ABSTRACT

HAMILTON, TODD KEITH. Synthesis, Characterization, Application, and Evaluation of Reactive Sizes for Cotton Warps. {Under the direction of Dr. Abdelfattah M. Seyam}.

The purpose of this research has been to investigate the weaving properties of cotton yarns treated with three newly developed reactive sizing agents. N-methlyocarbamoylethyl starch, N-methlyocarbamoylethyl starch + poly vinyl alcohol (PVA), and N-methlyocarbamoylethyl starch + PVA + melamine formaldehyde have been studied as durable, non-removable sizing agents.

Extensive work has been conducted to accomplish the objectives of the current research. The experimental activities have included: developing new reactive sizes; spinning a range of cotton yarns with three levels of twist multiple; preparing and applying size solution; testing mechanical and physical characteristics of sized yarns to evaluate their weavability; evaluating the stability of the sizing agents in woven structures; and performing statistical analyses to investigate the effect of size type, twist level, and solid concentration in the size bath on several yarn properties.

SYNTHESIS, CHARACTERIZATION, APPLICATION, AND EVALUATION OF REACTIVE SIZES FOR COTTON WARPS

By

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BIOGRAPHY

Todd Keith Hamilton was born on January 1, 1976 in Gastonia, North Carolina. After graduating from Ashbrook High School in 1994, he furthered his education at North Carolina State University. After studying abroad in West Yorkshire, England at the University of Leeds during the spring of 1998, he returned to NC State University to graduate with a Bachelor of Science degree in Textile Technology. In January of 2000, he again enrolled at the NC State College of Textiles to enter the Textile Management and Technology master's program.

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1. INTRODUCTION

Warp sizing, although non-productive, is one of the most significant processes in preparing warp yarns to withstand the complex field of stresses experienced by yarns during weaving. Ideally, the sizing process should be eliminated to reduce weaving manufacturing cost. This is, however, not currently practical since the structures of most spun warp yarns require sizing to achieve maximum weaving efficiency and highest quality of woven fabrics. In most cases, size material has to be removed from the fabric by a process known as de-sizing. The removed size material may be recovered by complex filtration processes or wasted. The latter case is not desirable due to increasing pressure to protect the environment from industrial waste, and eventually will not be permitted globally by environmental legislation.

For many years, starch-based agents have been the major component used in the slashing (sizing) of cotton yarns. The most relevant disadvantage of conventional slashing operations is the requirement of a considerable amount of time and energy in preparation of size solution. Starch-based size solution requires “cooking time”, and a constant bath temperature must be maintained throughout the entire sizing process in order to get constant size pick-up.

One solution to such complex problems is to consider the sizing process, other than its essential objective of strengthening warp yarns to withstand weaving stresses, as a finishing step by applying dual-purpose finishing/sizing agents to cotton warps instead of fabric. It is the main goal of the project to pursue such a solution by developing sizing agents that are reactive to cotton fibers. Such research work has the potential of

eliminating the need for the de-sizing process. The recognized advantages following this philosophy are:

- Reducing cooking time needed in case of starch-based agents.
- Eliminating/reducing heat energy required for cooking starch.
- Reducing cost by eliminating de-sizing and size material recovery.
- Protecting the environment from size waste.

This would be a successful route if the elimination of the de-sizing process does not adversely impact but may enhance:

- The resulting fabric properties (hand, comfort, wear, crease recovery, etc.).
- Finishing processes such as dyeing, printing, etc.

The de-sizing process may be eliminated if reactive sizes can form chemical bonding through introduction of functional groups to the size molecules. The functional groups are designed to form permanent bonds with the cellulose molecules of cotton fibers. Successful reaction should lead to durable and non-removable sizing agents.

2. LITERATURE REVIEW

The primary purpose of warp sizing is to create weavability in the warp or to conquer the inherent deficiencies of yarn to withstand the stresses and strains of weaving [10]. Although sizing diminishes the amount of breaks due to abrasion or fatigue, it sometimes increases the amount due to lumps and lack of extensibility. It is therefore crucial to provide yarns with a coating that not only increases its strength and abrasion resistance, but one that will be smooth, uniform, and sustain the property of elongation. However, the primary role of the material is to protect the warp yarns from the abrasive effects of weaving [17]. Although sizing is an important step in the weaving process, explicit consideration must be taken in regards to its impact on the environment. Prevention of both air pollution and water pollution from sizing effluents is key to the overall success of the textile industry.

2.1 Importance of Size Formula

The perfect size formula should protect and add strength to the warp yarns. It can add weight to the fabric if needed, and can allow the fabric to be finished to a desired hand or stiffness. The size formula should be stable, uniform, and economical. The major considerations in choosing a size formulation are the following:

- Tensile Strength—as the size bonds the fibers together, the yarn strength should increase.
- Adhesion—the ability of the size to surround, protect, and cling to warp yarns.
- Flexibility—size film should resist breakage during weaving.

- Fiber Lay—provides a smooth, compact yarn that results in less yarn-to-yarn and yarn-to-machine part friction.
- Abrasion Resistance—size must form a continuous film that protects the yarn being abraded by loom parts.
- Penetration—the size must penetrate the yarn enough to adhere to it.
- Viscosity—controls the penetration of the size at given squeeze roll pressures and size solution temperatures.
- Elongation—the amount of stretch the yarn will undergo before it breaks; this must be the same for the yarn and the size.
- Elasticity—allows the yarn to return to its original length after tension is released [18].

Two essential properties that sizing must produce in cotton yarns to achieve good weaving performance are: (1) adequate fiber-to-fiber bonding to create enough tensile in the yarn to protect weak areas, and (2) adequate fiber-to-fiber bonding to prevent excessive hairiness and clinging through abrasion of the yarn with itself and with loom parts. These properties must be maintained under the continuous stretch/relaxation cycle of weaving as well as varying weave room conditions. Thus, the performance of warp yarns on a loom is directly measurable in terms of fiber-to-fiber bonding. This is the case whether considering yarn tensile strength, abrasion resistance, elongation, or any other necessary function of warp yarns to weave properly [10].

Any individual property (such as film strength of the size agent itself) is relative, and its value, in terms of good weaving, must be related to other factors influencing its fiber-to-fiber bonding. For example, a size may have excellent tensile strength with

insufficient adhesion properties. This will cause sizing material to disconnect from the fiber, and little benefit is gained from its tensile strength. On the other hand, good adhesion properties may be obtained, but if the tensile elongation of the size is too low compared to yarn elongation, film separation will occur, and the advantages of the adhesion properties are lost. Therefore, other factors affecting the fiber-to-fiber bonding of a size include its specific adhesion to a particular fiber; percent encapsulation of the surface area of the fibers receiving maximum stress and strain; the level and type of stress and strain imparted during weaving; and how these are affected by humidity or other weaving conditions. Observably, the surface fibers in a yarn receive maximum stresses during weaving. The degree to which these fibers are adhered is a high measure of their performance throughout the loom. Thus, the location (or distribution) of the size in and on the surface of the yarn is very important. For example, if a yarn distributed with size such that only 25 percent of the surface fibers are adhered, a very poor performance during weaving would be expected in terms of clinging and soft warps. In contrast, the same quantity of size with 75 percent adherence would perform very well with less clinging and warp breaks [10].

2.2 Characteristics of Adhesion

Adhesion is the ability of the size material to adhere persistently to the fiber. Good adhesion properties are maintained by strong attraction between the size material and the fiber. This attraction can be either chemical or physical.

The chemical facet of adhesion can be summarized by the old expression, “like attracts like”. For example, observing the chemical structure of starch and PVA clearly

shows why these materials are excellent sizing agents for cotton yarns. Starch and PVA share a similar chemical structure with cellulose; therefore, these materials possess a strong chemical attraction for each other. The primary force that gives starch and PVA a strong attraction for cellulose is hydrogen bonding. When a size and fiber have this type of bonding, the strongest form of association occurs between the two materials, that is, they interact chemically with one another. Such size materials mix readily with water. Water also has a role in the hydrogen bond interactions by allowing the fiber surface to be easily "wet out" by the size-water mixture [12].

The physical surface of the fiber also affects adhesion of the size material. The smooth round surface of most man-made fibers repels the wetting action of a size mixture and the size does not adhere to the fiber. However, fibers that have irregular or rough surfaces (e.g. cotton) tend to pick up and retain more size mixture. An irregular surface has more frictional forces operating between fiber and size. This increases size add-on and makes the wet size more difficult to remove by the squeeze roll. Another way to increase adhesion is to increase the viscosity of the size formulation. This makes the size less fluid so that the rate of "run-off" is decreased. This action overcomes the effect of the physical element of adhesion to produce higher size add-on to the yarn. [12].

The chemical and physical aspects of adhesion become more active after the size has been dried on the yarn. In yarn encapsulation, the frictional forces of the physical constituent can be enough to prevent size removal for cotton or staple man-made fibers. Because filament fibers have minimal size add-on, a strong chemical interaction between fiber and size must be present to prevent loss of yarn reliability during weaving [12].

2.3 Influences on Warp Quality

Weaving machine performance is directly related to the demands placed on the quality of the warp—the higher the loom performance, the higher the quality of warp yarns needed. Optimum warp quality can be expressed through the causes of yarn loading and disturbance during weaving. These causes are:

- Abrasion stress on the back rest, reed, drop wires, and heddle wires, as well as yarn-to-yarn abrasion.
- Dynamic tensile loading through shed formation and reed beat-up.
- Clinging warp ends during shed formation due to hairy yarn.
- Disturbances caused by dust and crossed ends.

A warp that completely overcomes these conditions can be considered to have good or optimum quality. Some of the most important influences on warp quality are size add-on, dynamic stretch recovery, yarn hairiness, and drying section configuration and temperature distribution [19].

2.3.1 Size Add-on

Either too much or too little sizing contributes to increased warp breaks at the loom. Too little size does not give yarn sufficient abrasion resistance to overcome rubbing during weaving. Too much size creates a rigid, inelastic yarn that quickly fatigues, breaks, and fails at the loom [24]. The sizing agent must penetrate and cover the yarn uniformly. The quality of the size add-on can be evaluated by its degree of penetration and encapsulation. Size penetration has an optimum level between 20 and 30 percent; a significant loss in yarn flexibility occurs beyond 40 percent. Size

encapsulation reveals the amount of size covering the surface of the yarn. Optimum coating should be no less than 270° around the yarn, while ideal maximum encapsulation is 360° [8].

2.3.2 Elongation

Warp yarn breaks are dependent on the elongation of sized yarns during weaving. Too much sizing makes yarn stiff and brittle. This deters yarn elongation and causes premature breaking. On the other hand, a yarn that elongates further than its natural capacity will also break. The elongation of sized yarns is dependent on:

- The elasticity of the yarn.
- The sizing product used.
- The warp tension or stretch set.
- The moisture content in the yarn during weaving.

In order to gain good stretch recovery in sized yarns, the sizing machine must do the following:

- Monitor and adjust the yarn tension or stretch over the entire length to be sized.
- Reproduce the settings when a re-order takes place [19].

2.3.3 Yarn Hairiness

Yarn hairiness (protruding fibers) leads to warp ends clinging together during weaving. Fiber protrusion is particularly critical in staple fibers when woven on air-jet looms [19]. A high level of hairiness is destructive to the weaving process and is the major cause in warp-related filling stops in air-jet weaving. This creates an adverse

effect on weaving efficiency. Thomas [25] explains the following as the three major warp-relate stops due to yarn hairiness:

1. "Clinging yarns in the region between heddles and dropwires of the loom in which protruding hairs cause similar entanglements to those mentioned in the case of front shed clings above, or in which a "hair" fiber can be swung out violently from the originating yarn to capture and entangle other warp yarns during shed changing. In either case, the clings that occur behind the heddles must be severe enough to prevent the yarns from separating even under increasing tension during the weaving so that one or more of the yarns finally breaks.

2. Clinging yarns in the front shed, in which protruding "hair" fibers on warp yarn surfaces cause two or more warp yarns to become so tightly joined together that they cannot move into the required positions for shedding. It is impossible that one or more of the yarns will break because of excess tension, but more frequently the failed shedding action will cause one or more of the clinging warp yarns to interfere with the insertion of the weft.

3. Abraded yarn surfaces, in which fibers become progressively stripped out of the yarn until the yarn loses enough of them to experience tensile failure, or a lint ball formation, in which the fibers that are stripped out of the yarn by abrasion collect into ball shaped structures attached to the yarn and eventually become too large to pass through the narrow passage points in the loom accessories or obstruct movement by an adjacent end."

Thus, the primary causes of warp failures are the lack of abrasion resistance and adequate elasticity. Poor abrasion resistance is related to yarn hairiness. Poor elastic properties in a yarn may cause fatigue and breakage. It is therefore necessary to reduce hairiness and improve the elasticity of yarns through the application of a sufficient size material.

2.3.4 Drying

The configuration of the drying section also plays an important role in reducing yarn hairiness. When yarns separates into two or more sheets through multiple size

boxes, it is important that they be completely dried in order to minimize adhesion of the moist size film when returning all the ends into a single sheet. If each yarn sheet is not appropriately pre-dried, size bridges form and yarn hairiness increases because of yarn snatching in the dry leasing section. Therefore, it is critical to have proper cylinders for the yarn style, moisture content, and speed requirements of the size machine [14].

"Separated end drying" is an additional step toward reducing yarn hairiness. In this configuration, yarns leaving the size box separate into two sheets, so that each sheet can be pre-dried separately before being combined. This allows the yarn spacing to increase in each sheet, thus minimizing the formation of size bridges. They are joined again into one sheet after they are sufficiently dry. This provides a yarn sheet that disjoins easily in leasing, which decreases fiber removal and yarn tears [14].

2.4 Environmental Aspects of Sizing

Innovative developments such as foam sizing, solvent sizing, and hot-melt sizing have made strides toward the reduction of sizing pollutants in the environment. Although these methods prove to reduce some of the hazardous waste released by textile finishing plants, they have not been completely successful. The focus of the industry today is size reclamation or recovery, and even the possible elimination of the de-sizing process. Recovery of PVA has been practiced over 20 years, and in some cases, 90 percent of size can be recovered. This process is generally done by ultrafiltration systems [21]. More recently, researchers have been studying the possibility of completely eliminating the de-sizing process through the use of "permanent" sizing agents.

Sizing and de-sizing operations are extremely large contributors to waste and pollution. In some cases, 50 percent of pollutants come from finishing operations. Size materials vary greatly in pollutant characteristics. The primary chemical wastes from these operations are enormous volumes of water, fiber finishes and surfactants, and the size materials themselves. The resulting pollutants are biological oxygen demand (BOD) and chemical oxygen demand (COD) [23].

The de-sizing process causes about half the COD load from a finishing plant. However, this quantity of COD is merely contained in a small portion of the effluent stream. By eliminating the size from this relatively small percentage of the total quantity of effluents, the COD load of all combined effluents may be reduced by 50 percent [15].

When discharged into the environment, pollutants restrict the oxygen level in the receiving streams. The BOD values of size materials vary greatly. Typically, starch sizes have BOD values of 500,000 to 600,000 parts per million (ppm); alginate and modified starch, 100,000 to 500,000 ppm; and synthetic sizes (PVA, CMC, etc…), about 10,000 to 30,000 ppm. Also, starches are usually removed with enzymes that generally have BOD values over 10,000 ppm. De-sizing of most synthetic sizes is mainly accomplished with water, surfactants, and small amounts of alkali. Thus, the de-sizing system for synthetic sizes does not contribute much to the BOD level. In addition, synthetic sizes can be reclaimed from wastewater streams. Therefore, the use of synthetic sizes instead of starch can result in a BOD reduction of over 90 percent [23].

2.4.1 Size Recovery

To allow recycling by means of ultra-filtration, the size agents suitable for recovery have to show the following characteristics:

- Water soluble
- Mechanical and thermal stability
- Biological resistance
- Good washout characteristics
- Low average viscosity.

Past experiences with several manufacturers show that a number of PVA size agents, certain acrylate size agents, CMC size agents, and mixtures of these components meet these demands [20].

Ultra-filtration is a simple and distinct process. The size washed out in a countercurrent procedure and dispersed in washing liquor is removed from the first countercurrent section and cleared of impurities. It is then pumped into a buffer tank and sent to the ultra-filtration plant. Here, the washing liquor is circulated over membranes by pumps and water is removed. The water is reused for the washing process. Size molecules are retained from the membrane so upgrading can occur. A flow refractometer monitors the final concentration. The discharged size concentrate is stored in a buffer tank until reuse in the sizing department. The concentrate is then reused either by itself or with fresh original size [20]. Advantages of size recovery are:

- Reduction of environmental pollution
- Reduction of costs for waste water and fresh water
- Cost savings by multiple use of size agents

- Economical application of high quality size agents
- Improvement of efficiency in the weaving department
- High profitability due to short amortization period of the plant [20].

In many cases, plants face the high cost of upgrading treatment facilities to meet new regulations. Therefore, another alternative is increasing the percentage of PVA in a PVA/Starch size formulation. This also decreases pollution and creates lower add-on to attain mandatory yarn properties, further lowering BOD in wastewater [6].

Using 100% PVA formulations for certain fabrics greatly lowers BOD levels compared to starch, and also allows opportunities for size reclamation and reuse. Although the cost of PVA is more than starch, and the initial capital investment for a reclamation system is high, the avoided wastewater treatment upgrades and raw material savings can equal a fast payback. Also, in most cases, one pound of PVA can replace several pounds of starch. This substantially decreases the cost gap between the two materials.

