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EFFECT OF DIFFERENTIAL FOAM APPLICATION OF DURABLE PRESS AND FLUOROCHEMICAL FINISHES TO COTTON FABRIC

A Thesis

Presented for the

Master of Science

Degree

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Pi-Shin Wey

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DEDICATION

This thesis is dedicated to my parents, Mr. Long-Tu Wey and Mrs. Chen-Yuan C. Wey.

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ABSTRACT

In this study, the effect of differential foam finishing on the aesthetics and funtional properties of durable press (DP) and fluorochemical (FC) treated cotton fabrics was investigated. Effects of pretreatment and fluorochemical level on properties were also studied.

The 100% cotton print cloths were pretreated separately by two different preparatory process, bleaching (BL) only, and bleaching, followed by caustic mercerization/liquid ammonia treatment (BL-CM/AT). Two different pretreated fabrics were finished separately by one and two-sided foam application at 25% wet pick-up with various levels of FC (0.75, 1.00, and 1.25%) and fixed DMDHEU resin (4.00%) add-on, and at 21% wet pick-up with 1.00% FC/3.80% DMDHEU add-on. Performance properties (water and oil repellency, abrasion resistance, wrinkle recovery, durable press appearance rating, tearing and tensile strengths) of foam finished cotton fabrics were examined.

One-sided foam application of FC and DP finishes resulted in excellent water and oil repellency on the applied side while leaving the nonapplied side absorbent. At the same time resulted in similar durable press properties on both sides.

The combination pretreatment of caustic mercerization and liquid ammonia treatment appreciably improved tearing and tensile strength of both one and two-sided foam finished fabrics. Furthermore, abrasion resistance was also substantially improved by the combination pretreatment.

One-sided foam application generally resulted in higher tearing strength than two-sided foam application. However, one-sided foam treatment usually resulted in lower tensile strength than double-sided foam application.

Generally, abrasion resistance on the nonapplied side of differentially FC and DP finished fabric appeared to be better than on the applied side. As the fluorochemical add-on was increased from 0.75 to 1.25%, abrasion resistance, tensile strength and tearing resistance all unexpectedly decreased.

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CHAPTER I

INTRODUCTION

The cost of energy and the present market demand for special finishing effects have resulted in a number of machinery innovations that have changed the character of textile finishing. It has been well documented that the chief energy demands in the textile industry are for heating water during wet processing and evaporating water during drying (1, 2). The diminishing sources of energy coupled with increasing energy costs have motivated the textile industry to develop new techniques for reducing energy consumption. Toward this end, several low wet pick-up methods have been implemented to reduce the amount of water which has to be subsequently removed during drying steps. The most popular low wet pick-up techniques include: air jet assisted squeeze rolls, kiss rolls, engraved rolls, pad-vacuum extraction, spray systems and foam application (1-6). Foam finishing is widely accepted and much progress is being made in foam dyeing and printing technology.

The primary purpose of the water in conventional pad operations in as a medium for evenly distributing the desired chemicals throughout the fabric. In foams, air instead of water serves as the diluent, replacing a

large portion of the water with air, which reduces energy consumption for drying. Also air increases the volume of the chemical mixture thereby improving its ability to penetrate the fabric uniformly (5). Various researchers have reported that poor performance properties are caused by nonuniform distribution of finishes. For example, Rowland et al. (7, 8) noted that nonuniform application of durable press finishes caused nonuniform crosslinking throughout fabric, and thus resulted in low durable press ratings and poor abrasion resistance. Low wet pick-up results in less migration of chemical to the fabric surface during drying and should result in more uniform properties (2, 5). Experimental evidence (4, 6) exists that softer hand, reduced dusting, better wrinkle recovery and improved abrasion resistance with more efficient utilization of chemicals can be achieved with foam finishing due to less migration of chemicals.

Although the energy savings resulting from utilizing foam finishing techniques has been well documented (3, 4, 5, 9), the advantages of lower chemical usage and improved performance properties have not been investigated to the same extent (4, 6, 10, 11). Foam finishing has been shown to be more versatile by Wadsworth et al. (12). In their study, fluorochemical and durable press

resins were co-applied to one side only and to both sides of cotton fabrics. High levels of oil and water repellency were achieved on the treated side of the fabric while maintaining absorbency on the untreated side. Durable press appearance ratings and wrinkle recoveries were very similar from face to back of one-sided foam treated fabrics, and there were minimal differences between one and two-sided foam applications. Both tearing and tensile strength differed little between one and two-sided foam applications. The paper also compared effects of various pretreatments of bleaching, caustic mercerization, liquid ammonia treatment and a combination caustic mercerization/liquid ammonia treatment. At corresponding wet pick-up and resin add-on levels, the use of liquid ammonia and a combination caustic mercerization/liquid ammonia pretreatment improved wrinkle recovery and tearing and tensile strength. Thus, the combination of pretreatment and one-sided foam application resulted in more favorable repellency, crease recovery and fabric strength than with conventional finishing techniques. However, the optimum fluorochemical level was not determined. The distribution of fluorochemical and durable press finishes in the fabric and their effects on abrasion resistance were not fully investigated. The degree of improvement in strength and abrasion resistance resulting from the

combination pretreatment of caustic mercerization and liquid ammonia treatment were not quantitatively studied. Therefore, the purpose of this research was to more quantitatively determine the effects of differential foam finishing on aesthetics and performance properties of durable press and fluorochemical treated cotton fabrics.

The objectives of this research were:

1. To examine the effects on water and oil repellency and durable press properties (wrinkle recovery angle and durable press rating after repeated home launderings) of fluorochemical (FC) and durable press (DP) treated cotton fabric at a range of FC add-ons (0.75, 1.00, and 1.25%) with fixed DP resin (4.00%) by one and two-sided foam applications. The end-use of the finished fabric would be protective clothing for pesticide applicators.

2. To determine if the flex abrasion resistance of fabric treated on one side differs significantly from fabric finished on two sides.

3. To more fully substantiate the improved strength and abrasion resistance resulting from the combination pretreatment of caustic mercerization and liquid ammonia treatment compared to preparatory finishing through bleaching only.

CHAPTER II

REVIEW OF LITERATURE

Development of Low Wet Pick-Up

The textile industry presently consumes the equivalent of 55 million barrels of oil in energy each year. Wet processing (dyeing and finishing) requires between 55% to 60% of this total, and most of this is used for either heating or evaporation of water (13). Thus, in the face of the energy crisis since 1974, considerable efforts have been spent on developing methods for reducing the amount of energy consumed in the wet processing of textiles.

One approach has been to increase energy efficiency of conventional processes. Another is to reduce the amount of water required to apply finishes and colorants. Combining or elimination processes and reusing or recirculating the baths have also been proven feasible (3, 9, 14). The method to reduce the necessary amount of water is known as the low wet pick-up technique. Low wet pick-up techniques usually can be categorized into expression methods (saturate the substrate and subsequently remove the excess liquid, usually by squeezing or other mechanical means) and topical methods (apply a limited amount of treating liquor in the beginning) (4, 9).

According to Goldstein and Smith (4), the maximum water absorption of the fiber structure, also known as the critical moisture content or the water of imbibition value, represents the absolute lower limit of wet pick-up achievable with any expression method. They found that the water imbibition value varies from about 30% for cotton, to 10% for polyester staple and 1-3% for polypropylene and polyester filaments. If a wet pick-up lower than the water imbibition value of the fiber is needed, it would have to be achieved through a topical method of application. Topical treatment methods apply only a small amount of liquor corresponding to the low wet pick-up desired. Foam finishing is a topical method of low wet pick-up and is already well established in textile industry (2, 3, 4, 13).

Foam Finishing of Textiles

The function of water in finishing is to uniformly distribute relatively small amounts of chemicals throughout the fabric. In the foam system, air instead of water serves as the main diluent to transport the finishing agents or colorants (15, 16). Also, air increases the volume of the chemical mixture to ensure its ability to penetrate all interstices and surfaces of

the fabric. The only limitation on the amount of air that can replace water is the minimum amount of water that is required to produce a reasonably stable foam to facilitate the uniform spreading of the treating liquor over all fiber surfaces in the fabric. And if cellulosic fibers are being processed, enough liquid should be used to swell these fibers (5, 9, 11). To determine low wet pick-up, the amount of water already contained in the commercial chemicals should be considered. Acceptance of foam technology may result in more concentrated reagents being supplied by the chemical suppliers.

The ratio of the volume of the gas phase to that of the liquid phase can be measured by the "blow ratio," which is given by the following equation:

$$\text{Blow Ratio} = \frac{W_1}{W_2}$$

where

W_1 = the initial weight of a given volume of liquid before foaming,

W_2 = the weight of the same volume of foam.

The blow ratio may vary in textile applications from about 5:1 to 100:1 (5). Foams may also be characterized by density measurements. According to Cooke (5),

foam densities below 0.5g/cm^3 and often in the range of 0.01 to 0.03g/cm^3 are generally required. The density of the foam must be controlled to achieve uniform application.

Foams may be conceptuallized as a mass of distorted polyhedral bubbles dispersed in liquid. They can be produced by either condensation or dispersion methods (5). Condensation foams are made by generation or release of gas from within the liquid by either a chemical or physical change. Examples of condensation foam are chemically generated fire-fighting foams and foams in beer and soft drinks. Foams used in the textile finishing process are formed by the dispersion method. Dispersion formation is when the bubbles are foamed deliberately by introducing a gas into the liquid phase (9, 15). The bubbles are separated by thin films of liquid, and most of the volume is in the gas phase. The monodisperse foams (those consisting of bubbles of identical dimension) are necessary to obtain good quality dyeing and finishing (16).

Stability of a foam is critical in the foam finishing process. If the foam is too unstable, it will collapse too rapidly onto the fabric to permit control of add-on and uniformity. On the other hand, if the foam is too stable, it will not collapse evenly throughout the fabric, resulting in streaked and blotchy dyeing or

finishing (17). Also the foam may completely penetrate the fabric without collapsing. Foam stability can be measured by the drainage rate in which the increase in the amount of liquid separating from the foam is determined with time. The drainage of liquid from foam is caused by rearrangement of the lamellae between the bubbles, drainage of the lamellae by gravitational force, the diffusion of gas from smaller to larger bubbles, and the bursting of bubbles (5). After a foam is made, depending on its stability, it starts to drain. When it drains it forms a thinner film. Then gas diffuses from smaller to larger bubbles. This is dur to the gas pressure differential within the large and small bubbles. The pressure inside the small bubble is greater, so transfer of pressure occurs and the small bubble disappears. This process leads to the development of a reduced number of large bubbles having thin walls which are fragile and easily ruptured (16).

Distribution and movement of the foam through the fabric occur under a combination of hydrostatic, dynamic and capillary forces. Prior to collapse the foam merely sits on the surface of the fabric and only after collapse does it begin penetrating the fabric. Breakage of the foam and penetration of the liquor occur largely through the action of external forces such as pressure, heat or

other frictional forces. Once the foam is broken, the foam liquid is absorbed by the fibers, or is driven by capillary pressure for further distribution and penetration (9, 16).

The foam formulation is important to the characteristics of the foam and finished products. Some requirements that should be considered are as follow: (a) all of the chemicals should be compatible; (b) the foaming agents should not interfere with the desired finished qualities; and, (c) the finishing agents cannot inhibit the foam properties. Suitable foaming auxiliaries should be added to get the desired structure and stability of foam (9).

Application of a foam to the fabric may be achieved by several different methods. Although each method claims one or more distinctive features, they follow a common sequence of steps or physical transformations: (a) formulation to achieve foamability, (b) generation of foam, (c) application of foam to substrate, and (d) destruction of foam (9). The most common methods of producing foam for textile application are bubbling gas through small orifices into the solution, by the use of injectors, by agitation or by various other mechanical means as well as by chemical generation of gas in the liquid (15). The application is actually done using one

of several approaches which include knife over a roll, horizontal pad coating, floating knife coating, and extrusion coating. The method chosen depends on the fabric construction and the end effects desired (1, 3, 9, 13, 15).

The foam process is currently being used in plants for durable press finishing, shrinkage control, and softener applications to a variety of cotton and polyester/cotton blend fabrics (10). Gregorian and Namboodri (6) have reported that at equivalent dry add-ons, the durable press ratings of the foam finished cotton and cotton/polyester blend fabrics at low wet pick-up (10-30%) levels were significantly higher than the ratings of the fabrics finished by the conventional padding process at higher wet pick-up (60-100%) levels. They also pointed out that optimum performance for both fabrics was obtained at a wet pick-up level of about 30%. Gregorian et al. (13) have shown that foam processed durable press fabric exhibit less dusting, better wicking and higher durable press ratings than conventionally processed goods. Bryant (19) has indicated that the same performance properties may be obtained at lower chemical add-ons by foam application. In these researchers' opinions, the improved results with foam processing at low wet pick-up were probably due to less migration and

more uniform penetration throughout the fabrics. These comments were also made by several other researchers (4, 6, 11, 12).

Goldstein et al. (4) reported that with foam application, the abrasion resistance at 26-28% low wet pick-up appeared better than results obtained with padding. They found that foam application led to an improved relationship between wrinkle recovery and flex abrasion with liquid ammonia pretreatment of all wet pick-up levels. This may be due to less migration during drying by low wet pick-up process, and result in less surface deposition of resin.

Brown et al. (11) have employed both direct dye staining and fabric performance tests to study and define the uniform low wet pick-up limit for both single and double-nozzle application of semistable foams by Foam Finishing Technology (FFT). They found that when a single application nozzle was used, the face to back uniformity of applied resin, judged after direct dye staining of the treated fabric, was governed mainly by the foam wet pick-up. Wrinkle recovery value showed only a slight dependence on wet pick-up. When the durable press resin was applied by using the two-nozzle FFT application system, excellent face-to-back resin uniformity was observed, and high wrinkle recovery values were maintained over the same range of wet pick-up.

Preparation of fabric for foam application is very important. The ability of the fabric to absorb quickly and uniformly will ensure consistent end use properties of the fabric (9). Heap (19) has pointed out that by liquid ammonia pretreatment, migration of chemicals is more or less eliminated, which will result in more uniform crosslinking and less crosslinking on the surface of the fabric. Thus, Goldstein and Smith (4) found that foam durable press finishing resulted in superior performance if cotton fabrics were pretreated with liquid ammonia. In their experiments, ammonia pretreatment imparted better abrasion resistance to durable press foam finished cotton fabric. Wadsworth et al. (12) obtained similar results with liquid ammonia pretreatment and a combination pretreatment with caustic mercerization/ liquid ammonia which enhanced wrinkle recovery, tearing and tensile strength. The proper combination of pretreatment and foam application techniques resulted in more favorable repellency, crease recovery and fabric strength than conventional finishing techniques.

Foam processing is versatile. By control of foam penetration properties and application conditions, different finishing chemicals can be combined to apply them predominately to one side of a fabric. It is also apparent that various performance properties can be

achieved simulatneously on either side of the fabric (9) A study conducted by Wadsworth et al. (12) confirmed these observations. In that study, the simultaneous application of both fluorochemical and durable press finishes from one side of the fabric only by foam technique was shown to result in high oil and water repellency on the treated side and absorbency on the opposite side while achieving excellent and equal durable press properties on both sides of the fabric. Strength differed little for one and two-sided foam applications.

Durable Press Treatment with DMDHEU

"Durable press" refers to the ability of a fabric to retain an attractive appearance during wear and to retain its original smooth surface and shape after laundering. With durable press finishing, excellent wrinkle recovery, crease retention, and dimensional stability were made possible (20).

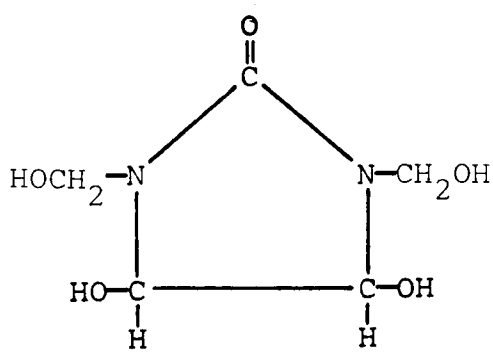
The common synthetic fibers are not prone to wrinkling, due to their high percentage of crystallinity and high work of recovery after deformation (21). Protein fibers resist creasing and wrinkling because polymer chain are bound together by covalent chemical bonds, such as salt linkages and disulphide (cystine) linkages. These bonds cause the fiber to revert to its

original shape and position. Cellulose fibers do not shrink, but their inherent disadvantage is their tendency to wrinkle badly during wear and maintenance. There are two explanations for the ease of wrinkling of cellulosic fibers. First, the weaker Van der Walls forces and hydrogen bonds in the cellulose fiber are incapable of pulling the fiber back to its original form. Second, fibers containing a high proportion of amorphous regions are markedly prone to crease because of the absence of intermolecular binding forces (22). Therefore, the widespread market penetration of durable press garments since 1964 has had a dramatic impact on cotton wash-wear research.

The mechanism of imparting durable press characteristics to fabric depends primarily upon the interaction of the textile resin and cellulosic component of the fabric. The early idea of durable press finishing was the polymer-stuffing theory, which indicates that if the amorphous region of the cellulose fiber filled with some other molecules such as resins, the fiber would become more rigid and crush resistant. Nowadays, the main theory about durable press finishing is that the finishing agent reacts chemically with cellulose molecules lying parallel to one another so that they become cross-linked and enhance resilience (22, 23).

