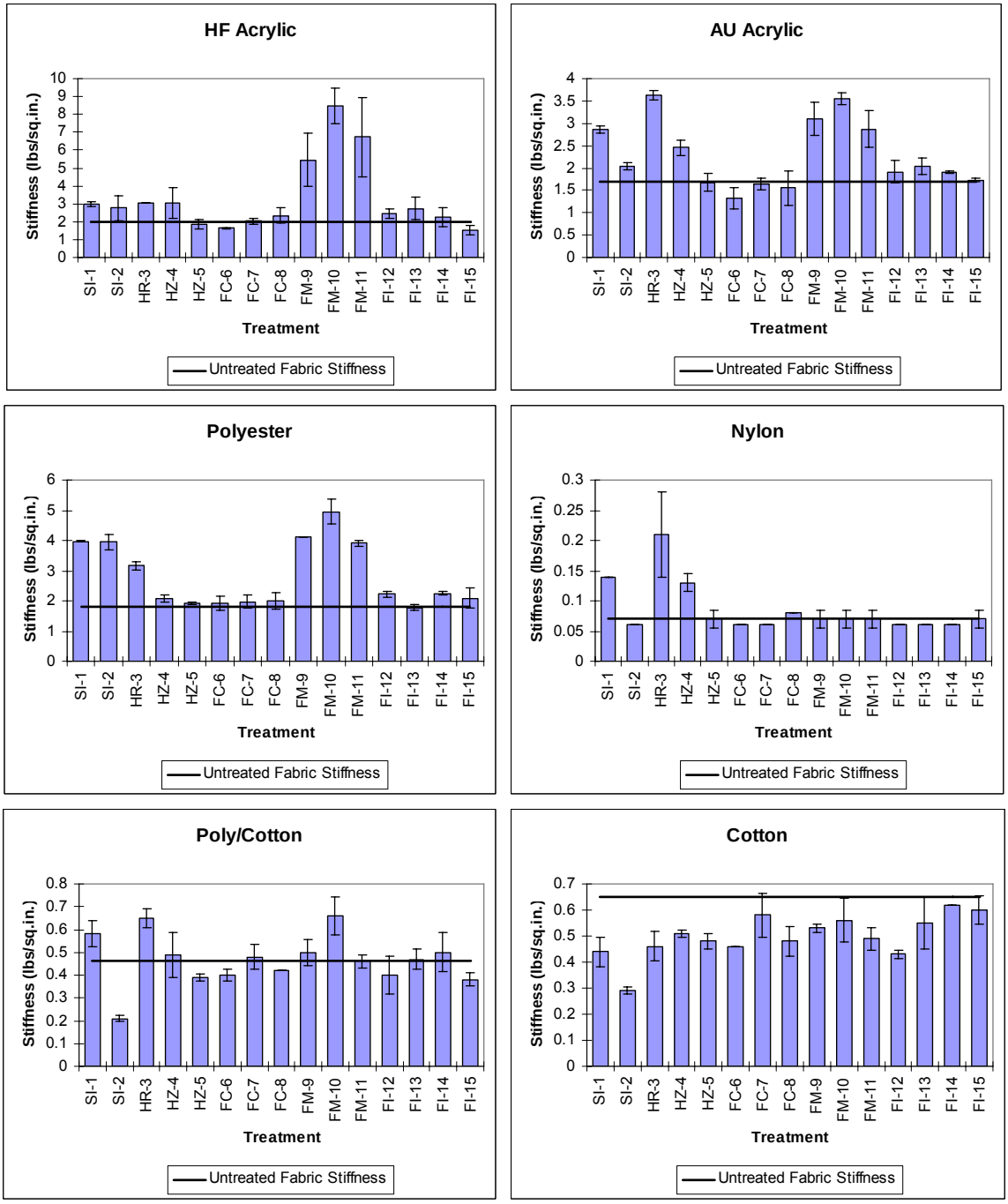


Figure 19: Average stiffness value of two replicates in lbf/sq.in. Horizontal line represents the stiffness of the untreated fabric.

Spray Test

The Spray Test was performed after five wash/dry cycles as previously described. Average values for three replicates are shown in Figure 20. As expected, hydrocarbon wax-based finishes showed the poorest short term durability across all six substrates. On synthetic fabrics, treatments using AsahiGuard E-061 show a greater decline than the other FC finishes tested, though the product still yields a higher rating than fabrics treated with Fluorochem A1. Melamine and isocyanate resins show no apparent effects on the performance of FC type finishes at this level durability.

Finishes applied to the cotton fabric all showed significantly poorer durability than is apparent in the other fabrics tested. This is potentially due to the swelling of the cotton fibers during washing. The finishing treatments act by covering the surface of fibers and forming a polymer network. The swelling of cotton fibers when wet can disturb this network and reduce the repellency by disrupting the repellent sheath which is formed around the fibers.

As can be seen in the graphical data, the performance of the Fluorochem A1-treated samples showed slight improvements in performance after five wash cycles. This may indicate some contamination of the finishing bath. This finish is discussed specifically in the Discussion of Sample Selection section for long term durability.

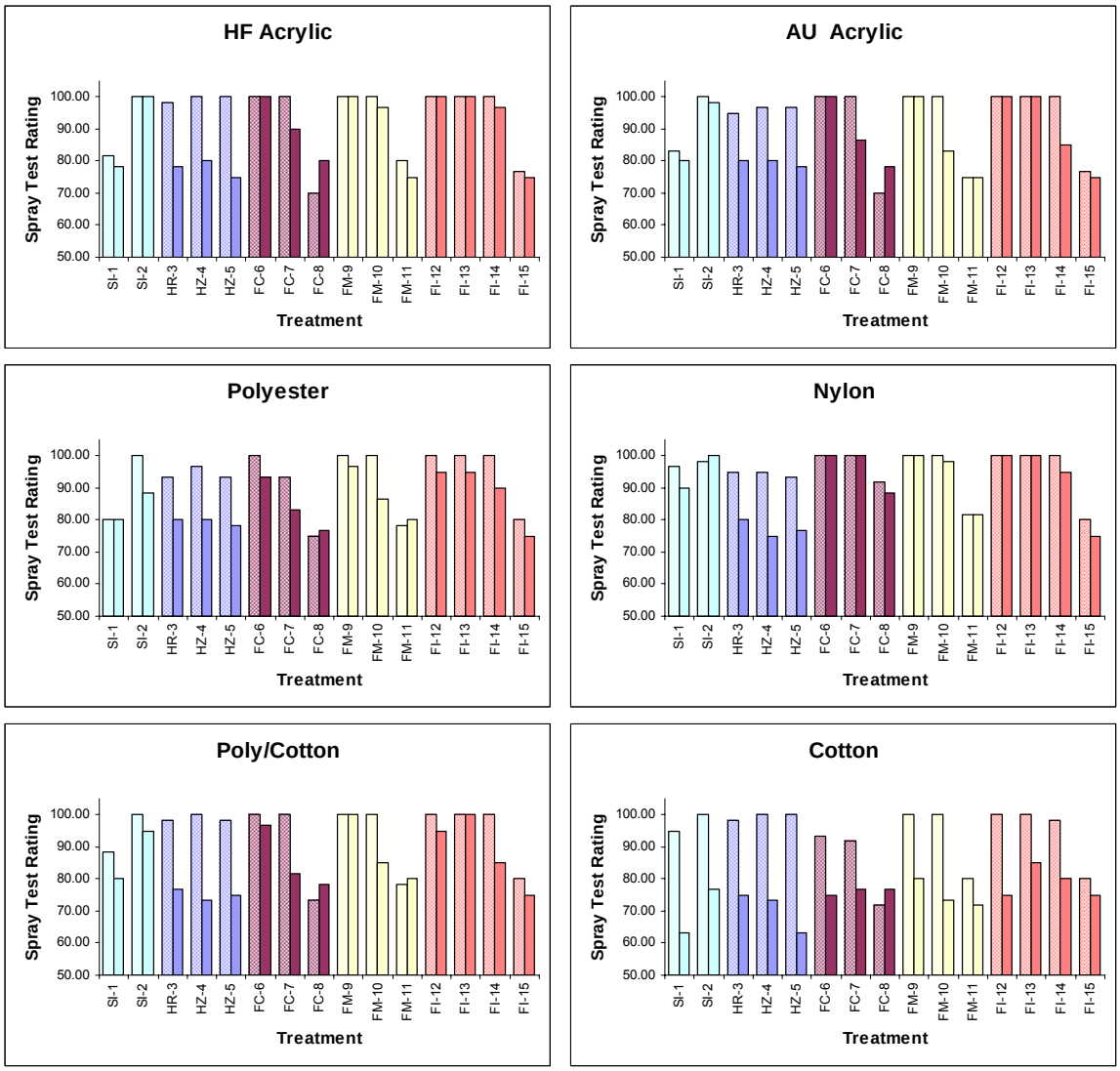


Figure 20: Average Spray Test ratings for three samples of continuous application treatment combinations after five washes. Decreases in performance after washing are noted by the shaded bars.

Aqueous Liquid Repellency

The Water/Alcohol Solution Resistance Test was performed as previously described. Shown in Figure 21 are the reported values for Aqueous Liquid Repellency after five wash/dry cycles. Complete results for this test can be found in Appendix C.

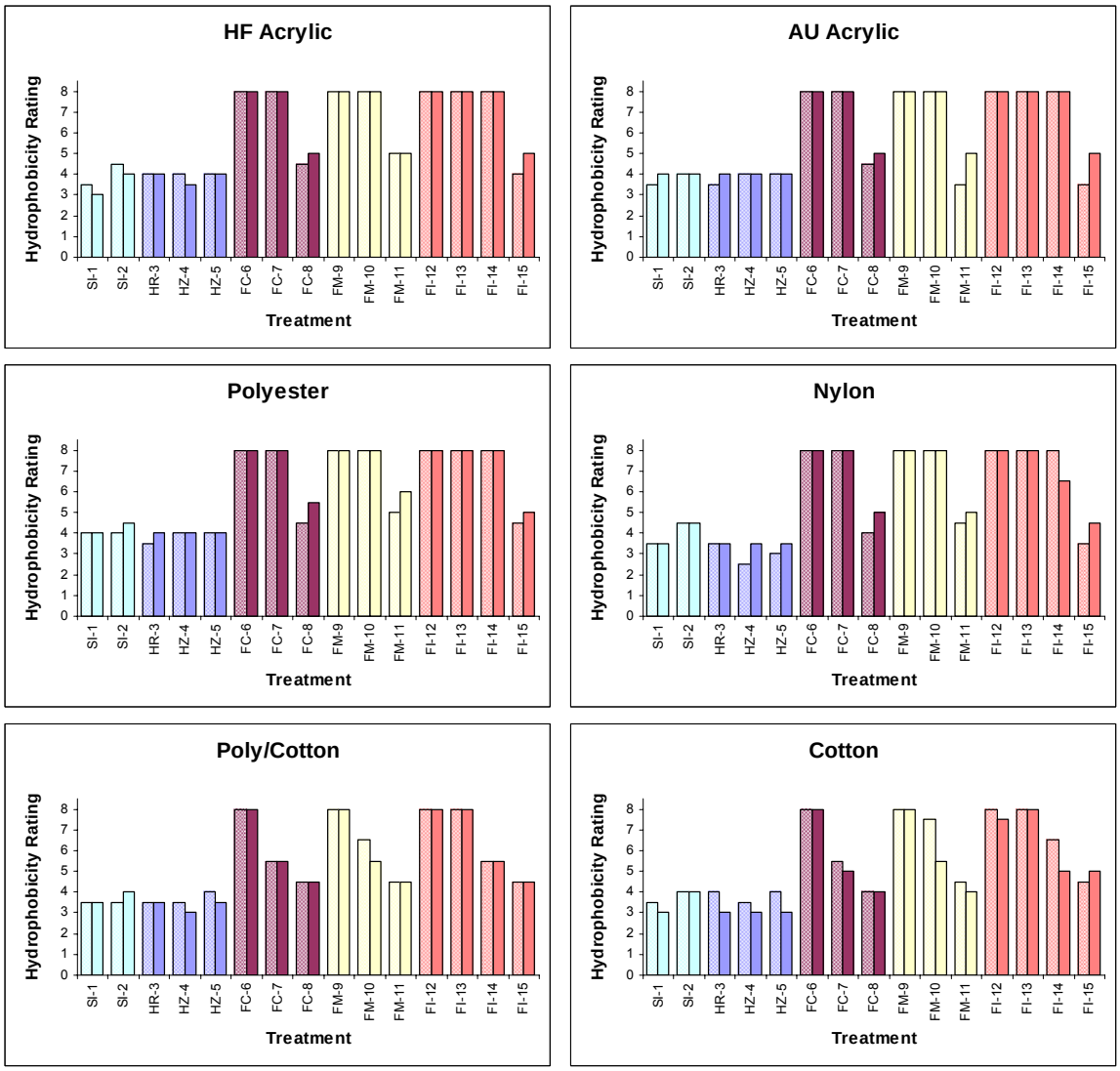


Figure 21: Aqueous liquid repellency test results for continuous application treatment combinations after five wash/dry cycles. Decreases in performance are shown by shaded bars, representing the performance of unwashed samples.

Very little decrease is performance is noted among the samples at this level of durability. Some slight changes were observed in among the synthetic fabric samples. The cotton substrate showed a more consistent loss of durability among the various finishing options. This result is potentially explained by swelling of the cotton fibers during washing.

Oil Repellency Test

The Hydrocarbon Resistance Test was performed on fabric samples after 5 washes as described in Section 3.3.4. Complete results of this test are provided in Appendix C. Reported ratings are shown in Figure 22, with secondary bars to indicate any performance loss after washing. As with the unwashed samples, polysiloxane- and hydrocarbon wax-based finishes are not shown since they do not provide oil repellency and received a rating of zero.

Slight decreases in performance are noted from a number of the samples tested. Nylon fabrics treated with AsahiGuard E-061 (FC-7, FM-10, and FI-14) showed marked performance reduction compared to what was seen from other fabric samples. Several of the fabrics treated with Fluorochem A1 performed better after washing, which may indicate contamination of the treatment bath. This is discussed further in the Discussion of Sample Selection section. No improvement is seen from the addition of melamine or isocyanate resin to the finishing bath at this level of durability.

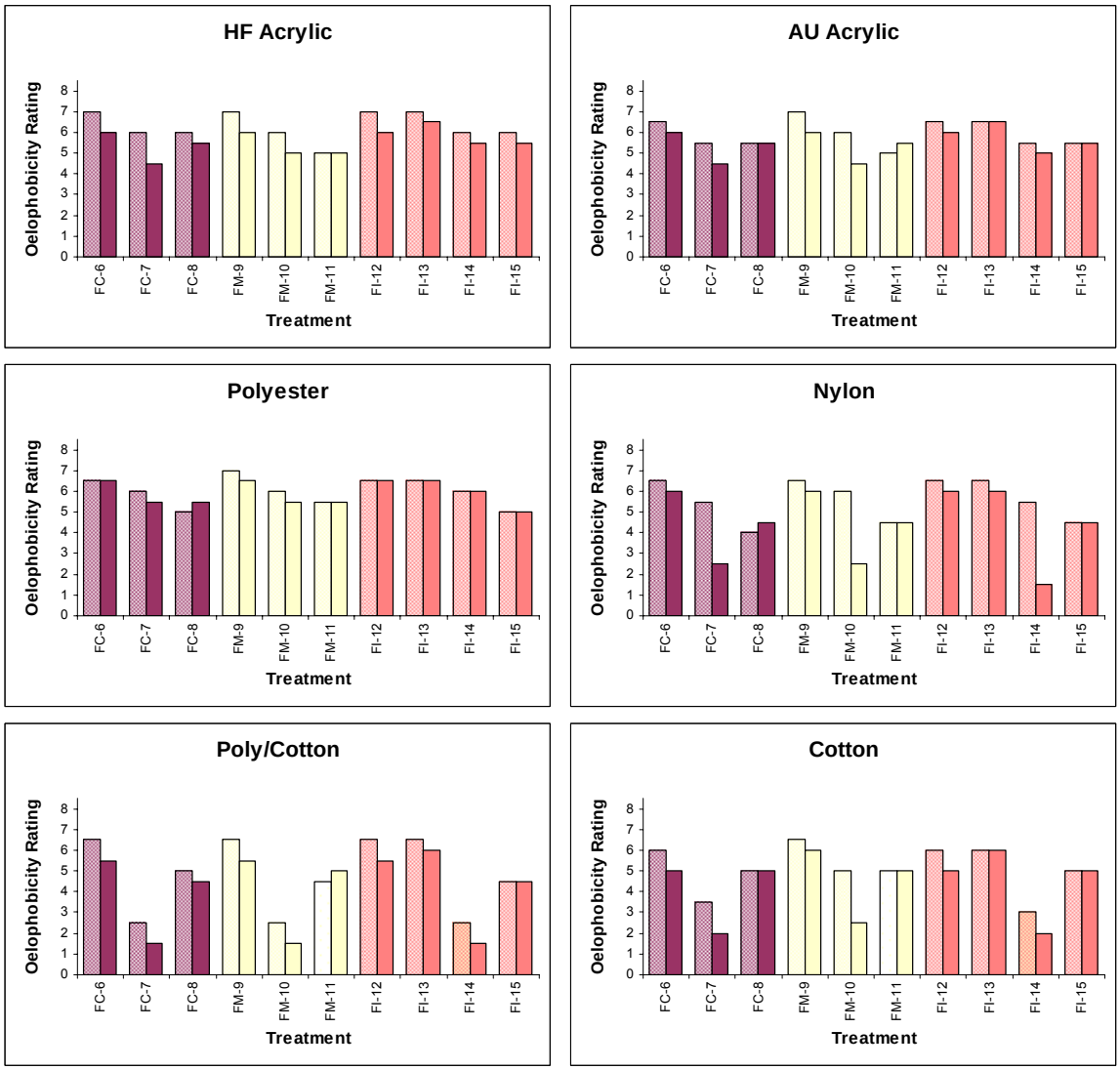


Figure 22: Oil Repellency Ratings for continuous application treatments after five wash/dry cycles. Shaded bars indicate performance reduction after washing.