In addition, 100% PVA is the only warp sizing formulation that is reclaimed with constant success, a technology first developed in the 1970s. Reclamation by ultra-filtration can recover approximately 60 to 80 percent of the PVA for reuse [6]. However, the recovered PVA comes with the disadvantages of being used and dirty. Therefore, it is necessary to treat the reclaimed concentrate with a small amount of bactericide after it leaves the buffer tank. This is important in order to prevent any biodegradation of the regenerated material [20].

2.4.2 Permanent Sizing Agents

The primary advantage of starch is its low cost. However, because it is readily biodegradable, its contribution to the BOD level can be significantly high. The total oxygen demand (TOD) of starch waste is also high due to the vast amounts of starch needed to protect yarn during the weaving process [5]. Therefore, polymers used as permanent sizing agents create opportunities to counterattack this effect.

2.4.2.1 Cross-linking

According to Blanchard, Lofton, Harper, and Graham [5], reducing the amount of material to be removed, and hence, the TOD, is achieved by making CMC, PVA, or starch insoluble. In this process a specified sizing agent and cross-linking agent, such as dimethylol dihydroxyethyleneurea (DMDHEU), is applied to the yarn as it passes through the size formulation. The yarn is partially cured as it travels over dry cans at 120°C. The degree of curing that occurs depends on the temperature of the dry cans and the number of cans used.

In wastewater from uncured samples, the TOD for samples sized with CMC is lower than those sized with PVA, and highest for those sized with starch. However, after curing and then de-sizing, the TOD values of the wastes are about the same. This proves that the amount of material typically removed from fabric during the de-sizing operation is considerably reduced by adding a small amount of cross-linking agent and catalyst to the warp size formulation [5].

By combining water-soluble sizing agents with permanent warp-sizing materials, such as polyacrylates and polyurethanes, improved durable-press properties are

obtained—sometimes without the need for de-sizing. Furthermore, the total amount of sizing material removed from the fabric during a de-sizing operation is greatly decreased, thus reducing pollution problems related with the application and disposal of de-sized waste [5].

Polymeric sizing agents have been researched in the past at the Southern Regional Research Center (SRRC) in New Orleans, Louisiana. The primary study dealt with durable-press (DP) fabrics with enhanced properties. A cross-linking agent was used in the sizing formulation with a polymer. When the polymer was used alone, the resin was later put on the fabric as a DP finish. These treatments, applied to course and medium yarns (65.6 tex, 49.2 tex, 32.8 tex), possessed an extra benefit by functioning as permanent sizing treatments. However, conventional sizing agents including CMC or PVA were added to the size bath for good weavability [16].

During this research, it became necessary that laboratory methods be used to screen various polymers as potential sizing agents for further high scale evaluation. Yarn treatments on a laboratory single-end slasher indicated that certain polyacrylates showed the most potential for full-scale investigation. The evaluations of polymer film, yarn hairiness, and abrasion resistance proved to be the most useful in demonstrating the weavability of the treated yarns. The overall physical properties of the treated fabrics proved favorable after testing. They fabrics showed increased breaking strength and abrasion resistance with good retention of elongation. Fabric stiffness increased compared to the untreated samples, but overall hand was acceptable for medium weight fabric. Wicking and absorbency were decreased, but did not adversely affect dyeing in the laboratory. Also, the presence of the polymer did not inhibit laboratory durable-press

finishing or raw mercerization. Infrared analysis showed that the polymer still remained present on warp yarns in mercerized and dyed samples [16].

These polymers offer potential as permanent sizing agents if complete functionality can be achieved. The benefits are increased fabric weight, improved fabric properties, less environmental pollution, energy savings, and no change in existing sizing equipment [16].

2.4.2.2 Reactive Carbohydrates

In general, carbohydrate materials such as starch and starch derivatives are used in textile finishing. Physical forces (primarily hydrogen bonding) are responsible for the attachment of these carbohydrates to textile fabrics and are considered as temporary finishes. When carbohydrates are removed during washing, the weight and hand of the fabrics change; also, the pollution level of de-sizing increases [13].

Past studies have assessed the technical practicability of synthesizing reactive carbohydrates and their application to cotton fabric. Starch, carboxymethyl starch, and CMC have been used as initial materials for the reactive carbohydrates. Carbohydrates were first reacted with acrylamide either by grafting or etherification, followed by the conversion of the resulting amide groups to methylol groups. The methylol groups were then reacted with the hydroxyl groups of cotton cellulose in a process similar to typical reactant resins [13].

A more recent study (in the past decade) was performed at the National Research Center in Cairo, Egypt. This experiment was geared toward studying the technical ability of converting starch and hydrolyzed starches to reactive carbohydrates by creating

pendant double bonds in the molecular structure of starch. This allowed the reactive starch to be chemically attached to cotton fabric through reaction of the pendant double bond with the hydroxyl groups of the cellulose. In this case, starch and hydrolyzed starches were reacted in an acid medium with N-methylolacrylamide to produce acrylamidomethyl starch. This starch was reacted with cotton cellulose in an alkaline medium to yield chemical bonding between the cellulose and the starch-based finish. Evaluation of the results obtained from this study showed that the most effective conditions for preparation of this reactive starch involved treating hydrolyzed starch with N-methylolacrylamide (20% b. o. w. of starch) in presence of NH_4Cl catalyst (12 g/l) at 140°C for seven minutes using a material-to-liquor ratio 1:2.5. Fabric samples were padded in a solution containing the reactive starch finish (75 g/l), KOH (10 g/l) to a set pick up of ca 90%. Finally, they were dried for three minutes at 100°C and cured for five minutes at 150°C. These conditions allowed the finished fabric to achieve optimal physical properties (increase in weight, crease recovery, elongation at break, and tensile strength) [13].

Carbamoylethyl starch (CES) is obtained by the reaction of starch with acrylamide in the presence of alkali. The reaction is a nucleophilic substitution reaction at the double bond site of acrylamide as follows:

1. $St\text{-}OH + HO^{-} \longrightarrow St\text{-}O + H_2O$

2. $St\text{-}O^{-} + CH_2{=}CHCONH_2 \longrightarrow St\text{-}O\text{-}CH\text{-}CHCONH_2$ [22].

In an experiment at the National Institute for Standards in El Harem, Egypt, carbamoylethyl starch was applied as a sizing agent for cotton and cotton/polyester yarn. Solutions of 10% solid starch and CES containing various nitrogen, carboxyl, and total ether levels, as well as dual mixture with PVA, were autonomously cooked at 90°C. The solutions were then applied to sample yarns [22].

Results of the study proved that regardless of the size composition and type of sized yarn, the tensile strength increased with the sizing, while the elongation decreased. It was guessed that this is due to an increase in yarn cohesiveness, the deterrence of fiber slippage, and the decrease in yarn elasticity. Tensile strength and elongation at break varied among the different types of sizing agents. This was expected due to differences in these film-forming polymers such as: type and ingredients of the substituted derivative (carbomoylethylated and/or carboxyethylated); viscosity of size formulation; placement and size depth of penetration; degree of encapsulation; adhesion and cohesion properties; film-forming properties; and coatability of the sized sample yarns [22].

3. OBJECTIVES

The main objective of this research is to develop non-removable, environmental friendly sizes, which have dual functions, namely sizing and finishing agents. Additional goals include:

- To apply the reactive sizes to cotton yarns
- To investigate the tensile properties of the sized yarns.
- To examine the durability of the sizes on woven fabrics.

4. EXPERIMENTAL COMPOSITION

4.1 Fundamentals and Preparation of Reactive Sizes

Examining reactive sizes allows one to understand the materials belonging to the conventional sizing materials containing reactive functional groups. For example, starch is a conventional sizing material for cotton warps. Modification of starch via physical or chemical methods may convert it to a reactive size. One of these methods is the reaction of starch with acrylamide to yield carbamoylethyl starch, a material that is easily soluble in hot or cold water depending on the degree of substitution (DS) of the reaction product. Lower degree of substitution (DS = 0.1 to 0.5) gives a material that is soluble in water at a temperature higher than or equal to 50^{o}C, while a higher degree of substitution (DS = 0.6 to 0.9) gives a material that is soluble in water at room temperature.

The reaction of acrylamide with starch follows the root of the well-known Michael addition reaction according to the following equation:

Starch + Acrylamide $\xrightarrow{OH^-}$ Carbamoylethyl Starch

or

St- O H + CH_2=CH-CO-NH_2 $\xrightarrow{OH^-}$ St-O-CH_2-CH_2-CO-NH_2

In spite of the fact that the reaction is so simple, it is highly sensitive and the product may be highly affected if the reaction conditions are not completely controlled. Many experiments have been carried out to optimize the reaction conditions needed for the preparation of carbamoylethyl starch. For example, if the reaction is carried out in an

aqueous media, a paste will form, especially at higher content levels of carbamoylethyl groups (higher DS); and the handling of the product is not easy—neither during purification nor during transportation. Moreover, the presence of the aqueous alkaline medium may affect the chemical stability of the resultant carbamoylethyl starch. To overcome such difficulty, preparation of carbamoylethyl starch has been tried in presence of an electrolyte (20% aqueous sodium sulfate: Na_2SO_4). Here, the resultant product remains in the granular form, especially at lower DS of the carbamoyl content. At higher DS the product swells and becomes very viscous, so its separation and purification from the electrolyte is not an easy task.

To overcome such difficulties, a new technique has been developed where the starch material, acrylamide, and sodium hydroxide are mixed together at different temperatures in presence of different amounts of alcohol and water for different periods of time. Recently, researchers have studied the effect of these different factors extensively at NRC, Cairo, Egypt, and the results showed that preparation of carbamoylethyl starch with a DS of 0.75 might result from mixing one mole starch and one mole acrylamide in presence of 1-5% solid sodium hydroxide (NaOH) based on weight of starch. Spraying the reaction mixture with a 20% mixture of alcohol and water based on weight of starch (e.g. isopropanol and water, at a ratio of 3:1) followed by heating at 50^oC for 60 minutes yields a product with DS of ca 0.75 (carbamoylethyl starch; DS = 0.75).

Nitrogen content analysis showed that from the one mole acrylamide used, 0.75 mole of this monomer has been reacted with starch (1 mole) and 0.25 mole of the acrylamide is still in the free form (un-reacted monomer). The un-reacted monomer may

be consumed in a later step during methylolation to form N-methylolacrylamide that is capable of reacting with both starch and cellulose macromolecules in the cotton fibers. However, to benefit from this amount of free acrylamide, it was thought feasible that addition of an equivalent amount of a primary or a secondary amine to the free acrylamide will transform it to a saturated amine-amide adduct. This insures that there will be no damage to the environment due to the presence of the un-reacted acrylamide. The exhaustion of the acrylamide is represented in the following reaction:

Diethylamine + Acrylamide $\longrightarrow$ N,N-Diethylamino-propionamide

or

$(C_2H_5)_2NH + CH_2=CH\text{-}CO\text{-}NH_2 \longrightarrow (C_2H_5)_2N\text{–}CH_2\text{–}CH_2\text{-}CO\text{-}NH_2$

This amine-amide adduct behaves exactly like carbamoylethyl starch in chemical reactions, and in its reactive form (methylolated form), it may react with the hydroxyl groups of starch as well as those of cellulose. Moreover, this amine-amide adduct contains a tertiary nitrogen atom capable of binding a hydrogen proton in acidic aqueous solution, so the resulting positively charged moiety will be able to attract negative anions to its neighborhood. Such a behavior can be used in the fixation of acid dyes or other dye molecules containing sulphonic acid groups onto the surface of the reactive size. Such a behavior may be ascribed as an internal marker that may serve two purposes. On one hand it can be used to follow up the stability of the reactive size on cotton yarns or fabrics. On the other hand, in case of fabrics with different woven or knitted patterns,

dyeing of such fabrics in a single dyeing bath might lead to colored fabrics with shade variations depending on the pattern type (differential dyeing).

Using the above finding, carbamoylethyl starch free of acrylamide monomer has been prepared using raw maize starch (high molecular weight, M_n, ca 3.2-8.6 x 10^6) and acid maize starch (partially hydrolyzed starch, M_n, ca 7.8 x 10^5). The resultant product is soluble in water at room temperature and yields an alkaline aqueous solution. Adjustment of the pH of the resultant aqueous solution to a value of 10.5-11.5, followed by addition of an equivalent amount of aqueous formaldehyde at 30°C-40°C for 60 minutes, transforms the aqueous carbamoylethyl starch into N-methylolcarbamoylethyl starch. The latter material is acting as the reactive size material.

Carbamoylethyl starch + aqueous formaldehyde $\xrightarrow{\text{pH 10.5-11.5}}$ N-methylolcarbamoylethyl starch, or

$$St\text{-}O\text{-}CH_2\text{-}CH_2\text{-}CO\text{-}NH_2 + HCHO \xrightarrow{\text{pH 10.5-11.5}} St\text{-}O\text{-}CH_2\text{-}CH_2\text{-}CO\text{-}NH\text{-}CH_2\text{-}OH$$

Simultaneously, the amine-amide adduct (resulted from the reaction of un-reacted acrylamide with equivalent amount of a primarily or a secondary amine) will also be methylolated to yield a reactive material, namely, N,N-dialkylamine-N`-methylolpropionamide, as:

$$(R)_2NH + CH_2{=}CH\text{-}CO\text{-}NH_2 \longrightarrow (R)_2N\text{-}CH_2\text{-}CH_2\text{-}CO\text{-}NH_2 + HCHO \longrightarrow (R)_2N\text{-}CH_2\text{-}CH_2\text{-}CO\text{-}NH\text{-}CH_2\text{-}OH$$

Where R (alkyl rest) = $-CH_3$, $-CH_2\text{-}CH_3$, or $-CH_2\text{-}CH_2\text{-}OH$

It is noteworthy to mention that during methylolation the aqueous formaldehyde needed for the methylolation reaction should be added in amounts slightly less than the equivalent amount, so that after thermo-fixation the amount found of free formaldehyde should lie and remain within the permissible range. To leave the level of free formaldehyde far less than the permissible range, sodium sulphite or a reductant, such as thioureadioxide, should be added in minute amounts at pH 9.5-10.5 in the first rinsing or scouring bath for fabrics made of such sized yarns.

A preliminary experiment showed that a mixture of high, medium, or low molecular weight starches at a ratio of 1:1 would be highly appropriate as a sizing material for cotton yarns. However, addition of PVA to the size formula improved the performance of yarns treated with such mixture. To increase the degree of fixation of PVA in the size formula onto the sized yarn, it is feasible to add as little as 0.1 mole of a dibasic acid to the sizing formula for each mole of PVA. At higher temperatures (130^{o}C or higher), and in presence of the catalyst added for thermo-fixation, the carboxyl groups may condense with the hydroxyl groups of PVA and/or those of starch and cellulose to form ester bonding capable of fixing PVA on the sized cotton yarns.

The third and last method to improve the quality and stability of the reactive size is simply treating the reactive starch/PVA mixture with a small amount of melamine formaldehyde (ca 10 wt % based on weight of starch used). Melamine formaldehyde helps in lowering the fixation temperature, and in the mean time, increases the extent of fixation of the reactive size via poly-condensation reaction. The latter reaction will take place not only exclusively between the –OH groups of melamine formaldehyde, but also with the –OH groups of starch, reactive starch, cellulose and/or PVA.

Two kilograms of carbamoylethyl starch mixed with one mole industrial maize starch and one mole acrylamide were prepared at NRC/Cairo. Un-reacted acrylamide was transformed to an amine-amide adduct using diethanolamine. Two kilograms of carbamoylethyl starch based on acid starch were also prepared at NRC/Cairo under similar conditions. These materials were physically mixed at the laboratories at the College of Textiles, NCSU at a ratio of 1:1. Three samples, each weighing 0.75 kg, were prepared.

4.1.1 Preparation of Formula Containing N-methylolcarbamoylethyl Starch

The first sample (750 g) was dissolved in 5 liters tap water at room temperature to yield a 15% aqueous solution of pH 10.5. The pH was lowered to 5-6 by making use of acetic acid. The temperature of the solution was raised to 75°C, and a mixture containing 34 grams of commercial wax and 82 grams of commercial lubricant were added under good stirring. The mixture was kept at 75°C for 10 minutes for the sake of homogenization, followed by cooling down to 40°C. 235 ml 37% aqueous formaldehyde was added to the solution after readjusting the pH to 10.5-11.5 with sodium hydroxide. The mixture was left for 60 minutes followed by neutralization with acetic acid. Finally, 75 grams of ammonium chloride dissolved in 300 ml water was added under good stirring.

4.1.2 Preparation of Formula Containing N-methylolcarbamoylethyl Starch and PVA

The second sample (750 g) was dissolved in 5 L tap water at room temperature to yield a 15% aqueous solution of pH 10.5. The pH was adjusted to 6-7 using glacial acetic acid, and the temperature of the whole mixture was raised to 75^{o}C for 5-10 minutes. During this period, a mixture of 34 grams commercial wax and 82 grams commercial lubricant were added under good stirring. Cooling down to 40^{o}C and readjustment of the pH to 10.5-11.5 was followed by addition of 235 ml of 37% aqueous formaldehyde under good stirring. The temperature was kept at this level for 60 minutes. The pH of the solution was then decreased to 6-7 using glacial acetic acid (denoted SOLUTION A).

In another beaker 75 grams PVA was dissolved in 500ml water under good stirring followed by addition of 25 grams succinic acid dissolved in 100 ml water (denoted SOLUTION B). Solution (B) was added to solution (A) under good stirring followed by addition of 75 grams ammonium chloride dissolved in 300 ml water. After good mixing for 10 minutes, the sizing formula was ready for use**.**

4.1.3 Preparation of Formula Containing N-methylolcarbamoylethyl Starch, PVA and Melamine Formaldehyde

The third sample (750 g) was prepared exactly as mentioned for the second sample. But in addition, 75 grams melamine formaldehyde was added under good stirring. It is recommended that such formula should be used as soon as possible. So, the solution was subdivided into five portions, each of ca 1:1. Finally, the required melamine quantity of 15 grams was added.

