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Durability of Water-Repellent Finishes to Accelerated Laundering

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DURABILITY OF WATER-REPELLENT FINISHES

TO ACCELERATED LAUNDERING

BY

JESSICA ROSE BROOKS

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE

REQUIREMENTS FOR THE DEGREE OF

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OF

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ABSTRACT

Water repellency is a common requirement for textiles. Most outdoor garments, tents, and bags require a durable water-repellent finish (DWR). Some specifications often require these products to pass multiple home laundering tests, which can be expensive in terms of time and resources. The present study sought to determine a combination of factors in an accelerated laundering test of a DWR fabric using a Launder-ometer that correlated with the water and oil repellency ratings of fabric tested in a top-loader washing machine. Testing rounds were created based on AATCC Test Method 61: Colorfastness to Accelerated Laundering. Samples were washed and dried twenty cycles and their repellency tested according to AATCC Test Method 22 and AATCC Test Method 118. When the results did not correlate with the top-loading results after a round of tests one of the following conditions were adjusted: amount of liquor (detergent), quantity of steel balls, and temperature. To determine the effect of each alteration, the laundry cycles and tests were run again after the change. Several preliminary studies were performed to determine the evenness of color loss, the evenness of the water-repellent finish, and if residual detergent was affecting the water and oil repellency ratings.

Results showed that changing the conditions in the Launder-Ometer affects the durability of water-repellent finishes on 70 denier nylon fabric. None of these condition combinations produced both water and oil repellency test results consistent with home laundering in top-loader washing machines. Further research is needed using different fabrics and DWRs to determine if finish durability testing with a Launder-Ometer is capable of reliably correlating with results from home launderings.

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I would also like to thank my committee member, Dr. Sze Yang for his time and involvement to this project.

DEDICATION

I dedicate this project to my family and especially my husband Chad whose support and love has helped me become the person I am today.

TABLE OF CONTENTS

ABSTRACT	ii
ACKNOWLEDGEMENTS	iii
DEDICATION	iv
TABLE OF CONTENTS	v
LIST OF TABLES.....	vi
INTRODUCTION	1
PRELIMINARY STUDIES	5
MAIN STUDY.....	20
Textile Samples	20
Laundering Conditions: Top-Loader Washing Machine	20
Laundering Conditions: Accelerated Laundering in Launder-Ometer	22
Round 1.....	23
Round 2.....	24
Round 3.....	25
Aqueous Liquid Test.....	26
Round 4.....	27
Rinse Comparison.....	28
Round 4A.....	29
Round 4B.....	30
DISCUSSION & CONCLUSION	31
BIBLIOGRAPHY.....	33

LIST OF TABLES

TABLE	PAGE
Table 1. Cost of Energy Comparison.....	4
Table 2. Lot Finishing Parameters.....	6
Table 3. Laundry Conditions Used in Preliminary Study.....	7
Table 4. Spectrophotometer Evaluation of Colorfastness.....	11
Table 5. Top-Loader Washing Machine Results for S16718 & S16719.....	13
Table 6. Round A Wash Conditions in Launder-Ometer & Dryer.....	15
Table 7. Round A Results in Launder-Ometer.....	15
Table 8. Round B Wash Conditions in Launder-Ometer & Dryer.....	16
Table 9. Round B Results in Launder-Ometer.....	17
Table 10. Round C Wash Conditions in Launder-Ometer & Dryer.....	17
Table 11. Round C Results in Launder-Ometer.....	18
Table 12. Round D Wash Conditions in Launder-Ometer & Dryer.....	18
Table 13. Round D Results in Launder-Ometer.....	19
Table 14. Top-Loader Washing Machine Results for Lot S16063.....	22
Table 15. Round 1 Wash Conditions in Launder-Ometer & Dryer.....	23
Table 16. Round 1 Results in Launder-Ometer.....	24
Table 17. Round 2 Wash Conditions in Launder-Ometer & Dryer.....	24
Table 18. Round 2 Results in Launder-Ometer.....	25
Table 19. Round 3 Wash Conditions in Launder-Ometer & Dryer.....	26
Table 20. Round 3 Results in Launder-Ometer.....	26
Table 21. Water/Isopropyl Alcohol Ratio Water Droplet Test.....	27

Table 22. Round 4 Wash Conditions in Launder-Ometer & Dryer.....	27
Table 23. Round 4 Results in Launder-Ometer.....	28
Table 24. Rinse Comparison Results.....	29
Table 25. Round 4A Results in Launder-Ometer.....	29
Table 26. Round 4B Results in Launder-Ometer.....	30

INTRODUCTION

Water-repellent finishes on textiles typically should be durable to laundering. Testing for such durability requires multiple laundering cycles (wash/dry), which are expensive in terms of time and resources. Colorfastness to laundering is routinely tested with an accelerated method in which the color loss from 5 home launderings is approximated in a single small-scale 45-minute test. However, no parallel methods have been developed to test other parameters such as the durability of water-repellent finishes to laundering. This study sought to determine a combination of factors in an accelerated test that best approximates the durability of water-repellent finishes to multiple home launderings.

Specifications for some products require textiles treated with a water-repellant finish to maintain the repellency through one hundred launderings. Running tests to meet such specifications is time, labor, and energy intensive in addition to the large amount of water required in a top-loading washing machine. A Launder-Ometer is an accelerated laundering machine in which fabric samples are placed with steel balls and detergent in rotating closed canisters subjected to a temperature-controlled water bath for a specific time. These factors produce the color loss that occurs in home laundering at an accelerated rate.

An accelerated method to test water-resistant finishes would save industry both time and resources, especially during the product development stage. An example of such an application is the Army Physical Fitness Uniform, specification GL/PD 13-04. This requires running both water and oil repellency tests initially and after one hundred laundering cycles (Military Specification, 2013). A manufacturer developing

a product to meet GL/PD 13-04 can face countless product development lots that require the full one hundred launderings. This testing necessitates a considerable amount of time, as one wash and dry cycle can take approximately an hour.