Notes on Color Impact of Washing

After five wash/dry cycles, significant color changes were noted in the polyester and cotton substrates that were treated with zirconium wax finishes (HZ-4, and HZ-5). The white cotton samples obtained a blue tint and the polyester samples appeared significantly more yellow. This is likely the result of dye transfer from the polyester to

cotton fabric during the wash cycle. Table 47 shows L*, a*, and b* values for the samples in question, along with the values for an untreated, unwashed control. The values clearly show cotton samples that at darker, bluer, and greener than the control sample. Polyester samples show less change, but are lighter, yellower and redder than the control. It is unclear why the dye transfer only occurred between polyester and cotton fabrics treated with zirconium waxes.

Table 47: Spectrophotometric data for cotton and polyester fabrics treated with zirconium waxes after five wash cycles.

Sample	L*	a*	b*
Cotton Control	93.63	-0.51	3.68
Cotton HZ-4	82.22	-8.34	-5.13
Cotton HZ-5	79.64	-8.98	-7.20
Polyester Control	26.61	-0.41	-0.11
Polyester HZ-4	28.10	-0.06	2.63
Polyester HZ-5	27.70	0.92	1.90

Durability – Long Term

The long term durability of selected finishing treatments was evaluated after 25 wash/dry cycles. Repellency testing was conducted on the laundered samples. The complete test results for these tests are provided in Appendix C. The following section discusses the selection of the six treatment combinations for long term durability analysis. Results of repellency testing on the selected samples are then presented.

Discussion of Sample Selection

Prior to long term durability analysis, the fifteen treatment sets were narrowed to six. The selection process was based primarily on the Aqueous Liquid Repellency and Spray Test results at zero and five wash levels. In several cases, other factors were considered where these test results did not indicate a significant performance difference among the samples tested.

For the selection, it was decided to choose one polysiloxane, and one wax based finish, as well as a novel FC finish both with and without one of the resins. The traditional FC finish was included for comparison, both with and without additional resin as well.

On the strength of the test data, treatment SI-2, using Phobotone WS CONC and catalyst, was selected for further analysis. Among the waxes, no significant trend was observable in the test data. Selection in this case was made based on the significant color change observed. Treatment HR-3 was chosen to avoid this color change, and any performance impact it may have, in the long term durability study.

The isocyanate resin from Apexical, Apexosist 186 was selected from among the resin finishes. Since no substantial gains were noted resin extenders, the melamine product was eliminated after considering the ecological considerations associated with the formaldehyde-based crosslinking chemistry. Apexosist 186 was chosen rather than TMI[META] due to processing challenges, which were previously discussed in the section outlining Treatment FI-12, encountered with the latter.

The final selection was made to continue testing with AsahiGuard E-061 rather than Fluorochem A1. This decision was made on the strength of Aqueous Liquid

Repellency and Spray Test data, which indicated that fabrics treated with AsahiGuard E-061 outperformed those treated with Fluorochem A1.

The test results obtained for Fluorochem A1 indicate that further testing of this product may be beneficial. Samples treated with this product tend to yield Oil Repellency ratings that are equivalent to, or higher than, the corresponding Aqueous Liquid Repellency ratings (Figure 23). This result is unexpected considering the surface tensions of the test liquids in each series. Two possible explanations are proposed for this behavior.

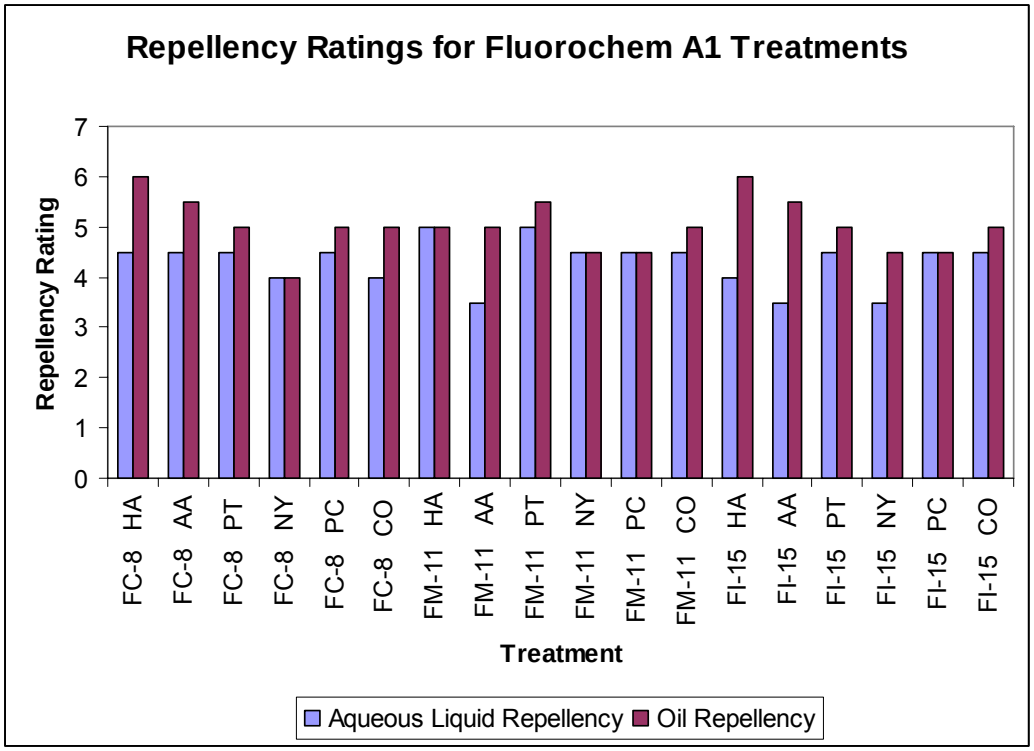


Figure 23: Comparison of oil repellency and aqueous liquid repellency ratings for unwashed samples treated with Fluorochem A1.

Contamination with a polar substance can result in a low Aqueous Liquid Repellency rating and relatively high Oil Repellency rating, as observed in the present

study due, to the polar interaction of the water/alcohol solutions. This theory is supported by the testing which indicates a performance improvement in many cases after five washes, as seen in Figure 24. Presumably, the polar contaminant could be removed in the wash cycle, which could improve the Aqueous Liquid Repellency by reducing the amount of polar interaction occurring.

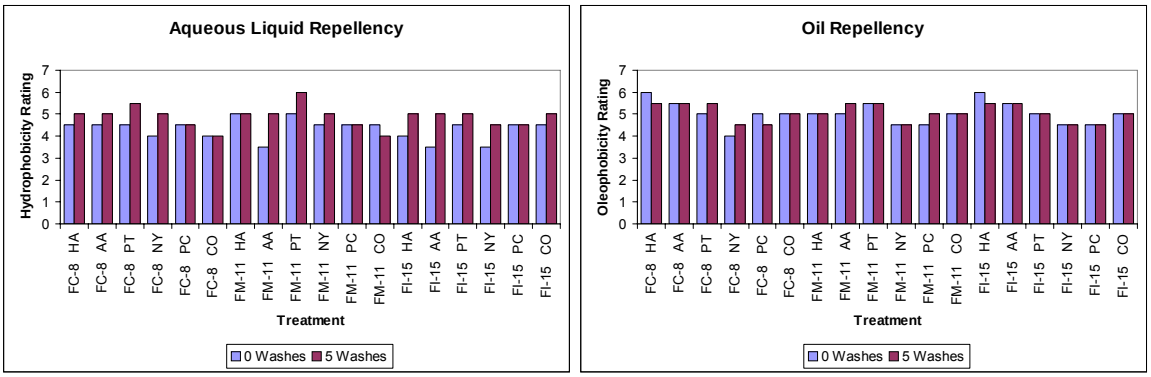


Figure 24: Repellency test ratings for zero and five wash samples of fabrics treated using Fluorochem A1.

The alternative hypothesis for this behavior is related to the short carbon chain present in this FC finish. The fluorine content analysis shows I high total level of fluorine which could explain the high Oil Repellency ratings. However, with FC finishes, the length of the carbon chain is important to ensure proper alignment of the fluorinated groups. A short carbon chain could have a high fluorine content and poor orientation relative to the surface. This scenario could theoretically produce a final product in which the fiber is not adequately covered by the fluorinated groups, allowing polar interactions with the fibers themselves, rather than a contaminant. This theory is supported by the results of the Fluorine Content Analysis.

Due to the questionable performance of the Fluorochem A1 treated samples, the final selection was made and treatments SI-2, HR-3, FC-6, FC-7, FI-13, and FI-14 were evaluated further for long term durability.

Spray Test

The Spray Test was performed as discussed in Section 3.3.5 on selected treatment samples after 25 wash cycles. Complete results from this testing can be found in Appendix C. Figure 25 shows the average spray test rating observed for the three replicates tested for each treatment.

The majority of the finishes tested showed a significant loss of performance after 25 wash cycles, though treatments using Waterproofon 246 (FC-6, and FI-13) largely retained their performance on polyester and nylon. The presence of isocyanate showed no significant impact on the synthetic fabrics, but was clearly beneficial to the durability of the FC finishes on the cotton substrate. The hydrocarbon wax and both FC types finishes yielded spray test ratings of zero, indicating complete wetting of the fabric.

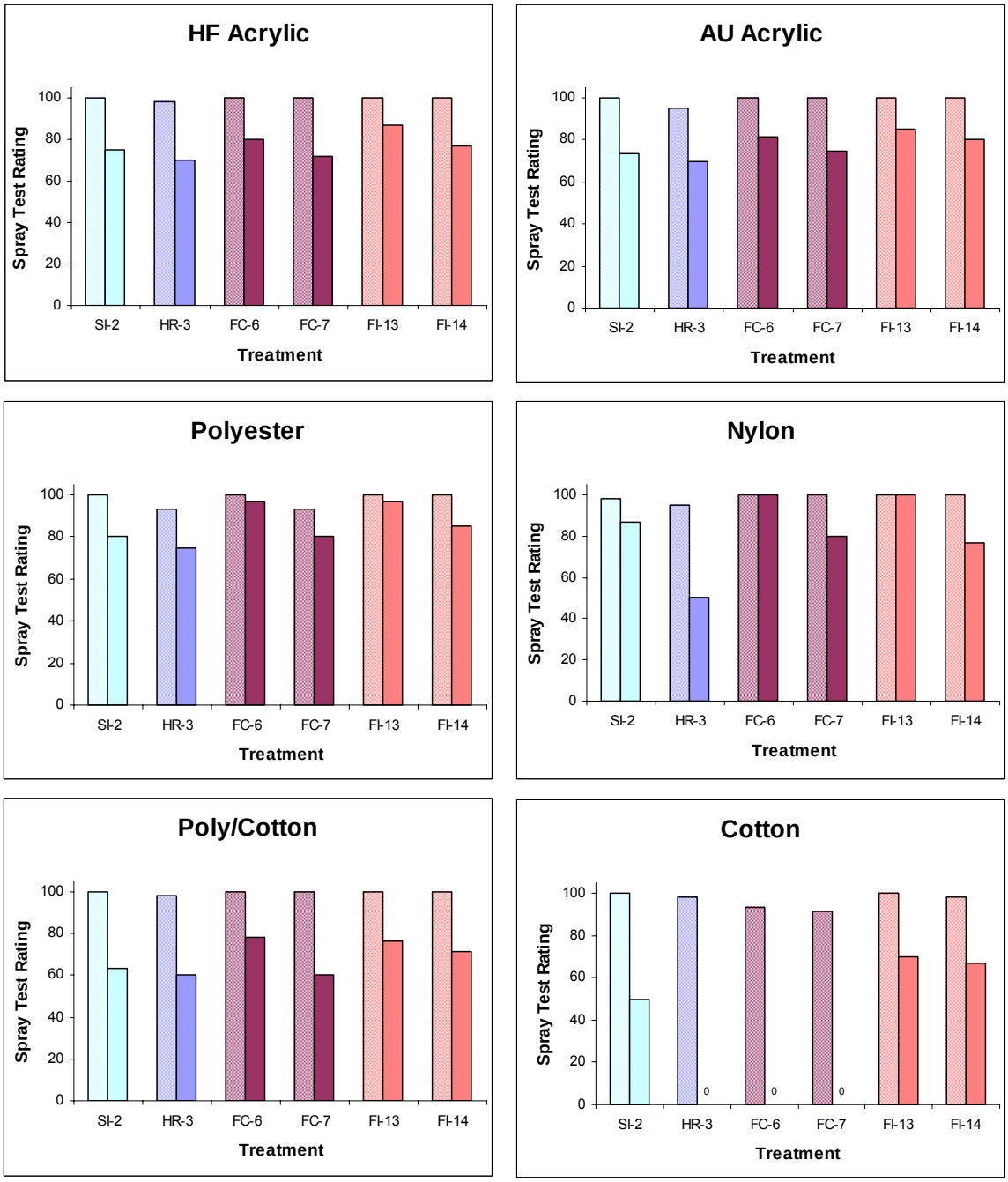


Figure 25: Spray Test results for selected samples after 25 wash cycles. Shaded bars show unwashed sample values to demonstrate loss of durability.

Aqueous Liquid Repellency

The Water/Alcohol Resistance test was performed on samples selected for long term durability analysis according to the procedure described in Section 3.3.3. Reported test

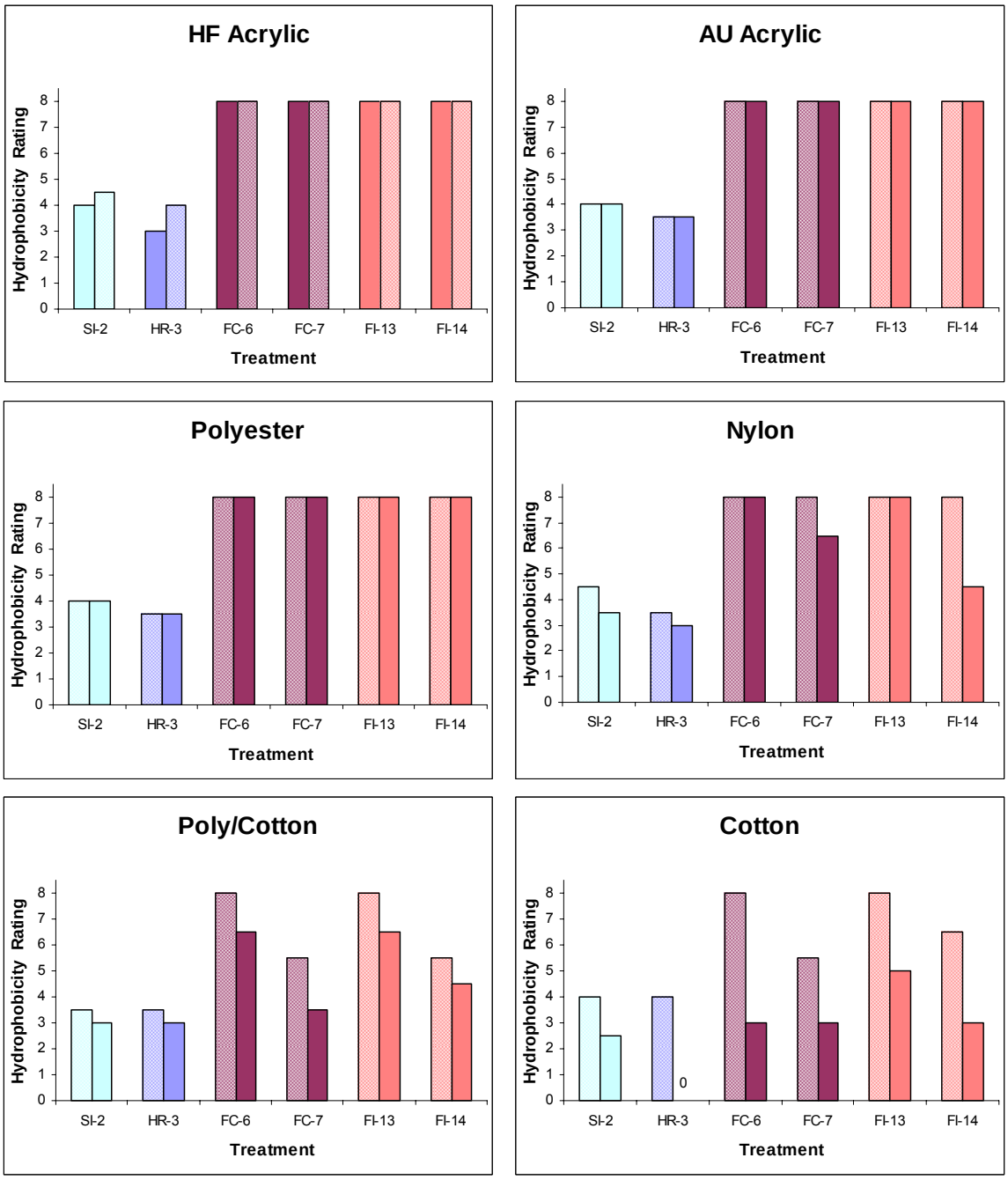


Figure 26: Aqueous Liquid Repellency ratings for 25 wash samples of selected fabrics. Shaded bars represent the unwashed sample results.

values are shown in Figure 26, below. Refer to Appendix C for a complete listing of repellency test results.

Results are discussed on a fabric specific level in the Interpretation and Discussion section, though it should be noted that no loss of performance was seen for the automotive acrylic and polyester substrates for any of the finishes selected.

Oleophobicity Test

The Hydrocarbon Resistance Test described in Section 3.3.4 was performed on samples selected for long term durability analysis. Resulting ratings are shown in Figure 27. For complete test results, refer to Appendix C. Tests show that traditional FC finish, Waterproofon 246, performs better than AsahiGuard E-061 for all fabrics both in terms of absolute repellency as well as durability for the fabrics tested.