4.2 Materials Used

4.2.1 Yarns

Three 30/1, 100% cotton ring-spun yarns were spun on a Rieter spinning frame at the College of Textiles at NCSU. The yarns have three different twist multipliers: 4.0, 4.2, and 4.4. These twist multipliers were used because they represent standard twist levels used by the textile industry for cotton warp yarns. Significant differences can be seen in such parameters as yarn strength, abrasion resistance, and size pick-up among yarns with different twist levels. For example, a yarn with low twist is typically weaker than a yarn with high twist to a certain twist level, after which, the yarn gets weaker.

4.2.2 Sizing Agents

The three prepared sizing agents were used to size the yarns. Each size was assigned a letter (A, B, and C) as indicated below.

- N-methylolcarbamoylethyl starch (*Size A*)
- N-methylolcarbamoylethyl starch + PVA (*Size B*)
- N-methylolcarbamoylethyl starch + PVA + melamine formaldehyde (*Size C*)

These sizing agents were prepared and applied to yarns separately. The purpose of using three different reactive agents was to compare and contrast their effects against each other and un-treated samples in order to discover which would be best for further research.

4.3 Design of Experiment

4.3.1 Experimental Design I

Full experimental design of three independent parameters (type of sizing agent, yarn twist multiple, and % size concentration in the size mix) was used to study the influence of these parameters and their interactions on responses of interest. Table 4.1 describes the independent variables used in the experiment.

Table 4.1 Experimental Design I

Independent Variable	**Levels**
Size Type	(3): Size A, Size B, Size C,
Yarn Twist Multiple	(3): 4.0 TM, 4.2 TM, 4.4 TM
% Solids	(5): Range: 5%-17%

The responses (dependent parameters) for Experimental Design I are:

- % Size add-on
- Yarn tensile properties before and after sizing (breaking force, breaking work, tenacity, and % elongation at break)
- Yarn hairiness
- Yarn-to-metal coefficient of friction
- Yarn abrasion resistance

The full experimental design adapted required a total of 48 runs: 3 runs for the un-treated yarns (one for each twist level) and 45 (3 x 3 x 5) runs for the sized yarns. Due to a lack of material, data for % size add-on, yarn hairiness, and the tensile properties are based on one observation for each. For the coefficient of friction and abrasion resistance, the data contains five replicates for each observation.

4.3.2 Experimental Design II

Full experimental design of three independent parameters (type of sizing agent, % size concentration in the size, and heat set temperature) was used to study the influence of these parameters and their interactions on responses of interest. Table 4.2 shows the independent variables used the experiment:

Table 4.2 Experimental Design II

Independent Variable	**Levels**
Size Type	(3): Size A, Size B, Size C
Launderings	(3): 15, 30, 45
Heat Set Temperature	(5): 160°F, 150°F, 140°F, 130°F, 0°F

The responses for Experimental Design II were measured in order to compare the stability of the sizing agents at different curing temperatures before and after repeated launderings of woven fabric samples in order to judge whether the sizes are removable. The responses are:

- % Weight loss
- Iodine Color Intensity

Due to the small size of the woven fabric, only three samples for each cured fabric could be taken. This allowed only one sample for each laundering level. The average of four color intensity measurements was observed for each iodine spot per sample.

4.3.3 Constant Parameters

Table 4.3 describes variables that were kept constant throughout the application of each sizing agent.

Table 4.3 Constant Parameters During Sizing Application

Variable	Value
Yarn Count	30/1
Machine Speed	112 meters/min
Drying Temperature	110°C
Heating Time	26 seconds

4.3.4 Preparation and Application of Materials

4.3.4.1 Size Solutions

Each sizing agent was applied to the yarn at five different % concentration (% solids) levels. They were diluted with tap water at room temperature to attain size solution concentrations between 5% and 17%. Because the viscosity and ingredients for the three sizing agents varied, the % solids used for each varied as well. For example, yarn applied with Size B at 17%, 16%, and 15% solids experienced frequent breaks due to its stickiness with different parts of the sizing machine. Therefore, it was necessary to use 13% solids as the highest level applied to Size B because the yarn ran well without breaking. The size application was performed on a single-end-sizing machine (ITOCHU-Yamada Corp., Osaka, Japan). The sizing speed and drying time remained constant as described in Table 4.3.

4.3.4.2 Woven Fabric Samples

Three 1-yard samples were woven at NCSU on a 24-inch AVL shuttle loom using 4.4 TM yarns containing the highest % concentration of each respective size type as

filling. The warp yarns were 16/2, 100% ring-spun cotton. The reed number on the loom was 16 dents/inch and the warp yarns were threaded 2 ends/dent. The woven samples had warp densities of 32 ends/inch and pick densities of 26 picks/inch measured off loom.

4.4 Responses

4.4.1 Definition of Responses

The 10 responses measured in the experiment are defined as:

- Size Add-on--the weight of solids left on a given weight of fabric after impregnation and drying [7].
- Breaking Force--the tensile force recorded at the moment of yarn rupture [7].
- Breaking Work--the total energy needed to rupture a yarn [9] represented by the area underneath the stress/strain curve; directly related to breaking force and elongation at break.
- Tenacity--the tensile force per unit linear density of an unstrained yarn [9].
- Elongation at Break--the increase in length when the last component of a yarn breaks [9].
- Hairiness--the total length of protruding fibers along one centimeter of yarn sample [11].
- Coefficient of Friction--the ratio of the tangential force needed to maintain relative motion between two contacting surfaces to the perpendicular force holding them in contact [2].

- Abrasion Resistance--the ability of a yarn to withstand surface rubbing and wear [9].
- Fabric Weight Loss--the difference in weight of a fabric sample before and after washing and drying.
- Color Intensity--the measurement of the color of a finish applied to a fabric sample expressed as the ratio of the absorption coefficient to the scattering coefficient [3].

4.4.2 Evaluation of Responses

4.4.2.1 Yarn Evaluation

The yarns were conditioned for at least 48 hours before testing under standard relative humidity and temperature. The sized and un-treated yarns were tested to evaluate their performance in weaving. The testers used to evaluate the yarns are:

- Uster Tensojet to measure yarn strength and elongation at break at high strain rates.
- Uster Tester 3 to measure yarn hairiness.
- Lawson Hemphill Friction Tester to measure yarn-to-metal coefficient of friction.
- Zweigle Abrasion Tester G 551 to measure yarn abrasion resistance.

The Uster Tensojet performs tensile tests on yarns at high strain rates similar to those that the warp yarns experience during weaving. There are four different strain rates available. The strain rate level 200 m/min was selected to simulate the strain on the warp yarns in low-speed weaving. The number of observations for each yarn was 1024 to account for yarn variability.

The Tensojet gives the mean values for breaking force, breaking elongation, tenacity, and breaking work. For each yarn a scatter graph showing all observations can be obtained. The total number of observations for each yarn is divided into 20 percentile groups to indicate the quality of the yarn and size application.

The Uster Tester 3 measures hairiness by optically monitoring the total length of protruding fibers along one centimeter of yarn sample. For example, a hairiness value of 3 corresponds to the total fiber length of 3 cm with reference to a sensing length of 1 cm [11]. This machine also provides the yarn count of each sample. This is needed for calculating the add-on percentage for each sized yarn. The % size add-on could not be controlled or measured during application. It could only be calculated after finding the actual yarn count of each yarn sample using the Uster Tester 3. The % size add-on can be found using treated and untreated sample counts and is expressed by the following equation:

$$\%\quad Add-on = \frac{(N_s - N_u)\times 100}{N_s}$$

where, N_s = count of sized yarn

N_u = count of untreated yarn.

The Lawson Hemphill Friction Tester measures the kinetic frictional properties of a moving yarn in contact with a solid material. The tester provides a direct measurement, meaning that the ratio of output tension to input tension is established directly and the coefficient of friction is indicated on a scale. ASTM D 3108-95 (Standard Test Method for Coefficient of Friction, Yarn to Solid Material) was used to measure the coefficient of friction [2].

The Zweigle Abrasion Tester G 551 consists of a rectilinear roller covered with emery paper that moves back and forth. Up to 20 pre-tensioned yarns (20g) are placed above the roller. All yarns are subjected to equal contact pressure and speed exertion by the roller. The abrasion roller slowly rotates about its own axis. Once a yarn has been worn out, it fails and activates an electronic evaluation unit that measures the amount of roller strokes for the break to occur.

4.4.2.2 Size Stability Evaluation

The fabric samples were conditioned for at least 48 hours before testing under standard relative humidity and temperature. The sized yarns were tested to evaluate the stability of the reactive sizes after multiple launderings. The apparatuses/testers used were:

- Launder-Ometer to wash the fabric samples. One run is equal to 5 home launderings.
- Datacolor SpectraFlash SF 300 to measure the color intensity of iodine drops placed on the samples.

Three 6”x1” samples were cut from each fabric (one to be washed 15 times, one to be washed 30 times and one to be washed 45 times). The samples were surged in order to avoid unraveling during the washing in the Launder-Ometer. The samples were washed and dried according to AATCC Test Method 61-2000 (Colorfastness to Laundering, Home and Commercial: Accelerated) [1]. After 15, 30, and 45 washes, the samples were weighed and the % weight loss was calculated using the following equation:

$$\%\ \ Weight\ \ \ Loss = \frac{(W_b - W_a) \times 100}{W_b}$$

where, W_b = weight before wash

W_a = weight after wash.

The SpectraFlash SF 300 is a dual channel spectrophotometer designed to measure color in both reflectance and transmittance mode at 10 mm intervals within the visual spectrum (360-700 ηm). For the purpose of this study, this instrument was used to measure the color intensity of 0.01% iodine solution droplets placed on the fabric samples according to the method described in reference [26] in order to detect the presence of starch. Starch produces a blue color in the presence of iodine and its spectral reflectance is determined by the spectrophotometer. The color intensity was measured at a wavelength of 540 nanometers because this setting provided the best results for each sample. Color intensity is expressed as:

$$K / S = \frac{(1-R)^2}{2R} - \frac{(1-R_o)^2}{2R_o}$$

where, R = the decimal fraction of the reflectance of the iodine substrate.

R_o = the decimal fraction of the untreated sample.

K = the absorption coefficient.

S = the scattering coefficient [3].

5. RESULTS AND DISCUSSION

In this chapter, the results of the two experimental designs in Table 4.1 and Table 4.2 are represented and discussed. For the first experimental design, the measured responses were size add-on, breaking force, tenacity, yarn elongation at break, breaking work, hairiness, yarn-to-metal coefficient of friction, and abrasion resistance. For the second experimental design, the measured responses were fabric sample weight reduction and iodine color intensity. The data sets for the experiments are shown in Tables A1-A4 in the appendix.

Statistical analyses using multiple regression models were performed to discover which parameters are significantly affecting each particular response. The multiple regression models were used to test for the main effects and, if any, their main interactions. Whenever an insignificant effect appeared in the effect test (e.g. a parameter effect with an F-ratio < 2), it was dropped from the model. Only the significant effects and/or interactions remained in a new model to be run again. All data were run in multiple regression analyses using JMP IN® 4 software in order to produce a prediction equation for each response [4].

The regression models were used to provide the predicted measurement for all three sizes at the same levels of solid concentration. These levels range from 5% to 20% increasing in increments of five. The presence of non-linear regressions was checked for each response.

For the predicted regressions shown in Figure 5.42 and Figure 5.48, the stability of the sizes are plotted as a function of the number of washings performed. The solid

concentration and twist multiple remain constant because they do not have a significant effect on the responses. All of the regressions were calculated using the prediction equation derived for each response. The predicted responses are expressed as:

$$\hat{Y}_i = Y_i - e_i \qquad i = 1, 2, 3, \ldots, n$$

where, $\hat{Y}_i$ = a corresponding predicted value

Y_i = a response observation

e_i = a residual.

The effect of the residuals on the predicted regressions explains the differences in the starting points for each predicted graph. In other words, the values for any particular response measured at zero % solids should be equal among the three size types. However, it can be seen that this is not the case in the graphs. This is due to the positive or negative deviation of the residuals from the response observations.

The abbreviations for the parameters are described as:

- ST_A = Size Type A
- ST_B = Size Type B
- ST_C = Size Type C
- ΔTM = Change in Twist Multiple = (TM – 4.0)
- PS = Percent Solids
- ΔW = Change in Number of Washes = (W – 15)

The transformations, ΔTM and ΔW, make the estimates more intuitively reasonable, but do not change the prediction values.

5.1 Size Add-on

5.1.1 Statistical Results for Size Add-on

Table 5.1 Model for Size Add-on

Response % Size Add-on
Whole Model
Summary of Fit

RSquare	0.85658
RSquare Adj	0.84164
Root Mean Square Error	1.493871
Mean of Response	5.267407
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	639.77065	127.954	57.3361
Error	48	107.11918	2.232	Prob > F
C. Total	53	746.88984		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ST	2	2	15.24998	3.4167	0.0410
ΔTM	1	1	13.46890	6.0354	0.0177
PS	1	1	588.20419	263.5737	<.0001
ΔTM*ΔTM	1	1	31.51440	14.1216	0.0005

Expanded Estimates

Nominal factors expanded to all levels

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	1.4588475	0.54842	2.66	0.0106
ST_A	-0.730307	0.288512	-2.53	0.0147
ST_B	0.5351266	0.290519	1.84	0.0717
ST_C	0.1951806	0.288034	0.68	0.5013
ΔTM	-3.058333	1.244892	-2.46	0.0177
PS	0.6429269	0.039601	16.23	<.0001
(ΔTM-0.2)*(ΔTM-0.2)	-40.51389	10.78108	-3.76	0.0005

Table 5.1 shows the multiple regression results for size add-on. *Size Type, ΔTM, % Solids,* and the interaction of *ΔTM* with itself show significant effects on the response. The R-Square value of 85.66% indicates a relatively good fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for size add-on is:

$$Y = 1.4588475 - 0.730307\mathbf{ST_A} + 0.5351266\mathbf{ST_B} + 0.1951806\mathbf{ST_C} - 3.058333\mathbf{\Delta TM} + 0.6429269\mathbf{PS} - [40.51389(\mathbf{\Delta TM}-0.2)(\mathbf{\Delta TM}-0.2)]$$

where, $ST_A = \begin{Bmatrix} 1 \\ 0, \quad otherwise \end{Bmatrix}$, $ST_B = \begin{Bmatrix} 1 \\ 0, \quad otherwise \end{Bmatrix}$,

$ST_C = \begin{Bmatrix} 1 \\ 0, \quad otherwise \end{Bmatrix}$.

5.1.2 Observation and Prediction Analyses for Size Add-on

Figures 5.1-5.3 depict the results for the actual observations of % add-on as a function of % solids for the three reactive sizes and yarns used. The 4.0 TM yarn treated with Size B has an unusually high % size add-on compared to the other two sizes at greater solid concentration levels. The overall trend suggests that % add-on increases as the solid concentration increases. However, some of the graphs reveal that % add-on increases, then decreases, and increases again as % solids increase. This is related to the variation of yarn count since the % add-on was calculated using the count before and after sizing (as measured by the Uster 3 tester). The most accurate method to determine the % add-on is by measuring the weight of a sized yarn and the weight of the same yarn after de-sizing. This method cannot be employed here because due to the difficulty involved in removing reactive sizes.

Figures 5.4-5.6 show linear regressions for size add-on as a function of solid content. Yarns treated with Size B show the best add-on at all three twist levels. Figure 5.7 shows a non-linear regression for all size types as a function of twist multiple. The size add-on initially increases as the twist multiple increases then decreases after 4.2 TM.

The example, based on predicted data at 10% solids, is typical at all levels of solid concentration.