As early as 1927 the anti-crease effect on cotton with phenol-formaldehyde in the presence of alkali was observed. Soon afterwards, the use of urea-formaldehyde in the presence of acid (22) was reported. Although good wash-wear performance can be obtained by the use of such resins, the urea-formaldehyde system suffers from the disadvantage that fabrics containing this resin absorb chlorine during hypochlorite bleaching in laundering. This is due to the formation of chloramines by the action of chlorine on free NH-groups remaining in the resin structure (24). Because of the low basicity of these NH-groups the chloramines formed on bleaching are readily decomposed by water under the hot, humid conditions of pressing or ironing, and the hydrochloric acid liberated by this decomposition causes severe tendering of the fabric (25). In order to overcome the chlorine-retention problem, melamine-formaldehyde was introduced by the textile industry. In melamine-formaldehyde resins, the triazine ring confers considerable basicity to the nitrogen atoms, with the result that, although chlorine is absorbed on bleaching with hypochlorite, the chloramines that are formed are stable to hydrolytic attack during any pressing or ironing operation. Unfortunately, the chloramines of melamine are yellow during bleaching and melamine-formaldehyde

imparts a stiff hand to the fabric. Dimethylolethylene-urea (DMEU) was used mostly in the 1950s. However, its use has diminished greatly in recent years, primarily because of its limited durability to commercial laundering and its retention of chlorine from bleaching agents (24). The popularity of the various resins seems to vary from time to time. The most important product at present is dimethyloldihydroxyethyleneurea (DMDHEU):



It is obtained by the reaction of glyoxal, urea, and formaldehyde. At elevated temperatures, not only can the methylol groups attached to the nitrogen react with the hydroxyl groups of cellulose to form and ether, but the two hydroxyl group attached to the ring carbon can also react with cellulose. This reactant, often referred to as a glyoxal-type reactant, is very stable under storage conditions after being applied to the fabric,

and it will not react until cured at high temperatures. Thus, it is used extensively in both postcure and precure applications. It also has the advantage of low free-formaldehyde content (26). Although DMDHEU is the most widely used resin for the production of durable press garments, it results in fabrics not only somewhat chlorine-retention, but also some degree of yellowing. This is the disadvantage common to all reactants derived from glyoxal, but the degree of yellowing is not sufficient to cause any major problem in the finishing of colored goods (25).

The crosslinking between cellulosic chains within the fiber can increase the resilence on the basis of stress-stain phenomena. This is accompanied by a reduction in elongation or embrittlement of the fibers. Embrittlement of the cotton fiber cause a reduction in tensile and tear strength and in abrasion resistance (21, 26). As a result, various investigations (25, 27) were conducted to improve the balance of textile performance properties of durable press fabrics. An important concept (25) has shown that retention of strength properties may be improved by minimizing the the strains that crosslinks fix within the fibers; this is accomplished by avoiding excessive crosslinking. Rowland et al. (27) reported that the penetration of

reagents into the less accessible regions in cotton fiber to relieve internal strains within the fibers is a critical factor in improving strength properties. They also pointed out that abrasion resistance is inversely proportional to surface crosslink density, but strength retention is related to the presence of a stronger untreated inner layer. After a series of experiments (7, 28), they concluded that the improved balances of strength properties to resilience are associated with the lowest reagent migration and more uniform distribution.

Proper preparation and low wet pick-up techniques may be combined to produce improved durable press cotton fabrics. Jones and his co-workers (29) have reported that both caustic mercerization and liquid ammonia treatment generally, but not always improved tensile strength. Liquid ammonia treatment contributed significantly to high durable press ratings and improved abrasion resistance. Raheel (30) found that liquid ammonia in conjuction with caustic soda pretreatment achieved a favorable balance of strength, abrasion resistance, and wrinkle recovery appearance in cotton fabric. Goldstein et al. (4) showed that ammonia mercerization and foam finishing results in better durable press performance for cotton fabrics. Wadsworth et al. (12) reported a similar conclusion.

At corresponding wet pick-up and resin add-on levels, the use of liquid ammonia and a combination caustic mercerization/liquid ammonia pretreatment resulted in better durable press properties and improved strength.

Water and Oil Repellency by Fluorochemicals

In studying the factors involved in the wetting of surfaces, Zisman and his co-workers (31, 32, 33, 34, 35) found that wettability of a surface was profoundly affected by the chemical nature or the physical chemistry of the atoms populating the surface layer. Specifically, they found that surface wettability was directly proportional to surface energy; the lower the furface energy, the lower the tendency to wet. The gross effect of both water and oil-repellent finishes is one of imparting hydrophobic and oleophobic surface characteristics to the materials and lowering their surface energy (32, 33).

Water repellents have long been applied commercially to textiles to provide protection from the wet weather and to impart resistance to water-borne stains, while maintaining air permeability. Since the 1950s, oil repellent finishes have been developed to decrease the adherence of oil-borne stains to textiles (32, 36). Wax emulsions, aluminum complexes, hyrophobic aminophasts, and compounds such as pyridinium compounds and long-chain fatty amides,

organo-chromium compounds, silicones, and fluorochemicals are the commercially important groups of water and oil repellency reagents (32, 37). Among these reagents, fluorochemicals are the most popular repellent finishes since they repel both water and oil-borne stains very efficiently, and require little add-on. They are also particularly durable to laundering and dry cleaning. However, they tend to be significantly more expensive. Thus, these finishes are supplied as two component systems including fluorochemical and extender (26, 32, 34).

Grajeck and Peterson (34) pointed out that all fluorochemical textile finishes available today will develop most of their potential initial oil and water repellency at concentration of 1% or less solids on the fabric. They employed Textile Chemical FC-208 (a nonionic latex of a modified fluorinated acrylic polymer produced by Minnesota Mining and Manufacturing Company) to study the relationship between concentration of fluorochemical and repellency properties. Both oil and water repellency increased dramatically as the concentration of FC-208 increased fro 0.2% to 1.0% on the fabric. Above the 1.0% level, repellency values were essentially constant thereafter. However, the durability of those finishes to laundering and drycleaning improved directly with increased concentration, and were it not

for the additional cost, concentrations of 2.0% or more on the fabric would be desirable.

A fluorochemical is defined as an organic compound in which a high percentage of the hydrogen attached to carbon has been replaced by fluorine. Not all fluorochemicals will impart water and oil repellency when applied to textiles. There are a number of conditions which must be satisfied to produce an effective textile finish. The major consideration is the need for a fluorinated carbon chain closely packed and evenly distributed on the fiber, with proper orientation of the perfluoro groups to present an essentially fluorinated low energy surface to repel the water and oil (32, 34).

A wide variety of fluorochemicals are mentioned in the patent literature. Philips et al. (33) have found that treatment with the chromium complexes of perfluoromonocarboxylic acids such as perfluoro-octanoic acid chromic chloride and poly (fluoro-esters of acrylic acid) such as 1,1-dihydroperfluorobutyl acrylate provided excellent repellency for cotton fabrics to oil and water and perhaps many other liquids. Furthermore, these chemicals decrease tear and breaking strength, elongation, and air permeability slightly, while flat abrasion resistance was improved. Goldstein (36) has shown that by the use of combination of fluorochemical and water

repellent, the resistance of cotton fabrics toward water, oil, washing, dry cleaning, and abrasion can be greatly increased. The combination finishes also imparted a high level of rot resistance to cotton, and just caused little or no yellowing.

Foam application of fluorochemical finishes to cotton fabrics has been studied by Wadsworth et al. (12). In that study, fluorochemical was applied alone and in combination with durable press resin to cotton print cloth by one and two-sided Foam Finishing Technology. Good water repellency of 70 to 80 spray ratings and oil repellency of 3 to 4 drop ratings can be obtained on the treated side. And the untreated side resulted in zero ratings for both repellency. These results proved that foam finishing of fluorochemical can be used to impart markedly different properties on each side of the fabric.

Foam agents for repellent finishes marketed as nonrewetting, and were intended to decompose during the curing step. Nevertheless, the small amount remaining has shown to adversely affect repellency (4). Work now in progress by Potnis and Wadsworth (unpublished Ph.D research) indicates that even higher oil and water repellency values can be obtained by reducing foaming agent concentration from the commercial level of 2-3% to 1% or less. However, modification in the equipment may be required.

CHAPTER III

METHODOLOGY

Materials

Fabrics Selection and Pretreatments

The 100% cotton fabric selected for this study was a 59W x 52F cotton print cloth having a weight of 5.7 oz/yd^2. The greige print cloth was prepared for finishing on conventional production equipment at Dan River Inc. The fabrics were all given a standard three-stage rope bleach preparation which included singeing, desizing, scouring and bleaching. Some of the bleached fabrics were mercerized with sodium hydroxide, and also treated with liquid ammonia by the Sanfor-Set Process. The pretreatments were performed as described in Table I. The prepared fabrics were neutralized with acetic acid and given a cold wash to ensure a uniform fabric pH before resin treatment.

Durable Press and Fluorochemical Formulations

Formulations developed and utilized in this experiment are shown in Table II. The concentration of resins, catalysts and agents were adjusted according to the desired wet pick-up levels and chemical add-ons. Information concerning chemical agents and suppliers is given in Table III.

Table I. Caustic Mercerization and Ammonia Treatment Conditions.

Treatment	Conditions
Caustic Mercerization	Fabric: Wet on Wet Saturator: 48° Tw NaOH Reaction Time: 30 sec. Washed on Frame/Open Width Washers Dried on Steam Heated Cans
Ammonia	Fabric: Dry (1% Moisture) Saturator: Anhydrous Liquid Ammonia Reaction Time: 1-2 sec. Removal: Dry Heat and Steam
Neutralization	1% Acetic Acid (Ambient Temperature) Washed Cold/Can Dried

Table II. Composition of Fluorochemical (FC) and Durable Press (DP) Formulations.

	FFT Formulations (Wt.%)			
	(25% WPU)			(21% WPU)
Agents	0.75%FC/ 4.00%DP	1.00%FC/ 4.00%DP	1.25%FC/ 4.00%DP	1.00%FC/ 4.00%DP
Mykon NRW-3	2.00	2.00	2.00	2.50
Norane 88	20.24	27.00	33.74	32.14
Scotchgard FC-208	10.71	14.29	17.86	17.01
Aerotex Reactant 900	35.56	35.56	35.56	40.21
Catalyst PRC-1	7.11	7.11	7.11	8.04
Palanthrene Violet B	0.025	0.025	0.025	-
Intralite Blue L	-	-	-	0.01

Table III. Sources of Commercial Finishing Agents.

Agent	Description	Supplier
Mykon NRW-3	Nonrewetting foaming Agent	Sun Chemical Corp.
Norane 88	Water Repellent and FC Extender	Sun Chemical Corp.
Scotchgard FC-208	Cationic Emulsion of Fluorochemical, 28% Solids	3M Co.
Aerotex Reactant 900	Unbuffered Dimethyloldihydroxy-Ethyleneurea (DMDHEU), 45.0% Active	American Cyanamid Co.
Catalyst PRC-1	Aluminum Salt Solution	Sun Chemical Corp.
Palanthrene Violet B	Vat Dye	BASF Wyandotte Corp.
Intralite Blue L	Direct Dye	Crompton & Knowles Corp.

The Foam Finishing Technology Process

The Foam Finishing Technology (FFT) process was developed by Union Carbide Corporation, and application and control equipment for the process was manufactured by Gaston County Dyeing Machine Corporation. The equipment is designed to apply chemical treatments to a moving textile substitute by means of a foam. In the system (Figure 1), the finishing liquid and compressed air are simultaneously metered to a radial head foamer to produce a homogeneous foam with an appropriate foam density and delivery rate. The liquid delivery is correlated with fabric weight and processing speed to give an accurate add-on of chemical. This system used a semi-stable type of foam under pressure. The pressurized foam is injected directly onto the substrate in precise quantities through a narrow slot over which the fabric passes. Foam contacting the fabric, breaking at the point of application and penetrating the substrate are being controlled by various application parameters (38, 39). As shown in Figure 2, foam can be applied by a single nozzle from one side of the fabric or by two nozzles to both sides.

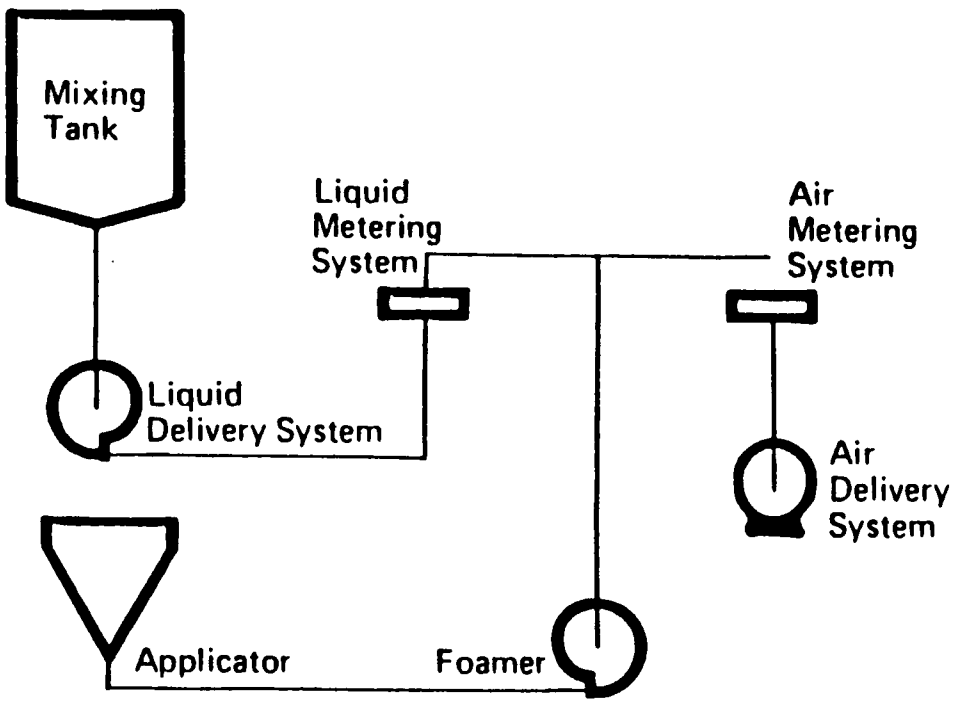


Figure 1. FFT System Schematic.

Source: Larry C. Wadsworth, "Foam Application of Durable Press and Fluorochemical Finishes to Cotton Fabrics", Textile Chemist and Colorist, Vol. 15, No. 12, December 1983, p. 241/27.

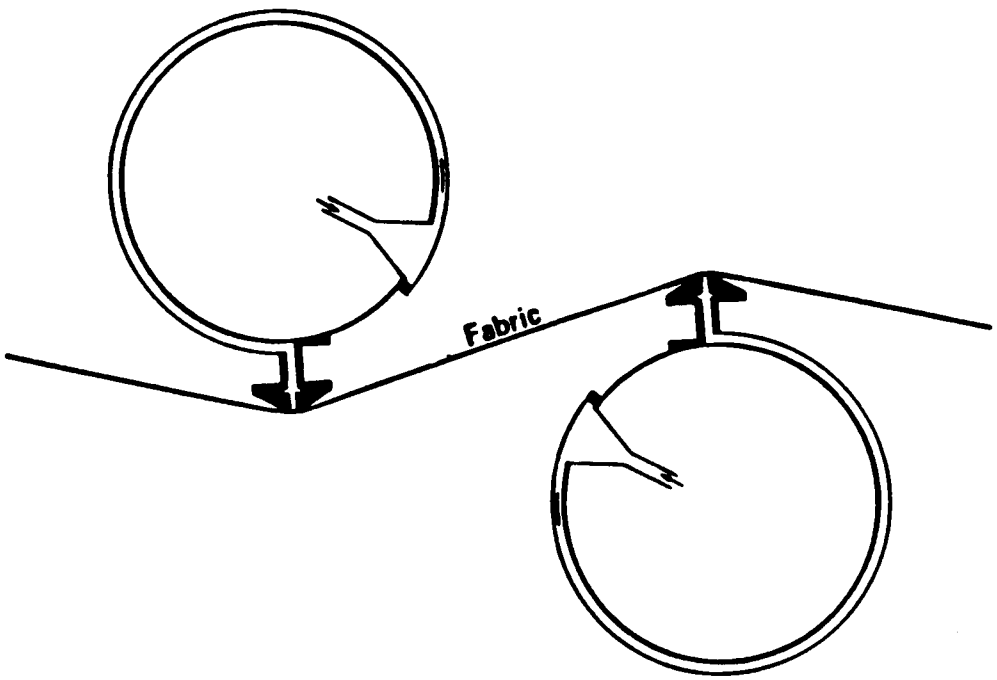


Figure 2. FFT Two-sided Application.

Source: Larry C. Wadsworth, "Foam Application of Durable Press and Fluorochemical Finishes to Cotton Fabrics", Textile Chemist and Colorist, Vol. 15, No. 12, December 1983, p. 241/27.