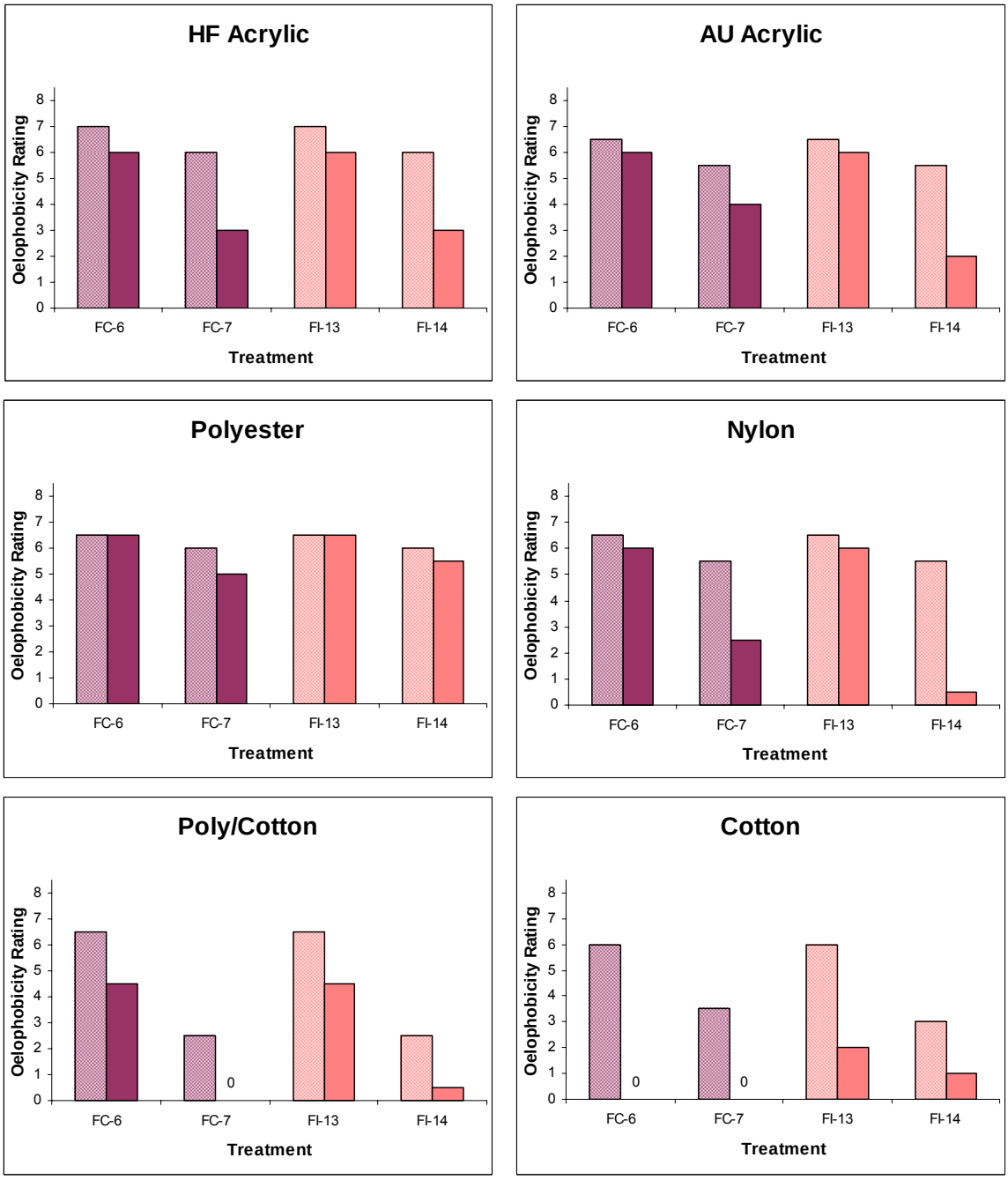


Figure 27: Oil Repellency results for selected samples after 25 wash cycles. Shaded bars represent the unwashed fabric samples and are provided as a means for comparison.

4.3.2 Interpretation and Discussion

The following sections evaluate the results of the testing provided above on a fabric specific level. The overall performance of the finishes tested is discussed for each fabric tested. As with the earlier fabric selection process, repellency data are the primary focus of the discussion, with the remaining physical testing results mentioned as appropriate.

Cotton

No finishing treatment tested provided durable repellency to the cotton substrate at the 25 wash level. However, there were substantial differences among the treatments tested. Between the two polysiloxane finishes tested, SI-2 outperformed SI-1 in both the Aqueous Liquid Repellency (ALR) Test and Spray Test at both zero and five wash levels. Treatment SI-2 also showed no decrease in performance on the ALR Test after five washes while SI-1 finish decreased from a rating of 3.5 to 3.0. Both polysiloxane finishes showed significant loss of performance in the spray test after five washes, decreasing from ratings of 95 for SI-1 and 100 for SI-2, to average ratings of 63, and 77, respectively, after washing. After 25 washes, treatment SI-2 achieved a Spray Test rating of 50, which was superior to three of the six finishes tested, including FC finishes without isocyanate resin. The performance difference between the polysiloxane finishes could be related to the add-on levels, as treatment SI-2 had a theoretical add-on of 2.88% compared to just 1.53% for SI-1.

The three hydrocarbon wax finishes performed comparable to finish SI-1 in terms of absolute repellency. All three showed loss of performance on both the ALR Test and Spray Test after five washes. They each received an ALR rating of 3.0 at this wash level.

On the spray test, HR-3 performed the best of the three after five washes, producing a Spray Test rating of 75. Considering the previously noted color change of zirconium wax-treated samples after washing, Treatment HR-3 using Norane 100, reactive wax, was the best performing wax finish tested. It should be noted that the theoretical add-on for this product was approximately twice that of the other wax finishes (2.59% compared to 1.80 and 1.78% for HZ-4, and HZ-5, respectively), a factor which likely contributed to the superior performance.

For the FC finishes, the testing clearly indicates that the novel FCs do not perform at the same level as the traditional product. Samples treated with AsahiGuard E-061 performed comparably to the Waterproofon 246 samples for the Spray Test and ALR Test with unwashed samples, but showed a significant performance decrease after five washes, especially in the ALR Rating. The samples treated with Fluorochem A1 showed poorer initial performance on ALR, but no decrease after five washes. For Oil Repellency Testing, Fluorochem A1 out performed AsahiGuard E-061 initially, and was the only one of the three products that did not show a decline in Oil Repellency after five washes. As previously discussed, the Oil Repellency and ALR ratings for Fluorochem A1 indicate that there is potential contamination in the finish which could skew these results.

For long term durability, the isocyanate crosslinker was shown to be beneficial, specifically for the Spray Test. Both treatments with FC finish alone were rated at the zero level after 25 washes, while those with the isocyantate crosslinker were rated 70 and 67 for the Waterproofon 246 and AsahiGuard E-061 treatments, respectively.

Significant yellowing was seen from all treatments tested, ranging from a minimum DEcmc value of 0.93, a borderline pass for Treatment FM-9, and a maximum of 3.22 for Treatment SI-2. Color shifts for all the treatments fall roughly along the yellow axis of the L*a*b* color space.

All samples tested showed slightly lower stiffness values and slightly higher tear strength values than the untreated control. Polysiloxane- and wax-based finishes showed the greatest increase in tear strength, though these values were only slightly higher than the untreated samples. Treatment SI-2 showed the lowest stiffness value of the samples tested.

Poly/Cotton

The poly/cotton substrate showed many of the same trends as the cotton fabric. One primary difference was that the short term and long term durability of the finishes was improved for most finishes, compared to the results of the cotton trials.

Treatment SI-2 again performed substantially better than SI-1 on the Spray Test both initially and after five washes. For the wax-based finishes, very little difference was seen among the three treatments in either the ALR or Spray Test.

FC finish testing also followed the same trends seen with the cotton substrate. Neither novel FC product performed at as high a level as the traditional FC finish. Of the novel finishes, AsahiGuard E-061 performed better on the ALR and Spray Tests, while Fluorochem A1 provided better oil repellency.

Long term testing showed that all-six treatments retained some level of durability, with a minimum spray test value of 60 and minimum ALR rating of 3.0 for the six treatments. The traditional FC product, Waterproofon 246, proved the most durable,

showing the highest ratings in all three repellency tests after 25 washes, and generating an Oil Repellency rating of 4.5. The addition of isocyanate crosslinker showed no impact on performance or durability for the FC finishes applied to the poly/cotton substrate.

Color shifts were much less significant for the poly/cotton substrate, with DEcmc values ranging from 0.48 for FM-11, to 1.18 for HR-3. Color differences were fairly scattered, though many still fell near the yellow axis in the color space.

Treatment SI-2 showed the lowest stiffness value recorded for Poly/Cotton substrates (0.014 lbf max) and highest tear strength (5.0 lbf). Wax finishes also produced tear strengths significantly greater than the untreated control sample.

Polyester

As with previous fabrics tested, SI-2 performed better than SI-1 in ALR and Spray Tests when at initial and five wash durability levels. Among wax finishes there is very little difference between the test values observed at initial and five wash levels.

For FC finishes, at initial and five wash levels, AsahiGuard E-061 performs comparably to Waterproofon 246 in the ALR Test, and only slightly poorer in the Spray Test. Unlike other fabrics tested, Fluorochem A1 does not perform better than AsahiGuard E-061 in Oil Repellency. At 25 washes, AsahiGuard E-061 shows a greater decrease in performance than Waterproofon 246. The use of the isocyanate crosslinker in Treatment FI-14 does appear to slightly improve the durability at 25 washes for the Asahi product, having a Spray Test rating five points higher than observed for Treatment FC-7, but shows no impact on the Waterproofon 246 treatments.

One negative impact of Waterproofon 246 on the polyester fabric is the color shift associated with the treatment. Treatments FC-6, FM-9, and FI-13 had DEcmc values of

1.26, 1.68, and 0.99, respectively, with the primary component of each shift being in the negative L* (darker) direction. Only three treatments failed at a 1.0 acceptance level for DEcmc, with the third being Treatment HZ-4 which was significantly lighter and yellower after the initial finish, according to the spectrophotometric data.

Stiffness testing indicates that polysiloxane finishes and FC finishes with melamine resin increase the fabric stiffness considerably relative to the untreated control fabric, with values more than twice those measured for control samples. Treatment HR-3 also showed a significant increase in fabric stiffness over the untreated control.

Tear strength data reveal that polysiloxane and wax finishes substantially improve tear strength compared to control samples. This increase is minimal for Treatment SI-1, but samples SI-2, HR-3, HZ-4, and HZ-5 result in tear strength values more than double the control value.

Nylon

Polysiloxane and wax finishes applied to polyester substrates followed the same trends described previously for cotton and poly/cotton fabrics. SI-2 performed substantially better than SI-1 in both the Spray Test and ALR Test. SI-2 had an ALR Rating of 4.5 at both unwashed and five wash samples, compared to 3.5 ratings for SI-1. For unwashed samples, HR-3 performed better than either HZ-4 or HZ-5, in both Spray Test and ALR Ratings, though HZ-4 and HZ-5 performed better in ALR rating after 5 washes, and all three treatments performed equally at this level. After 25 washes, Treatment SI-2 performed better than AsahiGuard E-061 on the Spray Test, but was lacking the oil repellency that the FC finish offers.

For FC finished samples, results were similar to what has been discussed with cotton and poly/cotton substrates. Fabrics treated with AsahiGuard E-061 performed comparably to Waterproofon 246 samples initially, but this performance was decreased after five washes. Prior to washing, AsahiGuard E-061 performed at a higher level than Fluorochem A1 for Oil Repellency, but after five washes, Fluorochem A1 showed no decline in performance and tested better than the Asahi product at the short term durability level.

At 25 washes, Waterproofon 246 showed no decrease in performance on any of the repellency tests, rating at 100, 8.0, and 6.0 at all durability levels for Spray, ALR, and Oil Repellency Tests respectively. Test results for AsahiGuard E-061 at five and 25 washes indicate that the addition of isocyanate decreases the performance and durability of the finish. Based on this finding, it is recommended to apply finishes to this fabric without additional resin.

With the exception of samples treated with melamine resin, nylon samples showed minimal impact of finishes on the color of the fabric. The three treatments utilizing melamine resin had DEcmc values averaging 3.5 units, primarily in the green and yellow directions of the color space. Other than the melamine treatments, only Treatment FI-12 failed at the 1.0 acceptance level, with an average DEcmc of 1.09.

Treatments SI-2, HR-3, HZ-4, and HZ-5 all returned tear strength values significantly higher than the untreated control. The value for SI-2, 13.22 lbf, was nearly five times higher than the control value. Stiffness data indicate that treatments SI-1, HR-3 and HZ-4 have higher stiffness than the untreated control, though these data may not be

significant, since the fabric was extremely thin, and values were only able to be recorded at one significant figure for most of the samples.

Automotive Acrylic

As seen with prior fabrics, SI-2 performed better that Treatment SI-1 on ALR and Spray tests initially and after five wash cycles. Only minimal differences were seen between wax-based finishes at initial and five wash levels. Wax-based finishes showed a significant decrease in performance on the Spray Test after five washes, but did not show a decline in ALR Test rating. After five washes, these polysiloxane and wax treated samples performed better than Fluorochem A1 treatments on Spray and ALR tests, though the FC finish provided a level of oil repellency.

For oil repellency, neither of the novel FC finishes performed as well as samples treated with Waterproofon 246, though AsahiGuard E-061 treatments were comparable on the Spray and ALR tests at zero washes. The Spray Test rating was significantly lowered for this product after five washes, however. At the initial treatment level, AsahiGuard E-061 performed slightly better than Fluorchem A1 for Oil Repellency, but after five washes this performance deteriorated, and the Fluorochem A1 treatments performed at a higher level.

None of the treatments evaluated at 25 washes were found to maintain their performance level on the Spray Test. However, ALR Test ratings remained unchanged from their initial levels for all six treatments evaluated. Oil Repellency data show a significant decline in the performance of AsahiGuard E-061 after washing (decreasing from 5.5 initially to 4.0), which was made worse by the addition of an isocyanate crosslinker (decreasing from 5.5 to 2.0). Waterproofon 246 samples showed only a slight

decline, from a rating of 6.5 to 6.0 after 25 washes, and were not impacted by the isocyanate crosslinker.

Color data show a trend along the yellow axis, though only one finish treatment failed at a 1.0 DEcmc acceptance level. Finish SI-2 had an average DEcmc value of 1.17, which was primarily from a shift in the positive b* (yellow) direction.

The three treatments containing melamine, FM-9, FM-10, and FM-11, as well as treatments SI-1, HR-3, and HZ-4 resulted in a significantly stiffer product than the control sample. All sample treatments except SI-1 and FI-12 resulted in higher tear strength than the control. The three treatments that produced the highest tear strength values were HZ-4, HZ-5, and FC-6, all of which were found to have tear strength more than double that of the control.

Home Furnishing Acrylic

Initial Spray Test ratings were at or near 100 for all sample treatments except SI-1, and FC finishes using Fluorochem A1, FC-8, FM-11, and FI-15. SI-2 performed better than SI-1 for Spray Test and ALR Test ratings at zero and five wash levels. Slight differences were noted among the wax based finishes, all of which showed poor durability on the Spray Test after five washes.

HF Acrylic samples treated with AsahiGuard E-061 showed a slight decrease in Spray Test performance after five washes, though the decrease was much less than what was found for the other substrates. Fluorochem A1 treatments perform well for Oil Repellency, but perform very poorly relative to the other FC finishes for ALR and Spray tests.

After 25 washes, the AsahiGuard E-061 treatments had lower Spray Test results than the Waterproofon 246 samples, and performed comparably to the polysiloxane and wax finishes tested. The addition of isocyanate resin improved the durability of both FC finishes, according to Spray Test results. For Oil Repellency and ALR tests, the isocyanate had no impact on the test results. AsahiGuard E-061 treatments showed decreased Oil Repellency at this level, while Waterproofon 246 samples performed comparably to the five wash samples.

Of all the fabrics, the Home Furnishing Acrylic showed the largest color shifts after finishing. None of the samples passed at the 1.0 DEcmc acceptance level. The DEcmc values ranged from a minimum of 1.43 for Treatment FC-6 to a maximum of 3.71 for Treatment SI-1. Color changes were primarily in the positive b* (yellow) direction of the color space.

Testing indicates that FC finishes with melamine resin resulted in samples that were more than 2.5 times stiffer than the control samples. Tear data show that all finishing treatments increased the tear strength compared with the control samples. The highest values were obtained by the three wax finishes, which resulted in tear strength values more than three times the control value.

Chapter 5 : Conclusions

The research presented has analyzed a variety of repellent finishing options for textile applications. After an initial product screening, fifteen treatment combinations utilizing polysiloxane, hydrocarbon wax, and FC finishes were evaluated for performance and durability on six different fabric substrates. In some treatments, the FC finishes were applied with a melamine resin or isocyanate crosslinker to evaluate the impact that such products have on both the performance and durability of such finishes. The treatments were applied using a continuous pad-dry-cure process, and durability was evaluated after five and 25 home laundering cycles. The results of this research have been presented and discussed. The results of the research are summarized in this chapter, and recommendations are made for future work in the area of repellent finishing.

5.1 Summary of Results

One of the primary goals of this research has been to evaluate the performance of novel FC finishes which have been created in response to concerns related to PFOA. Based on the present research, it has been shown that the products tested, AsahiGuard E-061 and Fluorochem A1, do not generate the same level of repellency as a traditional FC product, Waterproofon 246.

The use of isocyanate and melamine resins was found to have varying impact on both durability and performance of the FC finishes tested. At the five wash durability level, no substantial impact was noted, while at the 25 wash level, the isocyanate crosslinker tested was found to improve finish durability on cotton-containing fabrics. However, in the cases where an impact was noted, the change in performance was often

miminal. Additionally, the use of melamine resin, specifically, was found to increase the stiffness of many of the fabrics tested.

The two polysiloxane finishes tested showed significantly different performance characteristics, though this is very likely attributed to the difference in theoretical add-on between the finishing treatments. The polysiloxane products tested showed initial performance comparable to that of the hydrocarbon wax products, but were found to be more durable. Additionally, the use of both polysiloxane and hydrocarbon wax finishes were found to improve the tear strength of the fabrics tested.

Among the wax-based finishes, there was little difference noted in terms of performance and durability for the majority of fabrics tested. In general, the wax finishes showed significantly lower initial repellency characteristics that the FC finishes evaluated as well as the largest decrease in performance after home laundering at short term and long term levels.