A parallel situation occurs in the testing of color fastness to laundering, in which the color loss in five launderings needs to be assessed. Individual colors would need to be laundered separately five times, making the development of apparel products complicated and costly.

An AATCC test method to evaluate colorfastness using the Launder-Ometer was introduced in 1928 (AATCC Research Committee, 1928). Hugh Christison and William Appel, both members of the AATCC Committee on Research, developed the laundrometer in the 1920s (Clark, 2001). The prototype featured a large tub of water that rotated twenty glass jars in a bath of water held at a constant temperature. Like today's machine, samples were put into the jars with a specified amount of water, detergent, and balls to create consistent agitation and abrasion. In 1945, stainless steel balls replaced rubber balls (AATCC Research Committee, 1945).

In 1950, the first version of AATCC Test Method 61 using the Launder-Ometer to test colorfastness was published. "The tentative test method was made permanent in 1954, and in 1956, methods previously used for wool, silk, and manufactured fibers were discontinued" (Clark, 1996). As the method was further developed and revised, additional standardization designated the type of detergent and adding a multifiber strip to determine staining. These developments led to the method that is currently in place. AATCC Test Method 61: Colorfastness to Laundering:

Accelerated provides six tests with explanations on how each test relates to home laundering.

However, this test method was only established to predict color loss in laundering; the standard makes no claim of correlation between the Launder-Ometer and real laundering conditions for any other textile property, and, as far as can be determined, none has been researched. Instances when such a correlation is assumed are common, however, and typically are found in laboratory research. An example is the use of the Launder-Ometer to test the durability of a textile finished with a silver based antimicrobial (Geranio, et al, 2014). The washing experiments in these tests were based on the international standard ISO 105-C06: 1994 (procedure A1S) for colorfastness to domestic and commercial laundering, an accelerated method similar to that of the AATCC Test Method 61 (Lorenz, et al, 2012).

If a similar accelerated test were shown to approximate the effects of five launderings on the loss of a finish, it would allow 100-laundering durability testing in approximately 20 hours compared to over one hundred hours in a top-loader washer and dryer.

Additionally washing machines use resources such as water and energy. A top-loader machine can require twenty-three gallons of water per cycle, which after one hundred launderings, can equal approximately 2,300 gallons of water for one test lot (“Clothes Washers,” 2014) A Launder-Ometer uses a maximum of 200 mL of a combination of detergent and water per canister (AATCC Technical Manual, 2013). If used in place of a top-loader washing machine for one hundred washes, it would save approximately 2,295 gallons of water per test lot.

Multiple samples can be washed at one time in a Launder-Ometer, as each canister is sealed. In a top-loader washer, the respective lots must be washed separately to avoid any contaminants from one lot to the next. Being able to wash lots simultaneously is more efficient in terms of time and energy.

The cost of the energy required when running one hundred launderings is considerable, as shown in Table 1.

Table 1 – Cost of Energy Comparison

Machine	Wattage	Electricity Cost	Cost Per Hour	Cost for 100 Launderings	Combined Washing & Drying Cost
Launder-Ometer	11,500	\$0.10/kWh	\$1.15	\$17.25	\$34.25
Dryer	3,360		\$0.34	\$17.00	
Top-Loader Washing Machine	1200	\$0.10/kWh	\$0.12	\$6.00	\$38.00
Hot Water Heater	4,500		\$0.45	\$15.00	
Dryer	3,360		\$0.34	\$17.00	

Assuming the average cost of electricity is \$0.10 per kWh, testing one lot of fabric in the Launder-Ometer would save approximately \$3.75 per hour in energy alone. This does not take into account the cost of water, detergent, waste water treatment and labor. A test method using the Launder-Ometer would save industry time, labor, energy, and water.

PRELIMINARY STUDIES

Multiple preliminary studies were performed prior to the main study. The first was a preliminary study hereinafter referred to as the S16063 top-loader washing machine study where the researcher washed samples from lot S16063 one hundred times. After every ten launderings, three samples were tested and rated for water and oil-repellency. These results help establish a database in which the researcher compared the main study results from the Launder-Ometer to.

The second preliminary study was run to ensure an 8 x 8 in sample could be washed evenly in the Launder-Ometer canisters. AATCC Test Method 61 Test 2A requires a sample size of 2 x 4 in. However, an 8 x 8 in. sample was necessary to perform AATCC Test Method 22 and AATCC Test Method 118. The researcher then read the color on each of the samples using a spectrophotometer. These results helped determine if the finish on the larger sample size would reduce evenly when tested in the Launder-Ometer.

The third preliminary consisted of two lots of 70 denier nylon fabric similar to lot S16063 which was washed in the first preliminary study, except the two lots were finished with a C-6 durable water-resistant chemical. This preliminary study was set up exactly like the main study, with the top-loader washing machine water and oil repellency rating results serving as a database for the researcher to compare the Launder-Ometer results. The researcher used this third preliminary study as a guide to creating the main study testing rounds.

In preliminary study S16063 top-loader washing machine study, this researcher used AATCC Test Method 135 to establish a database of water and oil repellency

results through one hundred launderings in a top-loader washing machine. Four lots of a 70 denier nylon dyed black and finished with different concentrations of a durable C-8 fluorochemical oil- and water-repellent finish, DWR, were used in this study. A detailed description of each lot is listed in Table 2. A blocked isocyanate was added to the finish to increase its durability. After the finishes were applied, the lots were dried on a gas frame at specific temperatures and speeds that are listed in Table 2. Lots S15985, S16054 and S16077 each have two temperatures at which they were dried as there were two different ovens along the frame.

Table 2 – Lot Finishing Parameters

Lots	Finishing Process Description				Comment
	C-8 DWR	Extender (%)	Temperature (°F)	Speed (Yards Per Minute)	
S15985	12	4	350 & 360	27	Finished on same machine as S16054
S16054	10	2.5	350 & 360	30	Finished on a different machine than S16077
S16063	9	2.5	220	30	Finished on same machine as S16077
S16077	10	2.5	350 & 360	30	Finished on a different machine than S16054

Each lot of finished fabric was divided into two sets of ten quarter-yard cuts. One quarter-yard cut allowed for three water repellent testing replications and three oil repellent testing replications. The 80 samples were laundered in a top-loading Kenmore model 20442 washing machine with 66 grams of AATCC Standard Reference detergent. This choice of detergent is required by AATCC Test Method 135. Samples were tested for water and oil repellency after every ten launderings.