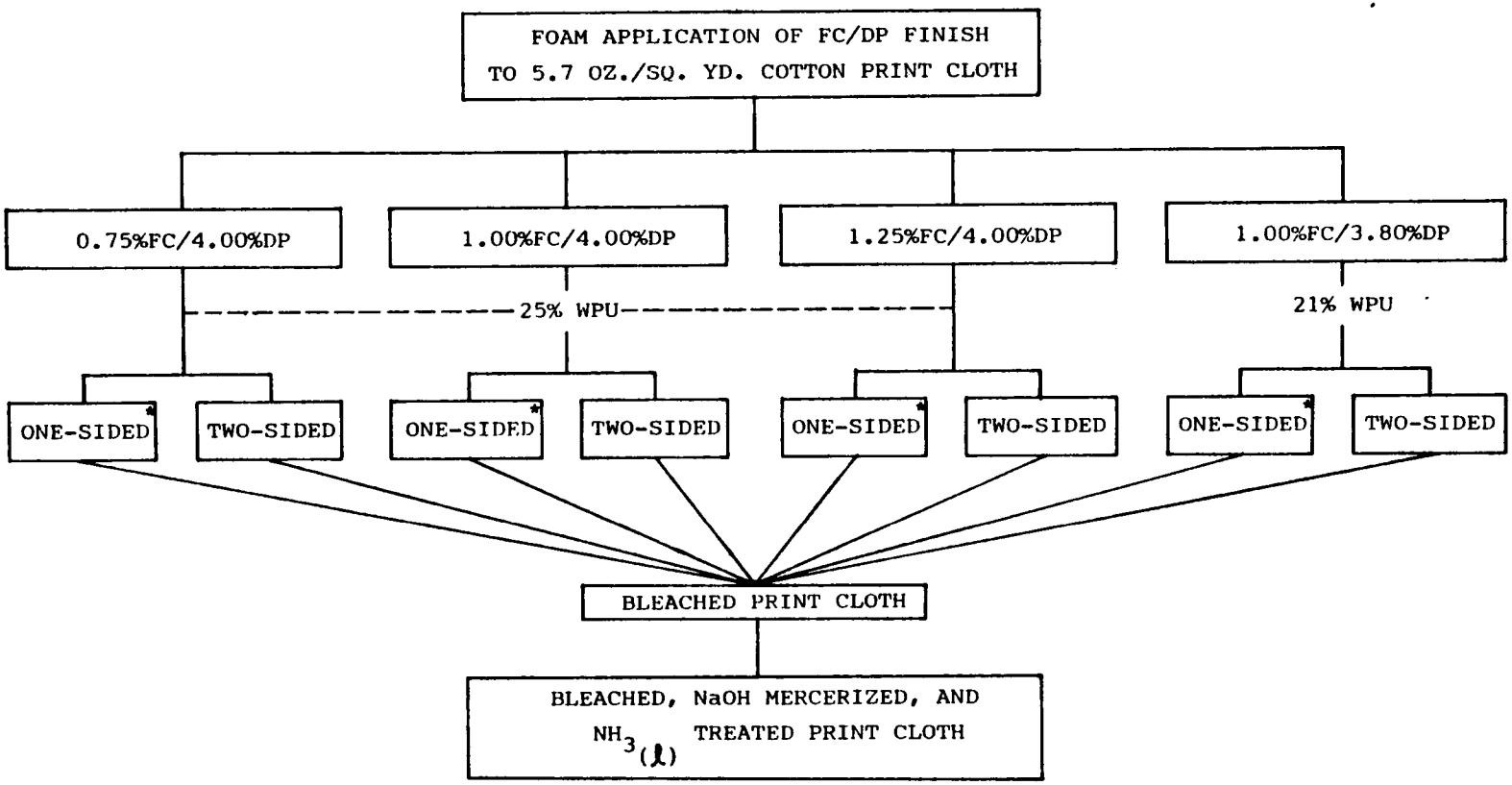
Methods

Experimental Design

The experimental design for the application of fluorochemical (FC) in combination with durable press (DP) resin to bleached (BL) only and bleached-caustic mercerized/ammonia treated (BL-CM/AT) cotton print cloth by one and two-sided foam treatments is shown in Figure 3.

Foam Application

All applications were made using an FFT laboratory applicator at Gaston County Dyeing Machine Corporation. The process unit is equipped with both top and bottom foam applicators (slot widths of 1/8 inch) for application across 18 inches of 20-inch wide fabric. In this research, the bottom applicator was utilized to apply foam to the fabric face with one-sided applications, and both top and bottom applicators were employed for two-sided applications. From 15 to 20 yards of each sample were treated at a fabric linear velocity of 10 yards/minute. Foamer revolution was set to maintain a uniformly consistent foam. Air flow rates were adjusted to provide foam densities of 0.05 gram/cubic centimeter (20/1 blow ratio) to 0.01 gram/cubic centimeter (10/1 blow ratio). This maintained sufficient



Drying: Temp.: 250°F
Time: 1 Min.

Curing: Temp.: 350°F
Time: 1.5 Min.

*One-sided Application to Fabric Face.

Figure 3. Experimental Design for Print Cloth (PC) Treatments.

volume and pressure of foam against the fabric to cover the fabric uniformly. Liquid flow rate and fabric speed were carefully monitored to insure that the desired wet pick-up was obtained. In addition, wet pick-ups were checked by weighing 12 x 15 inch samples of fabric cut immediately after foam application. To determine add-on, the specimen was weighed again after washing the finish out, drying and conditioning.

Fabrics were wound on a paper tube at the wind-up of the foam processing unit, and were then immediately dried on a tenter frame at 250°F for one minute and later cured at 350°F for one and half minutes.

Evaluation of Physical Properties

Performance properties of the finished fabrics were determined by the test methods listed in Table IV. Standard condition (65% $\pm$ 2% relative humidity and 70° $\pm$ 2°F) were used for testing.

Water Repellency. Water repellency was determined by AATCC Test Method 22-1980, "Spray Test" (40). The water spray ratings were utilized to determine the fabric surface repellency to water since pesticide applicators are exposed to pesticides which are distributed in water based aerosols. This test detects the resistance to surface wetting from a vertical spray apparatus. Six specimens 7 x 7 inch (17.8 c 17.8 cm) from each sample

Table IV. Test Methods.

Property	Method
Water Repellency: Spray Test	AATCC 22-1980
Oil Repellency: Drop Rating	AATCC 118-1978
Flex Abrasion	ASTM D1175-71
Wrinkle Recovery Angle	AATCC 66-1978
Appearance of Durable Press Fabrics after 5 Home Launderings	AATCC 124-1982
Tearing Resistance	ASTM D1424-81
One-Inch Raveled Strip Tensile Strength	ASTM D1682-64
Nitrogen Content after 5 Home Launderings	Kjeldahl Method

were sprayed with 250 ml of distilled water: three on face side and three on back side. Each specimen was evaluated by three individuals according to the AATCC Standard Spray Test Rating Chart.

Oil Repellency. Oil repellency was determined by the AATCC Test Method 118-1978, "Hydrocarbon Resistance Test" (41). Oil drop ratings were utilized to determine fabric repellency to oil because many pesticides are also applied in oil-based aerosols. The oil repellency of the fabric was determined by a series of hydrocarbon liquids of decreasing surface tension. The rating reflected the hydrocarbon liquid of the lowest surface tension which did not wet and wick into the fabric surface during 30 seconds. Six 8 x 8 inch (20 x 20 cm) specimens from each sample were evaluated: three on the face side and three on the back side. Each specimen was judged by three individuals.

Abrasion Resistance. Flex abrasion resistance was determined on the Flex Abrasion Tester using ASTM D1175-71 (42) to determine if one-sided foam application of FC and DP finishes resulted in improved abrasion resistance compared to two-sided application, and to determine the effect of FC content. One-sided finished fabrics were tested on both face side and back side respectively. Two-sided finished fabrics were tested

only on the face side. Ten one-inch ravelled strip specimens, 8 x 1.5 inch, were tested: five with their long dimension parallel to the warp and five with their long dimension parallel to the filling. The resistance to flexing and abrasion was measured by subjecting the specimen to unidirectional reciprocal folding and rubbing over a bar under one pound pressure and four pounds tension.

Wrinkle Recovery Angle. AATCC Test Method 66-1978 (43) was employed for measuring the wrinkle recovery to determine if acceptable crease recovery could be obtained by the co-application of FC and DP finishes. Six warp specimens and six filling specimens, 15 x 40 mm, were cut from each sample, and three were creased to the face and the other three to the back of the fabrics. The specimens were creased under a load of 500 grams for 5 min. $\pm$ 5 sec., and the angle of recovery from deformation was measured after 5 min. $\pm$ 5 sec. The warp and filling readings were averaged respectively, and then the averaged warp and filling angles were summed to give a total wrinkle recovery angle.

Appearance of Durable Press Fabrics. The durable press appearance ratings were judged by AATCC Test Method 124-1982, "Appearance of Durable Press Fabrics After Repeated Home Launderings" (44). Durable press ratings

and wrinkle recoveries are important parameters for assessing the permanent press performance of FC and DP finished fabrics. Three 15 x 15 inch (38 x 38 cm) specimens were washed in a Kenmore Automatic Washer, model number 7243.110, under the following conditions: total fabric weight of 4 pounds, 90 grams AATCC Standard Detergent 124, and 12-minute normal wash cycle, at 105 $\pm$ 5°F (41 $\pm$ 3°C) wash temperature. The fabric was tumbled dried at a temperature of 140°F to 160°F (60 to 70°C), in a Kenmore Electric Dryer. The procedure was repeated for a total of 5 cycles. The test specimens were conditioned for 2 hours and then judged for smooth appearance according to AATCC 3-D Durable Press Replicas.

Tearing Strength. Tearing strength was determined by ASTM Method D1424-81 (45), in order to assess the effects of the different finishes and pretreatments on fabric tearing resistance. Five warp specimens and five filling specimens (102 x 75 mm) were cut with a die from each fabric sample. Tearing resistance was determined on a falling pendulum Elmendrof Tear Tester with a capacity of 3,200 grams.

Tensile Strength. One-inch ravelled strip tensile breaking strength was determined by ASTM Designation D1682-64 (46). Five warp specimens and five filling specimens (8 x 1.5 inch) were ravelled to 1 inch width.

Instron Universal Testing Machine (Instron Table Model 1130 Tester) was used under the following conditions: 50-kg load cell, 200 mm/min cross head speed, 50 mm/min chart speed, and 50-kg chart range.

Nitrogen Contents. The percentage of nitrogen was determined by the Kjeldahl method on the fabrics after five home launderings to provide both an indication of the amount of DP finish and the durability to laundering. The test was done by Galbraith Laboratories, Inc., Knoxville, Tennessee.

Dyeing Stain Test

A dye stain test recommended by Turner (47) and later reported by Brown et al. (11) and Wadsworth et al. (12), involving the staining of resin treated fabric with Diphenyl/Brilliant Blue FF Supra (C.I. Direct Blue 1), was utilized to determine the location of resins which acted as a dye resist.

Statistical Analysis

The t-test was used to compare the difference between two sample group means based on the following equation (48):

$$t = \frac{(x_1 - x_2) - \delta}{\sqrt{(n_1 - 1)\, s_1^2 + (n_2 - 1)\, s_2^2}} \sqrt{\frac{n_1 n_2\, (n_1 + n_2 - 2)}{n_1 + n_2}}$$

where

x_1 = mean of the first sample group,

x_2 = mean of the second sample group,

δ = 0,

n_1 = size of the first sample group,

n_2 = size of the second sample group,

s_1 = standard deviation of the first sample group,

s_2 = standard deviation of the second sample group.

CHAPTER IV

RESULTS AND DISCUSSION

Water and Oil Repellency

One Versus Two-Sided Application

Water repellency was determined on both one and two-sided foam finished fabrics at various levels of FC (0.75, 1.00, and 1.25%) and fixed DMDHEU resin (4.00%) add-on to cotton print cloth as shown in Table V. In the application of 0.75, 1.00, and 1.25% FC and 4.00% DMDHEU to bleached (BL) only print cloth, one-sided foam application at 25% wet pick-up resulted in 70.0, 71.1, and 86.7 spray ratings, respectively, on the face treated side and zero spray ratings on the back untreated sides with all three finishes. Under the same conditions, the combination of bleaching-caustic mercerization/ammonia treatment (BL-CM/AT) preparation resulted in 71.1, 80.0, and 90.0 spray ratings, respectively, on the face treated side and also in zero on the back untreated side with each one-sided application. Thus, for both BL and BL-CM/AT fabrics, one-sided foam application yielded at least 70.0 spray ratings on the applied side and zero ratings on the nonapplied side. It is significant that

Table V. Water Repellency of Cotton Print Cloth Finished by FFT.

Preparatory Treatment	Finish Application			Water Repellency Spray Rating[c] (0-100)	
	Add-on/Finish	WPU(%)	Sides[b]	Face	Back
BL[a]	-	-	-	0	0
BL	0.75%FC/4.00%DP	25	1	70.0	0
			2	70.0	70.4
BL	1.00%FC/4.00%DP	25	1	71.1	0
			2	76.7	90.0
BL	1.25%FC/4.00%DP	25	1	86.7	0
			2	90.0	83.3
BL	1.00%FC/3.80%DP	21	1	93.3	0
			2	83.3	93.3

Table V (Continued)

Preparatory Treatment	Finish Application			Water Repellency Spray Rating[c] (0-100)	
	Add-on/Finish	WPU(%)	Sides[b]	Face	Back
BL-CM/AT[d]	-	-	-	0	0
BL-CM/AT	0.75%FC/4.00%DP	25	1	71.1	0
			2	80.0	73.3
BL-CM/AT	1.00%FC/4.00%DP	25	1	80.0	0
			2	82.2	80.0
BL-CM/AT	1.25%FC/4.00%DP	25	1	90.0	0
			2	87.8	90.0
BL-CM/AT	1.00%FC/3.80%DP	21	1	76.7	0
			2	80.0	83.3

[a]BL = desized-scoured-bleached.

[b]Bottom applicator utilized to apply foam to fabric face with one-sided application.

[c]Each value given in this table is the average of nine observations (three judgements on each of three specimens).

[d]BL-CM/AT = desized-scoured-bleached-caustic mercerized-liquid ammonia treated.

differential foam application can result in fabrics clearly hydrophobic on one side and hydrophilic on the opposite side.

At the same wet pick-up and chemical add-on, two-sided foam application appeared to result in better water repellency on both sides compared to the applied side of one-sided application. For example, with two-sided foam application BL fabrics resulted in spray ratings of 70.0, 76.7, and 90.0 on the face side and 70.4, 90.0, and 83.3 on the back side, corresponding to 0.75, 1.00, and 1.25% FC add-ons compared to 70.0, 71.1, and 86.7 on the applied side of the one-sided treatment. The resulting spray ratings of BL-CM/AT fabrics corresponding to 0.75, 1.00, and 1.25% FC were 80.0, 82.2, and 87.7 on the face side and 73.3, 80.0, and 90.0, on the back side compared to 71.1, 80.0, and 90.0, respectively, on the treated side of the one-sided application.

Oil repellency results are given in Table VI. Co-application of various FC add-ons (0.75, 1.00, and 1.25%) and fixed DMDHEU add-on (4.00%) by one-sided foam application resulted in drop ratings of 2.0, 3.3, and 4.0, respectively, on the face treated side of the BL fabrics, and 3.7, 4.0, and 4.3 on the face treated side of the BL-CM/AT fabrics. As with water spray ratings,

Table VI. Oil Repellency of Cotton Print Cloth Finished by FFT.

Preparatory Treatment	Finish Application			Oil Repellency Drop Rating[c] (0-8)[d]	
	Add-on/Finish	WPU(%)	Sides[b]	Face	Back
BL[a]	-	-	-	0	0
BL	0.75%FC/4.00%DP	25	1	2.0	0
			2	3.3	4.0
BL	1.00%FC/4.00%DP	25	1	3.3	0
			2	5.0	5.0
BL	1.25%FC/4.00%DP	25	1	4.0	0
			2	5.0	5.0
BL	1.00%FC/3.80%DP	21	1	3.0	0
			2	4.0	4.0

Table VI (Continued)

Preparatory Treatment	Finish Application			Oil Repellency Drop Rating[c] (0-8)[d]	
	Add-on/Finish	WPU(%)	Sides[b]	Face	Back
BL-CM/AT[e]	-	-	-	0	0
BL-CM/AT	0.75%FC/4.00%DP	25	1	3.7	0
			2	3.3	3.7
BL-CM/AT	1.00%FC/4.00%DP	25	1	4.0	0
			2	4.0	4.0
BL-CM/AT	1.25%FC/4.00%DP	25	1	4.3	0
			2	5.0	5.0
BL-CM/AT	1.00%FC/3.80%DP	21	1	3.0	0
			2	4.0	4.0

[a]See Footnote "a," Table V.

[b]See Footnote "b," Table V.

[c]See Footnote "c," Table V.

[d]Maximum oil drop rating in AATCC Test Method is "8," but the goal in this research was "5."

[e]See Footnote "d," Table V.

the back nonapplied side had no detectable oil repellency resulting in a zero drop rating. With two-sided foam application, BL fabrics yielded 3.3, 5.0, and 5.0 on the face side and 4.0, 5.0, and 5.0 on the back side, and BL-CM/AT fabrics yielded 3.3, 4.0, and 5.0 on the face side and 3.7, 4.0, and 5.0 on the back side. In general, oil repellency followed the same trend as did water repellency. One-sided foam application yielded drop ratings ranging from 3.0 to 5.0 (except for drop ratings of 2.0 on BL fabric at 0.75% FC/4.00% DP add-on) on the face treated side and zero on the back untreated side. Two-sided foam application resulted in drop ratings of 3.0 to 5.0 on both sides, and appeared to be slightly better than the one-sided samples. As with water repellency, these results also support research by Wadsworth et al. (12) that different effects can be achieved on both sides of the fabric with the foam application technique, thereby, giving the finishes much greater flexibility.