Figure 5.1 Size Add-on vs. Solid Content (Twist Multiple = 4.0)

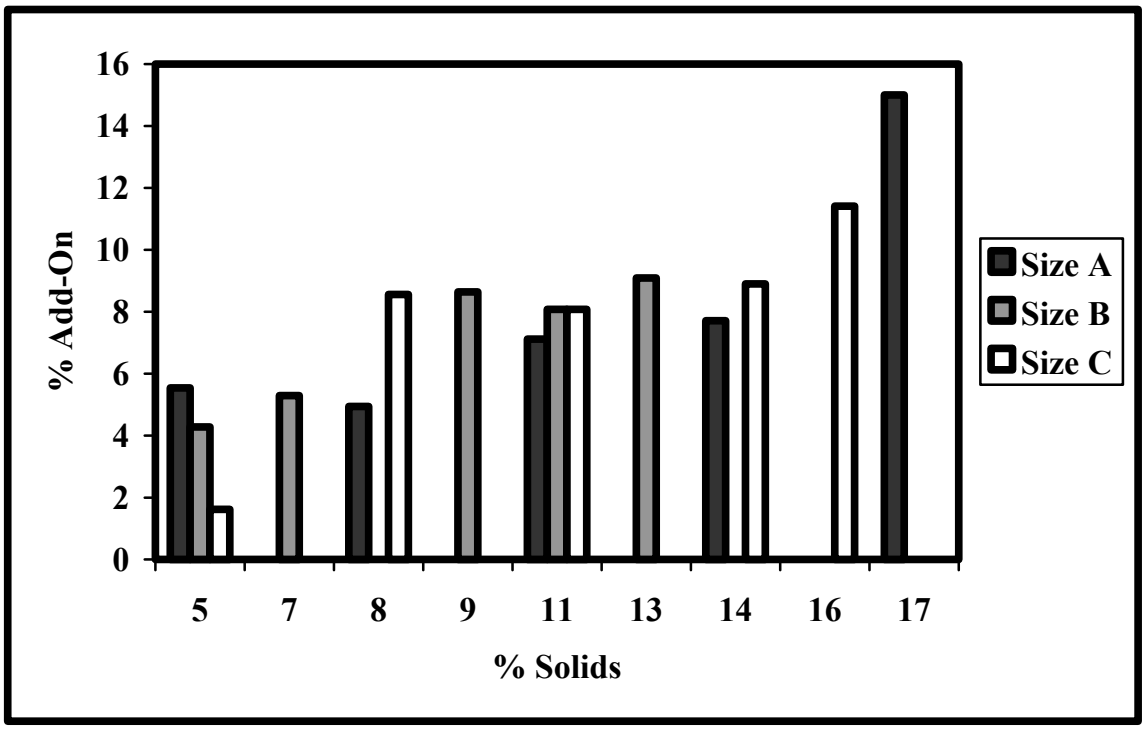


Figure 5.2 Size Add-on vs. Solid Content (Twist Multiple = 4.2)

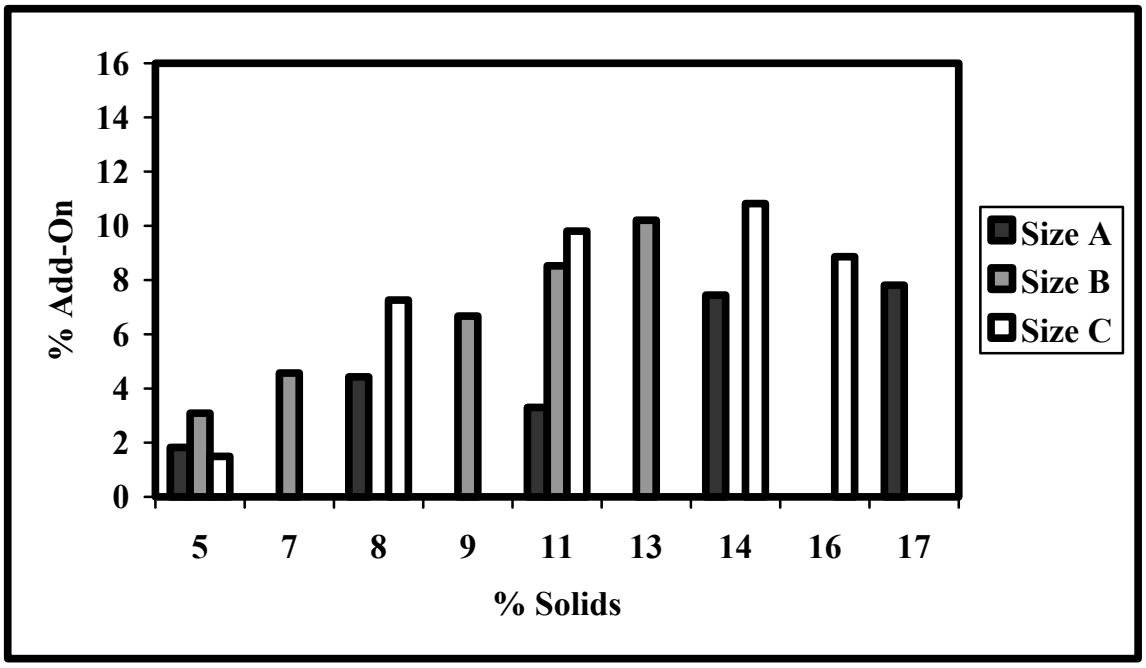


Figure 5.3 Size Add-on vs. Solid Content (Twist Multiple = 4.4)

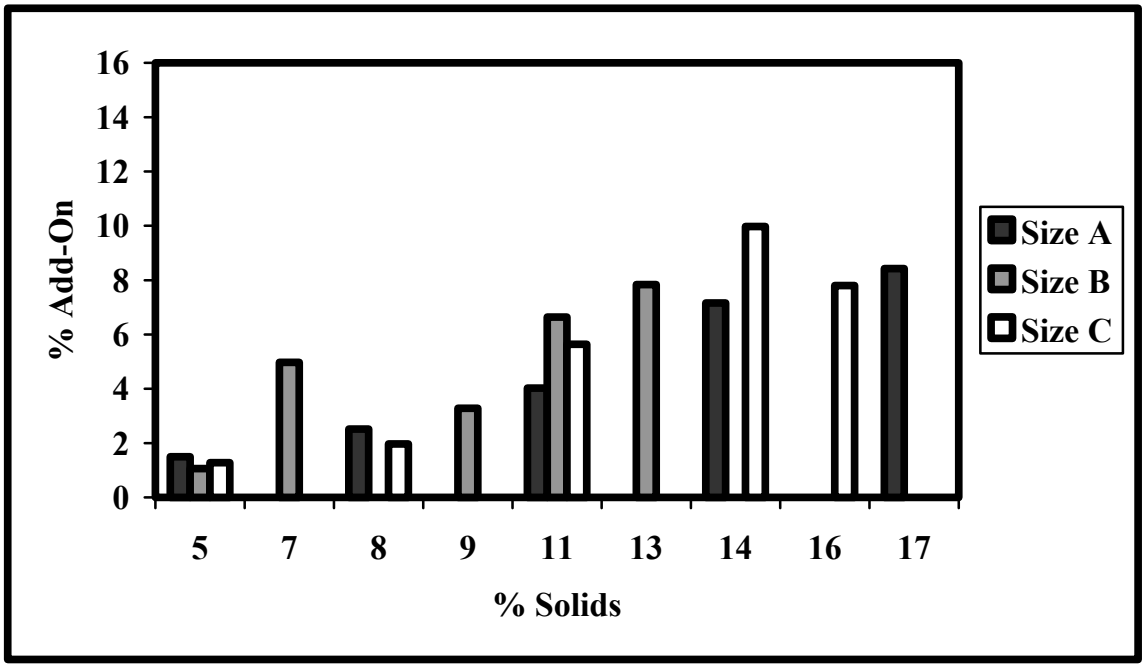


Figure 5.4 Predicted Size Add-on vs. Solid Content (Twist Multiple = 4.0)

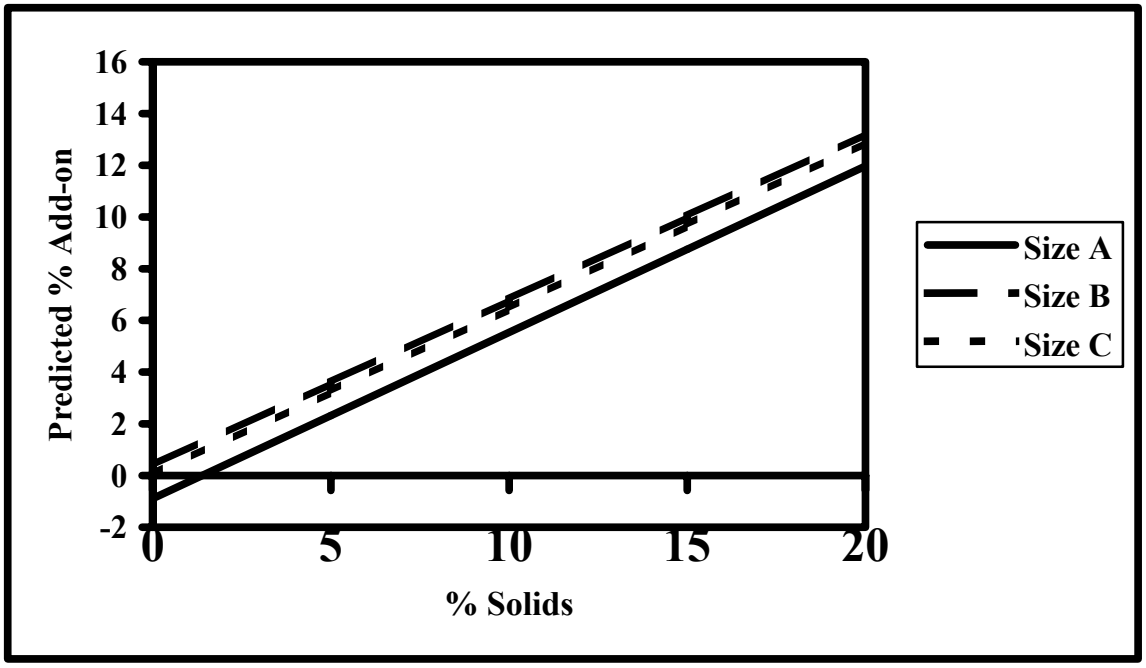


Figure 5.5 Predicted Size Add-on vs. Solid Content (Twist Multiple = 4.2)

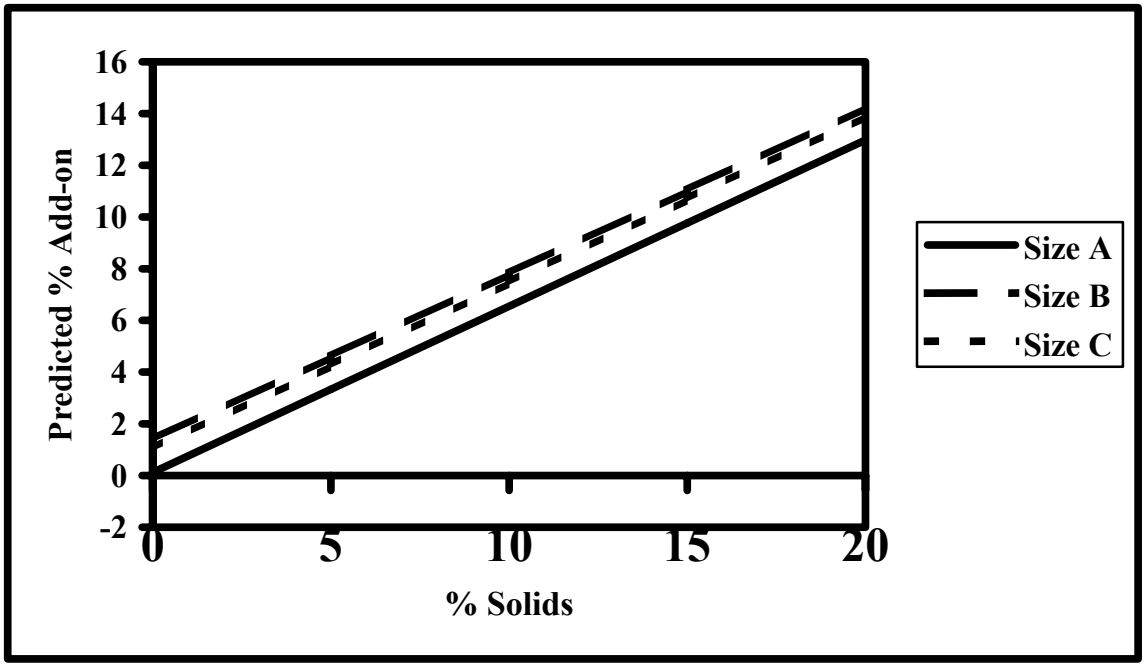


Figure 5.6 Predicted Size Add-on vs. Solid Content (Twist Multiple = 4.4)

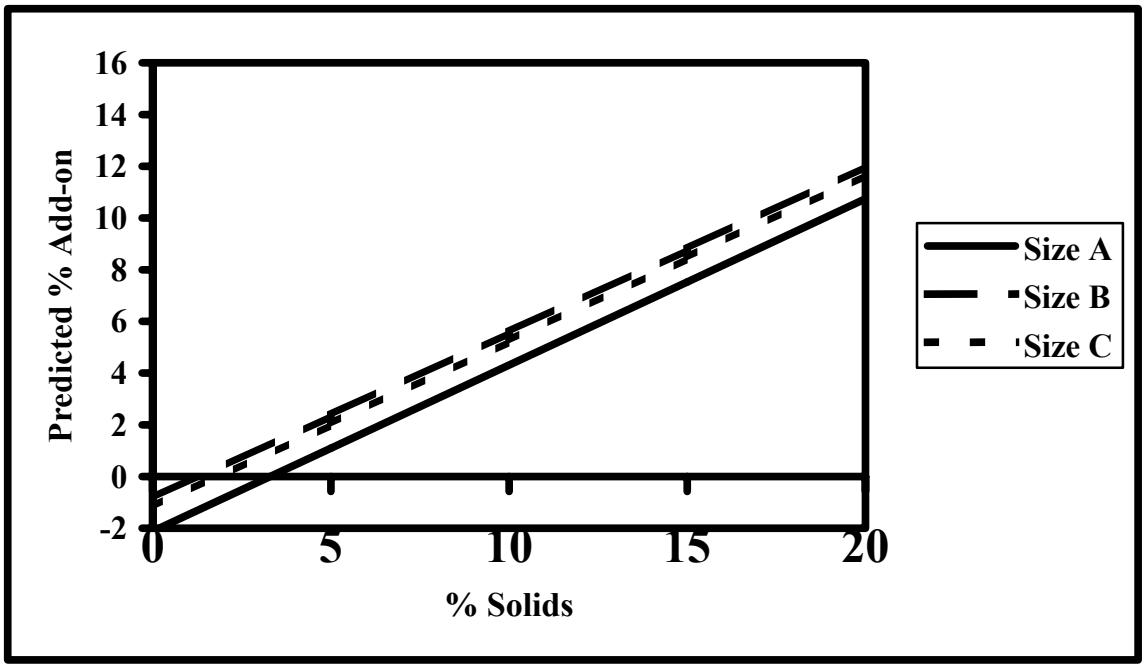
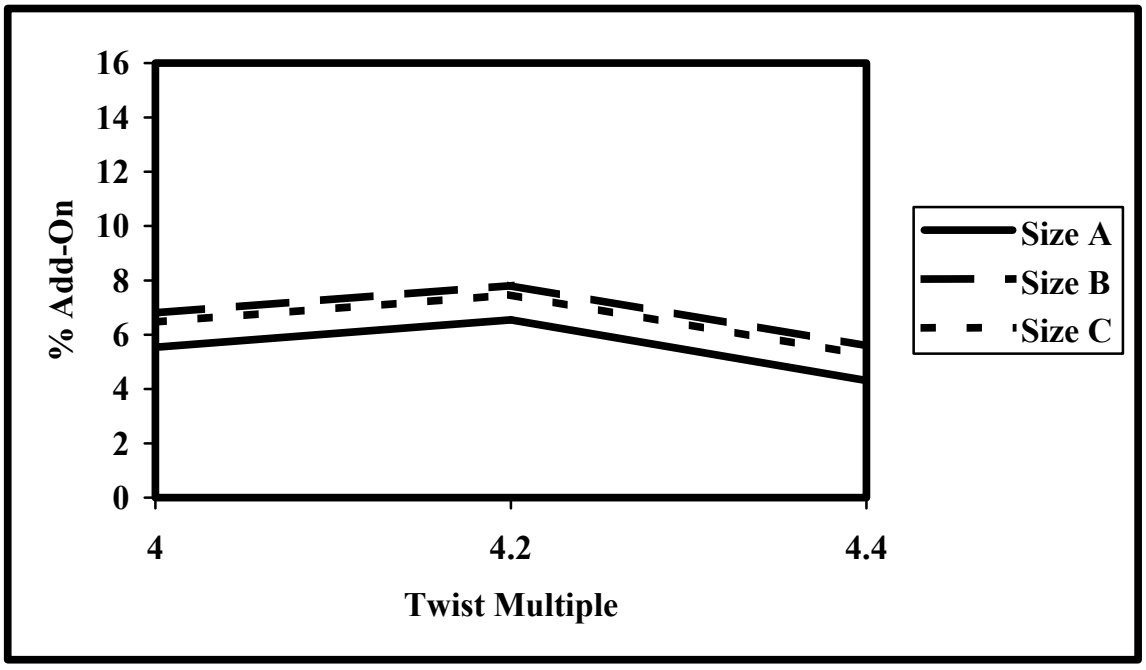


Figure 5.7 Predicted % Add-on vs. Twist Multiple (Solid Content = 10%)



5.2 Breaking Force

5.2.1 Statistical Results for Breaking Force

Table 5.2 Model For Breaking Force

Response B-Force (cN)
Whole Model
Summary of Fit

RSquare	0.793482
RSquare Adj	0.781091
Root Mean Square Error	8.802677
Mean of Response	292.0056
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	14886.012	4962.00	64.0365
Error	50	3874.356	77.49	Prob > F
C. Total	53	18760.368		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ΔTM	1	1	5176.8025	66.8085	<.0001
PS	1	1	9379.7772	121.0495	<.0001
PS*PS	1	1	1458.3736	18.8208	<.0001

Expanded Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	252.63424	3.176383	79.54	<.0001
ΔTM	59.958333	7.335564	8.17	<.0001
PS	2.5990539	0.236229	11.00	<.0001
(PS-8.55556)*(PS-8.55556)	0.1911042	0.04405	4.34	<.0001

Table 5.2 shows the multiple regression results for breaking force. Δ*TM, % Solids,* and the interaction of *% Solids* with itself show significant effects on the response. The R-Square value of 79.35% indicates a relatively good fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for breaking force is:

$$Y = 252.63424 + 59.958333\Delta TM + 2.5990539PS + [0.1911042(PS-8.55556)(PS-8.55556)]$$

5.2.2 Observation and Prediction Analyses for Breaking Force

Figures 5.8-5.10 depict the results of the actual observations for breaking force (or tensile strength at break). It can be seen from these figures that yarns treated with the Size C yield close results to the other sizing agents. An increasing trend in breaking force occurs as the solid concentration increases. The breaking force of Size B for the 4.0 TM yarn appears unusually high at 13% solids. The high size add-on depicted in Figure 5.1 for this particular yarn may explain this. At 5% and 11% solids, Size C is very competitive with the other two sizes for all twist levels.