AATCC Test Method 135: Dimensional Changes of Fabrics after Home Laundering specified permanent press wash cycle at 105 +/- 5°F, a large size wash load, and permanent press tumble dry at 155 +/- 10°F¹. The wash load consisted of eighteen gallons of water, the samples, and enough ballast to equal a 4-pound load. The standard offers a range of ten degrees for the drying temperature. To determine how the samples performed using both the middle and lower dryer temperatures of the specification, they were all washed at the same temperature, but one set was dried at the 145°F, and the other at 155°F. The dryer was equipped with a digital temperature to maintain a set temperature for the duration of the cycle. The laundry conditions for the samples are in Table 3.

Table 3 – Laundry Conditions Used In Preliminary Study

Lots	Home Laundering		
	Wash	Dry	
S15985	Permanent Press 105 +/- 5°F	Tumble Dry Permanent Press 145°F	Tumble Dry Permanent Press 155°F
S16054			
S16063			
S16077			

¹ AATCC publishes a number of different test methods involving laundering; many contain the same wash temperature and cycles as AATCC Test Method 135. AATCC Test Method 135 also features different types of washes other than the one specified in the preliminary study. In AATCC Test Method 124: Smoothness Appearance of Fabrics after Repeated Home Laundering, AATCC Test Method 150: Dimensional Changes of Garments after Home Laundering, and AATCC Test Method 179: Skewness Change in Fabric and Garment Twist Resulting from Automatic Home Laundering, samples are subjected to the same wash temperatures and cycles as AATCC Test Method 135. Because of the various methods, AATCC created Monograph 6 to establish a consistent set of test conditions for all test methods involving home laundering. (AATCC Technical Manual)

The researcher performed three AATCC Test Method 22: Water Repellency: Spray Test replications and three AATCC Test Method 118: Oil Repellency: Hydrocarbon Resistance Test replications after each cycle in the top-loading washing machine. The researcher and two observers evaluated the resistance of the samples. The data revealed the effect of laundering on durability of the lots, which is further explained in the main study.

AATCC Test Method 22 requires the sample (8 x 8 in.) to be mounted in a 6.0 in diameter hoop onto the AATCC Spray Tester. Prior to testing, calibration is performed by pouring 250 mL of distilled water at 80 +/- 2°F into the funnel of the tester, and the time it takes for the water to empty is measured. The spray time is required to be 25-30 seconds. If the spray is out of calibration, the nozzle is blown out with air or soaked in vinegar to remove any mineral build-up. Once calibration was completed, each sample was tested. After the water spray has completed, the hoop was tapped once against a solid object, rotated 180° and tapped again. Immediately after tapping, the samples were compared to the AATCC Test Method 22 rating chart and rated visually. A rating of 100 is defined as no sticking or wetting of the specimen face. A 95 is given if the sample has slight sticking of water droplets but no wetting of the specimen face. A 90 rating is defined a slight random sticking or wetting of the specimen face. An 80 rating is defined as wetting of specimen face at spray points. A 70 rating is partial wetting of the specimen face beyond the spray points. A 50 rating is complete wetting of the entire specimen face beyond the spray points. And the lowest rating is a 0, defined as complete wetting of the entire face of the specimen.

In AATCC Test Method 118, three 8 x 8 in. specimens were conditioned at 65 +/- 2% relative humidity and 70 +/- 2°F for four hours prior to testing. The samples were placed onto blotting paper laying on a smooth, horizontal surface. Beginning with the lowest test oil (AATCC Oil No. 1), five small drops (approximately 0.187 in. in diameter, or 0.05 mL in volume) were placed onto the sample along a line parallel to the filling yarns. Oils were rated from No. 1 to No. 6, with Oil No. 1 having the lowest surface tension, and Oil No. 6 having the highest. Drops were approximately 1.5 inches apart. Drops were observed for 30 +/- 2 seconds at a 45° angle. If no penetration or wetting was observed, the next oil was tested until the researcher reached Oil No. 6. A panel of the researcher and one additional respondent evaluated the specimens. The drops were rated using the AATCC Test Method 118 grading criteria. Rating A (clear well-rounded drop) and Rating B (rounding drop with partial darkening) were considered passing, and Rating C (wicking apparent and/or complete wetting) and Rating D (complete wetting) were considered failing. The researcher then continued to test the next highest oil until failure occurred. The number of the highest passing oil was then recorded.

A second preliminary study was run to determine if an 8 x 8 in sample could be washed evenly in the Launder-Ometer canisters. AATCC Test Method 61, Test 2A requires a sample size of 2 x 4 in. However, an 8 x 8 in. sample was necessary to perform AATCC Test Method 22 and AATCC Test Method 118 so four 8 x 8 in. samples and four 2 x 4 in. samples were run in the canisters. The samples were 70 denier nylon, no finish, and dyed channel blue. This particular sample lot was chosen as it is a similar substrate as the lot being used for the main study, and previous tests

established that the channel blue dye was not colorfast to laundering. It was assumed that even loss of color would indicate to even treatment within the Launder-Ometer. Color bleeding was a necessary trait to see if the color loss was even across the sample after being washed in the Launder-Ometer.