Though the novel FC finishes tested did not provide the same level of repellency as the traditional FC finish, they did offer a minimum level of oil repellency not achieved by polysiloxane or hydrocarbon wax finishes. Though the durability of the novel FC finishes was found to be comparable at the short term level, the novel finishes exhibited a greater decline in performance that the traditional products after 25 home launderings. .

All finishes tested yellowed the fabric samples slightly. The extent of yellowing was different for each fabric treatment, and no trends were identified in the yellowing behavior of any individual treatment type.

5.2 Future Work

Based on the present research, further studies can be proposed to more fully understand the performance of the available alternatives to FC finishes. This study has not fully evaluated the potential of Fluorochem A1 as a repellent finish, due to the inconsistencies in the test results. This product should be studied further to better understand the performance characteristics that can be obtained through its use.

The other novel FC finish, AsahiGuard E-061 showed some potential for durable repellency when used in conjunction with an isocyanate cross linker for several of the fabrics tested. The utility of such crosslinkers should be further examined to determine if improved durability can be obtained for novel FC finishes using this chemistry.

Finally, research should be conducted to examine several other novel finishing techniques discussed in the literature review which are not commercially available. Among these finishing options are the use of plasma treatment, application of silica nano-particles to achieve super-hydrophobicity, and the potential for fluorinated polysiloxane products.

The concern over the use of PFOA in textile finishing will continue to present challenges for repellent finishing. Work should be continued to find an appropriate replacement for traditional FC products that utilize this chemistry.

Chapter 6 : Literature Cited

(1) Kissa, E. In *Fluorinated surfactants and repellents;* Surfactant Science Series; Marcel Dekker: New York, 2001; Vol. 97, pp 615.

(2) Schindler, W. D.; Hauser, P. J.; Textile Institute In *Chemical finishing of textiles;* Crc; Woodhead: Boca Raton; Cambridge, England, 2004; , pp 213.

(3) Cassie, A. B. D.; Baxter, S. *Trans. Faraday Soc.* **1944**, *40*, 546.

(4) Patankar, N. A. *Langmuir* **2003**, *19*, 1249.

(5) Bico, J.; Marzolin, C.; Quere, D. *Europhys. Lett.* **1999**, *47*, 220.

(6) Johnson, R. E.; Dettre, R. H. *J. Phys. Chem* **1964**, *68*, 1744.

(7) Chen, Y.; He, B.; Lee, J.; Patankar, N. A. *Journal of Colloid and Interface Science* **2005/1/15**, *281*, 458.

(8) Zisman, W. A.; Fox, H. W. *J. Coll. Sci.* **1950**, *5*, 514.

(9) Zisman, W. In *Relation of the Equilibrium Contact Angle to Liquid and Solid Constitution;* Gould, R., Ed.; Contact Angle Wettability and Adhesion; American Chemical Society: Washington, DC, 1964; Vol. 43, 1.

(10) Kennedy, G. L.; Butenhoff, J. L.; Olsen, G. W.; O'Connor, J. C.; Seacat, A. M.; Perkins, R. G.; Biegel, L. B.; Murphy, S. R.; Farrar, D. G. *Crit. Rev. Toxicol.* **2004**, *34*, 351.

(11) Kissa, E. *International Fiber Science and Technology Series* **1984**, *2*, 143.

(12) Dupre, A. In *Theorie Mechanique de la Chaleur;* Gauthier-Villars: Paris, 1869; 369.

(13) Young, T. *Phil. Trans. Roy. Soc.* **1858**, *95*, 255.

(14) Bose, A. In *Wetting by Solutions;* Berg, J. C., Ed.; Wettability; Marcel Dekker: New York, 1993; Vol. 49, 149.

(15) Good, R. J. *Journal of Colloid and Interface Science* **1975**, *52*, 308.

(16) Wenzel, T. N. *J. Phys. Colloid Chem.* **1949**, *53*, 1466.

(17) Cassie, A. B. D. *Discuss. Faraday Soc.* **1948**, *3*, 11.

(18) Johnson, R. E.; Dettre, R. H. In *Wetting of Low Energy Surfaces;* Berg, J. C., Ed.; Wettability; Marcel Dekker: New York, 1993; Vol. 49, 1.

(19) Penn, L. S. *Journal of Colloid and Interface Science* **1980**, *77*, 574.

(20) Grottenmuller, R. *Meilland Textilberichte* **1998**, *79*, E195.

(21) Otto, P. *Meilland Textilberichte* **1991**, *72*, E155.

(22) Thumm, S. *International Textile Bulletin* **2000**, *46*, 56.

(23) Anon. *International dyer* **2000**, *185*, 9.

(24) Lammerman, D. *Meilland Textilberichte* **1991**, *72*, E380.

(25) Sahin, B. *International Textile Bulletin - Dyeing/Printing/Finishing* **1996**, *42*, 26.

(26) Nassl, W.; Sahin, B.; Schuierer, M. *Chemiefasern/Textilindustrie* **1992**, *42/94*, E45.

(27) Cerne, L.; Simoncic, B. *Textile Research Journal* **2004**, *74*, 426.

(28) Cole, M. D. *International Fiber Journal* **2004**, *19*, 18.

(29) Environmental Protection Agency. *High Production Volume (HPV) Challenge Program - Basic Information on PFOA.* http://www.epa.gov/opptintr/pfoa/pubs/pfoainfo.htm (accessed 3/15, 2007).

(30) Hinderliter, P. M.; DeLorme, M. P.; Kennedy, G. L. *Toxicology* **2006**, *225*, 195.

(31) Kannan, K.; Choi, J.; Iseki, N.; Senthilkumar, K.; Kim, D. H.; Masunaga, S.; Giesy, J. P. *Chemosphere* **2002**, *49*, 225.

(32) Olivero-Verbel, J.; Tao, L.; Johnson-Restrepo, B.; Guette-Fernandez, J.; Baldiris-Avila, R.; O'byrne-Hoyos, I.; Kannan, K. *Environmental Pollution* **2006,** *142*, 367.

(33) Calafat, A. M.; Needham, L. L.; Kuklenyik, Z.; Reidy, J. A.; Tully, J. S.; Aguilar-Villalobos, M.; Naeher, L. P. *Chemosphere* **2006**, *63*, 490.

(34) Karrman, A.; van Bavel, B.; Jarnberg, U.; Hardell, L.; Lindstrom, G. *Chemosphere* **2006**, *64*, 1582.

(35) Loveless, S. E.; Finlay, C.; Everds, N. E.; Frame, S. R.; Gillies, P. J.; O'Connor, J. C.; Powley, C. R.; Kennedy, G. L. *Toxicology* **2006**, *220*, 203.

(36) Yamada, T.; Taylor, P. H.; Buck, R. C.; Kaiser, M. A.; Giraud, R. J. *Chemosphere* **2005**, *61*, 974.

(37) Hauser, P. J. **2006**, *Discussion of Fluorochemical Textile Finishes*.

(38) Tantillo, T. **2006**, *Correspondence Regarding Novel Fluorochemicals*.

(39) Lee, M.; Nishi, K.; Jeong, D. S.; Tokuyama, T.; Itazu, T.; Miyaji, Y.; Wakida, T. *Sen'i Gakkaishi* **2005**, *61*, 309.

(40) Mahltig, B.; Bottcher, H. *J. Sol Gel Sci. Technol.* **2003**, *27*, 43.

(41) Mahltig, B.; Haufe, H.; Bottcher, H. *Journal of Materials Chemistry* **2005**, *15*, 4385.

(42) Shao, H.; Sun, J. Y.; Meng, W. D.; Qing, F. L. *Text. Res. J.* **2004**, *74*, 851.

(43) Boutevin, B.; Abdellah, L.; Dinia, M. N. *European Polymer Journal* **1995/11**, *31*, 1127.

(44) Gao, L. C.; McCarthy, T. J. *Langmuir* **2006**, *22*, 5998.

(45) Soeno, T.; Inokuchi, K.; Shiratori, S. *Applied Surface Science* **2004/10/15**, *237*, 539.

(46) Zhang, J.; France, P.; Radomyselskiy, A.; Datta, S.; Zhao, J. A.; van Ooij, W. *J Appl Polym Sci* **2003**, *88*, 1473.

(47) Hocker, H. *Pure and Applied Chemistry* **2002**, *74*, 423.

(48) Li, J.; Fu, J.; Cong, Y.; Wu, Y.; Xue, L.; Han, Y. *Applied Surface Science* **2006/1/15**, *252*, 2229.

(49) *AATCC Test Method 81-2001 pH of the Water-Extract from Wet Processed Textiles;* AATCC Technical Manual; 2005; Vol. 80, 105.

(50) *AATCC Test Method 97-1999 Extractable Content of Greige and/or Prepared Textiles;* AATCC Technical Manual; 2005; Vol. 80, 141.

(51) *AATCC Test Method 193-2004 Aqueous Liquid Repellency: Water/Alcohol Solution Resistance Test;* AATCC Technical Manual; 2005; Vol. 80, 376.

(52) *AATCC Test Method 118-2002 Oil Repellency: Hydrocarbon Resistance Test;* AATCC Technical Manual; 2005; Vol. 80, 191.

(53) *AATCC Test Method 22-2001, Water Repellency: Spray Test;* AATCC Technical Manual; 2005; Vol. 80, 65.

(54) American Society of Testing Materials. *Standard Test Method for Tearing Strength of Fabrics by the Tongue (Single Rip) Procedure (Constant-Rate-of-Extension Tensile Testing Machine)*; **2002**, *D 2261-96*. Retrieved Feb. 17, 2007 from IHS Specs and Standards database from www.ihs.com

(55) American Society of Testing Materials. *Standard Test Method for Stiffness of Fabrics by the Circular Bend Method*; **2001**, *D 4032-94*. Retrieved Feb. 20, 2007 from IHS Specs and Standards database from www.ihs.com.

APPENDIX

Appendix A : Chemical Vendors and Products

Product	Chemistry	Qualifier
3M www.3m.com		
Protective Material - PM 930	Fluorochemical	Short Chain
Apexical, Inc. www.apexical.com		
Gardapex 15	Fluorochemical	
Waterproofon 203	Fluorochemical	With Extender
Waterproofon 213	Fluorochemical	Low Cure
Waterproofon 244	Fluorochemical	
Waterproofon 246	Fluorochemical	
Waterproofon 247	Fluorochemical	
Waterproofon 265	Fluorochemical	Cellulosics
Apollo Chemical Co. LLC www.apollochemicalcom		
Barpel 2000	Fluorochemical	Olefin Blends
Barpel 2100	Fluorochemical	Cationic
Barpel 350	Fluorochemical	With Extender
Boehme-Filatex www.boehmefilatex.com		
Hipoguard FC-4000	Fluorochemical	
Clariant www.clariant.com		
Nuva 2064	Fluorochemical	
Nuva 2110	Fluorochemical	
Nuva 3049	Fluorochemical	Synthetics
Nuva C6	Fluorochemical	Short Chain
Nuva DWRX	Fluorochemical	Synthetics
Nuva FB	Fluorochemical	
Nuva FBK	Fluorochemical	
Nuva FBN	Fluorochemical	
Nuva FDS	Fluorochemical	Synthetics
Nuva FHN	Fluorochemical	Crosslinkable
Nuva FSN	Fluorochemical	Synthetics
Nuva HPC	Fluorochemical	
Nuva HPU	Fluorochemical	
Nuva N 2114	Fluorochemical	
Nuva TP	Fluorochemical	
Nuva TTC	Fluorochemical	

Product	Chemistry	Qualifier
Daikin www.daikin.com		
UNIDYNE TG-400 Series	Fluorochemical	Soft Hand
UNIDYNE TG-500 Series	Fluorochemical	Durable
UNIDYNE TG-656	Fluorochemical	After Market
Emerald Performance Materials www.emeraldmaterials.com		
Freepel 1225	Hydrocarbon	
Freepel 1235	Hydrocarbon	Concentrated
Freepel DTF	Hydrocarbon	
Freepel FC-30	Fluorochemical	
Freepel FX-1202	Fluorochemical	With Extender
Freepell CCS	Hydrocarbon	Zirconium
Huntsman www.huntsman.com/textile_effects		
Hydrophobol APK	Hydrocarbon	
Hydrophobol ZAN	Hydrocarbon	
Oleophobol 7596	Fluorochemical	Synthetics
Oleophobol 7713	Fluorochemical	
Oleophobol CM	Fluorochemical	Microfiber
Oleophobol CO	Fluorochemical	Cellulosics
Oleophobol S	Fluorochemical	Synthetics
Oleophobol SL	Fluorochemical	Syn + Wool
Oleophobol SLA NEW	Fluorochemical	Syn + Wool
Oleophobol SM	Fluorochemical	Synthetics
Oleophobol TF	Fluorochemical	Temp Resistant
Phobol 7811	Fluorochemical	Nonwovens
Phobol 7834	Fluorochemical	Nonwovens
Phobotex FLX	Fluorochemical	With Extender
Phobotex FMX	Fluorochemical	With Extender
Phobotex JVA	Hydrocarbon	Cellulosics
Phobotex RC	Fluorochemical	With Extender
Phobotex VFN	Hydrocarbon	Cellulosics
Phobotone WS CONC	Silicone	
Lanxess us.lanxess.com		
Baygard AFF	Fluorochemical	
Baygard RT	Silicone	Fluorinated
Baygard SF-A 02	Silicone	Fluorinated

Product	Chemistry	Qualifier
MIC Speciality Chemicals (Asahi) www.micchem.com		
AsahiGuard E-061	Fluorochemical	Short Chain
Repearl F-14 D-3	Fluorochemical	Acetone
Repearl F-335	Fluorochemical	Cationic
Repearl F-35	Fluorochemical	Cationic
Repearl F-3700	Fluorochemical	Low Cure
Repearl F-45	Fluorochemical	Cationic
Repearl F-505	Fluorochemical	Cationic
Repearl F-611	Fluorochemical	Low Cure
Repearl F-622	Fluorochemical	Low Cure
Repearl F-7005	Fluorochemical	
Repearl F-7105	Fluorochemical	
Repearl F-7605	Fluorochemical	Cationic
Repearl F-8025	Fluorochemical	Amphoteric
Repearl F-8095	Fluorochemical	Cationic
Repearl F-89	Fluorochemical	Low Cure
Repearl SR-1100	Fluorochemical	Soil Release
Omnova www.omnova.com		
Impregnole FH	Hydrocarbon	Zirconium
Norane 100	Hydrocarbon	Reactive
Norane Sil	Silicone	
Sequapel 417	Hydrocarbon	Paraffin
X-Cape AFC	Fluorochemical	Cationic
X-Cape DRC	Fluorochemical	
X-Cape GFC	Fluorochemical	Cationic
X-Cape LVN	Fluorochemical	Low Cure
X-Cape NFU	Fluorochemical	With Extender
X-Cape Ultra	Fluorochemical	
Rudolf Chemie www.rudolf.de		
RUCO-GUARD AFC	Fluorochemical	Cellulosics
RUCO-GUARD AFL	Fluorochemical	No Cure
RUCO-GUARD AFN	Fluorochemical	Microfiber
RUCO-GUARD AFR	Fluorochemical	
RUCO-GUARD AFS	Fluorochemical	Synthetics
RUCO-GUARD AFU	Fluorochemical	Upholstery
RUCOSTAR EEE	Fluorochemical	

Appendix B : Continuous Application Pad Bath Formulations and Oven Settings

Finishing Treatments used in the Continuous Application Process

Treatment	Primary Chemical	Concentration (g/L)	Secondary Chemical	Concentration (g/L)	Drying Set Point (°C)	Observed Min. Temp (°C)	Observed Max Temp. (°C)
SI-1	Norane Sil	50.0	Catalyst Sil	30.0	135	136	139
SI-2	Phobotone WS CONC*	30.0	Phobotone BC New	40.0	135	136	139
HR-3	Norane 100	120.0	--	--	135	138	138
HZ-4	Impregnole FH	100.0	--	--	135	134	140
HZ-5	Freepell CCS	70.0	--	--	132	135	137
FC-6	Waterproofon 246	25.0	--	--	132	134	135
FC-7	AsahiGuard E-061	25.0	--	--	132	134	135
FC-8	Fluorochem A1	25.0	--	--	132	135	137
FM-9	Waterproofon 246	25.0	Aerotex M3	7.5	132	135	136
FM-10	AsahiGuard E-061	25.0	Aerotex M3	7.5	132	133	136
FM-11	Fluorochem A1	25.0	Aerotex M3	7.5	132	136	137
FI-12	Waterproofon 246	25.0	TMI[META]	10.0	132	136	136
FI-13	Waterproofon 246	25.0	Apexosist 186	10.0	132	135	136
FI-14	AsahiGuard E-061	25.0	Apexosist 186	10.0	132	134	137
FI-15	Fluorochem A1	25.0	Apexosist 186	10.0	132	135	137