According to the regression model, size type does not have a significant effect on yarn breaking force. The prediction equation produces equal values for all three sizes at any solid content. The levels of twist and solid content have significant effects on breaking force depicted in the non-linear regressions in Figure 5.11. The breaking force increases at an increasing rate as % solids and twist multiple increase.

Figure 5.8 Breaking Force vs. Solid Content (Twist Multiple = 4.0)

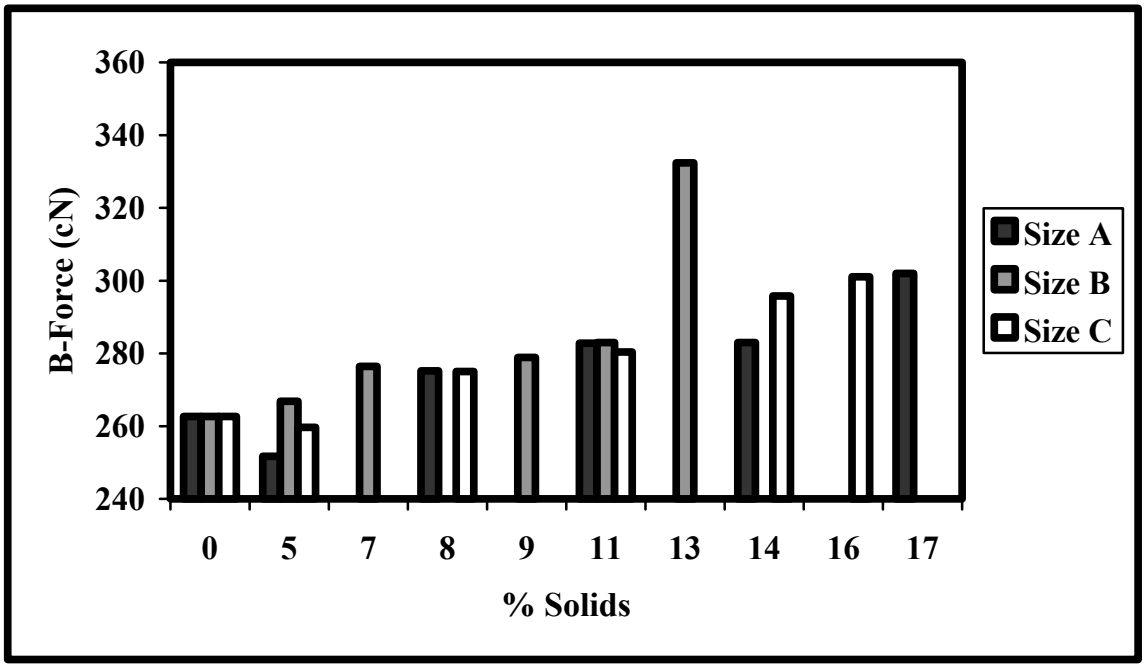


Figure 5.9 Breaking Force vs. Solid Content (Twist Multiple = 4.2)

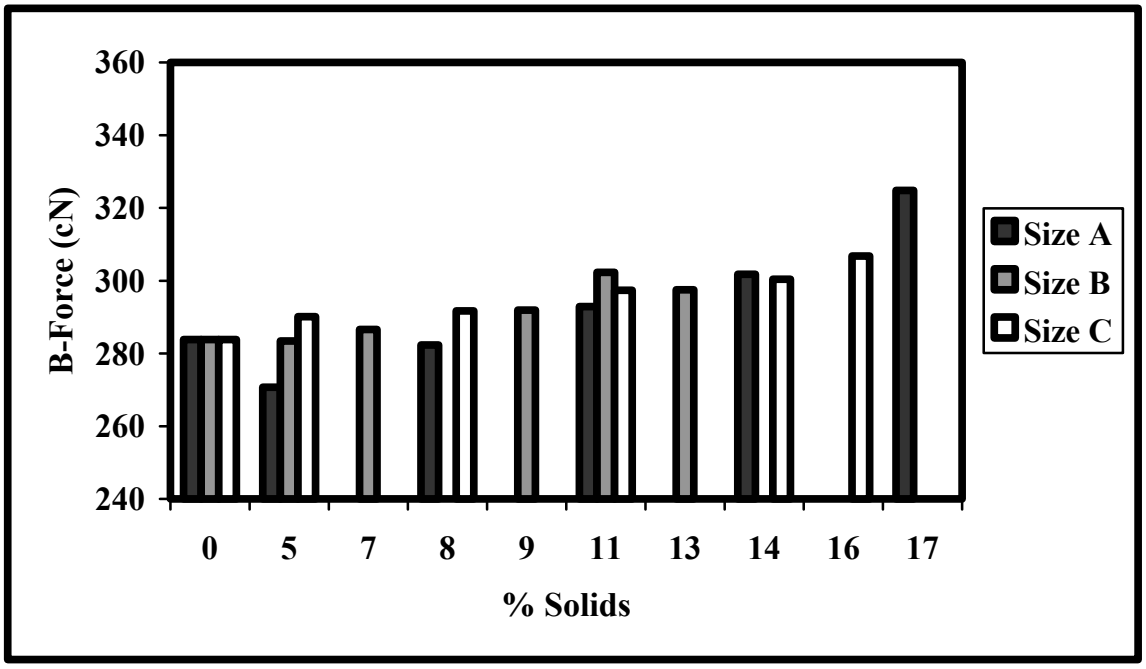


Figure 5.10 Breaking Force vs. Solid Content (Twist Multiple = 4.4)

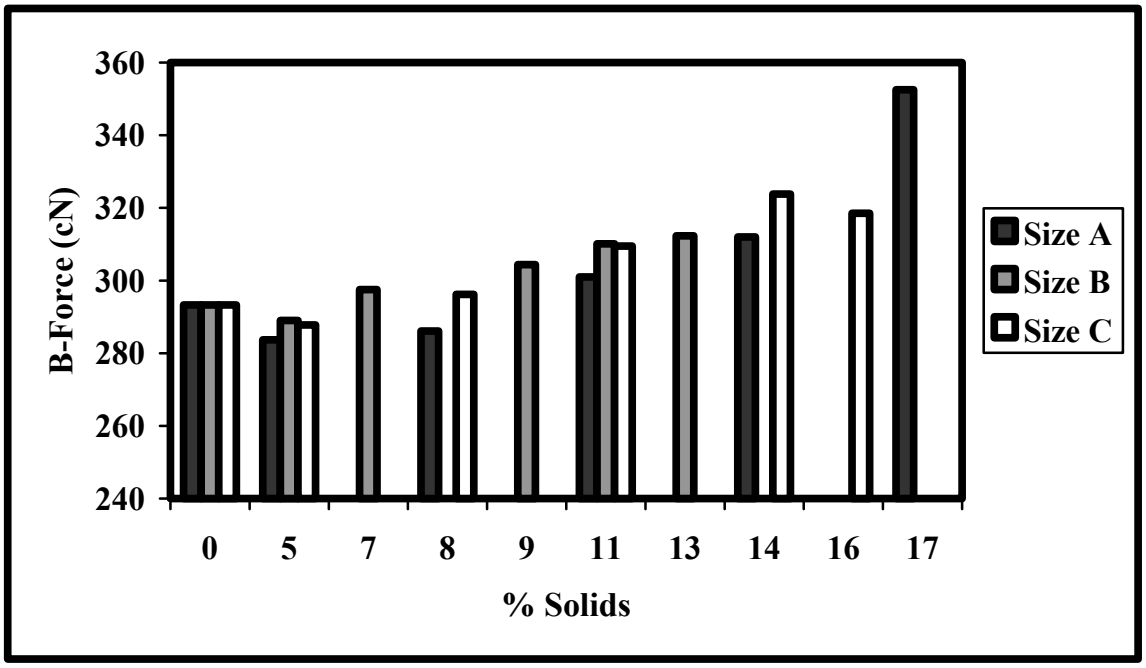
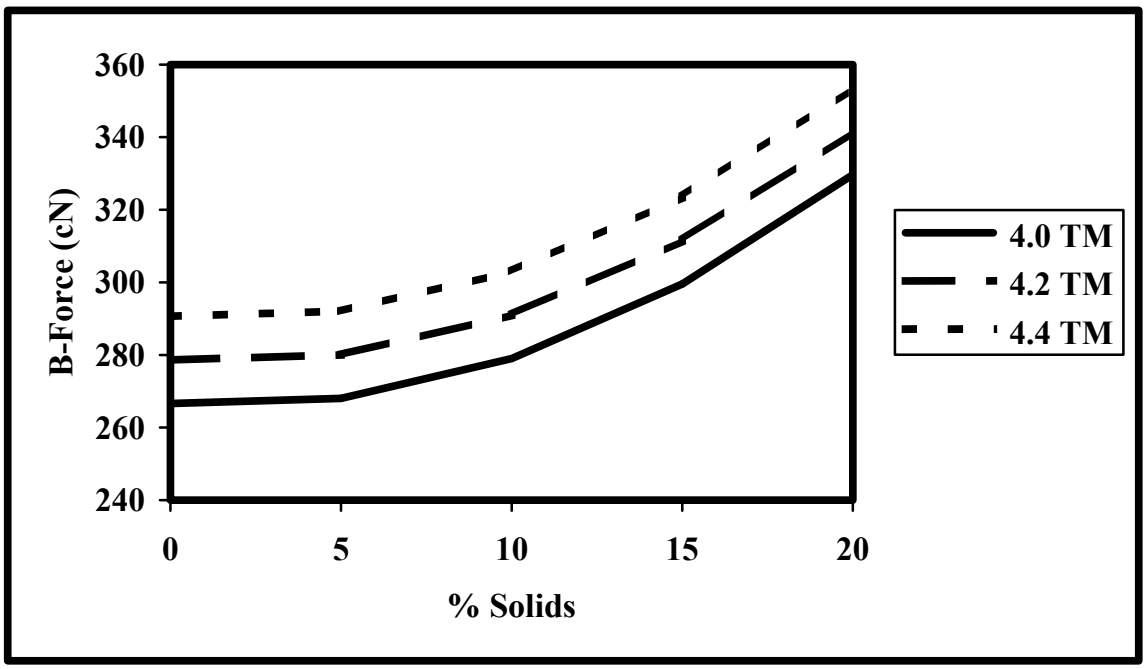


Figure 5.11 Predicted Breaking Force vs. Solid Content (Size Type Insignificant)



5.2.3 Weak Point Analysis for Breaking Force

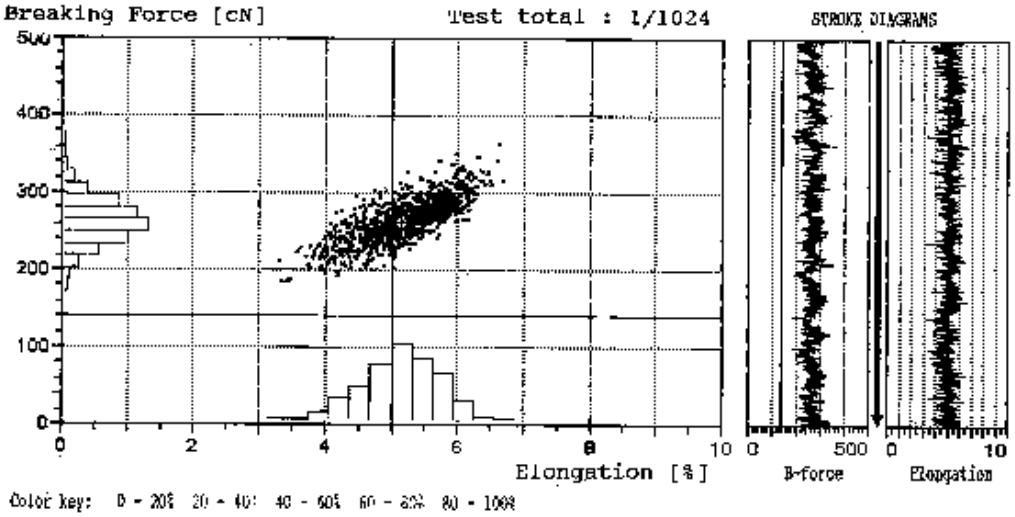
During weaving, the weakest point of a yarn is the point at which the yarn will break. Therefore, it is important to compare the weak points of yarn treated with sizing to the untreated yarns. This can be accomplished by studying the Uster Tensojet quality reports for the tested yarns. Each report contains a histogram positioned at the left in a graph. The histogram shows the distribution for yarn breaking force corresponding to the 1024 tested points in the center of the graph. Figures 5.12-5.14 represent the quality reports for the 4.0 TM, 4.2 TM, and 4.4 TM untreated yarns. The weakest point for all three yarns in terms of breaking force appears to be around 190 cN. Figures 5.15-5.17 show the quality reports for each size type at 11% solids (Size A with 4.0 TM, Size B with 4.2 TM, and Size C with 4.4 TM) as examples of comparison. Each figure shows that the weakest point for each respective sized yarn is at least above 200 cN. (This was typical for all treated yarns). This indicates that all three sizes increased the weakest point of the yarns, thus improving the overall breaking force for each sample.

Figure 5.12 Uster Quality Report for 4.0 TM, Untreated Yarn

USTER TENSOJET R3.31 FR 20-06-80 16:36 Operator: Todd Page: 1
N.C.STATE UNIVERSITY

Article number: Test number: 46 Mean Count: 20 tex TestCounter: 1.2R
Untreated, 4.0tm TesterNo: 4716-1024
Tests: 1/ 1024 V = 200 m/min. FV = 20.00 cN

USTER QUALITY REPORT



Color key: 0 - 20% 20 - 40% 40 - 60% 60 - 80% 80 - 100%

	B-force [cN]	Elongation [%]	Tenacity [cN/tex]	B-work [cN.cm]
Overall results: (total)				
1 Test / 1024 Single tests				
Mean values	262.7	5.15	13.14	373.9
CV%	10.19	11.09	10.19	19.02
s +/-	26.8	0.57	1.34	71.1
Q95% +/-	1.6	0.03	0.08	4.4
Max values	362.5	6.68	18.13	632.2
Min values	182.4	3.30	9.12	158.3
P0.01:(0)				
P0.05:(0)				
P0.1 :(1)	182.4	3.30	9.12	158.3
P0.5 :(5)	195.9	3.64	9.80	216.3
P1.0 :(10)	201.0	3.82	10.05	220.5

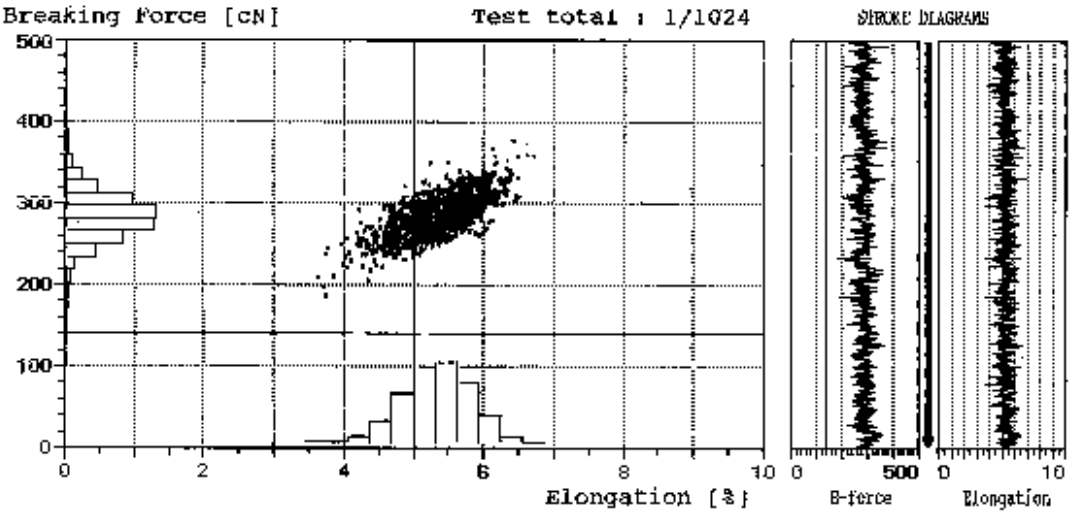
Weak points (140cN,5%) F = 0 E = 384 F+E = 0

Figure 5.13 Uster Quality Report for 4.2 TM, Untreated Yarn

USTER TENSOJET R3.31 FR 20-06-80 16:42 Operator: Todd Page: 1
N.C.STATE UNIVERSITY

Article number: Test number: 47 Mean.Count: 20 tex TestCounter: 1.2M
untreated, 4.2tm TesterNo: 4716-1024
Tests: 1/ 1024 V = 200 m/min. FV = 10.00 cN

USTER QUALITY REPORT



Color key: 0 - 20% 20 - 40% 40 - 60% 60 - 80% 80 - 100%

	B-force [cN]	Elongation [%]	Tenacity [cN/tex]	B-work [cN.cm]
Overall results: (total)				
1 Test / 1024 Single tests				
Mean values	283.8	5.33	14.19	416.9
CV%	9.09	9.45	9.09	16.54
s +/-	25.8	0.50	1.29	68.9
Q95% +/-	1.6	0.03	0.08	4.2
Max values	377.2	6.70	18.86	670.1
Min values	186.8	3.62	9.34	190.1
P0.01:(0)				
P0.05:(0)				
P0.1 :(1)	186.8	3.62	9.34	190.1
P0.5 :(5)	217.2	3.86	10.86	239.5
P1.0 :(10)	223.0	4.04	11.15	255.4