Effect of Pretreatment on Repellency

In general, at the same chemical add-on, the combination pretreatment of caustic mercerization/ammonia treatment yielded slightly higher water repellency than a bleaching only pretreatment. For example, at 25% wet pick-up and 1.00% FC/4.00% DP add-on, BL-CM/AT fabric

resulted in spray rating of 80.0 which is higher than BL fabric with spray rating of 71.1. Their ratings were significantly different at the 90% confidence level. As with water repellency, some BL-CM/AT fabric also appeared to result in slightly higher oil repellency than BL fabric. This result could be attributed to increased accessibility of cotton by caustic mercerization and liquid ammonia treatment. The enhancement in cotton accessibility has been reported by Rousselle et al. (49) and later by Wadsworth and co-workers (50). Rousselle et al. (49) found that both caustic and ammonia treatments resulted in increased accessibility as determined by increased moisture regain, iodine sorption, increased accessibility to deuterium exchange, decreased density and higher amorphous fractions shown in X-ray diffraction. Wadsworth et al. (50) also found that the either caustic or ammonia treatments by themselves resulted in this improvement. But additionally the combination of caustic and ammonia treatment with wood pulp was found to result in greatly enhanced moisture regain and increased amorphous fraction, as evidenced by the infrared crystallinity analysis. Thus, the increasing accessibility should theoretically increase uptake of a fluorochemical finish.

Effect of Fluorochemical Level on Repellency

At 25% wet pick-up, the effects of FC content on the water spray rating on face and back sides of cotton print cloth treated by one and two-sided foam applications are compared in Figure 4 and 5. As would be expected, water repellency increased with increasing FC content with two-sided foam application on both sides and on the applied side of the one-sided treatment; whereas, the back nonapplied side with one-sided application remained absorbent. Grajeck and Peterson (34) have reported that because fluorochemicals are expensive, to be commercially feasible they should result in acceptable oil and water repellency at concentration of one percent or less. This study did show that at least spray ratings of 80.0 can be achieved at 1.00% FC/4.00% DMDHEU add-on by one or two-sided foam application on the applied side, while average spray ratings of 90.0 were obtained with 1.25% FC/4.00% DMDHEU. Nevertheless, the higher FC levels evaluated in this study may be justified since these finishes are also being developed for protective apparel for pesticide applicators.

With both one and two-sided foam application, oil repellency on the face treated side increased with an increase in FC levels (Figure 6). The back treated side of the two-sided samples followed a similar trend as

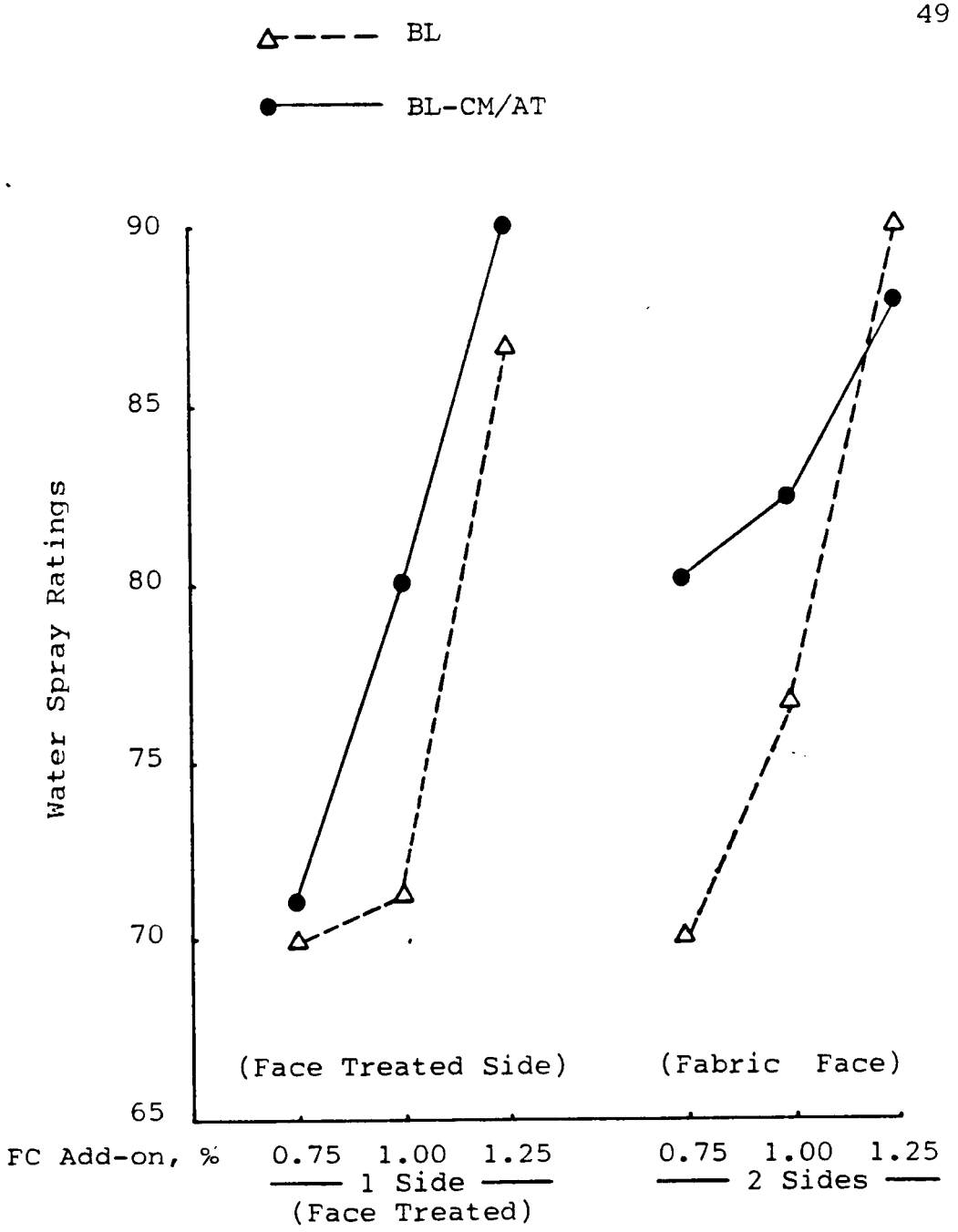


Figure 4. Water Spray Rating of Face Side of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

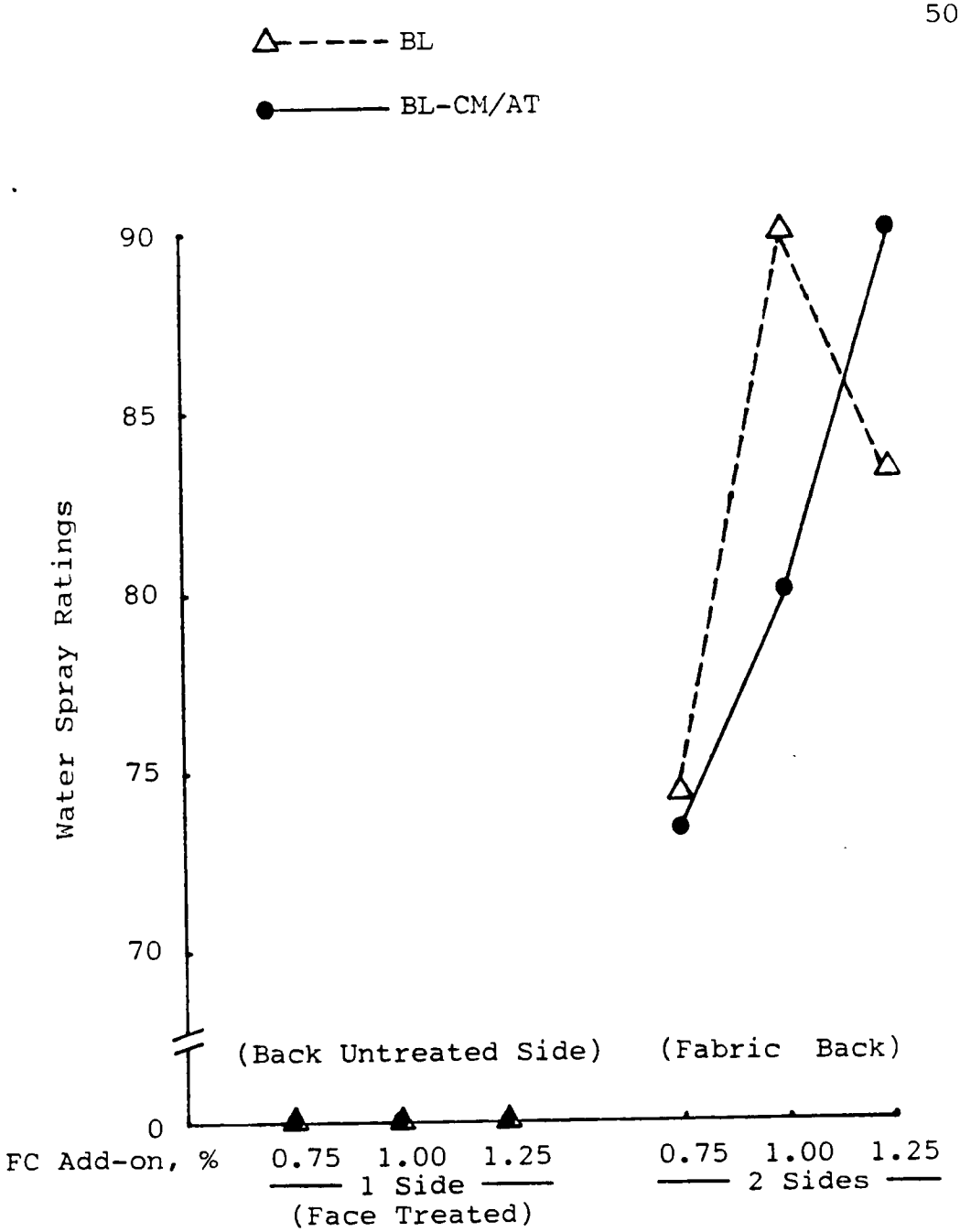


Figure 5. Water Spray Rating of Back Side of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

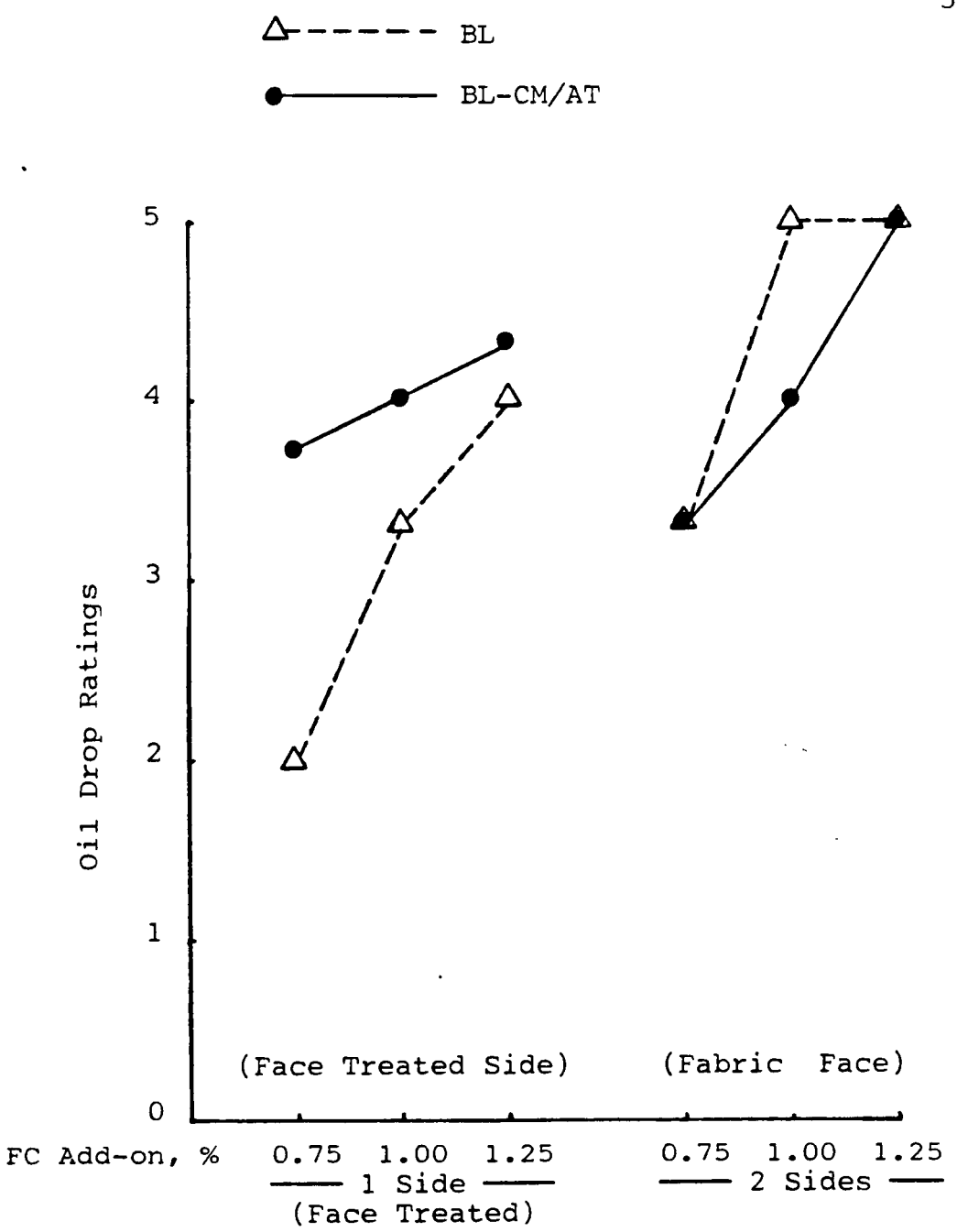


Figure 6. Oil Drop Rating of Face Side of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

shown in Figure 7. The highest oil repellency (5.0 drop ratings) of any of the applications occurred with 1.25% FC/4.00% DMDHEU. Thus, as would be expected, both water and oil repellency increased as the FC content (typical add-on is 0.3 to 0.5%) increased. However, it is recognized that such high levels of FC are too expensive for other than speciality product such as protective apparel for pesticide sprayers. In future work, attempts will be made to decrease the level of FC required by either decreasing the amounts or changing the chemical composition of foaming agents which may be detrimental to repellency, even though they are marketed as "non-rewetting."

Generally, at the same FC add-on, 21% wet pick-up resulted in almost identical water repellency as did 25% wet pick-up (Table V, page 41). Oil repellency showed the same result (Table VI, page 44).

Abrasion Resistance

One Versus Two-Sided Application

Comparison of abrasion resistance on the applied (face) side of the one-sided application to face side of the two-sided foam application are shown in Figure 8 and 9. For BL fabrics, one-sided foam application resulted in higher abrasion resistance in both warp and filling

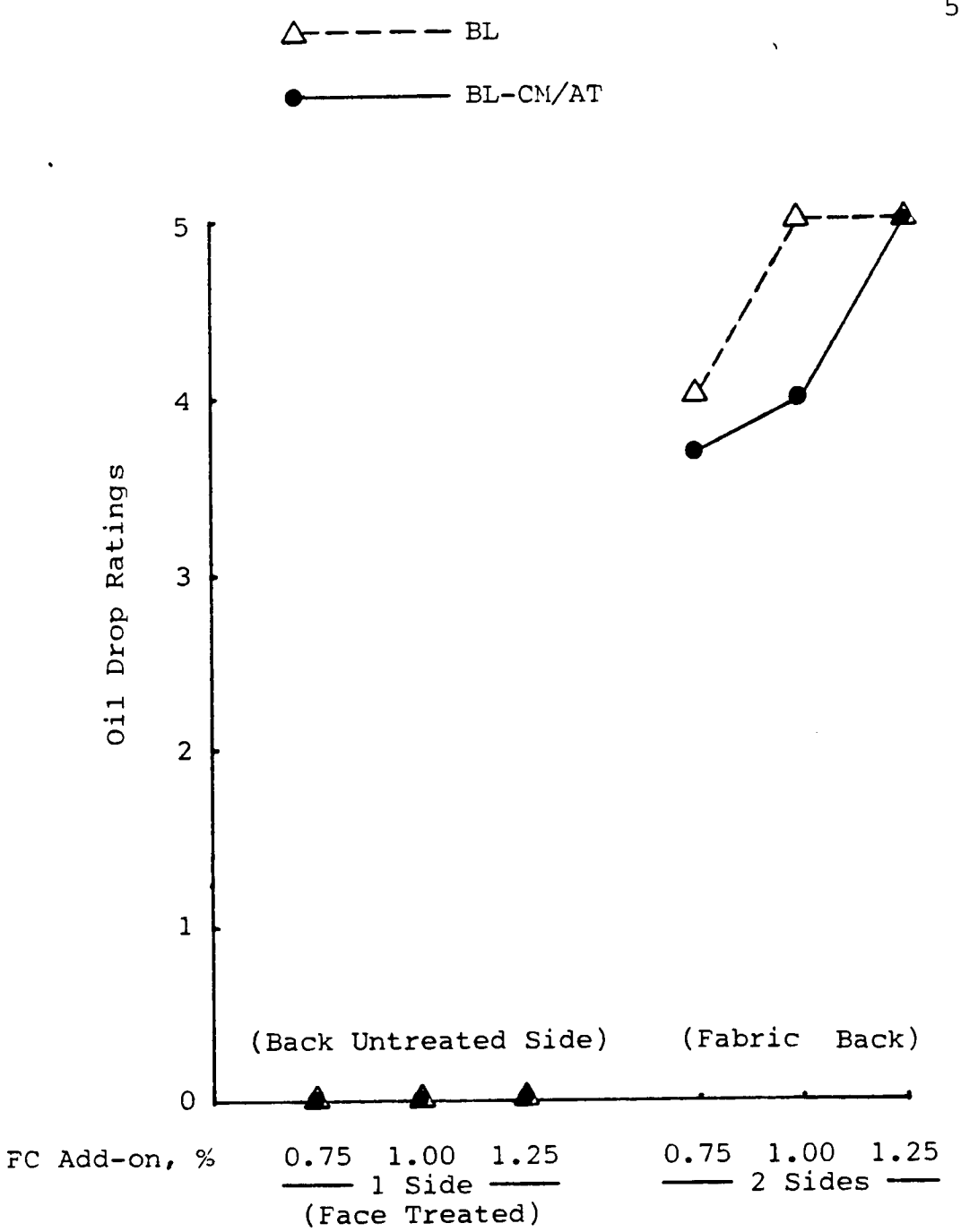


Figure 7. Oil Drop Rating of Back Side of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