* Note: Finish bath SI-2 also contained 5mL glacial acetic acid to control pH

Appendix C : Repellency Testing Results

Cotton Fabric - Repellency Test Results																
		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
0 Washes	Spray Test																
	Sample 1	95	100	100	100	100	90	90	70	100	100	80	100	100	100	80	0
	Sample 2	95	100	95	100	100	90	90	75	100	100	80	100	100	100	80	0
	Sample 3	95	100	100	100	100	100	95	70	100	100	80	100	100	95	80	0
	Average	95.00	100.00	98.33	100.00	100.00	93.33	91.67	71.67	100.00	100.00	80.00	100.00	100.00	98.33	80.00	0.00
	Oil Repellency																
	Sample 1	0	0	0	0	0	6	3.5	5	6.5	5	5	6	6	2.5	5	0
	Sample 2	0	0	0	0	0	6	4	5	6.5	5	5	6	6	3	5	0
	Sample 3	--	--	--	--	--	--	3.5	--	--	--	--	--	--	3	--	--
	Report Value	0	0	0	0	0	6	3.5	5	6.5	5	5	6	6	3	5	0
	Aqueous Liquid Repellency																
	Sample 1	3.5	4	3.5	3.5	4	8	5.5	4.5	8	7.5	4.5	8	8	6.5	4.5	0
	Sample 2	3.5	4	4	3.5	4	8	5.5	4	8	7.5	4.5	8	8	6.5	4.5	0
	Sample 3	--	--	4	--	--	--	--	4	--	--	--	--	--	--	--	--
	Report Value	3.5	4	4	3.5	4	8	5.5	4	8	7.5	4.5	8	8	6.5	4.5	0
5 Washes	Spray Test																
	Sample 1	65	75	75	75	60	75	80	80	80	75	70	75	85	80	75	--
	Sample 2	65	75	75	70	60	75	80	70	80	75	75	75	85	80	75	--
	Sample 3	60	80	75	75	70	75	70	80	80	70	70	75	85	80	75	--
	Average	63.33	76.67	75.00	73.33	63.33	75.00	76.67	76.67	80.00	73.33	71.67	75.00	85.00	80.00	75.00	--
	Oil Repellency																
	Sample 1	0	0	0	0	0	5	2	5	6	2.5	5	5	6	2.5	5	--
	Sample 2	0	0	0	0	0	5	2	5	6	2.5	5	5	6	2	5	--
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	2	--	--
	Report Value	0	0	0	0	0	5	2	5	6	2.5	5	5	6	2	5	--
	Aqueous Liquid Repellency																
	Sample 1	3	3.5	3	3	3	8	5	4	8	5	4	8	8	5	5	--
	Sample 2	3	4	3	3	3	8	5	4	8	5.5	3.5	7.5	8	5	5	--
	Sample 3	--	4	--	--	--	--	--	--	--	5.5	4	7.5	--	--	--	--
	Report Value	3	4	3	3	3	8	5	4	8	5.5	4	7.5	8	5	5	--
25 Washes	Spray Test																
	Sample 1	--	50	0	--	--	0	0	--	--	--	--	--	70	60	--	--
	Sample 2	--	50	0	--	--	0	0	--	--	--	--	--	70	70	--	--
	Sample 3	--	50	0	--	--	0	0	--	--	--	--	--	70	70	--	--
	Average	--	50.00	0.00	--	--	0.00	0.00	--	--	--	--	--	70.00	66.67	--	--
	Oil Repellency																
	Sample 1	--	0	0	--	--	0	0	--	--	--	--	--	2	1	--	--
	Sample 2	--	0	0	--	--	0	0	--	--	--	--	--	2	1	--	--
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	--	0	0	--	--	0	0	--	--	--	--	--	2	1	--	--
	Aqueous Liquid Repellency																
	Sample 1	--	1.5	0	--	--	3	3	--	--	--	--	--	4	3	--	--
	Sample 2	--	2.5	0	--	--	3	3	--	--	--	--	--	5	3.5	--	--
	Sample 3	--	2.5	--	--	--	--	--	--	--	--	--	--	5	3	--	--
	Report Value	--	2.5	0	--	--	3	3	--	--	--	--	--	5	3	--	--

Poly/Cotton Fabric - Repellency Test Results																	
		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
0 Washes	Spray Test																
	Sample 1	90	100	95	100	95	100	100	75	100	100	75	100	100	100	80	0
	Sample 2	90	100	100	100	100	100	100	75	100	100	80	100	100	100	80	0
	Sample 3	85	100	100	100	100	100	100	70	100	100	80	100	100	100	80	0
	Average	**88.33**	**100.00**	**98.33**	**100.00**	**98.33**	**100.00**	**100.00**	**73.33**	**100.00**	**100.00**	**78.33**	**100.00**	**100.00**	**100.00**	**80.00**	**0.00**
	Oil Repellency																
	Sample 1	0	0	0	0	0	6.5	2.5	5	6.5	3.5	4.5	6.5	6.5	2.5	4.5	0
	Sample 2	0	0	0	0	0	6.5	2.5	5	6.5	2.5	4.5	6.5	6.5	2.5	4.5	0
	Sample 3	--	--	--	--	--	--	--	--	--	2.5	--	--	--	--	--	--
	Report Value	**0**	**0**	**0**	**0**	**0**	**6.5**	**2.5**	**5**	**6.5**	**2.5**	**4.5**	**6.5**	**6.5**	**2.5**	**4.5**	**0**
	Aqueous Liquid Repellency																
	Sample 1	3.5	3.5	3.5	3.5	4	8	5.5	4.5	8	6.5	4.5	8	8	5.5	4.5	0
	Sample 2	3.5	3.5	3.5	3.5	4	8	5.5	4.5	8	6.5	4.5	8	8	5.5	4.5	0
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	**3.5**	**3.5**	**3.5**	**3.5**	**4**	**8**	**5.5**	**4.5**	**8**	**6.5**	**4.5**	**8**	**8**	**5.5**	**4.5**	**0**
5 Washes	Spray Test																
	Sample 1	80	95	75	70	75	95	80	75	100	85	80	95	100	85	75	--
	Sample 2	80	95	75	75	75	95	85	80	100	85	80	95	100	85	75	--
	Sample 3	80	95	80	75	75	100	80	80	100	85	80	95	100	85	75	--
	Average	**80.00**	**95.00**	**76.67**	**73.33**	**75.00**	**96.67**	**81.67**	**78.33**	**100.00**	**85.00**	**80.00**	**95.00**	**100.00**	**85.00**	**75.00**	--
	Oil Repellency																
	Sample 1	0	0	0	0	0	5.5	1.5	4.5	5.5	1.5	4.5	6	6	1.5	5	--
	Sample 2	0	0	0	0	0	5.5	1.5	2.5	5.5	1.5	5	5.5	6	1.5	4.5	--
	Sample 3	--	--	--	--	--	--	--	4.5	--	--	5	5.5	--	--	4.5	--
	Report Value	**0**	**0**	**0**	**0**	**0**	**5.5**	**1.5**	**4.5**	**5.5**	**1.5**	**5**	**5.5**	**6**	**1.5**	**4.5**	--
	Aqueous Liquid Repellency																
	Sample 1	3.5	4	3.5	3	3.5	8	5.5	4.5	8	5.5	4.5	8	8	5.5	4.5	--
	Sample 2	3.5	4	3.5	3	3	8	5.5	4.5	8	5.5	4.5	8	8	5.5	4.5	--
	Sample 3	--	--	--	--	3.5	--	--	--	--	--	--	--	--	--	--	--
	Report Value	**3.5**	**4**	**3.5**	**3**	**3.5**	**8**	**5.5**	**4.5**	**8**	**5.5**	**4.5**	**8**	**8**	**5.5**	**4.5**	--
25 Washes	Spray Test																
	Sample 1	--	70	60	--	--	80	60	--	--	--	--	--	75	70	--	--
	Sample 2	--	60	60	--	--	75	60	--	--	--	--	--	80	70	--	--
	Sample 3	--	60	60	--	--	80	60	--	--	--	--	--	75	75	--	--
	Average	--	**63.33**	**60.00**	--	--	**78.33**	**60.00**	--	--	--	--	--	**76.67**	**71.67**	--	--
	Oil Repellency																
	Sample 1	--	0	0	--	--	5	0	--	--	--	--	--	4.5	0.5	--	--
	Sample 2	--	0	0	--	--	4.5	0	--	--	--	--	--	4.5	0.5	--	--
	Sample 3	--	--	--	--	--	4.5	--	--	--	--	--	--	--	--	--	--
	Report Value	--	**0**	**0**	--	--	**4.5**	**0**	--	--	--	--	--	**4.5**	**0.5**	--	--
	Aqueous Liquid Repellency																
	Sample 1	--	3	3	--	--	6.5	3.5	--	--	--	--	--	6.5	4.5	--	--
	Sample 2	--	3	3	--	--	6.5	3.5	--	--	--	--	--	6.5	4.5	--	--
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	--	**3**	**3**	--	--	**6.5**	**3.5**	--	--	--	--	--	**6.5**	**4.5**	--	--

Polyester Fabric - Repellency Test Results																	
		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
0 Washes	Spray Test																
	Sample 1	80	100	90	90	95	100	100	75	100	100	75	100	100	100	80	0
	Sample 2	80	100	90	100	95	100	90	75	100	100	80	100	100	100	80	0
	Sample 3	80	100	100	100	90	100	90	75	100	100	80	100	100	100	80	0
	Average	80.00	100.00	93.33	96.67	93.33	100.00	93.33	75.00	100.00	100.00	78.33	100.00	100.00	100.00	80.00	0.00
	Oil Repellency																
	Sample 1	0	0	0	0	0	6.5	6	5	7	6	5.5	6.5	6.5	6	5	0
	Sample 2	0	0	0	0	0	6.5	6	5	7	6	5	6.5	6.5	6	5	0
	Sample 3	--	--	--	--	--	--	--	--	--	--	5.5	--	--	--	--	--
	Report Value	0	0	0	0	0	6.5	6	5	7	6	5.5	6.5	6.5	6	5	0
	Aqueous Liquid Repellency																
	Sample 1	4	4	3.5	3.5	4	8	8	4.5	8	8	5	8	8	8	4.5	0
	Sample 2	4	4	3.5	4	4	8	8	4.5	8	8	5	8	8	8	4.5	0
	Sample 3	--	--	--	4	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	4	4	3.5	4	4	8	8	4.5	8	8	5	8	8	8	4.5	0
5 Washes	Spray Test																
	Sample 1	80	85	80	80	75	90	85	75	95	85	80	95	95	90	75	--
	Sample 2	80	90	80	80	80	95	85	75	95	85	80	95	95	90	75	--
	Sample 3	80	90	80	80	80	95	80	80	100	90	80	95	95	90	75	--
	Average	80.00	88.33	80.00	80.00	78.33	93.33	83.33	76.67	96.67	86.67	80.00	95.00	95.00	90.00	75.00	--
	Oil Repellency																
	Sample 1	0	0	0	0	0	6.5	5.5	5.5	6.5	5.5	5.5	6.5	6.5	6	5	--
	Sample 2	0	0	0	0	0	6.5	5.5	5.5	6.5	5.5	5.5	6.5	6.5	6	5.5	--
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	5	--
	Report Value	0	0	0	0	0	6.5	5.5	5.5	6.5	5.5	5.5	6.5	6.5	6	5	--
	Aqueous Liquid Repellency																
	Sample 1	4	5	4	4	4	8	8	5.5	8	8	6	8	8	8	5.5	--
	Sample 2	4	4.5	4	4	4	8	8	5.5	8	8	6	8	8	8	5	--
	Sample 3	--	4.5	--	--	--	--	--	--	--	--	--	--	--	--	5	--
	Report Value	4	4.5	4	4	4	8	8	5.5	8	8	6	8	8	8	5	--
25 Washes	Spray Test																
	Sample 1	--	80	75	--	--	95	80	--	--	--	--	--	95	85	--	--
	Sample 2	--	80	75	--	--	95	80	--	--	--	--	--	95	85	--	--
	Sample 3	--	80	75	--	--	100	80	--	--	--	--	--	100	85	--	--
	Average	--	80.00	75.00	--	--	96.67	80.00	--	--	--	--	--	96.67	85.00	--	--
	Oil Repellency																
	Sample 1	--	0	0	--	--	6.5	5.5	--	--	--	--	--	6.5	5.5	--	--
	Sample 2	--	0	0	--	--	6.5	5	--	--	--	--	--	6.5	5.5	--	--
	Sample 3	--	--	--	--	--	--	5	--	--	--	--	--	--	--	--	--
	Report Value	--	0	0	--	--	6.5	5	--	--	--	--	--	6.5	5.5	--	--
	Aqueous Liquid Repellency																
	Sample 1	--	4	3.5	--	--	8	8	--	--	--	--	--	8	8	--	--
	Sample 2	--	4	3.5	--	--	8	8	--	--	--	--	--	8	8	--	--
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	--	4	3.5	--	--	8	8	--	--	--	--	--	8	8	--	--

Nylon Fabric - Repellency Test Results

		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
0 Washes	**Spray Test**																
	Sample 1	95	100	95	95	95	100	100	90	100	100	80	100	100	100	80	0
	Sample 2	95	100	95	95	95	100	100	90	100	100	80	100	100	100	80	0
	Sample 3	100	95	95	95	90	100	100	95	100	100	85	100	100	100	80	0
	Average	**96.67**	**98.33**	**95.00**	**95.00**	**93.33**	**100.00**	**100.00**	**91.67**	**100.00**	**100.00**	**81.67**	**100.00**	**100.00**	**100.00**	**80.00**	**0.00**
	Oil Repellency																
	Sample 1	0	0	0	0	0	6.5	5.5	4	6.5	6	4.5	6.5	6.5	5.5	4.5	0
	Sample 2	0	0	0	0	0	6.5	5.5	4.5	6.5	6	4.5	6.5	6.5	4	3.5	0
	Sample 3	--	--	--	--	--	--	--	4	--	--	--	--	--	5.5	4.5	--
	Report Value	**0**	**0**	**0**	**0**	**0**	**6.5**	**5.5**	**4**	**6.5**	**6**	**4.5**	**6.5**	**6.5**	**5.5**	**4.5**	**0**
	Aqueous Liquid Repellency																
	Sample 1	3.5	4.5	3	2.5	3	8	8	4	8	7.5	4.5	8	8	8	3	0
	Sample 2	3.5	4.5	3.5	3	3	8	8	4	8	8	4.5	8	8	8	3.5	0
	Sample 3	--	--	3.5	2.5	--	--	--	--	--	8	--	--	--	--	3.5	--
	Report Value	**3.5**	**4.5**	**3.5**	**2.5**	**3**	**8**	**8**	**4**	**8**	**8**	**4.5**	**8**	**8**	**8**	**3.5**	**0**
5 Washes	**Spray Test**																
	Sample 1	90	100	80	75	80	100	100	90	100	100	80	100	100	100	75	--
	Sample 2	90	100	80	75	75	100	100	90	100	100	80	100	100	90	75	--
	Sample 3	90	100	80	75	75	100	100	85	100	95	85	100	100	95	75	--
	Average	**90.00**	**100.00**	**80.00**	**75.00**	**76.67**	**100.00**	**100.00**	**88.33**	**100.00**	**98.33**	**81.67**	**100.00**	**100.00**	**95.00**	**75.00**	--
	Oil Repellency																
	Sample 1	0	0	0	0	0	6	2.5	4.5	6	1.5	4.5	6	6	0.5	4.5	--
	Sample 2	0	0	0	0	0	6	2.5	4.5	6	2.5	4.5	6	6	1.5	4.5	--
	Sample 3	--	--	--	--	--	--	--	--	--	2.5	--	--	--	1.5	--	--
	Report Value	**0**	**0**	**0**	**0**	**0**	**6**	**2.5**	**4.5**	**6**	**2.5**	**4.5**	**6**	**6**	**1.5**	**4.5**	--
	Aqueous Liquid Repellency																
	Sample 1	3.5	4.5	3.5	3.5	3.5	8	7.5	5	8	8	5	8	8	5.5	4.5	--
	Sample 2	3.5	4.5	3.5	3.5	3.5	8	8	5	8	7.5	5	8	8	6.5	4.5	--
	Sample 3	--	--	--	--	--	--	8	--	--	8	--	--	--	6.5	--	--
	Report Value	**3.5**	**4.5**	**3.5**	**3.5**	**3.5**	**8**	**8**	**5**	**8**	**8**	**5**	**8**	**8**	**6.5**	**4.5**	--
25 Washes	**Spray Test**																
	Sample 1	--	90	50	--	--	100	80	--	--	--	--	--	100	80	--	--
	Sample 2	--	90	50	--	--	100	80	--	--	--	--	--	100	75	--	--
	Sample 3	--	80	50	--	--	100	80	--	--	--	--	--	100	75	--	--
	Average	--	**86.67**	**50.00**	--	--	**100.00**	**80.00**	--	--	--	--	--	**100.00**	**76.67**	--	--
	Oil Repellency																
	Sample 1	--	0	0	--	--	5.5	2.5	--	--	--	--	--	6	0.5	--	--
	Sample 2	--	0	0	--	--	6	2.5	--	--	--	--	--	6	0.5	--	--
	Sample 3	--	--	--	--	--	6	--	--	--	--	--	--	--	--	--	--
	Report Value	--	**0**	**0**	--	--	**6**	**2.5**	--	--	--	--	--	**6**	**0.5**	--	--
	Aqueous Liquid Repellency																
	Sample 1	--	3.5	3	--	--	8	6.5	--	--	--	--	--	8	5	--	--
	Sample 2	--	3.5	3	--	--	8	6.5	--	--	--	--	--	8	4.5	--	--
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	4.5	--	--
	Report Value	--	**3.5**	**3**	--	--	**8**	**6.5**	--	--	--	--	--	**8**	**4.5**	--	--