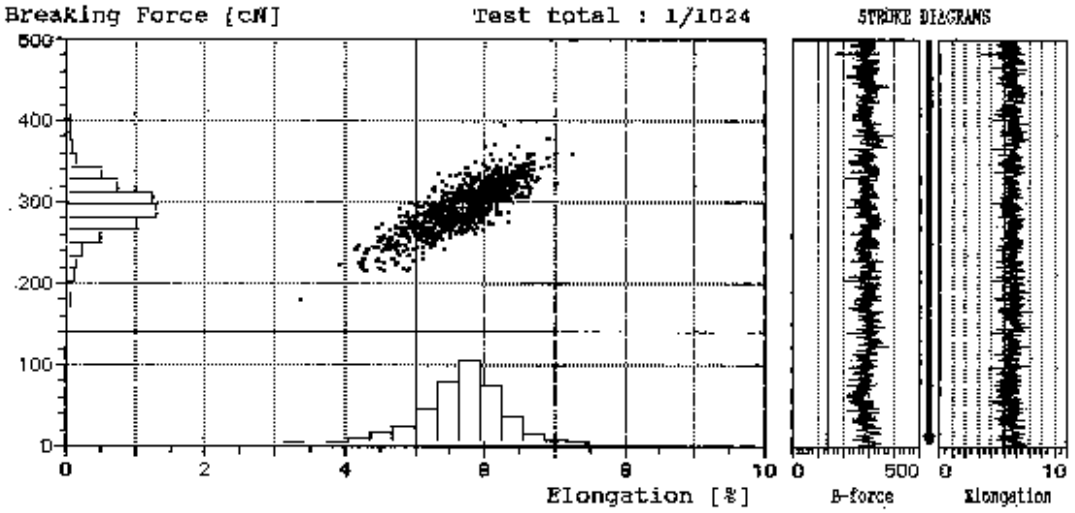
Weak points (140cN,5%) F - 0 E - 253 F+E - 0

Figure 5.14 Uster Quality Report for 4.4 TM, Untreated Yarn

USTER TENSOJET R3.31 FR 20-06-80 16:48 Operator: Todd Page: 1
N.C.STATE UNIVERSITY

Article number: Test number: 48 Mean Count: 20 tex TestCounter: 1.2M
untreated, 4.4tm TesterNo: 4716-1024
Tests: 1/ 1024 v = 200 m/min. fY = 10.00 cN

USTER QUALITY REPORT

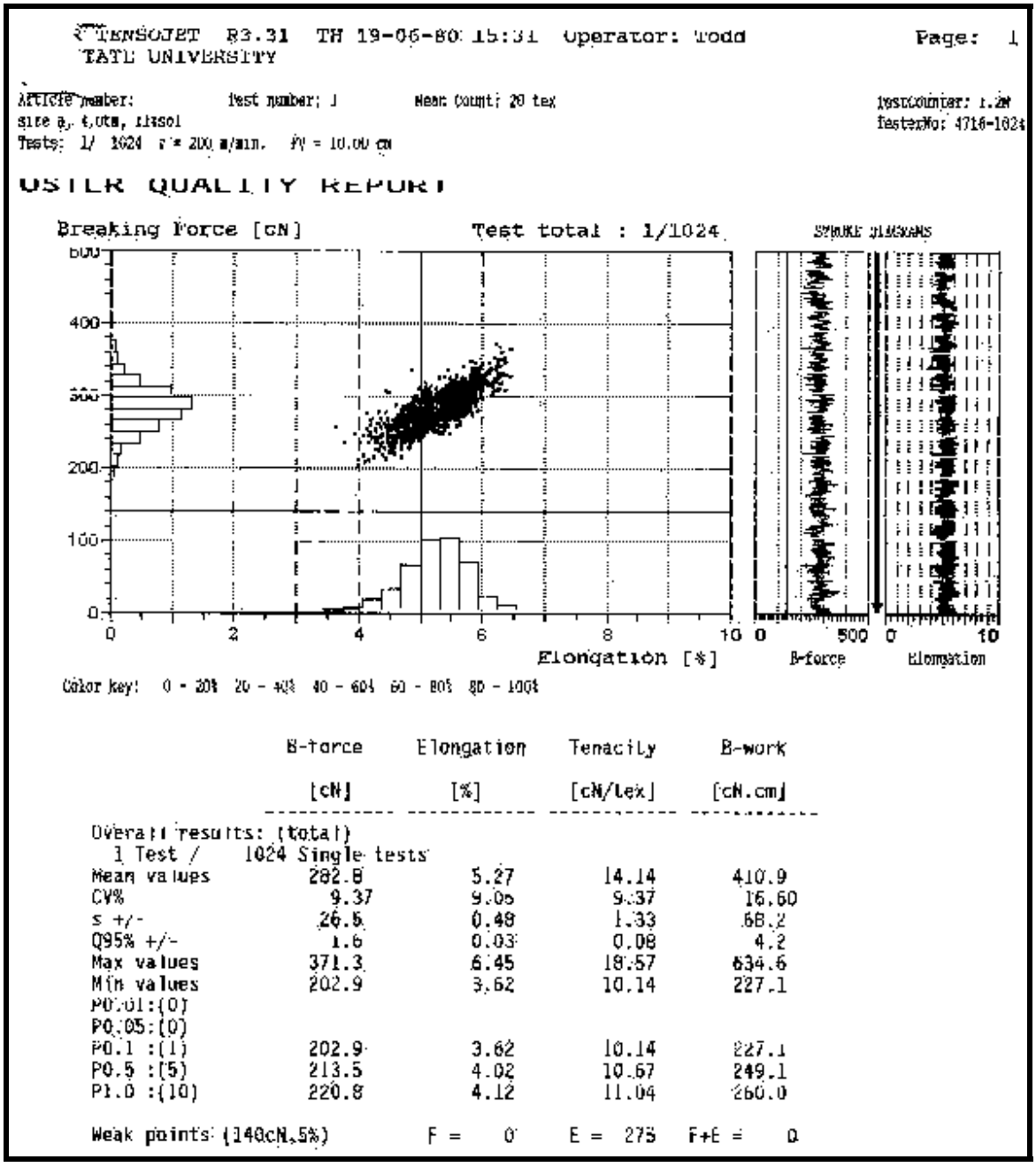


Color key: 0 - 20% 21 - 40% 40 - 60% 60 - 80% 80 - 100%

	B-force [cN]	Elongation [%]	Tenacity [cN/tex]	B-work [cN.cm]
Overall results: (total)				
1 Test / 1024 Single tests				
Mean values	293.3	5.68	14.66	458.0
CV%	9.34	9.07	9.34	16.44
s +/-	27.4	0.51	1.37	75.3
Q95% +/-	1.7	0.03	0.08	4.6
Max values	393.3	7.23	19.67	701.2
Min values	178.7	3.37	8.94	167.2
P0.01:(0)				
P0.05:(0)				
P0.1 :(1)	178.7	3.37	8.94	167.2
P0.5 :(5)	216.4	4.24	10.82	257.4
P1.0 :(10)	222.3	4.30	11.11	267.9

Weak points (140cN,5%) F = 0 E = 100 F+E = 0

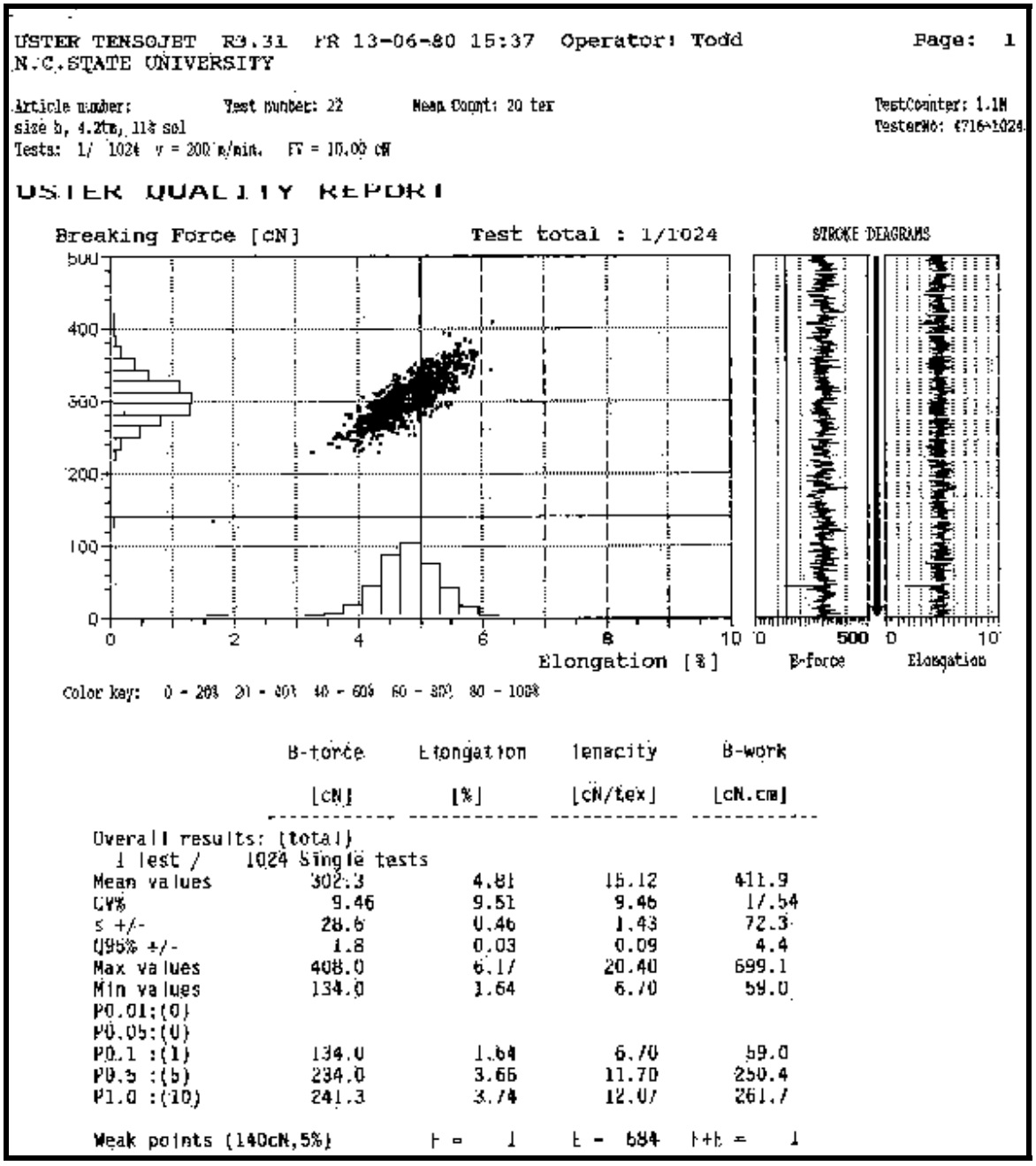
Figure 5.15 Uster Quality Report for 4.0 TM Yarn Treated with Size A (11% Solids)



	B-force [cN]	Elongation [%]	Tenacity [cN/tex]	B-work [cN.cm]
Overall results: (total)				
1 Test / 1024 Single tests				
Mean values	282.8	5.27	14.14	410.9
CV%	9.37	9.05	9.37	16.60
s +/-	26.5	0.48	1.33	68.2
Q95% +/-	1.6	0.03	0.08	4.2
Max values	371.3	6.45	18.57	634.6
Min values	202.9	3.62	10.14	227.1
P0.01:(0)				
P0.05:(0)				
P0.1 :(1)	202.9	3.62	10.14	227.1
P0.5 :(5)	213.5	4.02	10.67	249.1
P1.0 :(10)	220.8	4.12	11.04	260.0

Weak points (140cN,5%) F = 0 E = 275 F+E = 0

Figure 5.16 Uster Quality Report for 4.2 TM Yarn Treated with Size B (11% Solids)



USTER TENSOJET R3.31 FR 13-06-80 15:37 Operator: Todd Page: 1
N.C.STATE UNIVERSITY

Article number: Test number: 22 Mean Count: 20 ter TestCounter: 1.1M
size b, 4.2tm, 11% sol TesterNo: 6716-1024
Tests: 1/ 1024 v = 200 m/min. FT = 10.00 cN

USTER QUALITY REPORT

Breaking Force [cN] Test total : 1/1024 STROKE DIAGRAMS

Elongation [%] B-force Elongation

Color key: 0 - 20% 20 - 40% 40 - 60% 60 - 80% 80 - 100%

	B-force [cN]	Elongation [%]	Tenacity [cN/tex]	B-work [cN.cm]
Overall results: (total)				
1 Test / 1024 Single tests				
Mean values	302.3	4.81	15.12	411.9
CV%	9.46	9.51	9.46	17.54
s +/-	28.6	0.46	1.43	72.3
Q95% +/-	1.8	0.03	0.09	4.4
Max values	408.0	6.17	20.40	699.1
Min values	134.0	1.64	6.70	59.0
P0.01:(0)				
P0.05:(0)				
P0.1 :(1)	134.0	1.64	6.70	59.0
P0.5 :(5)	234.0	3.66	11.70	250.4
P1.0 :(10)	241.3	3.74	12.07	261.7

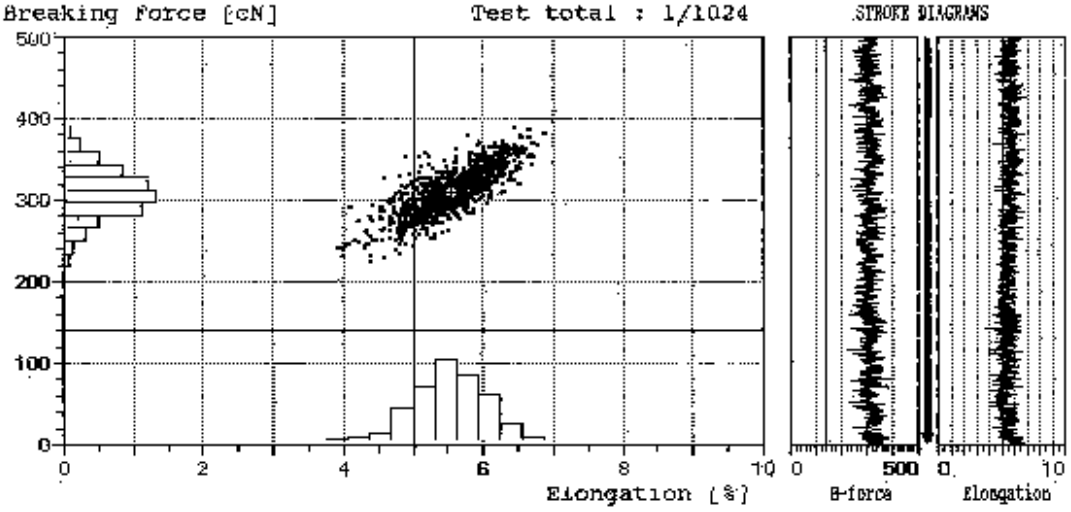
Weak points (140cN,5%) F = 1 E = 684 F+E = 1

Figure 5.17 Uster Quality Report for 4.4 TM Yarn Treated with Size C (11% Solids)

USTER TENSOJET R3.31 FR 20-06-80 16:18 Operator: Todd Page: 1
N.C.STATE UNIVERSITY

Article number: Test number: 45 Mean Count: 20 tex TestCounter: 1.2K
size c, 4.4tm, 11%sol TesterNo: 4/16-1024
Tests: 1/ 1024 v = 200 m/min. FF = 10.00 cN

USTER QUALITY REPORT



Color key: 0 - 20% 20 - 40% 40 - 60% 60 - 80% 80 - 100%

	B-force [cN]	Elongation [%]	Tenacity [cN/tex]	B-work [cN.cm]
Overall results: (Total)				
1 Test / 1024 Single tests				
Mean values	309.5	5.53	15.47	464.9
CV%	8.98	8.98	8.98	16.09
s +/-	27.8	0.50	1.39	74.8
Q95% +/-	1.7	0.03	0.09	4.6
Max values	388.2	6.86	19.41	689.4
Min values	224.9	3.90	11.24	248.8
P0.01:(0)				
P0.05:(0)				
P0.1 :(1)	224.9	3.90	11.24	248.8
P0.5 :(5)	237.7	4.08	11.88	270.3
P1.0 :(10)	244.3	4.30	12.21	289.8

Weak points (140cN,5%) F = 0 E = 148 F+E = 0

5.3 Tenacity

5.3.1 Statistical Results for Tenacity

Table 5.3 Model for Tenacity

Response Tenacity (cN/tex)
Whole Model
Summary of Fit

RSquare	0.725504
RSquare Adj	0.709034
Root Mean Square Error	0.505124
Mean of Response	14.57556
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	33.718625	11.2395	44.0507
Error	50	12.757508	0.2552	Prob > F
C. Total	53	46.476133		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ΔTM	1	1	12.484444	48.9298	<.0001
PS	1	1	20.617983	80.8072	<.0001
PS*PS	1	1	2.978919	11.6752	0.0013

Expanded Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	12.711679	0.18227	69.74	<.0001
ΔTM	2.9444444	0.420937	6.99	<.0001
PS	0.1218547	0.013556	8.99	<.0001
(PS-8.55556)*(PS-8.55556)	0.008637	0.002528	3.42	0.0013

Table 5.3 shows the multiple regression results for tenacity. Δ*TM, % Solids,* and the interaction of *% Solids* with itself show significant effects on the response. The R-Square value of 72.55% indicates a relatively good fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for tenacity is:

$$Y = 12.711679 + 2.9444444\boldsymbol{\Delta TM} + 0.1218547\boldsymbol{PS} + [0.008637(\boldsymbol{PS}-8.55556)(\boldsymbol{PS}-8.55556)]$$

5.3.2 Observation and Prediction Analyses for Tenacity

Figures 5.18-5.20 depict the results of the actual observations for tenacity. Overall, an increasing trend in tenacity occurs as the solid concentration increases. At 5% solids, the tenacity for yarn treated with Size A is consistently lower than the untreated yarn at all levels of TM. The 4.0 TM yarn treated with Size B shows an unusually high breaking load at 13% solids. The high % add-on for this particular concentration and twist level may be the cause.