The researcher washed the samples in the Launder-Ometer with the same conditions as AATCC Test Method 61, test 2A. One set, consisting of one 8 x 8 in and one 2 x 4 in. sample, was washed one cycle with one multifiber strip attached to each. The second set was washed without multifiber strips for one cycle. The third set was washed without multifiber strips for two cycles. The fourth set was washed without multifiber strips for three cycles. The multifiber strip was used as it is a required element to read stain for AATCC Test Method 61. However, the researcher performed the test without the multifiber as well, as the main study looking at water-repellent finishes would not be using multifiber strips attached to the 8 x 8 in. samples. The researcher performed testing using samples with and without a multifiber strip to ensure there were no obvious differences between using one, and not using a multifiber strip. While the Launder-Ometer reached a temperature of 120 +/- 4°F, samples were placed in individual 1200 mL, 3.5 x 8 in. canisters. Each canister had fifty steel balls and 150 mL of liquor (0.15% detergent) heated to 120 +/- 4°F. The detergent used was AATCC 1993 Standard Reference Detergent (without optical brighteners). Once locked, the canisters were placed into the Launder-Ometer and agitated for 45 minutes. After the washing cycle, the samples were air dried. The samples were each inspected visually for evenness of the color loss, and the color change read with a spectrophotometer using CIELAB. The change in color (DE) is

reported with the unwashed sample set as the standard. The following were also noted: DL is the difference in lightness or darkness, Da is the difference in red or green, and Db is the difference in yellow or blue. Results are listed in Table 4.

Table 4 – Spectrophotometer Evaluation of Colorfastness

Sample No.	# of Cycles	Sample Size	Multifiber Strip Used	Size of Can	DE	DL	Da	Db
1	Initial	N/A	N/A	N/A	N/A	24.39	2.98	-24.68
2	1	2x4 in.	YES	500 mL	0.42	0.34	-0.07	-0.24
3	1	2x4 in.	NO	500 mL	0.37	0.28	-0.13	-0.19
4	2	2x4 in.	NO	500 mL	0.7	0.52	-0.02	-0.48
5	3	2x4 in.	NO	500 mL	0.97	0.76	-0.01	-0.61
6	1	2x4 in.	YES	1200 mL	0.45	0.38	-0.1	-0.21
7	1	2x4 in.	NO	1200 mL	0.51	0.45	-0.11	-0.21
8	2	2x4 in.	NO	1200 mL	0.82	0.66	-0.05	-0.49
9	3	2x4 in.	NO	1200 mL	0.99	0.88	-0.05	-0.46
10	1	8x8 in.	YES	1200 mL	0.23	0.17	-0.02	-0.15
11	1	8x8 in.	NO	1200 mL	0.48	0.37	-0.12	-0.28
12	2	8x8 in.	NO	1200 mL	0.54	0.4	-0.08	-0.35
13	3	8x8 in.	NO	1200 mL	0.72	0.47	-0.04	-0.54

This preliminary study showed that the color change of the 2 x 4 in sample was very similar to the 8 x 8 in sample. The researcher assumed from this that washing the larger sample in the Launder-Ometer would allow the finish to reduce evenly.

Before the researcher could begin the main study, a third preliminary study was required by the researcher's company with two different lots of fabric. Lots S16718 and S16719 are the same 70 denier nylon fabric, but were finished with a C-6 durable water-resistant chemical, as opposed to the previous C-8 finish. The researcher washed S16718 and S16719 with the same conditions as the lots established for the main study; permanent press wash cycle at 105 +/- 5°F, a large size wash load, and permanent press tumble dry at 155 +/- 10°F. The wash load consisted of eighteen gallons of water, the samples, and enough ballast to equal a 4-pound load. The researcher performed AATCC Test Method 22 and AATCC Test Method 118 after every five laundering cycles. See Table 5 for water and oil resistance ratings of the two fabrics laundered in a top-loader washing machine.

Table 5 – Top-Loader Washing Machine Results for S16718 & S16719

# of Launderings	S16718						S16719					
	Water AATCC 22			Oil AATCC 118			Water AATCC 22			Oil AATCC 118		
Initial	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
5	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
10	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
15	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
20	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
25	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
30	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
35	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
40	100	100	95	#6	#6	#6	95	100	95	#6	#6	#6
45	95	95	95	#6	#6	#6	95	100	100	#6	#6	#6
50	95	95	95	#6	#6	#6	95	95	95	#6	#6	#6
55	90	90	90	#6	#6	#6	90	90	90	#6	#6	#6
60	90	90	90	#6	#6	#6	90	90	90	#5	#5	#5
65	90	90	90	#6	#6	#6	90	90	90	#5	#5	#5
70	90	90	90	#6	#6	#6	90	90	90	#5	#5	#5
75	80	80	80	#5	#5	#5	85	80	80	#5	#5	#5
80	80	80	80	#5	#5	#5	80	80	80	#5	#5	#5
85	80	80	80	#5	#5	#5	80	80	80	#5	#5	#5
90	70	70	70	#5	#5	#5	70	70	70	#5	#5	#5
95	70	70	70	#5	#5	#5	70	70	70	#5	#5	#5
100	70	70	70	#5	#5	#5	70	70	70	#5	#5	#5

Both lots performed similarly, starting out with spray ratings of 100 (no wetting or sticking) and ending with spray ratings of 70 (partial wetting of the specimen face beyond the spray points). Images of these ratings can be found in the AATCC Test Method 22, or on an AATCC Spray test rating chart. With AATCC Test Method 118, both lots began by passing oil #6 and ended passing oil #5.

Results were needed quickly by the researcher’s company, so while these samples were washed 100 times in the top-loader washing machine, the researcher simultaneously began a comparison in the Launder-Ometer. Then 8 x 8 in. samples were cut from each lot. These samples, after being washed in twenty Launder-Ometer

cycles (various milestones in between were taken for testing), were tested to AATCC Test Method 22 and AATCC Test Method 118. When conditions in the Launder-Ometer failed to produce comparable results to the home laundered samples, a variable was changed, and a new testing round of twenty cycles in the Launder-Ometer was performed. Variables included temperature, number of balls, and amount of detergent.

Using the water and oil repellency results from the top-loader washing machine as a database, the researcher began Round A in the Launder-Ometer with the same conditions as AATCC Test Method 61, test 2A. While the Launder-Ometer reached a temperature of 120 +/- 4°F, samples were placed in individual 1200 mL, 3.5 x 8 in. canisters. Each canister had fifty steel balls and 150 mL of liquor (0.15% detergent) heated to 120 +/- 4°F. The detergent used was AATCC 1993 Standard Reference Detergent (without optical brighteners). Once locked, the canisters were placed into the Launder-Ometer and agitated for 45 minutes. These conditions are outlined in Table 6. Between washing cycles, the samples were placed into a nylon mesh bag and dried in a front loading dryer at 155 +/- 10°F until dry, and then they were laundered again.