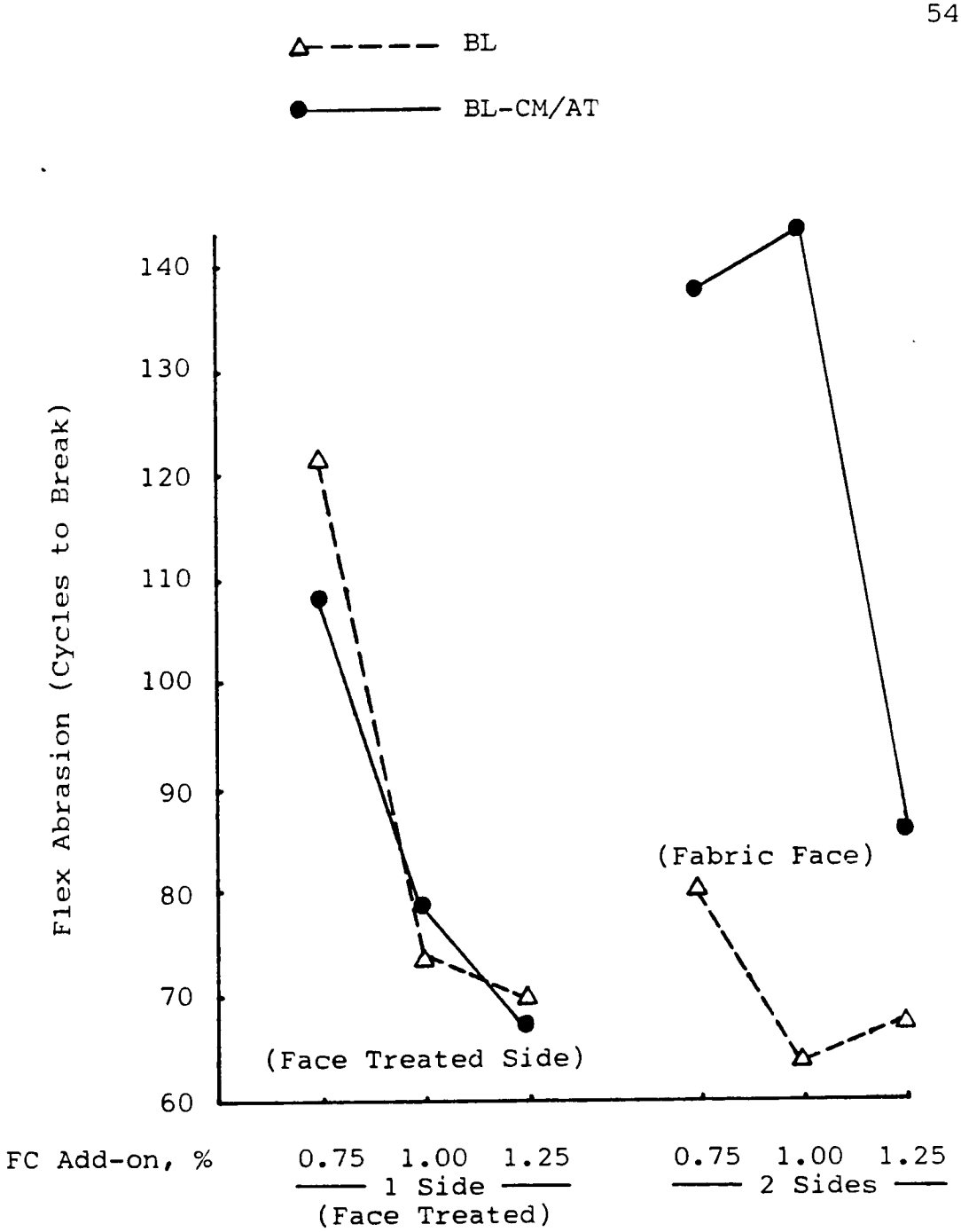


Figure 8. Warp Flex Abrasion of Face Side of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

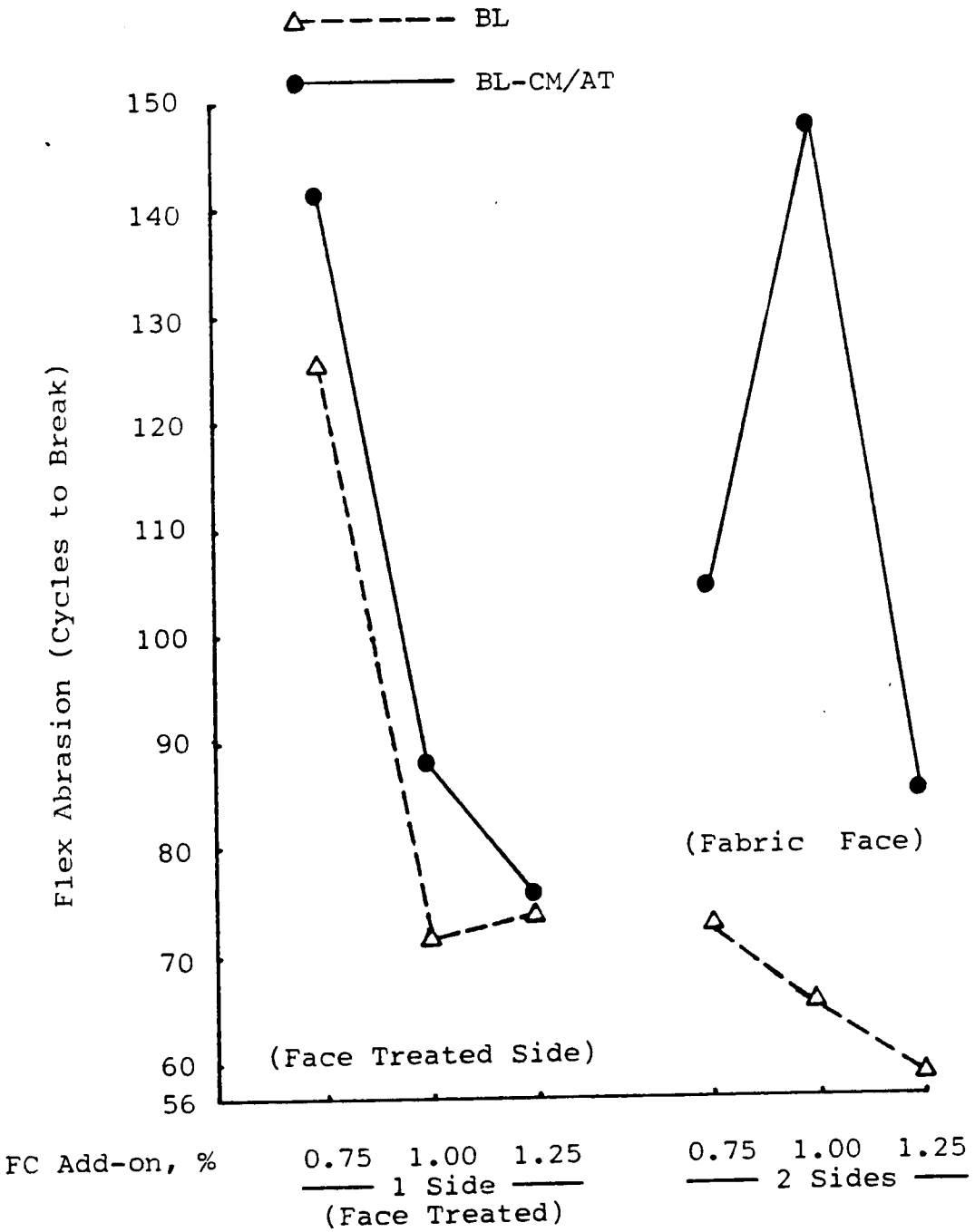


Figure 9. Filling Flex Abrasion of Face Side of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

directions than two-sided foam application over the range of FC levels (0.75, 1.00, and 1.25%) investigated. For example, at 0.75% FC add-on, the difference was found to be statistically significant at the 90% confidence level. However, for BL-CM/AT fabrics, the one-sided foam application resulted in lower abrasion resistance at all three FC levels except that the 0.75% FC add-on resulted in greater abrasion resistance in the filling direction.

Figure 10 illustrates the warp flex abrasion resistance of face and back sides of one-sided foam finished fabrics. The nonapplied side resulted in a greater number of cycles to break than the applied face side at the same FC add-on, although the differences were not statistically significant at 90% confidence level. Generally, one-sided foam application appeared to give better filling abrasion resistance (Figure 11) on the nonapplied side.

Effect of Pretreatment on Abrasion Resistance

The abrasion resistance data from the two different fabric preparations were analyzed statistically. With two-sided foam application, abrasion resistance of BL-CM/AT fabrics were much greater than that of BL fabrics at all FC levels. The differences were significant at least at the 90% confidence level. Generally, with

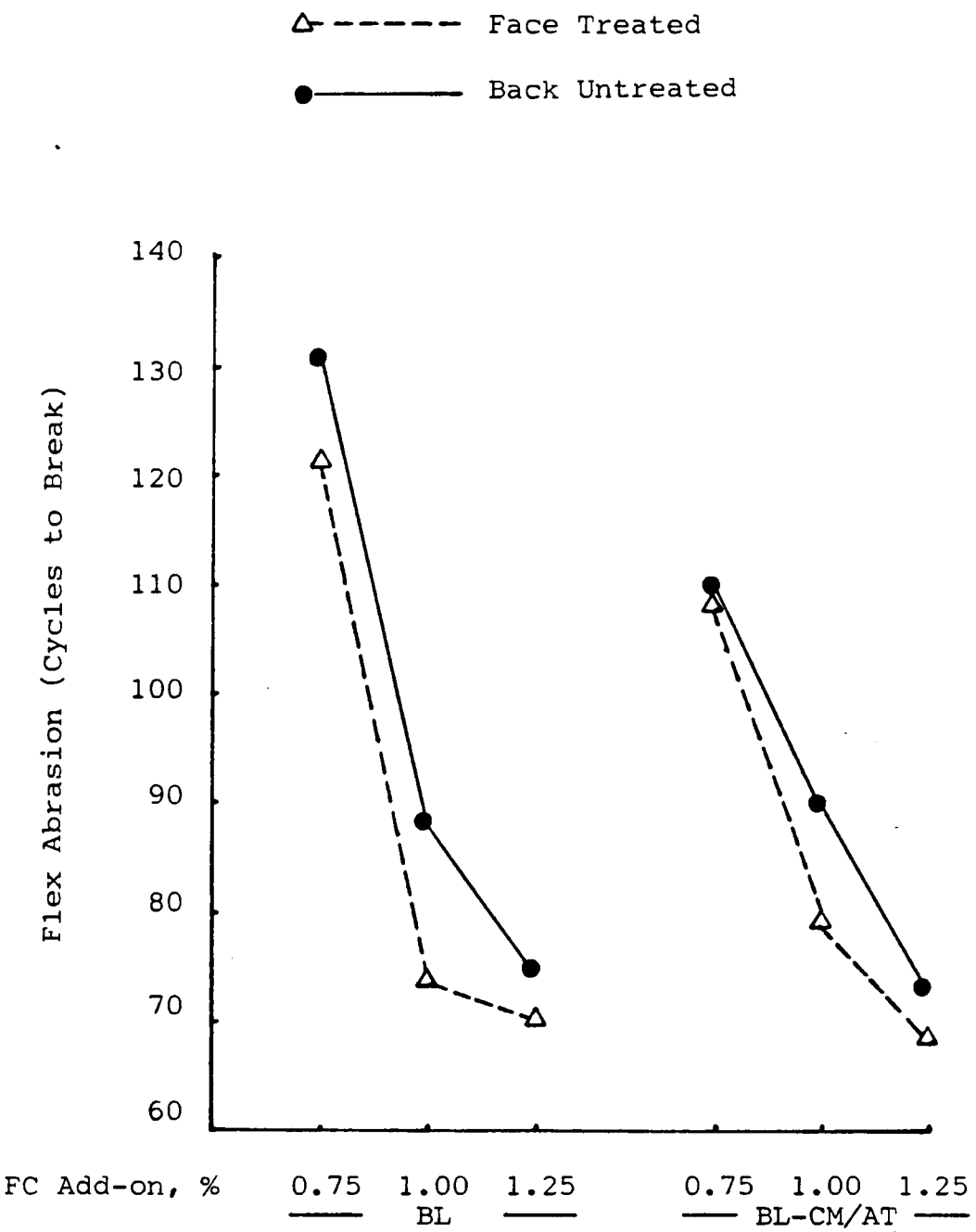


Figure 10. Warp Flex Abrasion of Face and Back Sides of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One-sided FFT Application at 25% WPU.

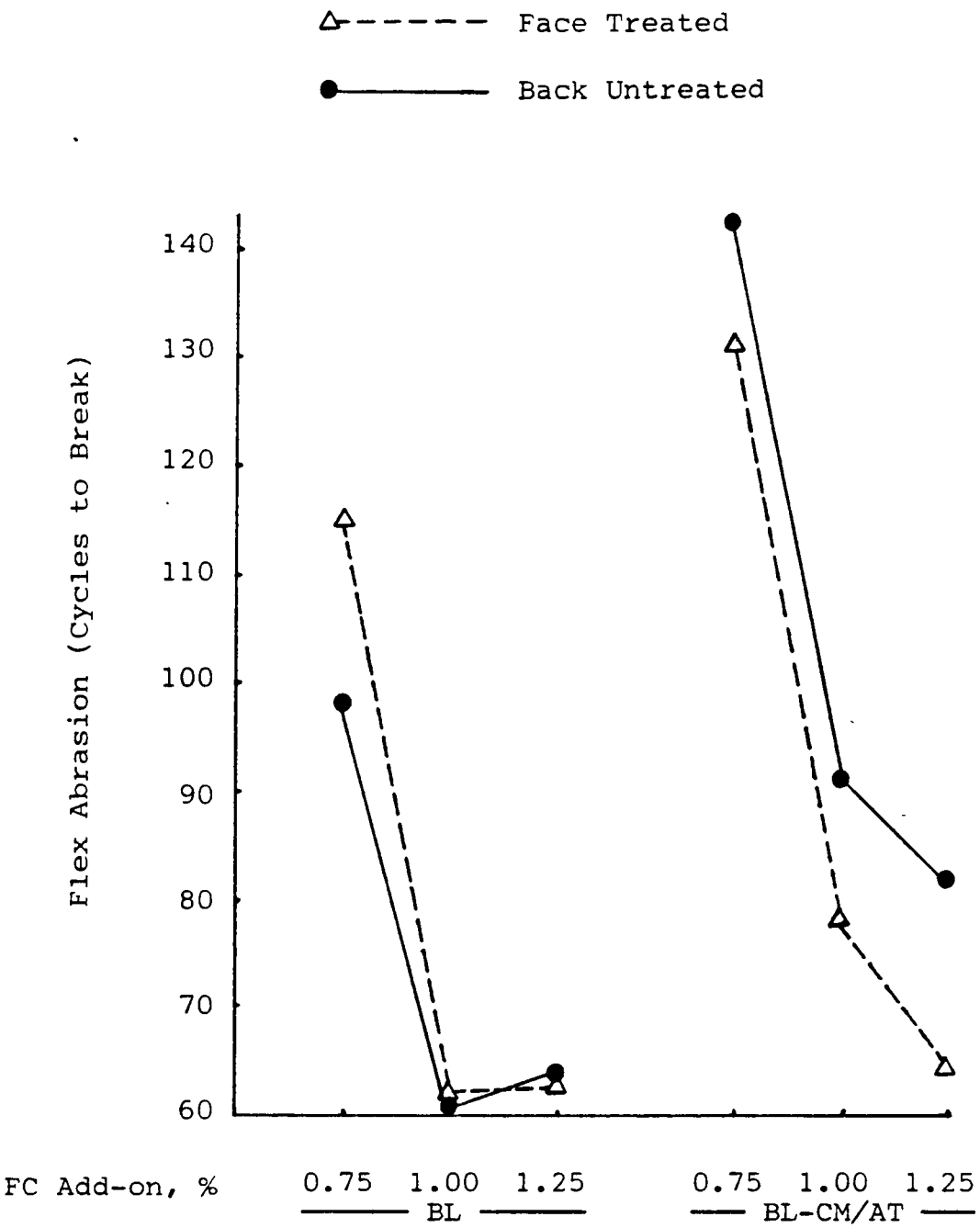


Figure 11. Filling Flex Abrasion of Face and Back Sides of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One-sided FFT Application at 25% WPU.

one-sided foam finishing, higher abrasion resistance was obtained on BL-CM/AT fabrics, although the differences were not statistically significant at the 90% confidence interval. This study did support the findings of Goldstein and Smith (4), and Jones et al. (29) that liquid ammonia would improve abrasion resistance of cotton fabric.