Automotive (AU) Acrylic Fabric - Repellency Test Results																
		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
0 Washes	**Spray Test**																
	Sample 1	85	100	90	95	95	100	100	70	100	100	75	100	100	100	75	0
	Sample 2	85	100	95	100	100	100	100	70	100	100	75	100	100	100	80	0
	Sample 3	80	100	100	95	95	100	100	70	100	100	75	100	100	100	75	0
	Average	**83.33**	**100.00**	**95.00**	**96.67**	**96.67**	**100.00**	**100.00**	**70.00**	**100.00**	**100.00**	**75.00**	**100.00**	**100.00**	**100.00**	**76.67**	**0.00**
	Oil Repellency																
	Sample 1	0	0	0	0	0	6.5	5.5	5.5	7	6	5	6.5	6.5	5.5	5.5	0
	Sample 2	0	0	0	0	0	6.5	5.5	5.5	7	6	5	6.5	6.5	5.5	5.5	0
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	**0**	**0**	**0**	**0**	**0**	**6.5**	**5.5**	**5.5**	**7**	**6**	**5**	**6.5**	**6.5**	**5.5**	**5.5**	**0**
	Aqueous Liquid Repellency																
	Sample 1	3.5	4	3.5	4	4	8	8	4.5	8	8	3.5	8	8	8	4.5	0
	Sample 2	3.5	4	3.5	3.5	4	8	8	4.5	8	8	3.5	8	8	8	3.5	0
	Sample 3	--	--	--	4	--	--	--	--	--	--	--	--	--	--	3.5	--
	Report Value	**3.5**	**4**	**3.5**	**4**	**4**	**8**	**8**	**4.5**	**8**	**8**	**3.5**	**8**	**8**	**8**	**3.5**	**0**
5 Washes	**Spray Test**																
	Sample 1	80	100	80	80	75	100	90	75	100	80	75	100	100	85	75	--
	Sample 2	80	100	80	80	80	100	85	80	100	85	75	100	100	85	75	--
	Sample 3	80	95	80	80	80	100	85	80	100	85	75	100	100	85	75	--
	Average	**80.00**	**98.33**	**80.00**	**80.00**	**78.33**	**100.00**	**86.67**	**78.33**	**100.00**	**83.33**	**75.00**	**100.00**	**100.00**	**85.00**	**75.00**	--
	Oil Repellency																
	Sample 1	0	0	0	0	0	6	4.5	5.5	6.5	5	5.5	6	6.5	5	5.5	--
	Sample 2	0	0	0	0	0	6	4.5	5.5	6	4.5	5.5	6	6.5	5	5.5	--
	Sample 3	--	--	--	--	--	--	--	--	6	4.5	--	--	--	--	--	--
	Report Value	**0**	**0**	**0**	**0**	**0**	**6**	**4.5**	**5.5**	**6**	**4.5**	**5.5**	**6**	**6.5**	**5**	**5.5**	--
	Aqueous Liquid Repellency																
	Sample 1	4	4	4	4	4	8	8	5	8	8	5	8	8	8	5	--
	Sample 2	4	4	4	4	4	8	7.5	5	8	8	5	8	8	8	5	--
	Sample 3	--	--	--	--	--	--	8	--	--	--	--	--	--	--	--	--
	Report Value	**4**	**4**	**4**	**4**	**4**	**8**	**8**	**5**	**8**	**8**	**5**	**8**	**8**	**8**	**5**	--
25 Washes	**Spray Test**																
	Sample 1	--	75	70	--	--	80	75	--	--	--	--	--	85	80	--	--
	Sample 2	--	75	70	--	--	85	75	--	--	--	--	--	85	80	--	--
	Sample 3	--	70	70	--	--	80	75	--	--	--	--	--	85	80	--	--
	Average	--	**73.33**	**70.00**	--	--	**81.67**	**75.00**	--	--	--	--	--	**85.00**	**80.00**	--	--
	Oil Repellency																
	Sample 1	--	0	0	--	--	6	4	--	--	--	--	--	6	2.5	--	--
	Sample 2	--	0	0	--	--	6	4.5	--	--	--	--	--	6	2	--	--
	Sample 3	--	--	--	--	--	--	4	--	--	--	--	--	--	2	--	--
	Report Value	--	**0**	**0**	--	--	**6**	**4**	--	--	--	--	--	**6**	**2**	--	--
	Aqueous Liquid Repellency																
	Sample 1	--	4	3	--	--	8	8	--	--	--	--	--	8	6.5	--	--
	Sample 2	--	4	3.5	--	--	8	8	--	--	--	--	--	8	8	--	--
	Sample 3	--	--	3.5	--	--	--	--	--	--	--	--	--	--	8	--	--
	Report Value	--	**4**	**3.5**	--	--	**8**	**8**	--	--	--	--	--	**8**	**8**	--	--

Home Furnishing (HF) Acrylic Fabric - Repellency Test Results

		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
0 Washes	**Spray Test**																
	Sample 1	80	100	100	100	100	100	100	70	100	100	80	100	100	100	75	0
	Sample 2	85	100	95	100	100	100	100	70	100	100	80	100	100	100	80	0
	Sample 3	80	100	100	100	100	100	100	70	100	100	80	100	100	100	75	0
	Average	**81.67**	**100.00**	**98.33**	**100.00**	**100.00**	**100.00**	**100.00**	**70.00**	**100.00**	**100.00**	**80.00**	**100.00**	**100.00**	**100.00**	**76.67**	**0.00**
	Oil Repellency																
	Sample 1	0	0	0	0	0	7	6	6	7	6	5	7	7	6	6	0
	Sample 2	0	0	0	0	0	7	6	6	7	6	5	7	7	6	6	0
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	**0**	**0**	**0**	**0**	**0**	**7**	**6**	**6**	**7**	**6**	**5**	**7**	**7**	**6**	**6**	**0**
	Aqueous Liquid Repellency																
	Sample 1	4	4.5	4	4	4	8	8	4.5	8	8	5	8	8	8	4	0
	Sample 2	3.5	4.5	4	3.5	4	8	8	4.5	8	8	5	8	8	8	4	0
	Sample 3	3.5	--	--	4	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	**3.5**	**4.5**	**4**	**4**	**4**	**8**	**8**	**4.5**	**8**	**8**	**5**	**8**	**8**	**8**	**4**	**0**
5 Washes	**Spray Test**																
	Sample 1	75	100	75	80	75	100	95	80	100	95	75	100	100	95	75	--
	Sample 2	80	100	80	80	75	100	85	80	100	95	75	100	100	100	75	--
	Sample 3	80	100	80	80	75	100	90	80	100	100	75	100	100	95	75	--
	Average	**78.33**	**100.00**	**78.33**	**80.00**	**75.00**	**100.00**	**90.00**	**80.00**	**100.00**	**96.67**	**75.00**	**100.00**	**100.00**	**96.67**	**75.00**	--
	Oil Repellency																
	Sample 1	0	0	0	0	0	6.5	4.5	5.5	6.5	5	5	6	6.5	5.5	5.5	--
	Sample 2	0	0	0	0	0	6	5	5.5	6	5	5	6	6.5	5.5	5.5	--
	Sample 3	--	--	--	--	--	6	4.5	--	6	--	--	--	--	--	--	--
	Report Value	**0**	**0**	**0**	**0**	**0**	**6**	**4.5**	**5.5**	**6**	**5**	**5**	**6**	**6.5**	**5.5**	**5.5**	--
	Aqueous Liquid Repellency																
	Sample 1	3	4	4	4	4	8	8	5	8	8	5	8	8	8	5	--
	Sample 2	3	4	4	3.5	4	8	8	5	8	8	5	8	8	8	5	--
	Sample 3	--	--	--	3.5	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	**3**	**4**	**4**	**3.5**	**4**	**8**	**8**	**5**	**8**	**8**	**5**	**8**	**8**	**8**	**5**	--
25 Washes	**Spray Test**																
	Sample 1	--	75	70	--	--	80	75	--	--	--	--	--	85	80	--	--
	Sample 2	--	75	70	--	--	80	70	--	--	--	--	--	90	75	--	--
	Sample 3	--	75	70	--	--	80	70	--	--	--	--	--	85	75	--	--
	Average	--	**75.00**	**70.00**	--	--	**80.00**	**71.67**	--	--	--	--	--	**86.67**	**76.67**	--	--
	Oil Repellency																
	Sample 1	--	0	0	--	--	6	4	--	--	--	--	--	6	4	--	--
	Sample 2	--	0	0	--	--	6	3	--	--	--	--	--	6	3	--	--
	Sample 3	--	--	--	--	--	--	3	--	--	--	--	--	--	2	--	--
	Report Value	--	**0**	**0**	--	--	**6**	**3**	--	--	--	--	--	**6**	**3**	--	--
	Aqueous Liquid Repellency																
	Sample 1	--	4	3	--	--	8	8	--	--	--	--	--	8	8	--	--
	Sample 2	--	4	3	--	--	8	8	--	--	--	--	--	8	8	--	--
	Sample 3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	Report Value	--	**4**	**3**	--	--	**8**	**8**	--	--	--	--	--	**8**	**8**	--	--

Appendix D : Fluorine Content Analysis by Ion Selective Electrode Results

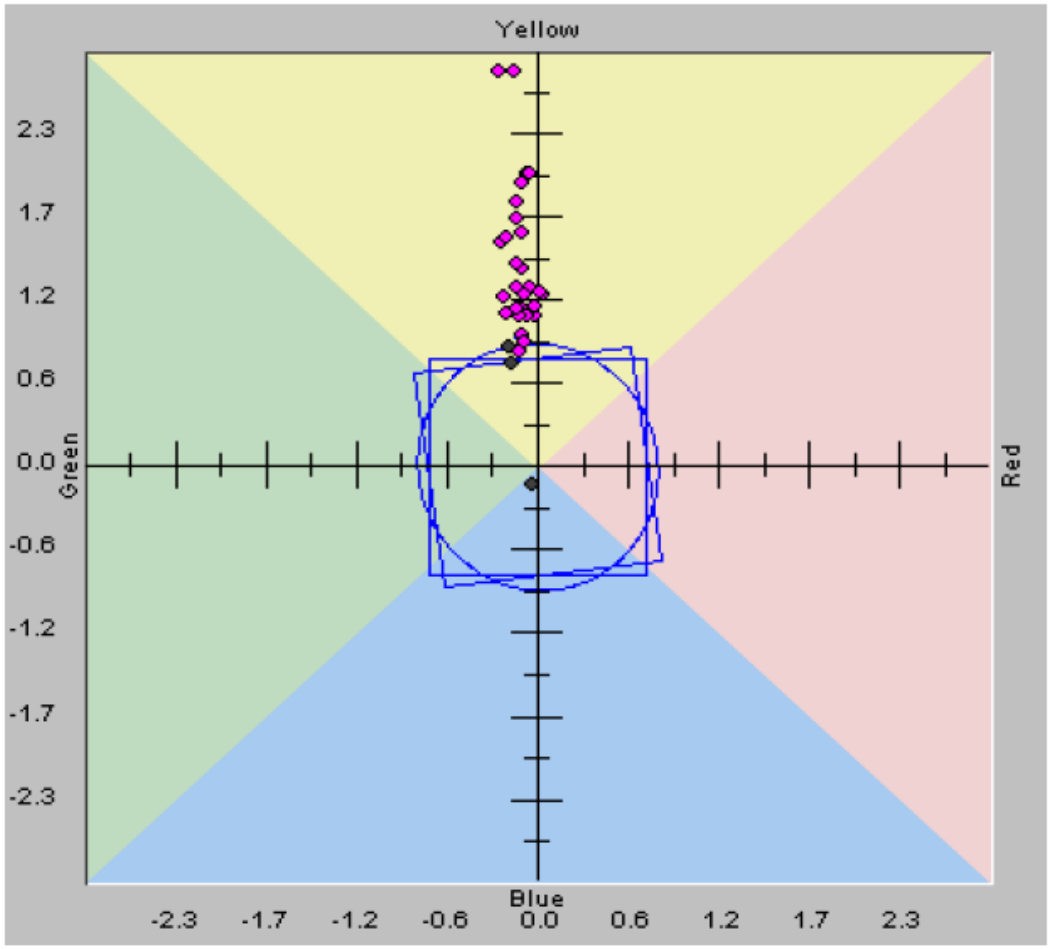
Total Fluorine Content by ISE (ppm)

Fabric	Waterproofon 246	AsahiGuard E-061	Fluorochem A1	Untreated Fabric
HF Acrylic	2871	1947	2826	<16
AU Acrylic	2699	1990	2717	<18
Nylon	2401	1909	2430	<18
Polyester	3621	2738	3046	21
Poly/Cotton	2031	1241	2380	<17
Cotton	3467	2620	4494	<17

Appendix E : Spectrophotometric Analysis of Color Shift due to Finishing

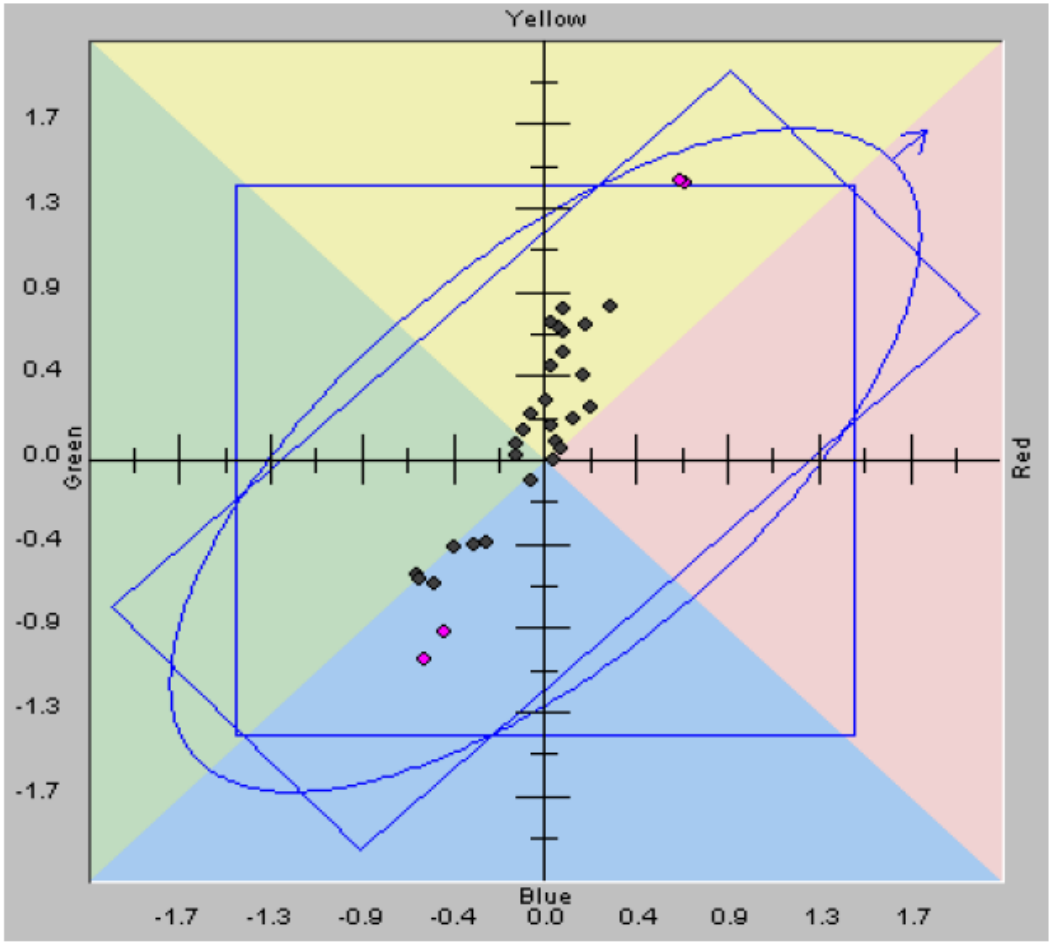
Color Change After Finishing Treatment - Cotton

--	L*	a*	b*	--
Standard	*93.63*	*-0.51*	*3.68*	--
Sample	DL*	Da*	Db*	Decmc
SI-1	-0.48	-0.10	1.38	**1.61**
SI-1	-0.30	-0.14	1.42	**1.65**
SI-2	-0.75	-0.24	2.77	**3.23**
SI-2	-0.71	-0.15	2.76	**3.21**
HR-3	-0.61	-0.13	1.84	**2.15**
HR-3	-0.76	-0.09	1.98	**2.31**
HZ-4	-0.75	-0.07	2.06	**2.40**
HZ-4	-0.68	-0.05	2.06	**2.40**
HZ-5	-0.55	-0.09	1.64	**1.91**
HZ-5	-0.42	-0.13	1.74	**2.02**
FC-6	-0.18	-0.13	1.25	**1.46**
FC-6	-0.24	-0.09	1.21	**1.41**
FC-7	-0.08	-0.11	1.06	**1.24**
FC-7	-0.14	-0.14	1.10	**1.28**
FC-8	-0.39	-0.10	1.09	**1.28**
FC-8	-0.39	-0.07	1.05	**1.23**
FM-9	-0.25	-0.17	0.73	**0.87**
FM-9	-0.25	-0.19	0.83	**0.99**
FM-10	-0.30	-0.21	1.18	**1.40**
FM-10	-0.31	-0.20	1.08	**1.27**
FM-11	-0.75	-0.09	0.93	**1.11**
FM-11	-0.66	-0.08	0.86	**1.03**
FI-12	-2.80	-0.02	1.12	**1.62**
FI-12	-1.81	-0.11	0.81	**1.13**
FI-13	-0.46	-0.01	1.05	**1.23**
FI-13	-0.50	0.02	1.22	**1.43**
FI-14	-0.36	-0.23	1.57	**1.85**
FI-14	-0.33	-0.19	1.60	**1.86**
FI-15	-1.18	0.03	1.20	**1.46**
FI-15	-0.67	-0.05	1.26	**1.48**



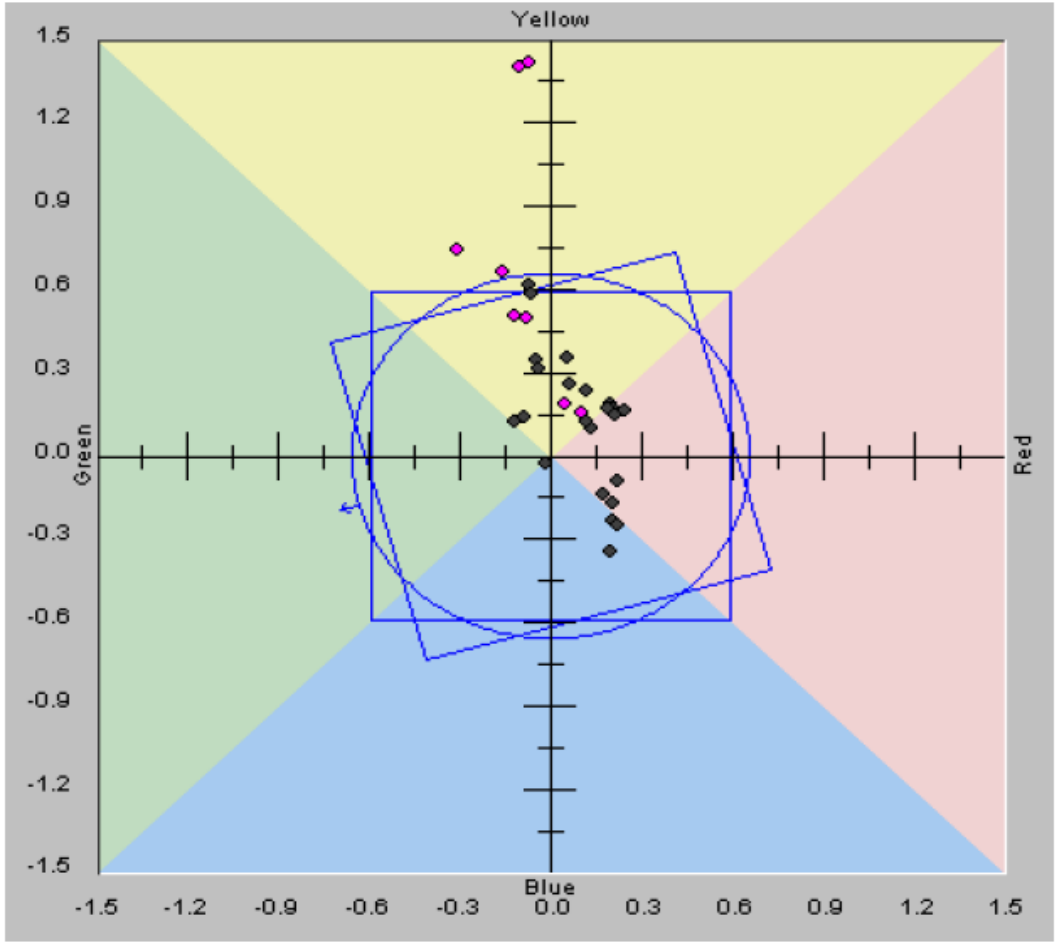
Color Change After Finishing Treatment - Poly/Cotton