Samples from the following cycles were tested against AATCC Test Method 22 and AATCC Test Method 118: 1 cycle (approximately five home launderings), 5 cycles (approximately 25 home launderings), 10 cycles (approximately 50 home launderings), 15 cycles (approximately 75 home launderings) and 20 cycles (approximately 100 home launderings).

Table 6 – Round A Wash Conditions in Launder-Ometer & Dryer

Solution Temp (°F)	Steel Balls (quantity)	Detergent Solution (mL) (0.15%)	Run Time (Minutes)	Dryer Temp (°F)
120	50	150	45	155 +/- 10

Before testing, the dry samples required conditioning in a conditioned lab at 70 +/- 2°F and 65 +/- 2% relative humidity for four hours. The researcher tested three samples per lot per cycle in each round. See Table 7 for the results from Round A.

Table 7 – Round A Results in Launder-Ometer

# of cycles	S16718						S16719					
	Water			Oil			Water			Oil		
1	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
5	95	95	95	#6	#6	#6	95	100	95	#6	#6	#6
10	95	95	95	#6	#6	#6	100	95	95	#6	#6	#6
15	95	90	95	#6	#6	#6	95	90	90	#6	#6	#6
20	95	90	90	#6	#6	#6	90	90	80	#6	#6	#6

Tests on both lots showed that the Round A conditions were not harsh enough compared to cleaning in the top-loading washing machine. If the chosen conditions had been satisfactory, the results would have matched spray ratings of 70 at cycle number twenty, instead of spray ratings of one 95, 90's and one 80, and oil ratings of less than oil number 6.

The researcher next created a Round B with the following conditions: a temperature of 120°F, a total liquor volume of 800 mL (0.15% detergent), 50 steel balls, and a time of 45 minutes. These conditions are outlined in Table 8. The total liquor volume was moved from 150 mL to 800 mL to see if this variable would make a difference. When running AATCC Test Method 61, the specified sample size for test

2A is 2.0 x 6.0 in., and the samples run in the test are 8.0 x 8.0 in. If the area of the 2A sample is 12 square inches, the liquor to sample size ratio is 12.5 mL to 1 square inch. The researcher determined that a sample with an area of 64 square inches could have an equivalent of 800 mL of liquor.

Table 8 – Round B Wash Conditions in Launder-Ometer & Dryer

Solution Temp (°F)	Steel Balls (quantity)	Detergent Solution (mL) (0.15%)	Run Time (Minutes)	Dryer Temp (°F)
120	50	800	45	155 +/- 10

After running Round B, the researcher tested the samples using AATCC Test Method 22 and AATCC Test Method 118. The samples did not achieve reduction in the water and oil repellency ratings to correlate with the home launderings, and this was possibly due to the 800 mL of liquid. The volume of water did not allow the samples to move within the canister as much. The spray ratings after 20 cycles for both lots were 100, and the oils passed Number 6. See Table 9 for the results for Round B.

Table 9 – Round B Results in Launder-Ometer

# of cycles	S16718						S16719					
	Water			Oil			Water			Oil		
1	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
5	100	100	100	#6	#6	#6	95	100	100	#6	#6	#6
10	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
15	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
20	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6

Reviewing the results from Round B, the researcher decided that the next variable to change would be the number of steel balls, as the increase of abrasion action might help lower the water and oil ratings. When running AATCC Test Method 61, the specified sample size for test 2A is 2.0 x 6.0 in, and the samples run in the test are 8.0 x 8.0 in. The researcher determined that a sample with an area of 64 square inches could have 267 steel balls. The researcher created Round C with the following conditions: a temperature of 120°F, a total liquor volume of 150 mL (0.15% detergent), 267 steel , and a time of 45 minutes. These conditions are in Table 10.

Table 10 – Round C Wash Conditions in Launder-Ometer & Dryer

Solution Temp (°F)	Steel Balls (quantity)	Detergent Solution (mL) (0.15%)	Run Time (Minutes)	Dryer Temp (°F)
120	267	150	45	155 +/- 10

After washing, the samples were conditioned and tested against AATCC Test Method 22 and AATCC Test Method 118. Of all the rounds, the results from Round C correlated the most with the home laundering results. After 20 cycles, lot S16718 had

a spray rating of 80 and lot S16719 had a spray rating of 70. This compares with the home laundering results in which after 100 launderings both lots S16718 and S16719 had spray ratings of 70. However the oils in Round C did not reduce from Number 6 to Number 5 like they did in the home laundering testing. Results can be found in Table 11.

Table 11 – Round C Results in Launder-Ometer

# of cycles	S16718						S16719					
	Water			Oil			Water			Oil		
1	100	100	100	#6	#6	#6	80	90	80	#6	#6	#6
5	90	90	90	#6	#6	#6	80	80	80	#6	#6	#6
10	90	90	90	#6	#6	#6	70	70	70	#6	#6	#6
15	90	90	90	#6	#6	#6	70	70	70	#6	#6	#6
20	80	80	80	#6	#6	#6	70	70	70	#6	#6	#6

Reviewing the results from Round C, the researcher determined that the next variable to change would be the temperature. The goal of the next testing round was to achieve reduction in the oil repellency rating. In AATCC Test Method 61, the next round up from test 2A is test 3A. This test requires a temperature of 160°F. The researcher created Round D with the following conditions: a temperature of 160°F, a total liquor volume of 150 mL (0.15% detergent), 50 steel balls, and a time of 45 minutes. Conditions can be found in Table 12.

Table 12 – Round D Wash Conditions in Launder-Ometer & Dryer

Solution Temp (°F)	Steel Balls (quantity)	Detergent Solution (mL) (0.15%)	Run Time (Minutes)	Dryer Temp (°F)
160	50	150	45	155 +/- 10

The samples were laundered, conditioned, and then tested to AATCC Test Method 22 and AATCC Test Method 118. Unfortunately, the increase in temperature did not degrade the samples enough to move from oil Number 6 to oil Number 5. See Table 13 for the results of Round D.