Effect of Fluorochemical Level on Abrasion Resistance

Results of flex abrasion resistance are listed in Table VII. Generally, both warp and filling abrasion resistance decreased with increased FC contents (Figure 8 and 9). With the one-sided foam treatment, the trend of decreased abrasion resistance was obvious and was most dramatic between 0.75 and 1.00% FC add-ons. It decreased from 121.6 to 73.4 cycles to rupture in warp and from 125.4 to 71.6 cycles to rupture in filling directions of the BL fabrics. The BL-CM/AT fabrics also decreased from 107.6 to 77.4 cycles to break in warp and from 141.6 to 87.4 cycles to break in filling directions at 0.75 and 1.00% FC add-ons. However, with two-sided foam application, there were no consistent trends. For example, BL-CM/AT fabrics resulted in the highest warp abrasion resistance of 143.4 cycles to rupture at 1.00% FC add-on, but in 137.8 and 85.4 cycles to rupture respectively at

Table VII. Flex Abrasion of Cotton Print Cloth Finished by FFT.

Preparatory Treatment	Finish Application			Flexing and Abrasion[c] (Repture Cycles)			
				(Face)		(Back)	
	Add-on/Finish	WPU(%)	Sides[b]	Warp	Filling	Warp	Filling
BL[a]	-	-	-	207.0	207.6	-	-
BL	0.75%FC/4.00%DP	25	1	121.6	125.4	130.2	107.6
			2	80.4	72.2	-	-
BL	1.00%FC/4.00%DP	25	1	73.4	71.6	87.0	71.2
			2	63.6	65.5	-	-
BL	1.25%FC/4.00%DP	25	1	69.8	73.0	74.0	74.2
			2	67.8	58.6	-	-
BL	1.00%FC/3.80%DP	21	1	85.6	94.4	93.2	75.8
			2	100.0	69.0	-	-

Table VII (Continued)

Preparatory Treatment	Finish Application			Flexing and Abrasion[c] (Repture Cycles)			
				(Face)		(Back)	
	Add-on/Finish	WPU(%)	Sides[b]	Warp	Filling	Warp	Filling
BL-CM/AT[d]	-	-	-	198.0	206.6	-	-
BL-CM/AT	0.75%FC/4.00%DP	25	1	107.6	141.6	109.4	153.0
			2	137.8	105.0	-	-
BL-CM/AT	1.00%FC/4.00%DP	25	1	77.4	87.4	89.4	100.6
			2	143.4	148.0	-	-
BL-CM/AT	1.25%FC/4.00%DP	25	1	66.8	74.8	73.0	91.6
			2	85.4	85.0	-	-
BL-CM/AT	1.00%FC/3.80%DP	21	1	79.8	92.0	89.8	97.2
			2	102.8	93.2	-	-

[a]See Footnote "a," Table V.

[b]See Footnote "b," Table V.

[c]Each value given in this table is the average of five test specimens.

[d]See Footnote "d," Table V.

at 0.75 and 1.25% FC add-ons. BL-CM/AT fabrics also yielded the best filling abrasion resistance of 148.0 cycles to break at 1.00% FC add-on, and fewer rupture cycles of 105.0 and 85.0 respectively at 0.75 and 1.25% FC levels. Whereas, the best abrasion resistance was obtained at 0.75% FC add-on in both warp and filling directions of the BL fabrics.

As would be expected, Pittman (35) noted that lower surface energy should decrease the coefficient of friction and thereby increase abrasion resistance. However, in this work as the surface energy of the fabric decreased with increasing FC content, abrasion resistance generally decreased. For example, with two-sided foam application, over the FC range of 1.00 to 1.25%, oil repellency on the face treated side of BL-CM/AT fabrics increased from 4.0 to 5.0 in drop ratings which corresponded to surface energies of 26.6 and 25.4 dynes/cm^2, respectively, at 20°C (51). At the same time, the abrasion resistance decreased from 143.4 to 85.4 cycles to rupture in the warp and from 148.0 to 85.0 cycles to rupture in the filling, respectively. A possible explanation may be derived from the findings of Soignet et al. (52) who used electron spectroscopy to determine the location of oil repellency finishes. This technique is sensitive to elemental concentration within the outer

100 Å of the surface of an organic matrix. They found when N-methylolated crosslinking reagents were incorporated into an oil repellent treatment, the fluoro type oil repellent coats the surface, and the cross-linking reagent is distributed evenly throughout the sample. Thus, the co-application of FC and DP finish in the present work may have nonuniformly deposited the FC on the fabric surface. An uneven coating of FC may have actually decreased overall abrasion resistance by producing a FC surface of varying thickness and with localized differences in coefficients of friction. This concept will be further explored in future work. Also, uneven distribution or localized deposition of crosslinks could be responsible.

At the same FC add-on, the 21% wet pick-up resulted in rather higher flex abrasion resistance on BL fabric than did the 25% wet pick-up. For example, at 1.00% FC level with two-sided foam application, the 21% wet pick-up resulted in 57% higher warp abrasion resistance and 5% higher filling abrasion resistance on face side of the BL fabric than did the 25% wet pick-up. However, there is no consistent trend for BL-CM/AT fabric. For example, under the same conditions (1.00% FC add-on and two-sided foam application), the 21% wet pick-up resulted

in 28% lower warp abrasion resistance and 37% lower filling abrasion resistance on the face side of the BL-CM/AT fabric than did the 25% wet pick-up.

Durable Press Performance

One Versus Two-Sided Applications

Wrinkle recoveries are summerized in Table VIII. With one-sided foam application, BL fabric yielded wrinkle recovery angles of 299.4, 295.0, and 295.7 degrees on the face applied side and 292.4, 293.4, and 287.6 degrees on the back nonapplied side, corresponding to 0.75, 1.00, and 1.25% FC and a constant 4.00% DMDHEU add-on at 25% wet pick-up. The BL-CM/AT fabric resulted in 298.3, 299.7, and 295.7 degrees on the face treated side, and 290.3, 287.6, and 291.0 degrees on the back untreated side. Thus, at 25% wet pick-up, there is no significant difference (even at the 90% confidence level) in wrinkle recoveries from the face applied side to the back nonapplied side of the one-sided foam application. And as shown in Figure 12, wrinkle recoveries between one and two-sided foam application techniques were not statistically different even at the 90% confidence level. This supports the findings by Wadsworth et al (12) that acceptable wrinkle recoveries could be obtained even though DP resin was applied from only one side of the

Table VIII. Wrinkle Recovery of Cotton Print Cloth Finished by FFT.

Preparatory Treatment	Finish Application			N(%)	Wrinkle Recovery Angle[c] (^{o})(W+F)	
	Add-on/Finish	WPU(%)	Sides[b]		Face	Back
BL[a]	-	-	-	0.01	153.3	149.0
BL	0.75%FC/4.00%DP	25	1	0.41	299.4	292.4
			2	0.54	298.4	295.0
BL	1.00%FC/4.00%DP	25	1	0.40	295.0	293.4
			2	0.60	292.7	293.3
BL	1.25%FC/4.00%DP	25	1	0.53	295.7	287.6
			2	0.57	296.0	291.7
BL	1.00%FC/3.80%DP	21	1	0.47	295.4	281.7
			2	0.71	297.0	289.0

Table VIII (Continued)

Preparatory Treatment	Finish Application				Wrinkle Recovery Angle[c] (°)(W+F)	
	Add-on/Finish	WPU(%)	Sides[b]	N(%)	Face	Back
BL-CM/AT[d]	-	-	-	0.02	204.0	209.3
BL-CM/AT	0.75% FC/4.00%DP	25	1	0.47	298.3	290.3
			2	0.44	294.7	294.3
BL-CM/AT	1.00%FC/4.00%DP	25	1	0.50	299.7	287.6
			2	0.43	289.7	284.0
BL-CM/AT	1.25%FC/4.00%DP	25	1	0.53	295.7	291.0
			2	0.54	301.0	294.7
BL-CM/AT	1.00%FC/3.80%DP	21	1	0.61	303.0	296.3
			2	0.55	303.3	297.6

[a]See Footnote "a," Table V.

[b]See Footnote "b," Table V.

[c]Three warp and three filling readings were averaged respectively, and the warp and filling angles were wummed to give a total wrinkle recovery angle.

[d]See Footnote "d," Table V.

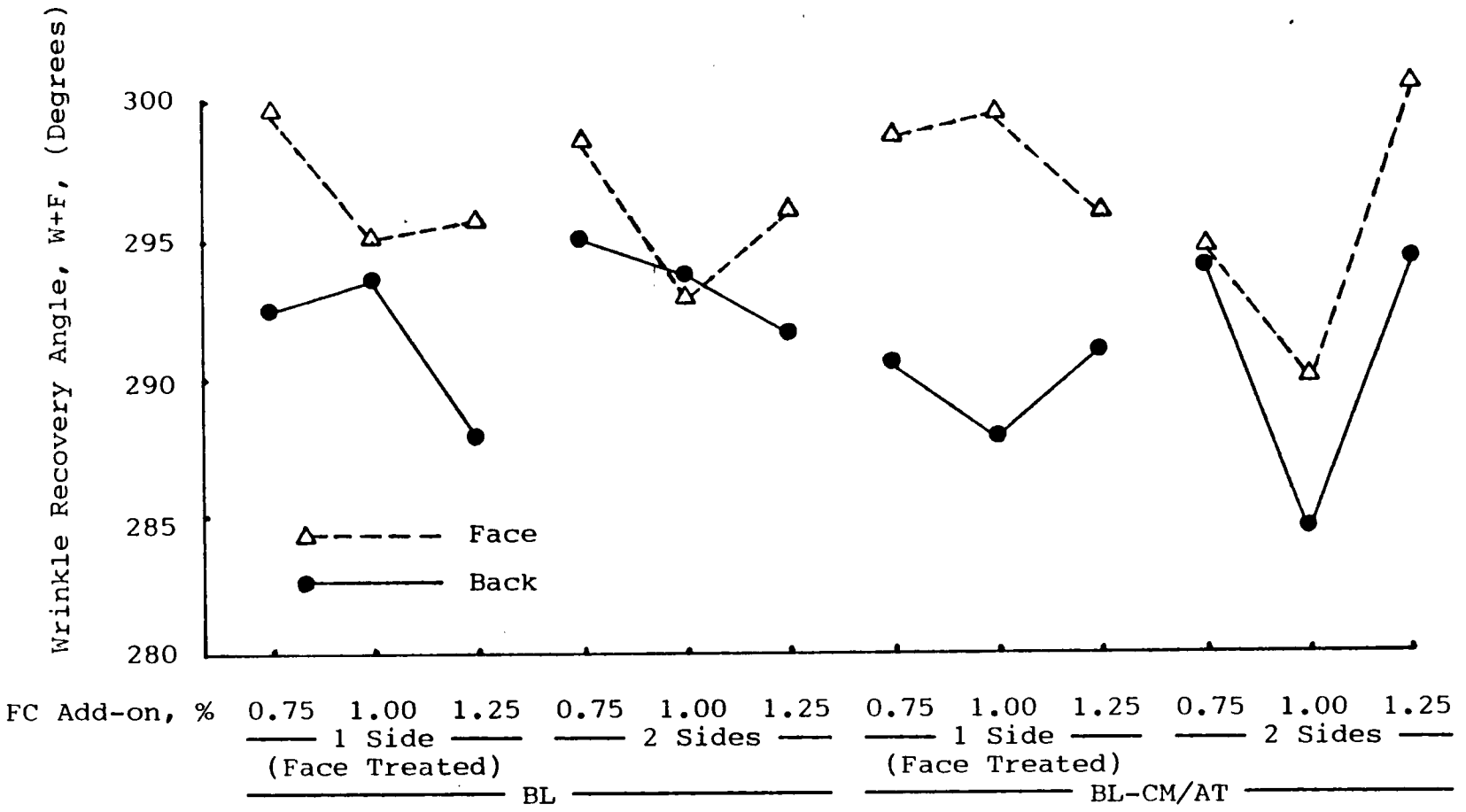


Figure 12. Wrinkle Recovery Angle of Face and Back Sides of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

fabric. Thus, the nonapplied side should theoretically be less harsh and more absorbent, and should be more comfortable than a conventionally finished fabric.

Dye stain test results of foam finished BL and BL-CM/AT fabrics are shown in Figure 13 and 14, respectively. For both BL and BL-CM/AT fabrics, one-sided foam application resulted in light staining on the face treated side and much darker staining on the back nonapplied side. Although the back sides were darker, both fabrics had a denim type appearance on the back untreated sides possibly resulting from some penetration through the pores in the fabric. With one-sided foam application, the 21% wet pick-up resulted in much more difference in staining between face applied side and back nonapplied side than did the 25% wet pick-up. Thus, lower wet pick-up showed less penetration through the fabric, thereby imparting the untreated side with darker staining. The same trend was also found in wrinkle recoveries (Table VIII) and durable press ratings (Table IX). With two-sided foam applications, both fabrics resulted in almost the same dye staining appearance on both sides.

Acceptable DP appearance ratings (over 3.5 ratings) were obtained with one and two-sided foam application of 0.75, 1.00, and 1.25% FC and 4.00% DMDHEU to cotton print

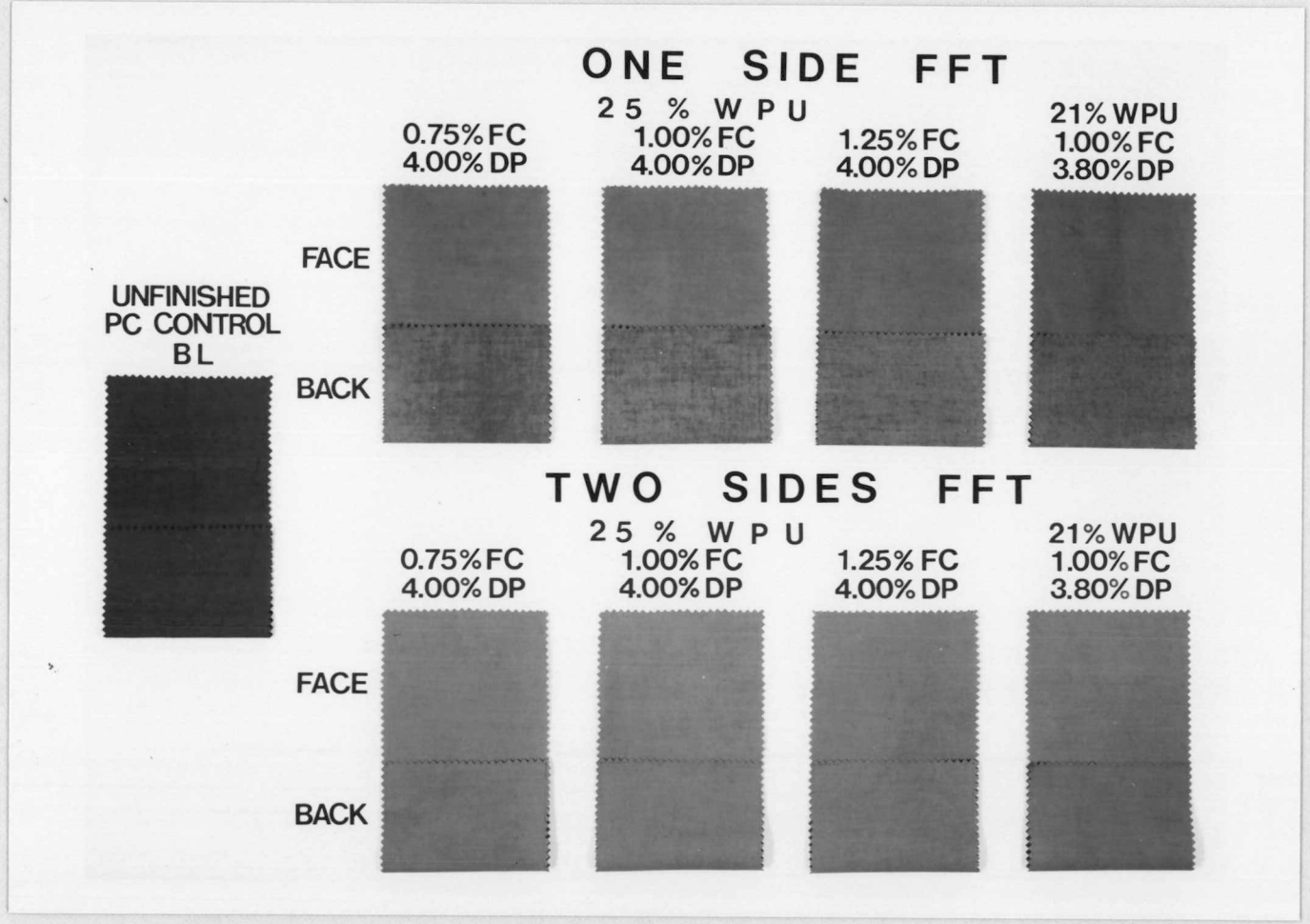


Figure 13. Comparison of Direct Dye Staining of Unfinished Control and FC/DP Finished BL Only Fabric by One and Two-sided FFT.