--	L*	a*	b*	--
Standard	*38.58*	*27.14*	*25.6*	--
Sample	DL*	Da*	Db*	Decmc
SI-1	-1.53	0.01	0.31	**0.85**
SI-1	-1.56	0.03	0.18	**0.84**
SI-2	-1.70	-0.59	-0.61	**0.98**
SI-2	-1.66	-0.52	-0.64	**0.96**
HR-3	-1.93	-0.48	-0.88	**1.16**
HR-3	-1.93	-0.57	-1.02	**1.20**
HZ-4	-1.30	-0.43	-0.44	**0.75**
HZ-4	-1.52	-0.61	-0.59	**0.89**
HZ-5	-1.22	-0.27	-0.42	**0.70**
HZ-5	-1.15	-0.34	-0.43	**0.66**
FC-6	-1.01	0.08	0.78	**0.79**
FC-6	-1.02	0.03	0.70	**0.77**
FC-7	-0.82	-0.06	0.23	**0.49**
FC-7	-1.02	-0.10	0.16	**0.57**
FC-8	-0.91	0.07	0.06	**0.48**
FC-8	-1.21	0.04	0.00	**0.64**
FM-9	-1.51	0.65	1.41	**1.18**
FM-9	-1.39	0.63	1.42	**1.15**
FM-10	-0.88	0.31	0.79	**0.68**
FM-10	-1.09	0.20	0.69	**0.74**
FM-11	-0.81	0.21	0.26	**0.46**
FM-11	-0.78	0.19	0.43	**0.50**
FI-12	-1.54	0.03	0.48	**0.90**
FI-12	-1.39	0.09	0.55	**0.84**
FI-13	-0.96	0.09	0.65	**0.70**
FI-13	-0.89	0.07	0.68	**0.69**
FI-14	-1.20	-0.14	0.09	**0.66**
FI-14	-1.17	-0.13	0.02	**0.63**
FI-15	-1.11	0.14	0.21	**0.60**
FI-15	-1.09	0.05	0.09	**0.58**



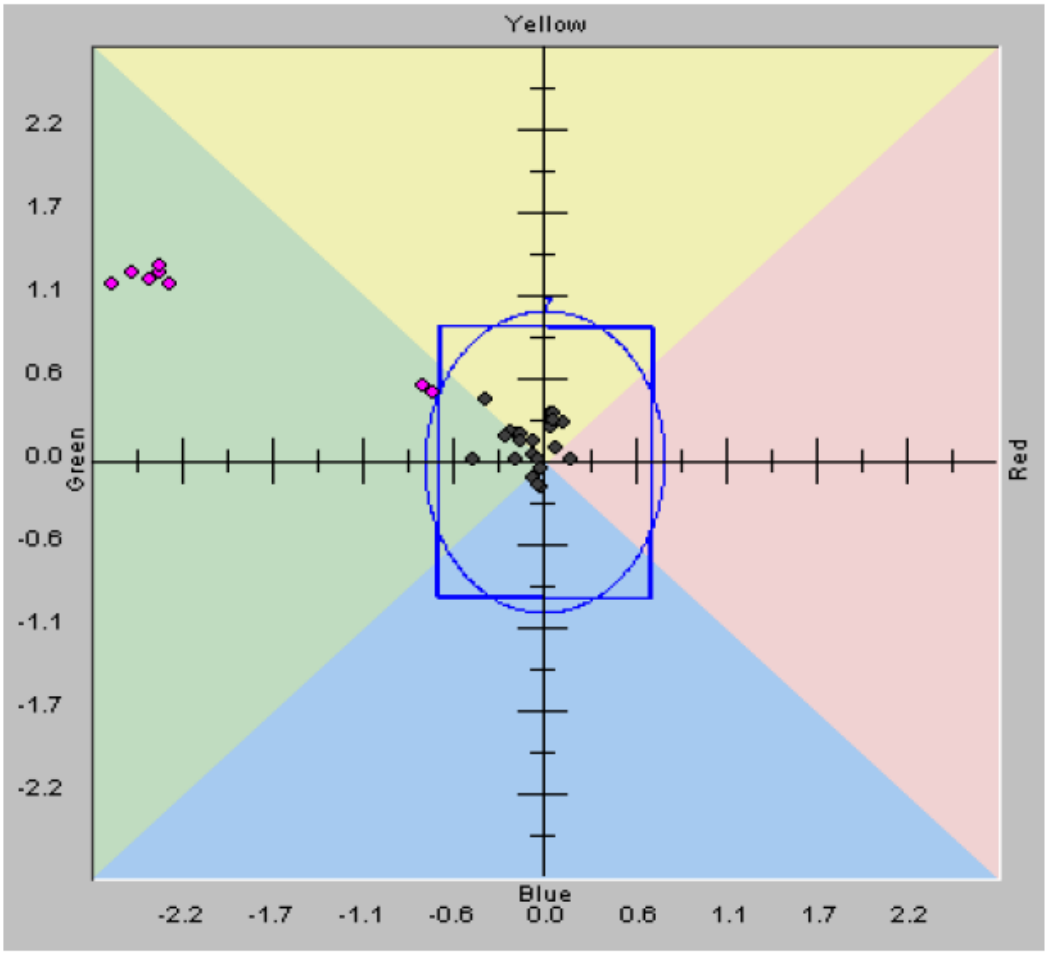
Color Change After Finishing Treatment - Polyester

--	L*	a*	b*	--
Standard	*26.61*	*-0.41*	*-0.11*	--
Sample	DL*	Da*	Db*	Decmc
SI-1	-1.20	-0.12	0.51	**1.13**
SI-1	-1.13	-0.08	0.50	**1.08**
SI-2	-1.07	0.05	0.35	**0.90**
SI-2	-1.04	0.07	0.26	**0.81**
HR-3	-0.21	0.20	-0.35	**0.62**
HR-3	-0.39	0.22	-0.25	**0.57**
HZ-4	1.50	-0.07	1.43	**2.38**
HZ-4	1.52	-0.10	1.42	**2.37**
HZ-5	-0.09	-0.07	0.62	**0.95**
HZ-5	-0.18	-0.06	0.59	**0.90**
FC-6	-1.80	0.04	0.19	**1.25**
FC-6	-1.82	0.10	0.16	**1.26**
FC-7	-1.08	0.12	0.24	**0.83**
FC-7	-1.34	0.21	0.15	**0.98**
FC-8	-1.07	0.21	-0.23	**0.87**
FC-8	-1.17	0.17	-0.14	**0.85**
FM-9	-1.83	-0.16	0.67	**1.61**
FM-9	-1.85	-0.31	0.75	**1.75**
FM-10	-0.83	-0.05	0.35	**0.77**
FM-10	-1.05	-0.04	0.31	**0.85**
FM-11	-0.46	-0.12	0.12	**0.41**
FM-11	-0.62	-0.09	0.14	**0.49**
FI-12	-1.06	0.14	0.10	**0.76**
FI-12	-1.24	0.12	0.12	**0.88**
FI-13	-1.35	0.21	0.17	**0.99**
FI-13	-1.34	0.19	0.17	**0.98**
FI-14	-1.16	0.25	0.17	**0.90**
FI-14	-1.24	0.20	0.18	**0.93**
FI-15	-0.99	0.21	-0.17	**0.78**
FI-15	-1.03	0.22	-0.09	**0.78**



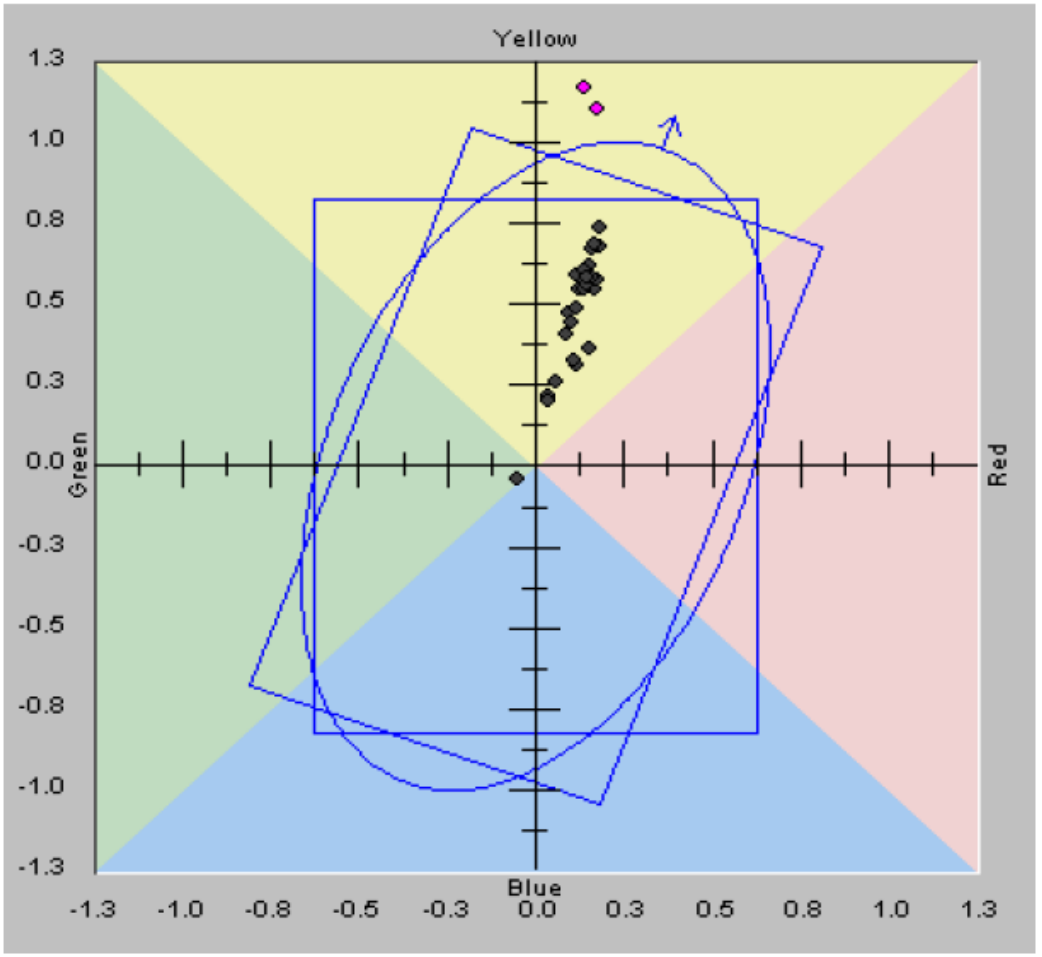
Color Change After Finishing Treatment - Nylon

--	L*	a*	b*	--
Standard	*57.83*	*0.09*	*6.17*	--
Sample	DL*	Da*	Db*	Decmc
SI-1	-0.47	-0.44	0.02	**0.63**
SI-1	-0.34	-0.03	-0.15	**0.21**
SI-2	-2.25	-0.03	-0.05	**0.96**
SI-2	-1.73	-0.02	-0.17	**0.76**
HR-3	-0.12	-0.17	0.01	**0.24**
HR-3	-0.27	-0.24	0.17	**0.38**
HZ-4	-0.13	-0.15	0.19	**0.28**
HZ-4	-0.01	-0.15	0.13	**0.25**
HZ-5	0.65	0.04	0.23	**0.36**
HZ-5	0.73	0.06	0.27	**0.42**
FC-6	0.56	0.04	0.26	**0.35**
FC-6	1.09	0.05	0.33	**0.57**
FC-7	2.25	0.15	0.01	**0.98**
FC-7	1.56	0.07	0.10	**0.68**
FC-8	0.94	0.12	0.27	**0.51**
FC-8	0.43	0.03	0.33	**0.38**
FM-9	2.08	-2.28	1.17	**3.34**
FM-9	1.86	-2.34	1.30	**3.43**
FM-10	1.11	-2.64	1.18	**3.70**
FM-10	1.05	-2.52	1.25	**3.57**
FM-11	0.41	-2.40	1.21	**3.39**
FM-11	0.87	-2.35	1.25	**3.35**
FI-12	-0.29	-0.75	0.51	**1.14**
FI-12	-0.19	-0.68	0.46	**1.03**
FI-13	1.05	-0.36	0.41	**0.78**
FI-13	1.71	-0.20	0.20	**0.80**
FI-14	0.32	-0.06	0.05	**0.17**
FI-14	0.49	-0.03	0.02	**0.21**
FI-15	-0.24	-0.16	0.18	**0.30**
FI-15	-0.77	-0.07	0.14	**0.37**



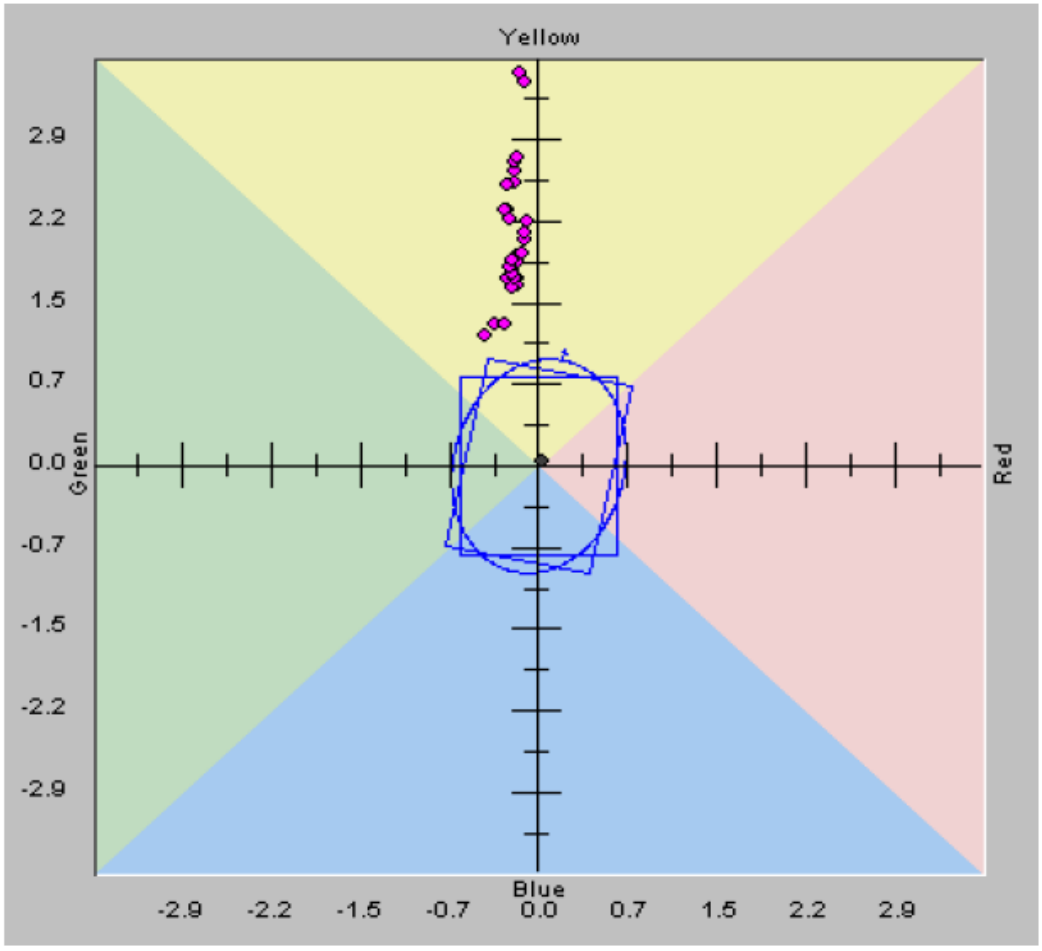
Color Change After Finishing Treatment - Automotive Acrylic

--	L*	a*	b*	--
Standard	*41.65*	*2.62*	*7.16*	--
Sample	DL*	Da*	Db*	Decmc
SI-1	-0.05	0.14	0.62	**0.60**
SI-1	-0.24	0.15	0.60	**0.59**
SI-2	-0.59	0.18	1.14	**1.14**
SI-2	-0.46	0.14	1.21	**1.20**
HR-3	-0.59	0.12	0.33	**0.44**
HR-3	-1.06	0.16	0.37	**0.66**
HZ-4	0.46	0.04	0.22	**0.32**
HZ-4	0.52	0.03	0.20	**0.33**
HZ-5	0.12	0.12	0.32	**0.33**
HZ-5	0.36	0.06	0.27	**0.32**
FC-6	0.38	0.17	0.61	**0.62**
FC-6	0.27	0.16	0.64	**0.63**
FC-7	0.44	0.12	0.61	**0.63**
FC-7	0.28	0.14	0.63	**0.62**
FC-8	0.12	0.15	0.57	**0.55**
FC-8	0.23	0.15	0.58	**0.57**
FM-9	-0.12	0.14	0.63	**0.61**
FM-9	-0.28	0.14	0.63	**0.62**
FM-10	-0.15	0.16	0.69	**0.67**
FM-10	-0.37	0.18	0.71	**0.71**
FM-11	0.65	0.90	0.42	**0.52**
FM-11	0.53	0.11	0.46	**0.52**
FI-12	0.68	0.10	0.49	**0.58**
FI-12	0.73	0.12	0.50	**0.61**
FI-13	0.59	0.13	0.57	**0.62**
FI-13	0.51	0.14	0.59	**0.63**
FI-14	-0.02	0.19	0.70	**0.68**
FI-14	0.05	0.19	0.76	**0.73**
FI-15	0.10	0.17	0.56	**0.55**
FI-15	-0.23	0.18	0.59	**0.59**