Table 13 – Round D Results in Launder-Ometer

# of cycles	S16718						S16719					
	Water			Oil			Water			Oil		
1	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
5	100	100	100	#6	#6	#6	100	100	100	#6	#6	#6
10	90	90	90	#6	#6	#6	80	80	90	#6	#6	#6
15	90	90	90	#6	#6	#6	80	80	80	#6	#6	#6
20	90	90	90	#6	#6	#6	80	80	80	#6	#6	#6

At this point in the preliminary study, the researcher was no longer required to test lots S16718 and S16719. The researcher used this preliminary data to design the rounds from the main study. The preliminary study showed that changing conditions can affect the durability of water-repellent textiles on 70 denier nylon fabric. None of these combinations of conditions produced both water and oil test results consistent with home laundering in top-loader washing machines. Dramatically increasing the number of steel balls to 267 reduced the finish on one lot enough to reach home laundering levels for water-repellency, but did not change the oil-repellency rating. Finally, increasing the temperature of the detergent solution affected the water-repellency of one lot to a lesser degree than increasing the number of steel balls, but that loss was still less than the home laundering test results.

MAIN STUDY

The researcher used the information from the preliminary studies to develop the main study. The colorfastness study using the spectrophotometer resulted in the 2 x 4 in. samples and the 8 x 8 in. samples looking level. The researcher assumed that the even color loss corresponded with even treatment. The preliminary study using the C-6 finish helped shape the conditions used to begin the main study.

Textile Samples:

The researcher started with samples from lot S16063, as it had the least durable water-resistant finish of the four fabrics found in Table 2. Lot S16063 was a 70 denier nylon dyed black lot finished with a C-8 durable water-resistant chemical. Once optimal conditions were determined, the researcher planned to continue with the additional three lots. Ultimately, no set of optimal conditions were determined, so the remaining three lots were not prepared for testing.

Laundering Conditions: Top-Loader Washing Machine

Each lot of finished fabric was divided into two sets of ten quarter-yard cuts. One quarter-yard cut allowed for three water-repellent testing replications and three oil-repellent testing replications. The 80 samples were laundered in a Kenmore model 20442 top-loading washer with 66 grams of AATCC Standard Reference detergent. This choice of detergent is required by AATCC Test Method 135. Three samples were tested after every ten launderings. AATCC Test Method 135: Dimensional Changes of Fabrics after Home Laundering specified permanent press wash cycle at 105 +/- 5°F, a large size wash load, and permanent press tumble dry at 155 +/- 10°F. The wash load

consisted of eighteen gallons of water, the samples, and enough ballast to equal a 4-pound load. The specification offers a range of ten degrees for the drying temperature. To determine how the samples performed using both the middle and lower dryer temperatures of the specification, they were all washed at the same temperature, but one set was dried at the 145°F, and the other at 155°F. The dryer was equipped with a digital temperature to maintain a set temperature for the duration of the cycle. The laundry conditions for the samples are in Table 3.

In the S16063 top-loading machine study, the researcher performed one hundred launderings in a top-loader washing machine and tested the water and oil repellency after every ten launderings. Table 14 gives the test results that served as a correlative database from S16063 to compare with the Launder-Ometer results. Once the ratings were determined, the researcher used these results as the specification to compare the Launder-Ometer water and oil repellency results. If the samples from one of the testing rounds was worse than the same number of launderings in the S16063 top-loading machine study, the sample was marked as failing.

Table 14 – Top-Loader Washing Machine Results for Lot S16063

S16063- Black (MOD-3 DWR)												
	Water						Oil					
Cycles	Dried @ 145°F			Dried @ 155°F			Dried @ 145°F			Dried @ 155°F		
Initial	100	100	100	N/A	N/A	N/A	#6	#6	#6	N/A	N/A	N/A
10	95	100	100	100	100	100	#6	#6	#6	#6	#6	#6
20	100	100	95	100	100	100	#6	#6	#6	#6	#6	#6
30	95	95	95	100	100	100	#6	#6	#6	#6	#6	#6
40	95	100	95	100	100	100	#6	#6	#6	#6	#6	#6
50	95	95	95	100	100	95	#6	#6	#6	#6	#6	#6
60	95	95	95	95	95	95	#6	#6	#6	#6	#6	#6
70	90	90	90	95	95	95	#6	#6	#6	#6	#6	#6
80	80	80	90	95	90	95	#6	#6	#6	#6	#6	#6
90	80	80	70	90	90	90	#6	#6	#6	#6	#6	#6
100	70	70	70	90	90	90	#6	#6	#6	#6	#6	#6

Laundering Conditions: Accelerated Laundering in Launder-Ometer

Textile samples were washed using conditions in a Launder-Ometer based on AATCC Test Method 61: Colorfastness to Laundering: Accelerated. Each testing round consisted of twenty cycles, with samples being tested after one, five, ten, fifteen, and twenty cycles. Once the samples were run through one washing cycle in the Launder-Ometer, each canister was emptied into beakers, keeping each test specimen in a separate beaker. Each sample was then rinsed three times in beakers with deionized water at 105 +/- 5°F for one minute each with occasional agitation. The samples were then dried in a front-loading dryer at 145°F. After the preparation of the fabric, AATCC Test Method 22 and AATCC Test Method 118 were performed to correlate the Launder-Ometer results with the S16063 top-loading machine results found in Table 14. All water was purified using an ion-exchange system. Launder-Ometer conditions were determined as the study progressed by comparing the results

of AATCC Test Method 22 and AATCC Test Method 118 with the S16063 top-loading machine study. When the results did not correlate after the round, one of the following variables were adjusted: amount of liquor (detergent), quantity of steel balls, or temperature. To determine the effect of that alteration, the laundry cycles and tests were run again after each change.

Round 1:

The researcher established the test conditions for 8 x 8 in. samples of fabric S16063 laundered in a top-loading washing machine using the conditions from Round D of the preliminary study.