The regression model shows that size type does not have a significant effect on yarn tenacity. The prediction equation produces equal values for all three sizes at any solid content. The levels of twist and solid concentration have significant effects on tenacity depicted. The non-linear regressions in Figure 5.21 show that the tenacity of the yarns increases at an increasing rate as % solids and twist multiple increase.

Figure 5.18 Tenacity vs. Solid Content (Twist Multiple = 4.0)

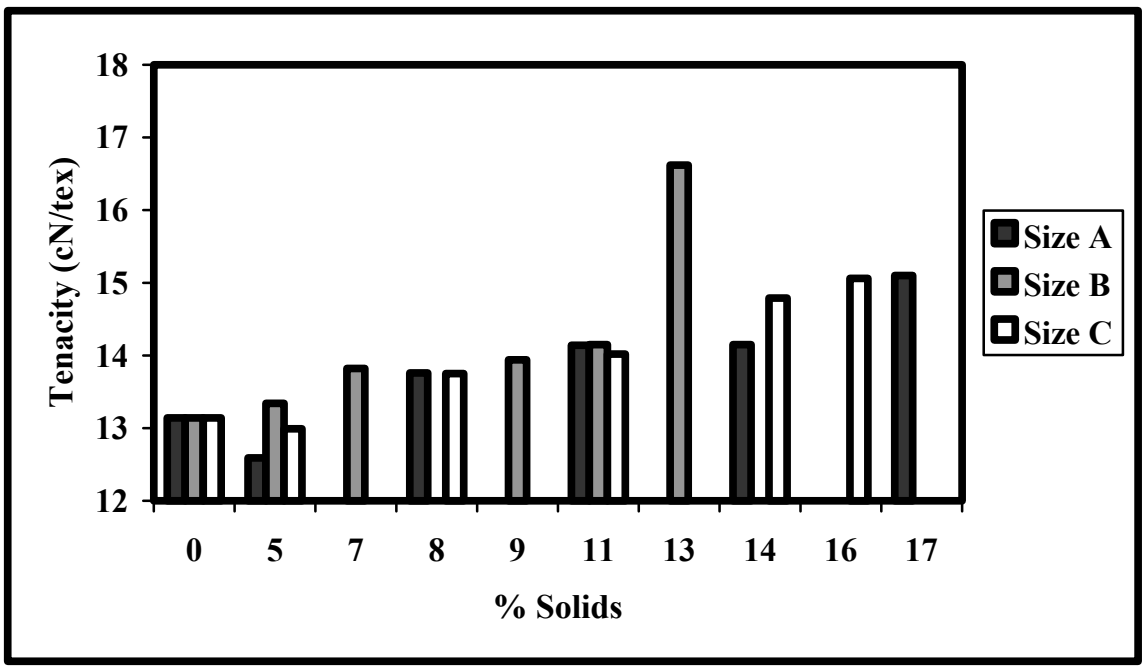


Figure 5.19 Tenacity vs. Solid Content (Twist Multiple = 4.2)

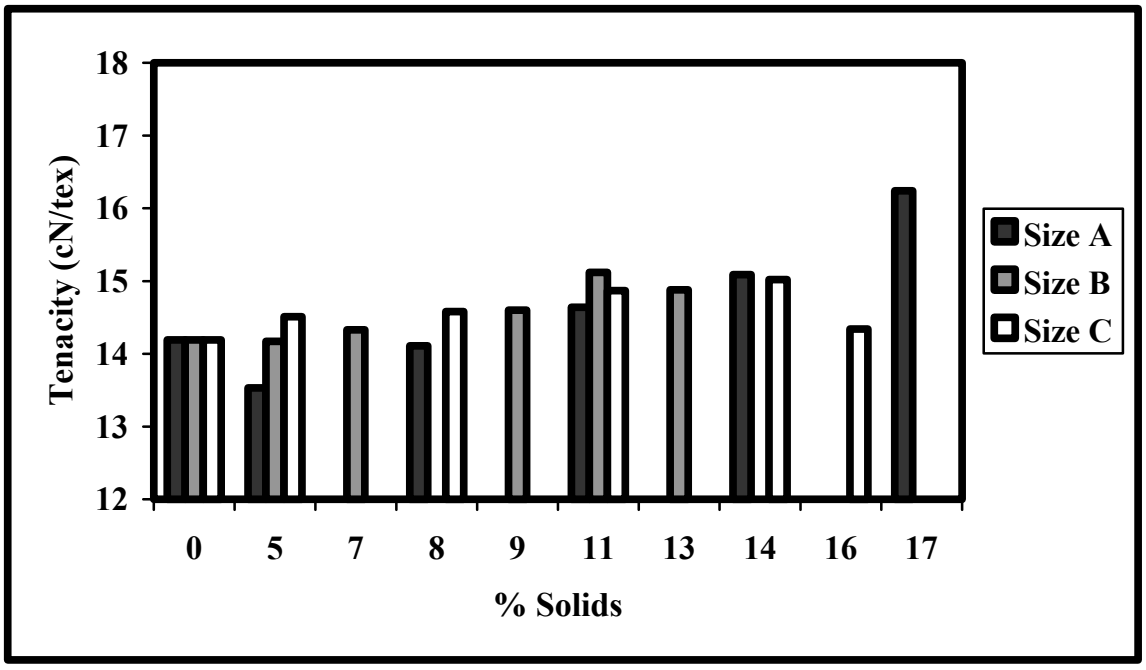


Figure 5.20 Tenacity vs. Solid Content (Twist Multiple = 4.4)

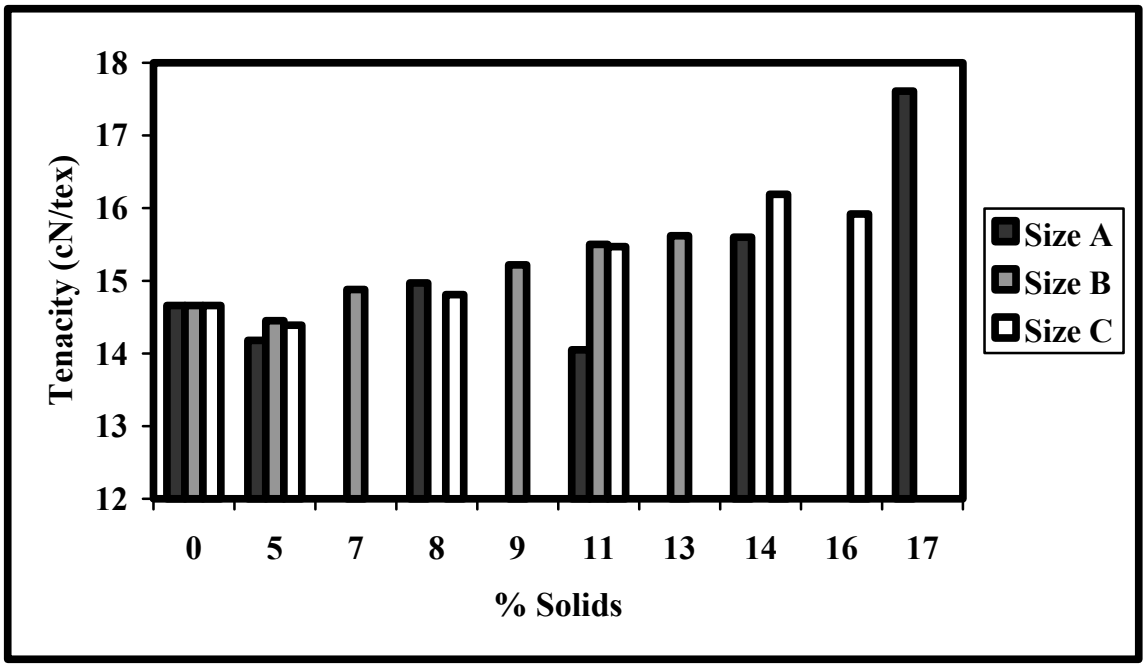
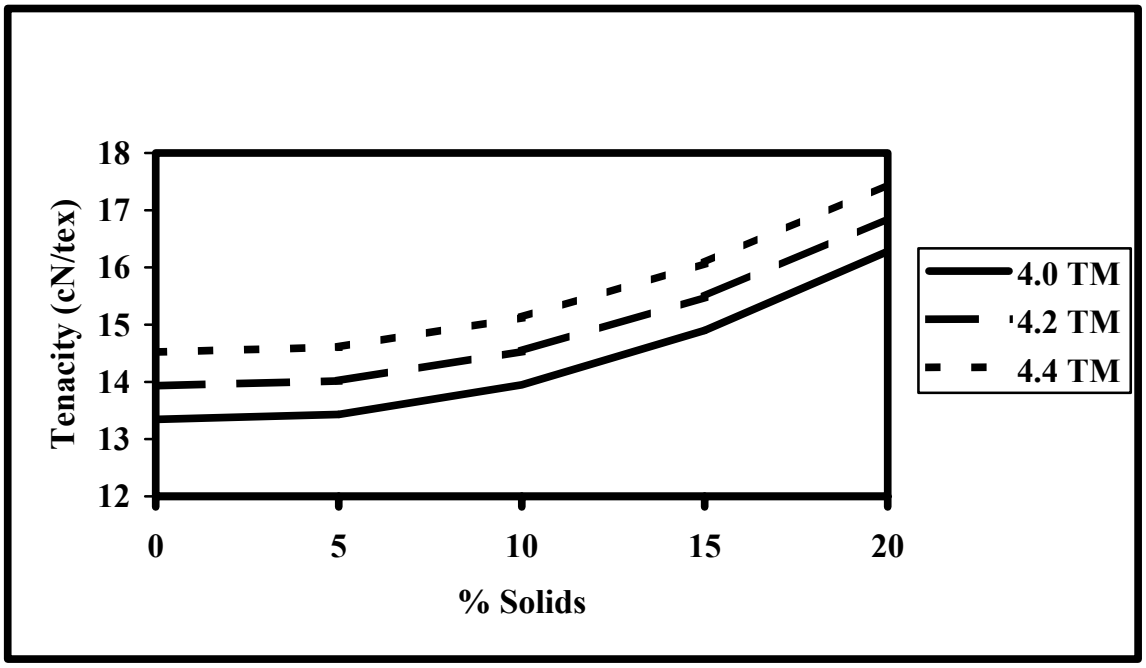


Figure 5.21 Predicted Tenacity vs. Solid Content
(Size Type Insignificant)



5.4 Yarn Elongation at Break

5.4.1 Statistical Results for Yarn Elongation at Break

Table 5.4 Model for Yarn Elongation at Break

Response Elong. (%)
Whole Model
Summary of Fit

RSquare	0.584115
RSquare Adj	0.559161
Root Mean Square Error	0.206276
Mean of Response	5.237593
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	2.9880887	0.996030	23.4085
Error	50	2.1274984	0.042550	Prob > F
C. Total	53	5.1155870		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ΔTM	1	1	1.6298778	38.3050	<.0001
PS	1	1	0.2086651	4.9040	0.0314
PS*PS	1	1	1.3097503	30.7815	<.0001

Expanded Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	4.7657999	0.074433	64.03	<.0001
ΔTM	1.0638889	0.171897	6.19	<.0001
PS	0.0122587	0.005536	2.21	0.0314
(PS-8.55556)*(PS-8.55556)	0.005727	0.001032	5.55	<.0001

Table 5.4 shows the multiple regression results for yarn elongation at break. *ΔTM, % Solids*, and the interaction of *% Solids* with itself show significant effects on the response. The R-Square value of 58.41% indicates a moderate fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for % elongation is:

$$Y = 4.7657999 + 1.0638889\boldsymbol{\Delta TM} + 0.0122587\boldsymbol{PS} + [0.005727(\boldsymbol{PS}-8.55556)(\boldsymbol{PS}-8.55556)]$$

5.4.2 Observation and Prediction Analyses for Yarn Elongation at Break

Figures 5.22-5.24 show the results for the actual observations for yarn elongation at break. It is difficult to determine the overall trend for all of the sized yarns. Breaking length ultimately increases the most at all twist levels for yarns treated with Size A. All three sizing agents are competitive at the 5% concentration level. At 11% solids, the 4.2 and 4.4 yarns treated with Size B compete well against those with Sizes A and C. However, this is not the case for the yarn with a twist multiple of 4.0. Most of the breaking elongation measurements are lower for the yarns treated with Sizes B and C than the un-sized yarns, while most of the yarns treated with Size A show greater elongation than the untreated yarns.

The predicted % elongation for the yarns shown in Figures 5.25 provides a better understanding of the results. The effects of twist multiple and solid content on the treated yarns can all be seen. The % elongation initially decreases then increases at an increasing rate after 10% solids. The % elongation also becomes higher with an increase in twist multiple.

Figure 5.22 Yarn Elongation vs. Solid Content (Twist Multiple = 4.0)

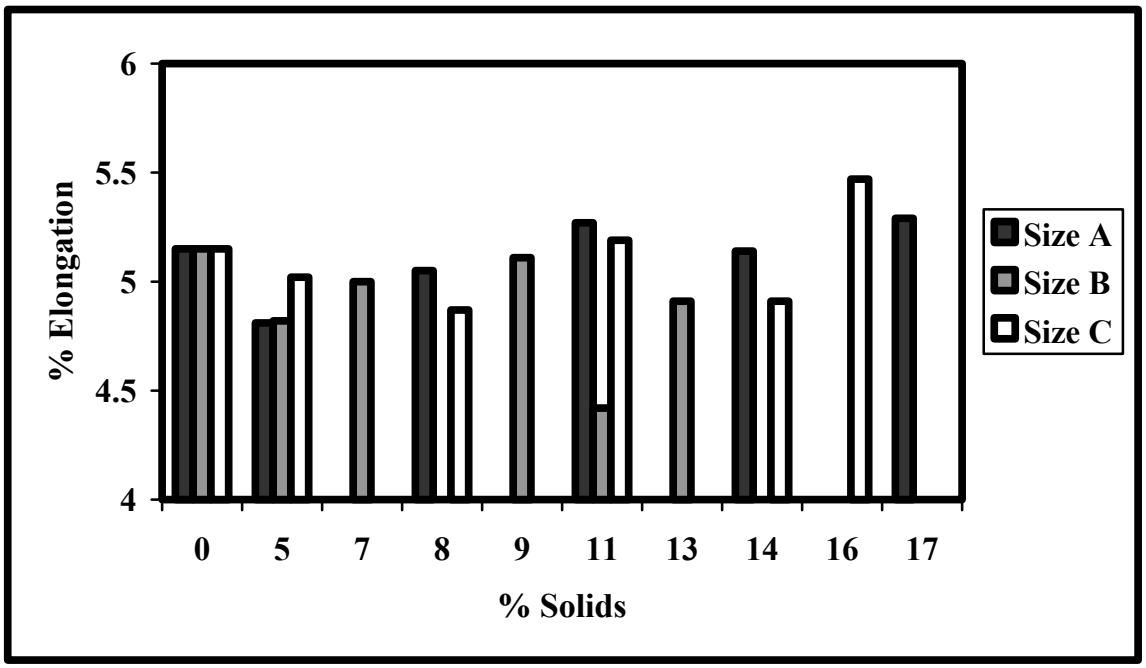


Figure 5.23 Yarn Elongation vs. Solid Content (Twist Multiple = 4.2)

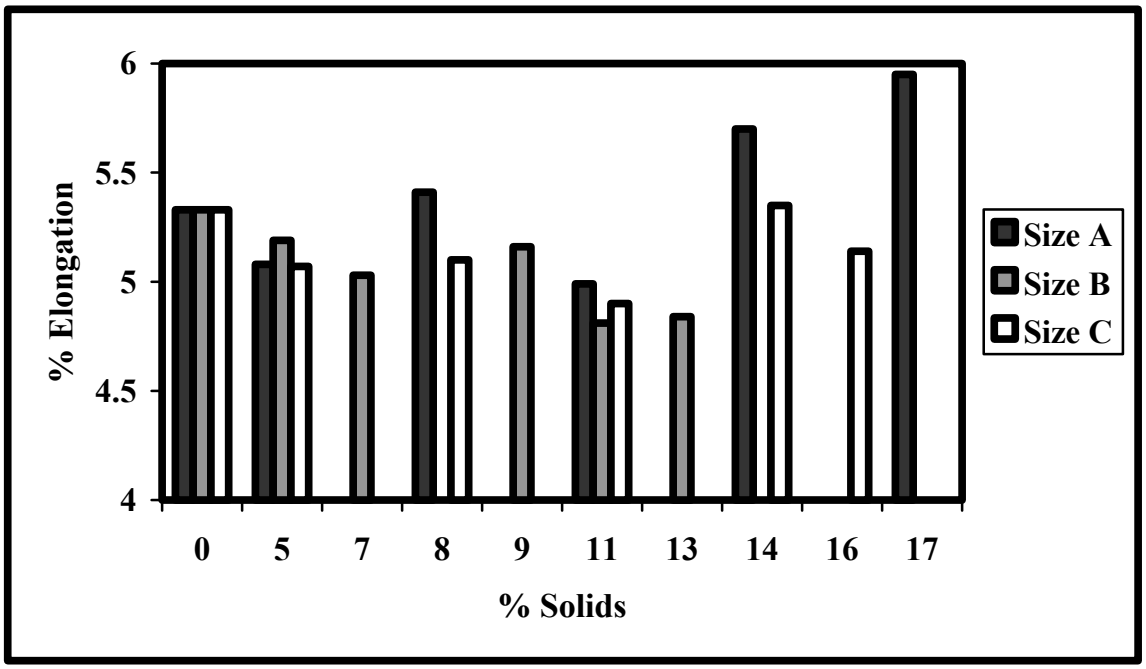


Figure 5.24 Yarn Elongation vs. Solid Content (Twist Multiple = 4.4)