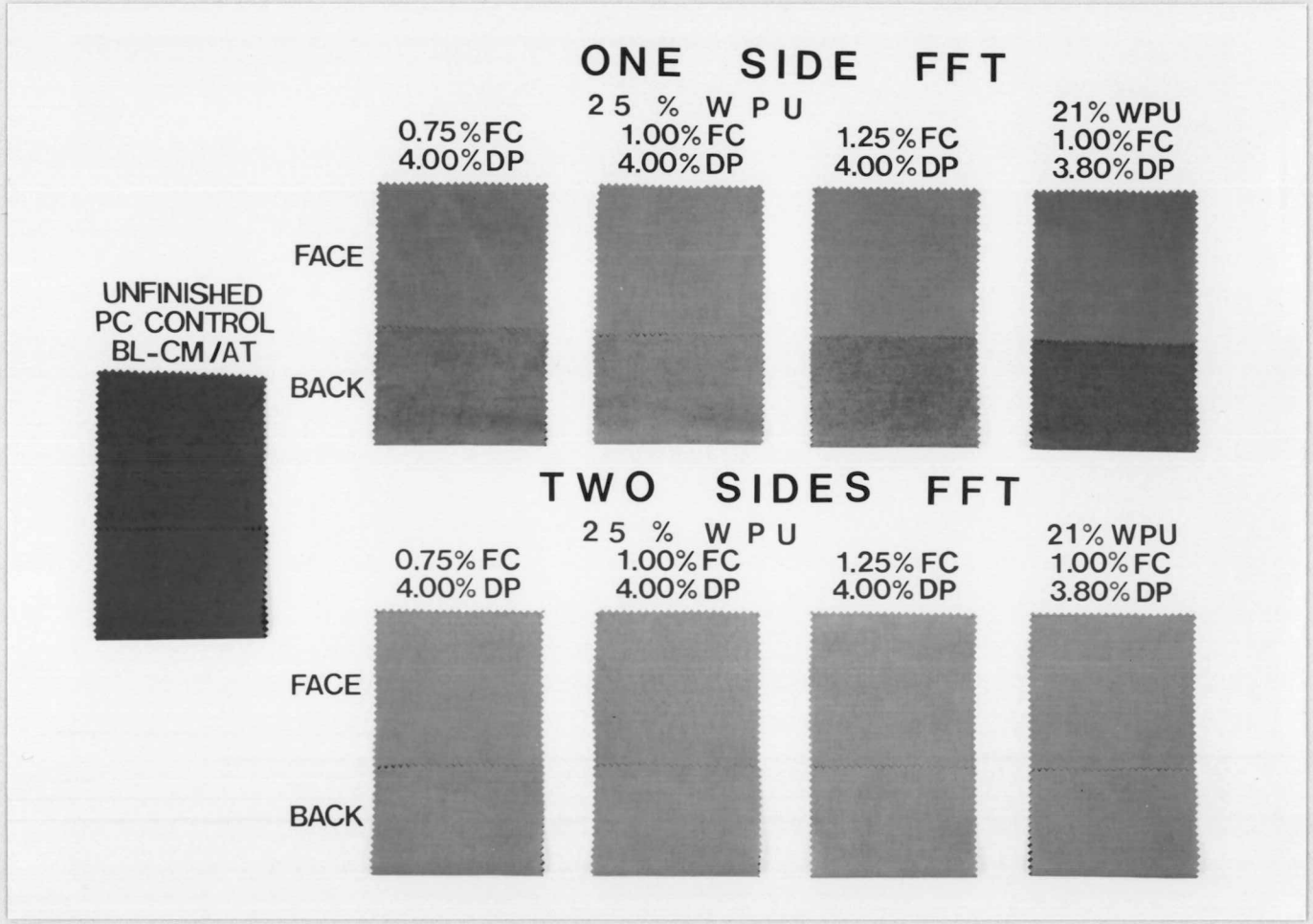


Figure 14. Comparison of Direct Dye Staining of Unfinished Control and FC/DP Finished BL-CM/AT Fabric by One and Two-sided FFT.

Table IX. Durable Press of Cotton Print Cloth Finished by FFT.

Preparatory Treatment	Finish Application			N(%)	Durable Press Rating[c]	
	Add-on/Finish	WPU(%)	Sides[b]		Face	Back
BL[a]	-	-	-	0.01	1.4	1.2
BL	0.75%FC/4.00%DP	25	1	0.41	4.3	4.3
			2	0.54	3.9	4.0
BL	1.00%FC/4.00%DP	25	1	0.40	4.3	4.3
			2	0.60	3.9	3.6
BL	1.25%FC/4.00%DP	25	1	0.53	4.2	4.2
			2	0.57	4.0	4.0
BL	1.00%FC/3.80%DP	21	1	0.47	4.7	4.1
			2	0.71	4.0	4.0

Table IX (Continued)

Preparatory Treatment	Finish Application			N(%)	Durable Press Rating[c]	
	Add-on/Finish	WPU(%)	Sides[b]		Face	Back
BL-CM/AT[d]	-	-	-	0.02	2.1	2.4
BL-CM/AT	0.75%FC/4.00%DP	25	1	0.47	4.9	4.7
			2	0.44	4.0	3.9
BL-CM/AT	1.00%FC/4.00%DP	25	1	0.50	4.3	4.0
			2	0.43	4.0	4.1
BL-CM/AT	1.25%FC/4.00%DP	25	1	0.53	3.8	3.8
			2	0.54	4.0	3.9
BL-CM/AT	1.00%FC/3.80%DP	21	1	0.61	4.3	4.1
			2	0.55	3.8	3.7

[a]See Footnote "a," Table V.

[b]See Footnote "b," Table V.

[c]See Footnote "c," Table V.

[d]See Footnote "d," Table V.

cloth (Table IX). As with wrinkle recoveries, the DP ratings were almost identical from face to back for both one and two-sided foam applications (Figure 15).

Effect of Pretreatment on Durable Press Performance

The differences in wrinkle recoveries between the two pretreatments are also compared in Figure 12, page 67. Surprisingly, there was no significant difference in wrinkle recoveries between BL and BL-CM/AT fabrics even though nitrogen contents after five home launderings indicated an interesting trend. In every case, the two-sided application resulted in higher nitrogen content with the BL only fabrics; whereas, one-sided application resulted in almost always higher nitrogen content with the BL-CM/AT pretreatment. Since wrinkle recoveries obtained by both application techniques on the two fabrics were already quite high (i.e. greater than 285.0 degrees of warp + filling), the differences in nitrogen contents may not have been reflected as significant differences in wrinkle recoveries. Although DP ratings differed more between BL and BL-CM/AT fabrics (some samples were significantly different at 90% confidence level), there was also no notable relationship among DP ratings, resin fixations and pretreatments.

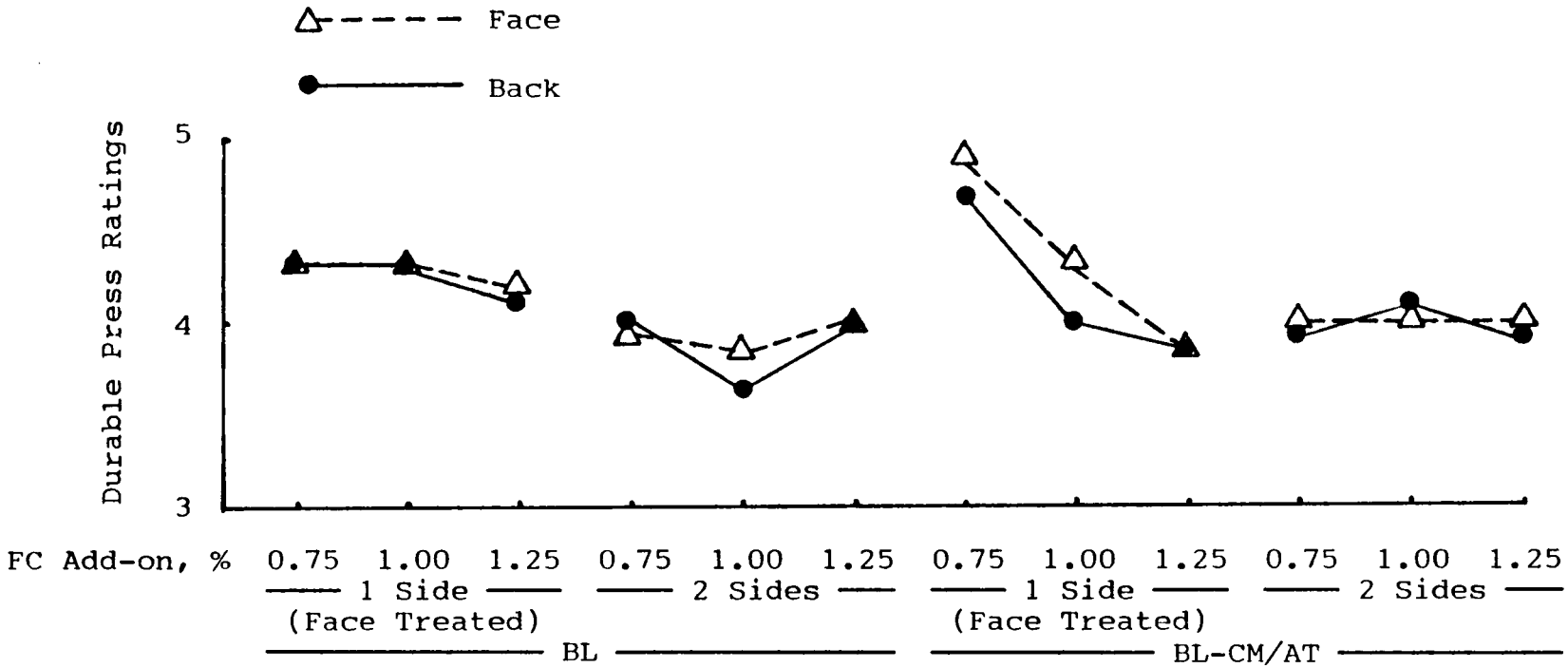


Figure 15. Durable Press Rating of Face and Back Sides of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

Effect of Fluorochemical Level on Durable Press Performance

As observed in Figure 12 (page 67), there was no notable relationship between wrinkle recoveries and FC contents. Neither was there any relationship between DP ratings and FC contents (Figure 15). These results are consistent with previous findings that both acceptable repellency and durable press properties can be achieved by co-application of FC and DP chemicals.

Strength Properties

One Versus Two-Sided Application

Tearing strength of foam finished fabrics are shown in Table X. Generally, one-sided foam application, for both BL and BL-CM/AT fabrics, resulted in significantly higher tearing strength (at 90% confidence level) than with a two-sided foam application. For example, at 25% wet pick-up and 0.75% FC/4.00% DMDHEU add-on, BL fabric with one-sided foam application resulted in a tearing strength of 1,040 grams in the warp and 576 grams in the filling directions. Two-sided foam treatment resulted in a lower tearing strength of 972 grams in the warp and 490 grams in the filling. Under the same conditions with BL-CM/AT fabric, one-sided foam application

Table X. Tearing Strength of Cotton Print Cloth Finished by FFT.

Preparatory Treatment	Finish Application			Tearing Strength[c] (g.)	
	Add-on/Finish	WPU(%)	Sides[b]	Warp	Filling
BL[a]	-	-	-	1734	1194
BL	0.75%FC/4.00%DP	25	1	1040	576
			2	972	490
BL	1.00%FC/4.00%DP	25	1	1020	640
			2	896	564
BL	1.25%FC/4.00%DP	25	1	982	620
			2	940	526
BL	1.00%FC/3.80%DP	21	1	1038	570
			2	1030	534

Table X (Continued)

Preparatory Treatment	Finish Application			Tearing Strength[c] (g.)	
	Add-on/Finish	WPU(%)	Sides[b]	Warp	Filling
BL-CM/AT[d]	-	-	-	1766	1274
BL-CM/AT	0.75%FC/4.00%DP	25	1	1356	684
			2	1250	592
BL-CM/AT	1.00%FC/4.00%DP	25	1	1266	668
			2	1294	674
BL-CM/AT	1.25%FC/4.00%DP	25	1	1196	592
			2	1228	566
BL-CM/AT	1.00%FC/3.80%DP	21	1	1274	612
			2	1254	526

[a]See Footnote "a," Table V.

[b]See Footnote "b," Table V.

[c]See Footnote "c," Table VII.

[d]See Footnote "d," Table V.

resulted in a tearing strength of 1,350 grams in the warp and 684 grams in the filling, and two-sided foam application again resulted in a lower tearing strength of 1,250 grams in the warp and 592 grams in the filling direction.

However, as shown in Table XI, one-sided foam treatment generally resulted in slightly lower tensile strength (not statistically significant at the 90% confidence level) than did the two-sided foam treatment. For example, at 25% wet pick-up and 1.00% FC/4.00% DMDHEU add-on to BL fabric, one-sided foam application resulted in a tensile strength of 16.0 kilograms in the warp and 12.2 kilograms in the filling directions compared to 17.3 and 12.6 kilograms in the warp and filling directions obtained by two-sided foam application. Similar results were obtained with the BL-CM/AT.

Effect of Pretreatment on Strength Properties

As was found by Wadsworth et al. (12), the combination pretreatment of caustic mercerization and ammonia treatment appreciably improved the tearing strength. At each chemical add-on, the BL-CM/AT fabric resulted in significantly higher warp tearing strength at the 90% confidence level than BL fabric (Figure 16). For example, with two-sided foam application at 25% wet pick-up and

Table XI. Tensile Strength of Cotton Print Cloth Finished by FFT.

Preparatory Treatment	Finish Application			Tensile Strength I Inch Strip[c] (Kg.)	
	Add-on/Finish	WPU(%)	Sides[b]	Warp	Filling
BL[a]	-	-	-	40.1	31.2
BL	0.75%FC/4.00%DP	25	1	16.5	12.9
			2	17.9	12.1
BL	1.00%FC/4.00%DP	25	1	16.0	12.2
			2	17.3	12.6
BL	1.25%FC/4.00%DP	25	1	15.1	11.9
			2	16.7	13.0
BL	1.00%FC/3.80%DP	21	1	17.2	11.4
			2	17.0	9.4

Table XI (Continued)

Preparatory Treatment	Finish Application			Tensile Strength 1 Inch Strip[c] (Kg.)	
	Add-on/Finish	WPU(%)	Sides[b]	Warp	Filling
BL-CM/AT[d]	-	-	-	39.2	32.0
BL-CM/AT	0.75%FC/4.00%DP	25	1	15.4	11.3
			2	18.6	12.3
BL-CM/AT	1.00%FC/4.00%DP	25	1	13.9	14.0
			2	20.3	14.3
BL-CM/AT	1.25%FC/4.00%DP	25	1	16.9	12.2
			2	17.6	12.6
BL-CM/AT	1.00%FC/3.80%DP	21	1	16.7	13.1
			2	15.0	9.9

[a]See Footnote "a," Table V.

[b]See Footnote "b," Table V.

[c]See Footnote "c," Table V.

[d]See Footnote "d," Table V.

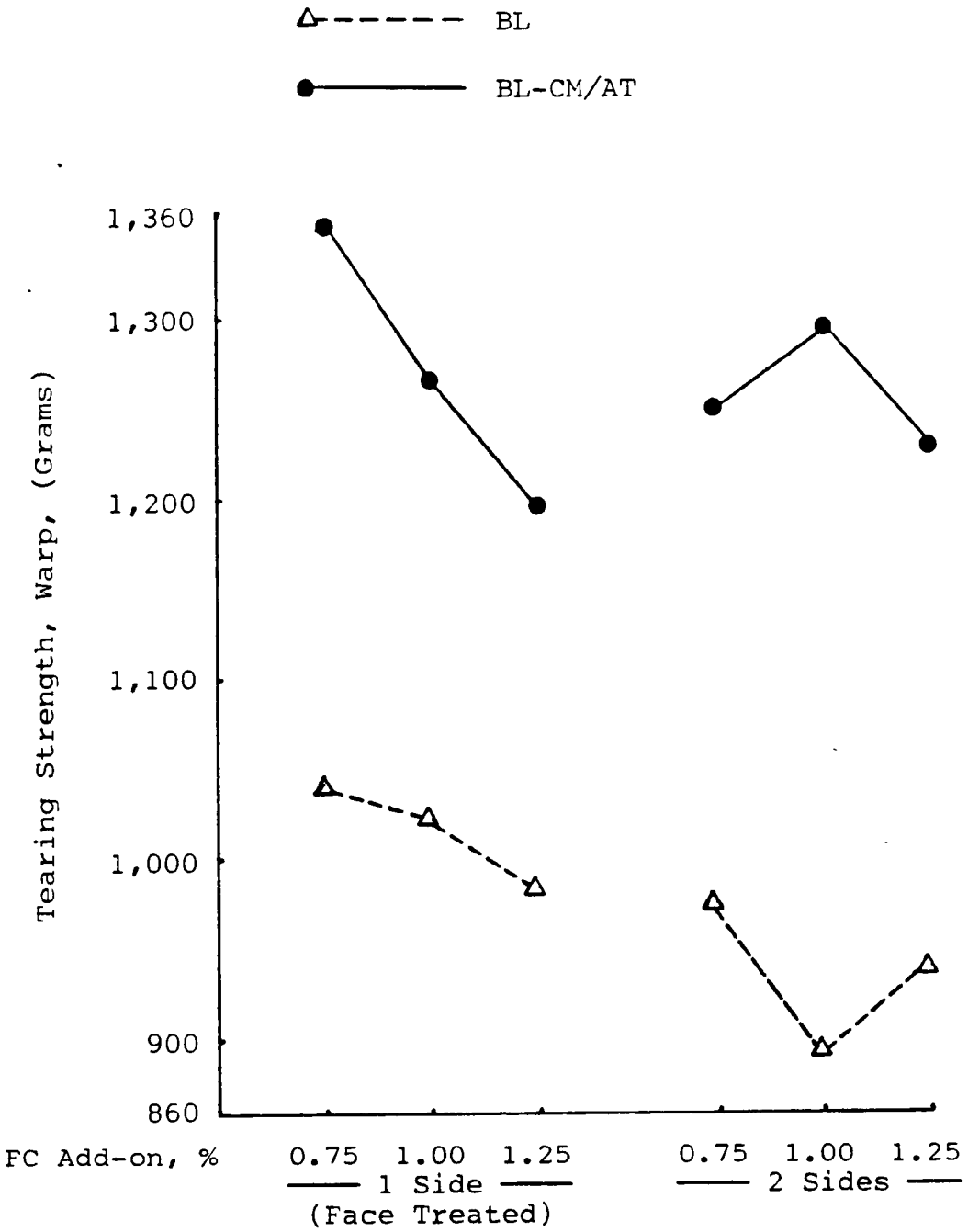


Figure 16. Warp Tearing Strength of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

1.00% FC/4.00% DMDHEU add-on, BL-CM/AT fabric had 44% higher warp tearing strength than did BL fabric. Likewise, the filling tearing strength of the BL-CM/AT pretreated fabric was 20% higher than the BL only pretreated fabric (Figure 17). With one-sided foam application at 25% wet pick-up and 1.00% FC/4.00% DMDHEU add-on, the BL-CM/AT fabric resulted in 24% higher tearing strength in warp, but in only 4% higher tearing strength in the filling direction.