While the Launder-Ometer reached a temperature of 160 +/- 4°F, samples were placed in individual 3.5 x 8 in. canisters. Each canister had 267 steel balls and 150 mL of liquor (0.15% detergent) heated to 160 +/- 4°F. The detergent used for all rounds was AATCC 1993 Standard Reference Detergent (without optical brighteners). Once locked, the canisters were placed into the Launder-Ometer and agitated for 45 minutes. These conditions are outlined in Table 15. Between washing cycles, the samples were dried using a front loading dryer at 145°F. In the preliminary study the samples dried at 155°F had better repellency than those dried at 145°F, so for this study, the lower temperature was used to produce a wider range in results.

Table 15 – Round 1 Wash Conditions in Launder-Ometer & Dryer

Solution Temp (°F)	Steel Balls (quantity)	Detergent Solution (mL) (0.15%)	Run Time (Minutes)	Dryer Temp (°F)
160	267	150	45	145

Once the 20 cycles in the Launder-Ometer with these settings were complete, the water and oil repellency of samples were tested using standards AATCC Test Method 22 and AATCC Test Method 118. See the preliminary studies for a detailed description of both water and oil repellency test methods.

The results from Round 1 are listed in Table 16. Unexpectedly, both the water and oil repellent ratings dropped after ten cycles. Based on this disparity, the researcher decided to use fewer steel balls in Round 2.

Table 16 – Round 1 Results in Launder-Ometer

# of cycles	Water			Oil		
1	100	100	100	#6	#6	#6
5	100	100	100	#6	#6	#6
10	95	100	100	#6	#6	#6
15	50	50	50	Fail #1	Fail #1	Fail #1
20	50	50	50	Fail #1	Fail #1	Fail #1

Round 2:

The researcher created Round 2 with the following conditions: a temperature of 160°F, a total liquor volume of 150 mL (0.15% detergent), 50 steel balls, and a time of 45 minutes. Conditions for Round 2 are listed in Table 17.

Table 17 – Round 2 Wash Conditions in Launder-Ometer & Dryer

Solution Temp (°F)	Steel Balls (quantity)	Detergent Solution (mL) (0.15%)	Run Time (Minutes)	Dryer Temp (°F)
160	50	150	45	145

The researcher reduced the number of steel balls, as the water and oil repellent results from Round 1 did not correlate with the top-loader washing machine results. After the Launder-Ometer was run and the samples conditioned, the researcher tested the samples from Round 2. These results are in Table 18.

Table 18 – Round 2 Results in Launder-Ometer

# of cycles	Water			Oil		
1	100	100	100	#6	#6	#6
5	90	90	90	#6	#5	#5
10	50	50	50	Fail #1	Fail #1	Fail #1
15	50	50	50	Fail #1	Fail #1	Fail #1
20	50	50	50	Fail #1	Fail #1	Fail #1

The researcher compared the results to those from the top-loader washing machine in Table 14 and found that unlike the top-loader experiment that had no failures, the repellency ratings failed between five and ten washings samples and achieved a reduction in the oil and water repellency tests. Compared to Round 1, these samples from Round 2 performed worse, failing after between 5-10 cycles.

Round 3:

The researcher next created Round 3. The finish on samples from both Round 1 and Round 2 degraded much quicker than anticipated, so Round 3 was created with the following conditions: a lower temperature of 120°F, a total liquor volume of 150 mL (0.15% detergent), back to 267 steel balls, and a time of 45 minutes. Conditions for Round 3 are listed in Table 19.

Table 19 – Round 3 Wash Conditions in Launder-Ometer & Dryer

Solution Temp (°F)	Steel Balls (quantity)	Detergent Solution (mL) (0.15%)	Run Time (Minutes)	Dryer Temp (°F)
120	267	150	45	145

To rule out the temperature variable, the researcher made Round 3 with the same number of balls as Round 1, but have the lower temperature that was used in the S16063 top-loader machine study. After being washed and conditioned, the samples were tested against AATCC Test Method 22 and AATCC Test Method 118. Results are in Table 20.

Table 20 – Round 3 Results in Launder-Ometer

# of cycles	Water			Oil		
1	100	100	100	#6	#6	#6
5	70	70	70	#5	#5	#5
10	50	50	50	#3	#3	#3
15	50	50	50	Fail #1	Fail #1	Fail #1
20	50	50	50	Fail #1	Fail #1	Fail #1

Round 3 samples reduced water and oil repellency ratings even quicker than Rounds 1 and 2. The researcher questioned the evenness of the finish.

Aqueous Liquid Test:

The researcher performed AATCC Test Method 193: Aqueous Liquid Repellency: Water/Alcohol Solution Resistance Test to ensure that the durable water resistant finish was even across the fabric. The researcher cut three 8 x 8 in. samples from Lot S16063. On each of the samples, the researcher performed three determinations of

three different water/isopropyl alcohol ratios. The results, listed in Table 21, showed that the finish was even across the samples.

Table 21 – Water/Isopropyl Alcohol Ratio Water Droplet Test

Samples	Detr.	Water/Isopropyl Alcohol		
		40/60	50/50	60/40
1	1	Pass	Pass	Pass
	2	Pass	Pass	Pass
	3	Pass	Pass	Pass
2	1	Pass	Pass	Pass
	2	Pass	Pass	Pass
	3	Pass	Pass	Pass
3	1	Pass	Pass	Pass
	2	Pass	Pass	Pass
	3	Pass	Pass	Pass

The above test was performed exactly like AATCC Test Method 118, except the researcher used drops of water/isopropyl alcohol instead of oil. All of the samples passed the three different ratios, showing that the finish was even across the samples.

Round 4:

Round 4 was created with the following conditions: a temperature of 120°F, a total liquor volume of 150 mL (0.15% detergent), 50 steel balls, and a time of 45 minutes. Conditions for Round 4 are listed in Table 22.