Color Change After Finishing Treatment - Home Furnishing Acrylic

--	L*	a*	b*	--
Standard	*81.54*	*1.09*	*5.36*	--
Sample	DL*	Da*	Db*	Decmc
SI-1	-1.50	-0.15	3.51	**3.75**
SI-1	-1.59	-0.11	3.44	**3.67**
SI-2	-2.03	-0.20	2.72	**2.98**
SI-2	-2.14	-0.18	2.74	**3.02**
HR-3	-2.22	-0.11	2.09	**2.36**
HR-3	-2.43	-0.09	2.19	**2.48**
HZ-4	-1.56	-0.22	1.77	**2.00**
HZ-4	-1.41	-0.22	1.83	**2.04**
HZ-5	-1.50	-0.21	1.74	**1.96**
HZ-5	-1.38	-0.21	1.71	**1.91**
FC-6	-0.37	-0.28	1.27	**1.43**
FC-6	-0.32	-0.27	1.27	**1.43**
FC-7	-0.29	-0.21	1.59	**1.73**
FC-7	-0.32	-0.20	1.67	**1.81**
FC-8	-0.36	-0.17	1.68	**1.81**
FC-8	-0.40	-0.18	1.62	**1.75**
FM-9	-1.38	-0.11	2.03	**2.21**
FM-9	-1.12	-0.14	1.91	**2.07**
FM-10	-1.71	-0.20	2.63	**2.86**
FM-10	-1.34	-0.25	2.51	**2.73**
FM-11	-1.15	-0.23	2.20	**2.40**
FM-11	-1.49	-0.19	2.52	**2.74**
FI-12	-0.68	-0.36	1.27	**1.50**
FI-12	-0.66	-0.43	1.17	**1.44**
FI-13	-0.27	-0.23	1.66	**1.80**
FI-13	-0.46	-0.25	1.66	**1.82**
FI-14	-0.59	-0.25	2.29	**2.47**
FI-14	-0.82	-0.27	2.29	**2.48**
FI-15	-0.73	-0.18	1.87	**2.02**
FI-15	-0.73	-0.18	1.81	**1.96**



Appendix F : Physical Testing Results

Cotton Fabric - Physical Testing

		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
Tear Strength	Peak Load (lbf) [Ave of 3]	6.42	5.52	5.66	6.34	6.03	5.74	4.65	4.55	5.08	4.63	4.75	5.48	5.26	5.15	5.11	4.90
	Load at Tear (lbf) [Ave of 3]	5.14	4.87	4.13	4.99	5.02	4.62	3.42	3.79	4.32	3.82	3.36	4.17	3.90	4.76	4.09	2.46
	Five Peaks*(lbf) - Sample 1	6.13	4.97	4.94	5.87	5.55	4.61	4.06	4.26	4.59	3.95	4.34	4.28	5.04	4.42	4.31	4.42
	Five Peaks (lbf) - Sample 2	5.98	5.32	4.91	4.99	5.78	4.97	3.95	3.73	4.39	4.23	4.13	4.58	4.51	4.68	4.48	4.06
	Five Peaks (lbf) - Sample 3	5.28	4.91	5.38	5.30	5.37	4.70	3.99	4.24	4.39	4.19	4.47	4.68	4.57	4.53	4.64	4.13
	Five Peak Ave (lbf)	**5.80**	**5.07**	**5.05**	**5.39**	**5.57**	**4.76**	**4.00**	**4.07**	**4.45**	**4.12**	**4.31**	**4.51**	**4.71**	**4.55**	**4.48**	**4.20**
	Std. Dev.	**0.45**	**0.22**	**0.26**	**0.45**	**0.21**	**0.19**	**0.06**	**0.30**	**0.11**	**0.15**	**0.17**	**0.21**	**0.29**	**0.13**	**0.16**	**0.19**
Stiffness	Sample 1 (lbs/sq/in.)	0.48	0.30	0.42	0.52	0.48	0.46	0.52	0.52	0.52	0.62	0.52	0.44	0.48	0.62	0.64	0.72
	Sample 2 (lbs/sq.in.)	0.40	0.28	0.50	0.50	0.50	0.46	0.64	0.44	0.54	0.50	0.46	0.42	0.62	0.62	0.56	0.58
	Average (lbs/sq/in.)	**0.44**	**0.29**	**0.46**	**0.51**	**0.48**	**0.46**	**0.58**	**0.48**	**0.53**	**0.56**	**0.49**	**0.43**	**0.55**	**0.62**	**0.60**	**0.65**
	Std. Dev.	**0.057**	**0.014**	**0.057**	**0.014**	**0.028**	**0.000**	**0.085**	**0.057**	**0.014**	**0.085**	**0.042**	**0.014**	**0.099**	**0.000**	**0.057**	**0.099**

Poly/Cotton Fabric - Physical Testing

		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
Tear Strength	Peak Load (lbf) [Ave of 3]	3.99	5.62	5.17	5.16	5.30	3.74	3.35	3.37	3.38	3.53	3.54	3.96	3.99	3.49	3.59	3.62
	Load at Tear (lbf) [Ave of 3]	2.49	5.03	3.96	3.77	4.00	2.88	2.54	2.80	2.68	2.61	3.29	3.15	2.70	2.17	3.23	1.92
	Five Peaks*(lbf) - Sample 1	3.71	5.10	4.71	4.54	5.15	3.29	3.05	3.18	3.27	3.14	2.88	3.48	3.38	2.99	3.11	2.93
	Five Peaks (lbf) - Sample 2	3.65	5.10	4.43	4.74	4.48	3.39	3.22	2.87	3.39	3.07	3.10	3.58	3.40	3.00	2.86	2.75
	Five Peaks (lbf) - Sample 3	3.63	4.79	4.68	4.54	4.96	3.24	304.00	3.05	3.03	3.05	3.11	3.52	3.59	3.48	3.13	3.11
	Five Peak Ave (lbf)	**3.66**	**5.00**	**4.60**	**4.61**	**4.86**	**3.30**	**3.10**	**3.03**	**3.23**	**3.09**	**3.03**	**3.53**	**3.46**	**3.15**	**3.03**	**2.93**
	Std. Dev.	**0.04**	**0.18**	**0.15**	**0.11**	**0.34**	**0.07**	**0.10**	**0.15**	**0.18**	**0.04**	**0.13**	**0.05**	**0.11**	**0.28**	**0.15**	**0.18**
Stiffness	Sample 1 (lbs/sq/in.)	0.62	0.20	0.62	0.56	0.40	0.38	0.44	0.42	0.46	0.60	0.48	0.46	0.44	0.56	0.36	0.44
	Sample 2 (lbs/sq.in.)	0.54	0.22	0.68	0.42	0.38	0.42	0.52	0.42	0.54	0.72	0.44	0.34	0.50	0.44	0.40	0.48
	Average (lbs/sq/in.)	**0.58**	**0.21**	**0.65**	**0.49**	**0.39**	**0.40**	**0.48**	**0.42**	**0.50**	**0.66**	**0.46**	**0.40**	**0.47**	**0.50**	**0.38**	**0.46**
	Std. Dev.	**0.057**	**0.014**	**0.042**	**0.099**	**0.014**	**0.028**	**0.057**	**0.000**	**0.057**	**0.085**	**0.028**	**0.085**	**0.042**	**0.085**	**0.028**	**0.028**

Polyester Fabric - Physical Testing

		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
Tear Strength	Peak Load (lbf) [Ave of 3]	32.95	48.33	46.74	56.65	51.77	30.05	26.92	28.72	29.75	23.95	30.32	29.45	30.71	24.15	28.24	21.24
	Load at Tear (lbf) [Ave of 3]	32.95	48.33	43.19	54.64	33.10	30.05	26.85	28.72	29.75	23.92	30.32	29.24	30.26	22.54	28.24	16.78
	Five Peaks*(lbf) - Sample 1	26.86	45.99	41.54	53.01	44.07	27.85	22.99	25.41	26.62	21.73	26.25	26.19	29.79	23.66	24.20	19.10
	Five Peaks (lbf) - Sample 2	27.69	44.00	43.75	51.01	51.61	25.80	24.00	25.68	26.84	22.55	27.17	27.95	29.38	22.44	24.06	19.66
	Five Peaks (lbf) - Sample 3	26.54	40.26	46.11	55.30	43.86	27.85	24.00	25.37	25.75	21.71	26.53	26.46	28.22	22.64	25.94	18.19
	Five Peak Ave (lbf)	**27.03**	**43.42**	**43.80**	**53.11**	**46.51**	**27.17**	**23.66**	**25.48**	**26.41**	**22.00**	**26.65**	**26.87**	**29.13**	**22.91**	**24.73**	**21.24**
	Std. Dev.	**0.60**	**2.91**	**2.29**	**2.15**	**4.41**	**1.18**	**0.59**	**0.17**	**0.57**	**0.48**	**0.47**	**0.95**	**0.81**	**0.65**	**1.05**	**4.04**
Stiffness	Sample 1 (lbs/sq/in.)	3.96	3.78	3.26	2.16	1.94	2.08	2.14	2.20	4.12	5.24	3.84	2.28	1.70	2.30	1.86	2.16
	Sample 2 (lbs/sq.in.)	3.98	4.14	3.06	2.00	1.90	1.74	1.82	1.82	4.12	4.66	3.98	2.16	1.86	2.20	2.32	1.44
	Average (lbs/sq/in.)	**3.97**	**3.96**	**3.16**	**2.08**	**1.92**	**1.91**	**1.98**	**2.01**	**4.12**	**4.95**	**3.91**	**2.22**	**1.78**	**2.25**	**2.09**	**1.80**
	Std. Dev.	**0.014**	**0.255**	**0.141**	**0.113**	**0.028**	**0.240**	**0.226**	**0.269**	**0.000**	**0.410**	**0.099**	**0.085**	**0.113**	**0.071**	**0.325**	**0.509**

Nylon Fabric - Physical Testing

		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
Tear Strength	Peak Load (lbf) [Ave of 3]	3.46	15.11	6.56	6.07	5.25	4.31	3.77	4.07	3.90	4.16	3.87	4.32	4.16	4.04	4.61	3.63
	Load at Tear (lbf) [Ave of 3]	3.44	15.11	5.72	5.99	5.25	3.91	3.77	4.07	3.90	3.84	3.51	4.30	3.77	3.79	4.46	3.63
	Five Peaks*(lbf) - Sample 1	2.80	12.98	5.87	5.06	4.86	4.16	--	3.07	2.68	3.84	3.68	3.65	3.94	2.93	2.98	2.63
	Five Peaks (lbf) - Sample 2	2.68	13.64	6.10	5.37	4.60	3.98	3.31	3.11	2.95	3.79	2.72	3.34	3.97	3.46	3.02	2.65
	Five Peaks (lbf) - Sample 3	3.16	13.04	6.21	5.47	4.54	3.78	2.82	2.94	3.29	3.62	3.32	3.65	3.95	3.00	3.04	2.61
	Five Peak Ave (lbf)	**2.88**	**13.22**	**6.06**	**5.30**	**4.66**	**3.98**	**3.07**	**3.04**	**2.97**	**3.75**	**3.24**	**3.55**	**3.95**	**3.13**	**3.01**	**2.63**
	Std. Dev.	**0.25**	**0.36**	**0.17**	**0.22**	**0.17**	**0.19**	**0.35**	**0.09**	**0.30**	**0.12**	**0.49**	**0.18**	**0.02**	**0.29**	**0.03**	**0.02**
Stiffness	Sample 1 (lbs/sq/in.)	0.14	0.06	0.16	0.12	0.08	0.06	0.06	0.08	0.06	0.06	0.06	0.06	0.06	0.06	0.06	0.08
	Sample 2 (lbs/sq.in.)	0.14	0.06	0.26	0.14	0.06	0.06	0.06	0.08	0.08	0.08	0.08	0.06	0.06	0.06	0.08	0.06
	Average (lbs/sq/in.)	**0.14**	**0.06**	**0.21**	**0.13**	**0.07**	**0.06**	**0.06**	**0.08**	**0.07**	**0.07**	**0.07**	**0.06**	**0.06**	**0.06**	**0.07**	**0.07**
	Std. Dev.	**0.000**	**0.000**	**0.071**	**0.014**	**0.014**	**0.000**	**0.000**	**0.000**	**0.014**	**0.014**	**0.014**	**0.000**	**0.000**	**0.000**	**0.014**	**0.014**

*Five Peaks = Average value of the five highest peaks during tear propagation from 30mm to 110mm

Automotive Acrylic Fabric - Physical Testing																
		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
Tear Strength	Peak Load (lbf) [Ave of 3]	8.21	10.79	14.77	15.89	16.58	11.67	10.67	10.91	12.66	10.78	4.75	10.50	11.33	9.48	9.73	6.26
	Load at Tear (lbf) [Ave of 3]	7.33	10.79	14.24	12.53	14.15	11.67	10.39	10.49	12.21	10.17	3.36	7.90	7.48	8.92	8.03	3.42
	Five Peaks*(lbf) - Sample 1	7.74	7.91	12.89	16.12	14.52	9.77	9.21	9.61	9.53	9.42	4.34	10.33	10.15	8.74	8.87	6.07
	Five Peaks (lbf) - Sample 2	7.91	8.34	13.13	13.90	16.46	9.88	9.59	9.42	9.37	9.04	4.13	10.16	10.55	8.94	9.17	5.81
	Five Peaks (lbf) - Sample 3	7.65	8.20	13.29	14.22	13.68	9.71	8.96	9.61	10.44	9.66	4.47	9.79	9.71	8.51	8.69	6.18
	Five Peak Ave (lbf)	**7.77**	**8.15**	**13.10**	**14.74**	**14.89**	**9.79**	**9.25**	**9.55**	**9.78**	**9.37**	**4.31**	**10.09**	**10.14**	**8.73**	**8.91**	**6.02**
	Std. Dev.	**0.13**	**0.22**	**0.20**	**1.20**	**1.43**	**0.09**	**0.32**	**0.11**	**0.58**	**0.31**	**0.17**	**0.27**	**0.42**	**0.21**	**0.24**	**0.19**
Stiffness	Sample 1 (lbs/sq/in.)	2.80	1.98	3.70	2.58	1.54	1.16	1.74	1.82	2.84	3.64	2.58	2.10	2.16	1.92	1.70	1.76
	Sample 2 (lbs/sq.in.)	2.92	2.10	3.56	2.34	1.82	1.50	1.56	1.28	3.36	3.46	3.16	1.74	1.90	1.88	1.76	1.64
	Average (lbs/sq/in.)	**2.86**	**2.04**	**3.63**	**2.46**	**1.68**	**1.33**	**1.65**	**1.55**	**3.10**	**3.55**	**2.87**	**1.92**	**2.03**	**1.90**	**1.73**	**1.70**
	Std. Dev.	**0.085**	**0.085**	**0.099**	**0.170**	**0.198**	**0.240**	**0.127**	**0.382**	**0.368**	**0.127**	**0.410**	**0.255**	**0.184**	**0.028**	**0.042**	**0.085**

Home Furnishing Acrylic Fabric - Physical Testing																	
		SI-1	SI-2	HR-3	HZ-4	HZ-5	FC-6	FC-7	FC-8	FM-9	FM-10	FM-11	FI-12	FI-13	FI-14	FI-15	UNTR
Tear Strength	Peak Load (lbf) [Ave of 3]	6.74	11.43	17.71	19.60	18.09	11.91	10.58	9.81	12.46	11.23	10.66	12.71	10.84	10.73	10.02	5.70
	Load at Tear (lbf) [Ave of 3]	5.07	11.14	16.16	17.39	13.56	11.17	10.10	8.85	11.92	8.37	3.34	12.71	8.66	7.60	9.17	2.85
	Five Peaks*(lbf) - Sample 1	6.35	9.35	17.56	18.98	15.11	10.06	9.20	9.03	11.54	9.64	9.60	11.03	10.65	10.15	8.95	5.17
	Five Peaks (lbf) - Sample 2	6.81	10.51	14.50	16.03	17.39	10.36	9.42	9.15	11.09	10.56	10.08	10.12	10.63	9.34	9.19	5.59
	Five Peaks (lbf) - Sample 3	6.59	10.51	16.16	17.28	15.77	10.24	9.46	8.40	11.33	10.08	9.61	10.61	10.12	9.61	--	5.42
	Five Peak Ave (lbf)	**6.58**	**10.12**	**16.07**	**17.43**	**16.09**	**10.22**	**9.36**	**8.86**	**11.32**	**10.09**	**9.76**	**10.59**	**10.47**	**9.70**	**9.07**	**5.39**
	Std. Dev.	**0.23**	**0.67**	**1.53**	**1.48**	**1.18**	**0.15**	**0.14**	**0.40**	**0.23**	**0.46**	**0.27**	**0.46**	**0.30**	**0.41**	**0.17**	**0.21**
Stiffness	Sample 1 (lbs/sq/in.)	3.08	2.28	3.04	3.66	1.68	1.62	1.92	2.66	4.42	7.76	5.16	2.28	2.28	2.60	1.32	2.02
	Sample 2 (lbs/sq.in.)	2.90	3.26	3.06	2.42	2.04	1.66	2.12	2.02	6.50	9.18	8.30	2.64	3.20	1.86	1.70	1.94
	Average (lbs/sq/in.)	**2.99**	**2.77**	**3.05**	**3.04**	**1.86**	**1.64**	**2.02**	**2.34**	**5.46**	**8.47**	**6.73**	**2.46**	**2.74**	**2.23**	**1.51**	**1.98**
	Std. Dev.	**0.127**	**0.693**	**0.014**	**0.877**	**0.255**	**0.028**	**0.141**	**0.453**	**1.471**	**1.004**	**2.220**	**0.255**	**0.651**	**0.523**	**0.269**	**0.057**

*Five Peaks = Average value of the five highest peaks during tear propagation from 30mm to 110mm