As observed with tearing strength, tensile strength was also improved by the combination pretreatment of caustic mercerization and ammonia treatment (Figures 18 and 19). For example, at 25% wet pick-up and 1.00% FC/4.00% DMDHEU add-on, BL-CM/AT fabric finished by two-sided application resulted in 17% higher warp tensile strength and 13% higher filling strength than did BL fabric. But with one-sided foam application, BL-CM/AT fabric resulted in 13% lower warp tensile strength and 15% higher filling tensile strength than did BL fabric. Nevertheless, both tearing and tensile strengths were statistically improved by the combination pretreatment of caustic mercerization and ammonia treatment.

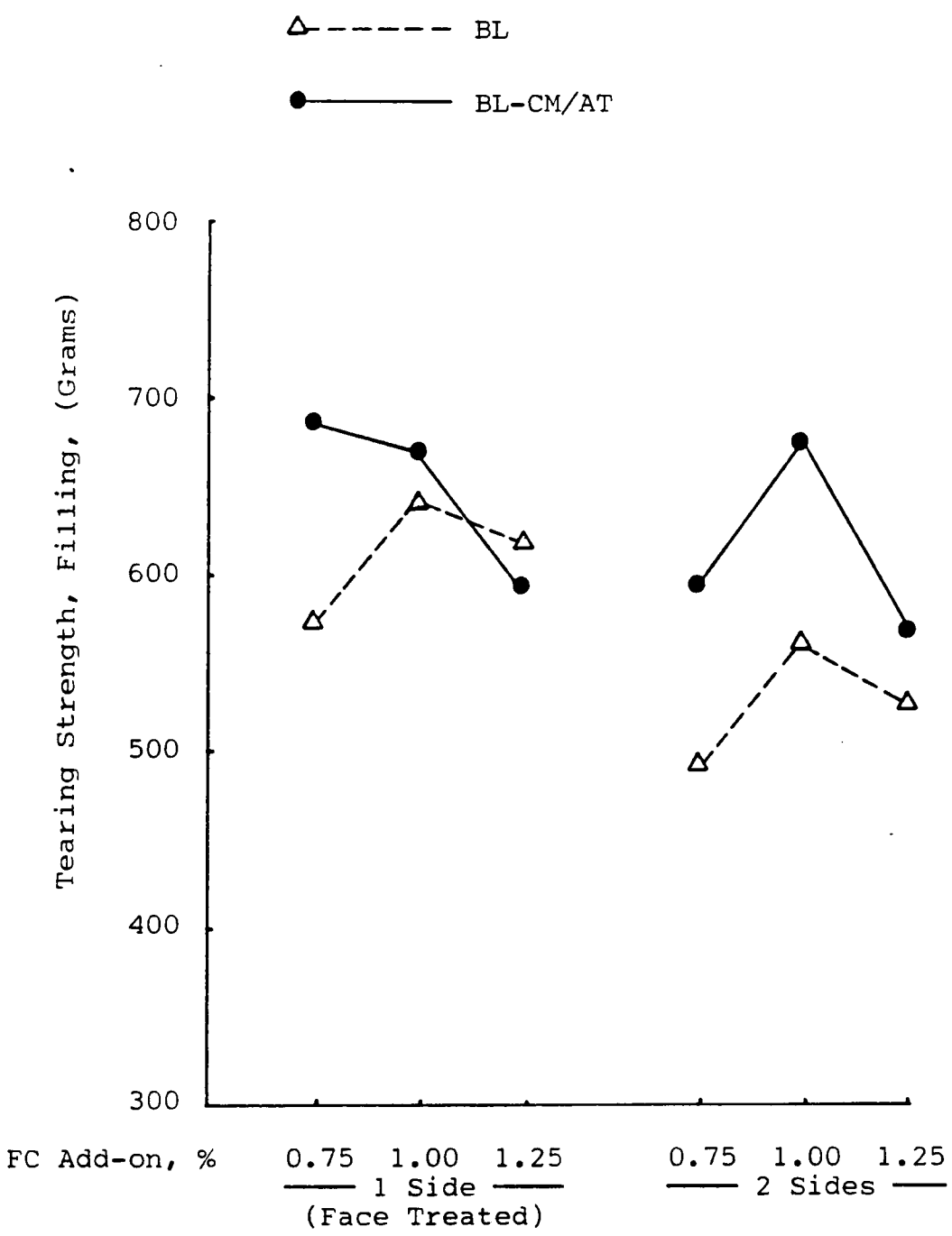


Figure 17. Filling Tearing Strength of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

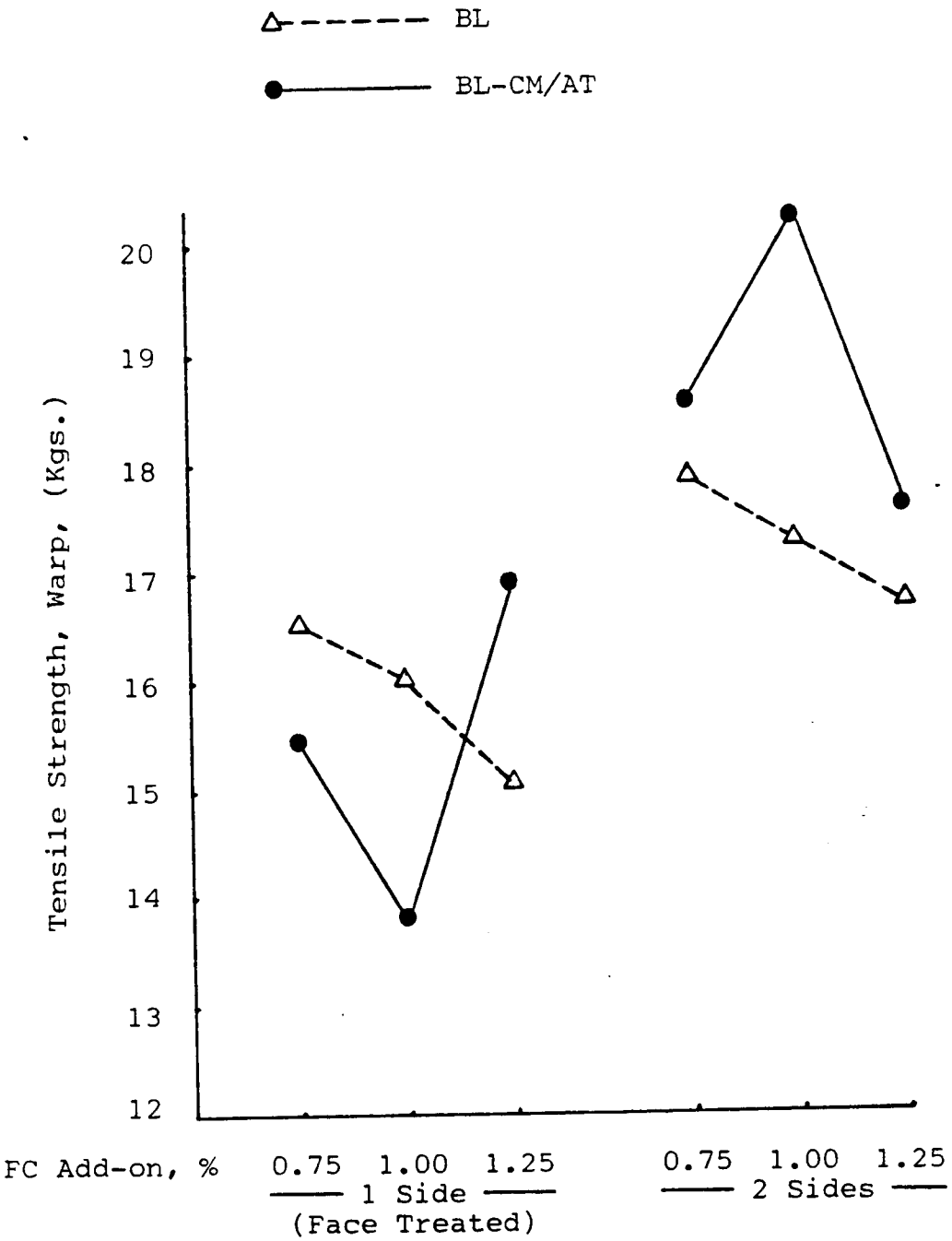


Figure 18. Warp Tensile Strength of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

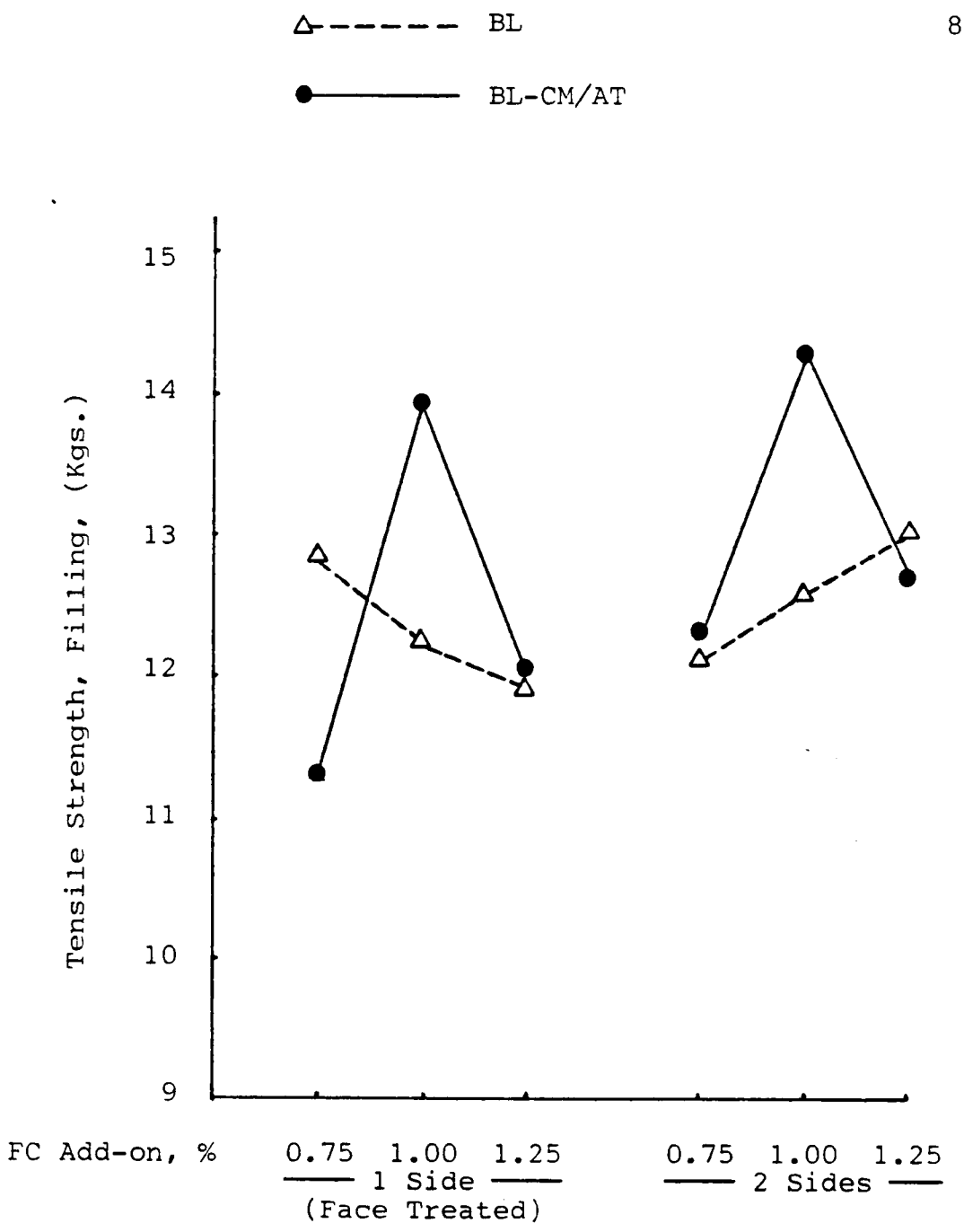


Figure 19. Filling Tensile Strength of Print Cloth Finished to Three Add-ons of FC and 4.00% DMDHEU by One and Two-sided FFT Application at 25% WPU.

Effect of Fluorochemical Level on Strength Properties

As shown in Figure 16, warp tearing strength decreased almost linearly (for most samples) with increasing FC add-on. Although the highest filling tearing strength usually resulted at 1.00% FC add-on over the range of 0.75 to 1.25% FC contents, filling tearing strength generally decreased with an increase of FC add-on (Figure 17). These results indicate that tearing strength may be favored by lower fluorochemical add-on, and support the findings by Philips et al. (33) that the tearing strength of fabric treated with fluorochemical decreased somewhat.

Generally, for BL fabrics, tensile strength decreased with increasing FC add-on (Figures 18 and 19). It is notable that with both one and two-sided foam applications, warp tensile strength of BL fabrics decreased dramatically over the FC range of 0.75 to 1.25% add-on. Filling tensile strength of BL fabric treated by one-sided foam application also appeared to decrease sharply over the range of FC contents. However, there was no consistent trend for BL-CM/AT fabrics. For example, with one-sided application, the lowest warp tensile strength of BL-CM/AT fabric was obtained at 1.00% FC level. At the same 1.00% FC add-on, BL-CM/AT fabric

with two-sided foam application resulted in the highest warp tensile strength. Filling tensile strength of BL-CM/AT fabric treated by both one and two-sided foam applications showed the same trend in that the highest filling strength was obtained at 1.00% FC level, followed by 0.75 and 1.25% FC add-ons.

CHAPTER V

SUMMARY AND CONCLUSIONS

The purpose for conducting this study was to investigate the effects of differential foam finishing on functional properties of durable press and fluorochemical treated fabris. The first objective was to examine the effects on performance properties of fluorochemical (FC) and durable press (DP) treated cotton fabric at a range of FC add-ons (0.75, 1.00, and 1.25%) with fixed DP resin (4.00%). The second objective was to determine if abrasion resistance of fabric treated on one side differs significantly from fabric finished on both sides. And, the third objective was to more fully substantiate the improved strength and abrasion resistance resulting from the combination pretreatment of caustic mercerization and liquid ammonia treatment compared to preparatory finishing through bleaching only.

Two pretreated 100% cotton print cloths were used in this study: bleached (BL) fabric and bleached-caustic mercerized/ammonia treated (BL-CM/AT) fabric. Two finishes: DMDHEU, a durable press resin, and fluoro-chemical, a water and oil repellent, were co-applied to pretreated cotton fabrics by one or two-sided foam

application. Four formulations were prepared: three for 25% wet pick-up (0.75% FC/4.00% DMDHEU, 1.00% FC/4.00% DMDHEU, and 1.25% FC/4.00% DMDHEU) and one for 21% wet pick-up (1.00% FC/3.80% DMDHEU).

The finished fabrics were examined for water repellency, oil repellency, flex abrasion resistance, wrinkle recovery angle, durable press ratings, tearing strength, tensile strength, and nitrogen content. Also, dye staining was utilized to determine the location of finishes.

The following conclusions were drawn from this study.

1. As was shown in the previous study by Wadsworth et al. (12), one-sided foam application of FC and DP finishes resulted in excellent water and oil repellency on the applied side while leaving the nonapplied side absorbent, and at the same time resulted in almost identical durable press properties on both sides.

2. It was also reaffirmed that the combination pretreatment of caustic mercerization and liquid ammonia treatment appreciably improved tearing resistance and tensile strength of both one and two-sided foam finished fabrics. Furthermore, abrasion resistance, which was not studied in detail in the previous work, was also substantially improved by the combination pretreatment.

3. One-sided foam application, as indicated in an earlier study, generally resulted in higher tearing strength than a two-sided foam application. However, one-sided foam treatment usually resulted in lower tensile strength than a double-sided foam application.

4. Generally, abrasion resistance on the nonapplied side of differentially DP and FC finished fabric appeared to be better than on the applied side.

5. As the fluorochemical add-on was increased from 0.75 to 1.25%, abrasion resistant, tensile strength and tearing resistance all unexpectedly decreased.

Although it has been conclusively shown that differential foam application of FC and DP finishes results in acceptable repellency, durable press, and strength properties, some unanswered questions suggest the need for further study:

1. To decrease the add-on of FC required by either decreasing the amount or changing the chemical composition of foaming agents (surfactants) which may be detrimental to repellency, even though they are marketed as "non-rewetting."

2. To develop a better understanding of why abrasion resistance decreases with increasing FC content.

3. To perform a wear study with differentially foam finished fabric to determine if the nonapplied side is perceived to be softer, more absorbent, that is more comfortable, than two-sided finished fabric.

4. To perform a soiling study to determine the relative soiling and soil release performance of differentially foam finished fabrics for one and two-sided foam applications.

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