Table 22 – Round 4 Wash Conditions in Launder-Ometer & Dryer

Solution Temp (°F)	Steel Balls (quantity)	Detergent Solution (mL) (0.15%)	Run Time (Minutes)	Dryer Temp (°F)
120	50	150	45	145

The researcher reduced the number of steel balls from 267 to 50 balls, which is the number of balls required in AATCC Test Method 61, test 2A. The results from Round 4 are in Table 23.

Table 23 – Round 4 Results in Launder-Ometer

# of cycles	Water			Oil		
	1	100	100	100	#4	#4
5	90	90	90	#3	#3	#3
10	50	50	50	Fail #1	Fail #1	Fail #1
15	50	50	50	Fail #1	Fail #1	Fail #1
20	50	50	50	Fail #1	Fail #1	Fail #1

The researcher determined that the results from this study and the results from the S16063 top-loading machine study do not correlate. In the S16063 top-loading machine study, the lots finished with a C-6 DWR performed better than the samples in the main study finished with a C-8 DWR. As C-8 durable water repellents are more robust than C-6, the study was not producing results comparable to top-loading washing machine.

Rinse Comparison:

The researcher next created a rinse comparison. As the samples are being laundered in the Launder-Ometer, the rinse cycle is performed by the researcher. If the samples are not rinsed sufficiently, the samples could have residual detergent present which might have a negative effect on the water and oil repellency ratings. The researcher created a rinse comparison with the following rinses: no rinse, 10 second rinse, 1 minute rinse, and a 3 minute rinse. The samples were washed one and five times in the Launder-Ometer, rinsed, dried, conditioned, and then tested for water repellency. The results are listed in Table 24.

Table 24 – Rinse Comparison Results

Cycles	No Rinse	10 sec rinse	1 min. rinse	3 min. rinse
Wash 1	50	50	70	80
	50	50	80	80
Wash 5	50	50	70	70
	50	50	70	70

Round 4A:

The researcher found that residual detergent reduced the water repellency of the samples. The less rinsing, the lower the water repellency rating of the sample. Therefore, the researcher decided to re-test Round 4, naming it Round 4A with increased rinsing with the following conditions: a temperature of 120°F, a total liquor volume of 150 mL (0.15% detergent), 50 steel balls, and a time of 45 minutes. Conditions for Round 4A are that same as Round 4, listed in Table 22.

After laundering the samples, the researcher tested them for water and oil repellency. The results can be found below in Table 25.

Table 25 – Round 4A Results in Launder-Ometer

# of cycles	Water			Oil		
1	100	100	100	#5	#5	#5
5	70	70	70	#4	#4	#4
10	50	50	50	#3	#3	#3
15	0	0	0	Fail #1	Fail #1	Fail #1
20	0	0	0	Fail #1	Fail #1	Fail #1

Comparing Round 4 and Round 4A, the results were not expected. Results from Round 4A were lower in rating than the ratings from Round 4. This was the first

round that received zero ratings for the water repellency. The rinse tests reported in Table 24 indicated that rinsing would help, but the results of Round 4A did not correlate. Therefore, the researcher decided to retest Round 4 again, creating Round 4B.

Round 4B:

Round 4B had the following conditions: a temperature of 120°F, a total liquor volume of 150 mL (0.15% detergent), 50 steel balls, and a time of 45 minutes. These conditions for Round 4B, the same as Round 4, are listed in Table 21.

The results from Round 4B are in Table 26. Once again, these results were unexpected.

Table 26 – Round 4B Results in Launder-Ometer

# of cycles	Water			Oil		
	1	90	100	100	#5	#4
5	80	80	70	#4	#4	#4
10	50	50	50	Fail #1	Fail #1	Fail #1
15	0	0	0	Fail #1	Fail #1	Fail #1
20	0	0	0	Fail #1	Fail #1	Fail #1

The results from Round 4B did not match the results from Round 4 or Round 4A, except for the oil resistance that was more durable, and the water resistance consistently failed between 5 and 10 cycles.

DISCUSSION & CONCLUSION

The main study showed that changing the conditions in the Launder-Ometer can affect the durability of water-repellent textiles on 70 denier nylon fabric. None of these conditions produced both water and oil test results consistent with home laundering in top-loader washing machines.

The researcher questioned the evenness of the finish application, but determined that it was even by performing AATCC Test Method 193 using different ratios of water/isopropyl alcohol. These results are in Table 21.

Round 1 failed both water and oil repellency ratings in comparison to the S16063 top-loading machine study after 10 cycles in the Launder-Ometer. When the researcher reduced the number of steel balls from 267 to 50 for Round 2, the finish failed after five cycles. In Round 3 the researcher dropped the bath and solution temperature from 160°F to 120°F, but the samples failed both water and oil repellency ratings again after five cycles.

In Round 4, the researcher reduced the number of balls so that the conditions would match AATCC Test Method 61, test 2A. These were the same conditions that were used in the first round in the C-6 durable water-resistant finish Launder-Ometer preliminary study. Yet the results from Round 4 failed oil ratings after one cycle and water ratings after five cycles. These results were unexpected, as S16063 (the lot of fabric being tested) was finished with a C-8 durable water-resistant finish that historically is more robust than the C-6 DWR. The researcher determined that rinsing does affect the water repellency of the fabric through a rinse comparison study. These

results are in Table 24. However when testing the samples from Round 4A and 4B, which had additional rinsing, the water and oil repellency ratings did not improve.

Round 4 was performed two more times, named 4A and 4B, and all three rounds produced different water and oil repellency ratings. Three rounds of the same conditions did not provide the same results. Further research is needed to determine what the cause of the variation is.

Further research is required to better understand the results from the various rounds. Additional retests of Round 4 would help prove if the steel balls are causing the inconsistencies in the water and oil repellency results.

In the preliminary study, the Launder-Ometer reduced the repellency less than a top-loader washing machine so the researcher made the conditions, more severe. In the main study, the researcher began with harsher conditions but had to reduce the severity of these when the water and oil repellency results failed when compared to the S16063 top-loader machine study. As the preliminary study was the opposite of the main study, further research is needed using different fabrics and DWRs to determine if the Launder-Ometer is capable of reliably correlating with home launderings for finish durability.

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