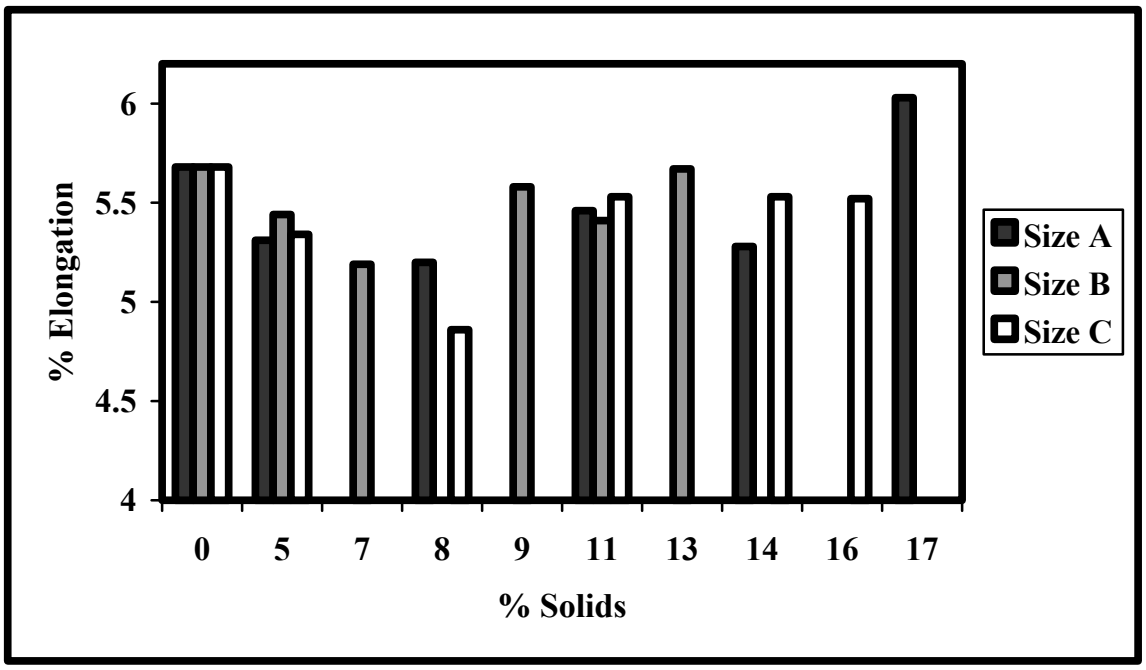
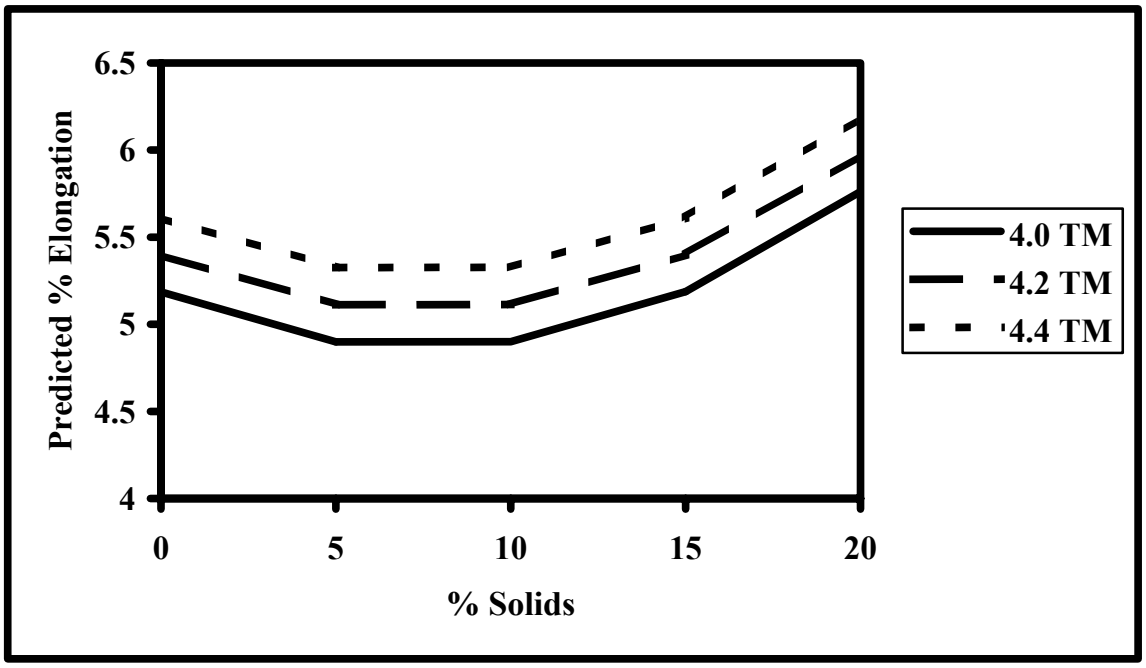


Figure 5.25 Predicted Yarn Elongation vs. Solid Content (Size Type Insignificant)



5.5 Breaking Work

5.5.1 Statistical Results for Breaking Work

Table 5.5 Model for Breaking Work

Response B-Work (cN.cm)
Whole Model
Summary of Fit

RSquare	0.807513
RSquare Adj	0.795964
Root Mean Square Error	18.88869
Mean of Response	423.0019
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	74838.115	24946.0	69.9194
Error	50	17839.134	356.8	Prob > F
C. Total	53	92677.250		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ΔTM	1	1	36144.347	101.3063	<.0001
PS	1	1	28347.649	79.4535	<.0001
PS*PS	1	1	18216.916	51.0589	<.0001

Expanded Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	334.48101	6.815849	49.07	<.0001
ΔTM	158.43056	15.74058	10.07	<.0001
PS	4.5183259	0.506898	8.91	<.0001
(PS-8.55556)*(PS-8.55556)	0.6754188	0.094523	7.15	<.0001

Table 5.5 shows the multiple regression results for breaking work. Δ*TM, % Solids,* and the interaction of *% Solids* with itself show significant effects on the response. The R-Square value of 80.75% indicates a relatively moderate fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for breaking force is:

$$Y = 334.48101 + 158.43056\boldsymbol{\Delta TM} + 4.5183259\boldsymbol{PS} + [0.6754188(\boldsymbol{PS}-8.55556)(\boldsymbol{PS}-8.55556)]$$

5.5.2 Observation and Prediction Analyses for Breaking Work

Figures 5.26-5.28 depict the results for breaking work. It can be seen that the breaking work for yarns treated with all size type tends to initially decrease at 5% solids, and then gradually increase. Overall, the work needed to break the yarns appears to increase as solid concentration rises. Slight difference in breaking work exists between all size types for the 4.2 TM and 4.4 TM yarns at 11% solids. But for the 4.0 TM yarn, Size B is not comparable to Sizes A nor C at this concentration. While the yarn with Size A at 17% solids outperforms Size B at 16% solids for higher twist levels, Size B provides the higher breaking work for the 4.0 TM yarn.

According to the regression model, size type does not have a significant effect on yarn breaking force. The prediction equation produces equal values for all three sizes at any solid content. The levels of twist and solid content have significant effects on breaking force depicted in the non-linear regressions in Figure 5.29. The breaking work initially decreases until reaching 5% solids, then increases at an increasing rate as % solids and twist multiple increase.

Figure 5.26 Breaking Work vs. Solid Content (Twist Multiple = 4.0)

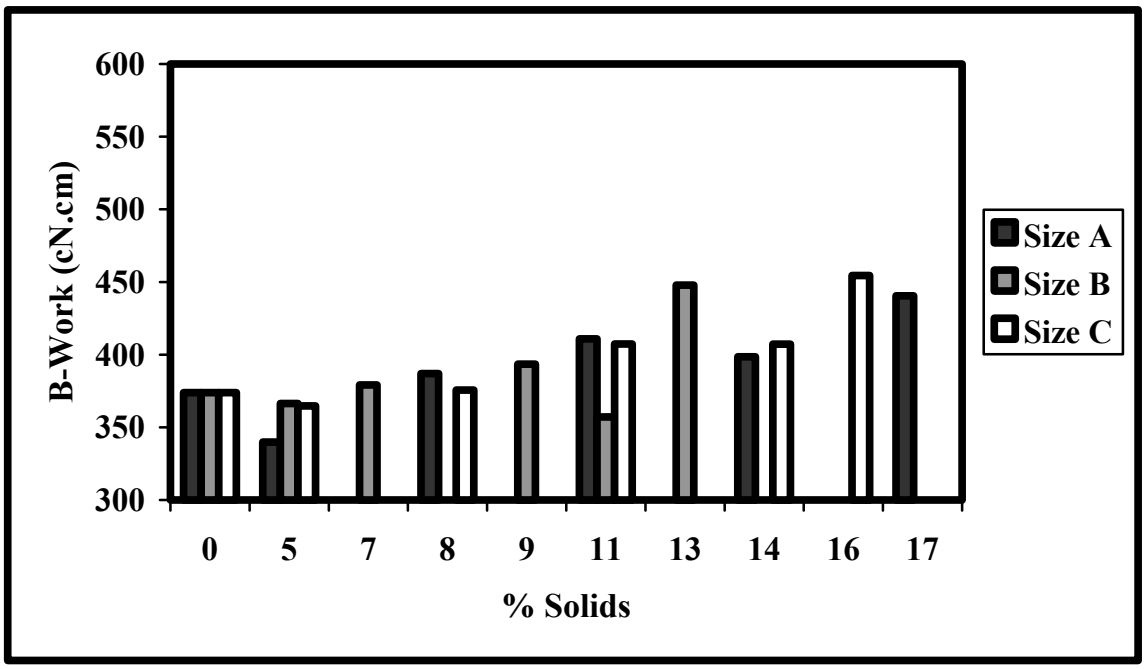


Figure 5.27 Breaking Work vs. Solid Content (Twist Multiple = 4.2)

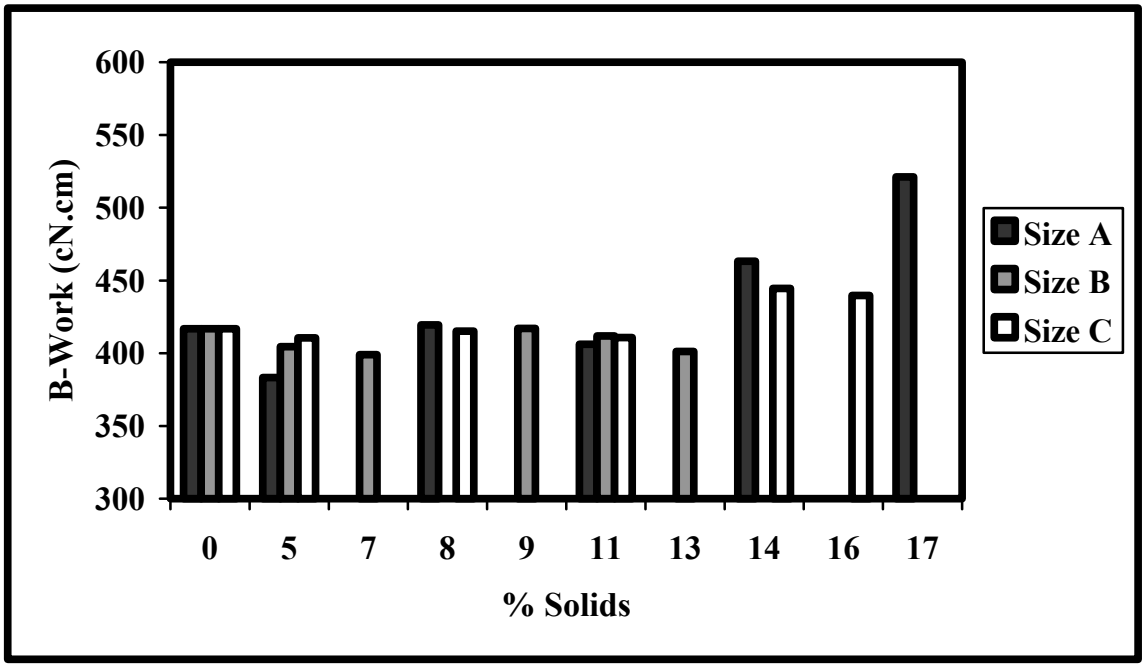


Figure 5.28 Breaking Work vs. Solid Content (Twist Multiple = 4.4)

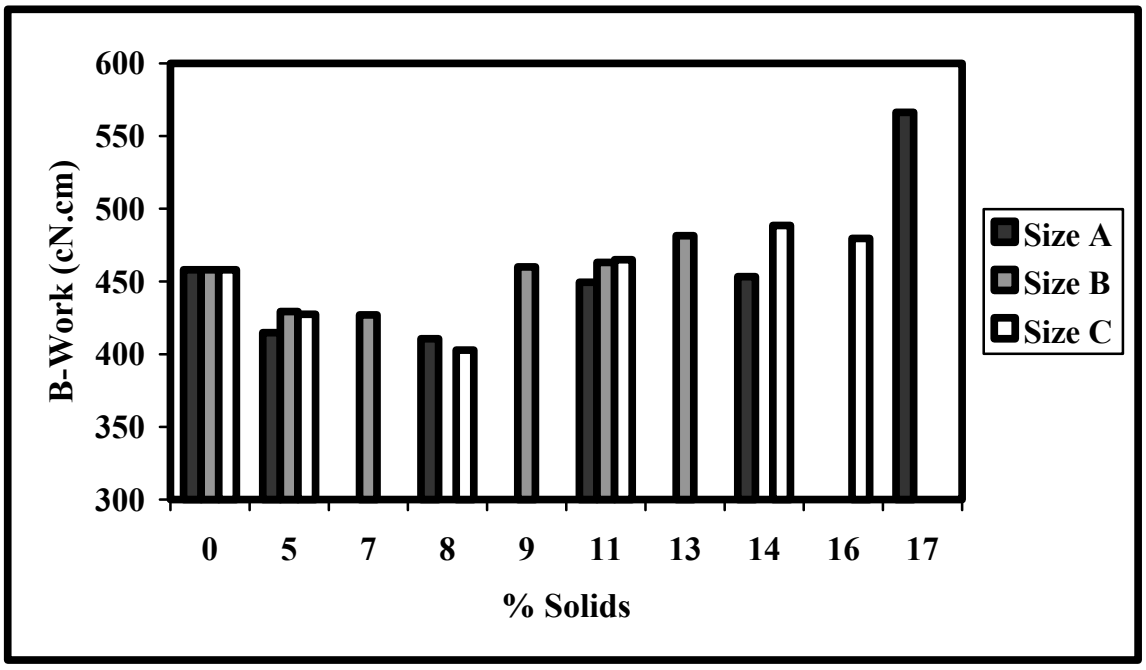
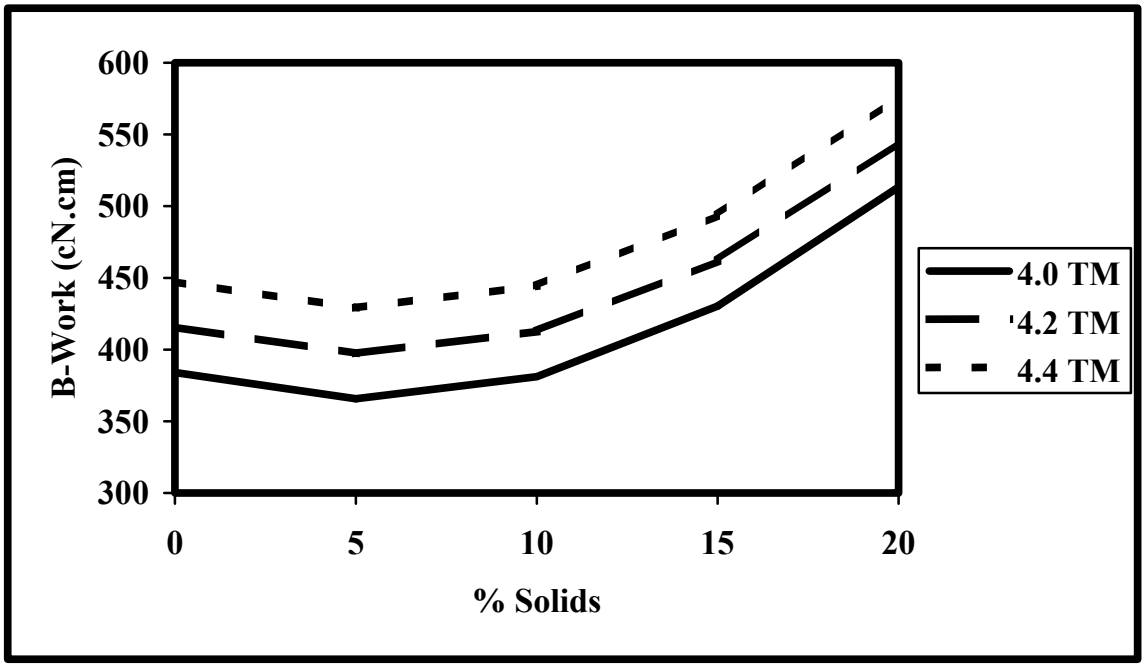


Figure 5.29 Predicted Breaking Work vs. Solid Content
(Size Type Insignificant)



5.6 Yarn Hairiness

5.6.1 Statistical Results for Yarn Hairiness

Table 5.6 Model for Yarn Hairiness

Response Hairiness
Whole Model
Summary of Fit

RSquare	0.788996
RSquare Adj	0.762059
Root Mean Square Error	0.32696
Mean of Response	5.084815
Observations (or Sum Wgts)	54

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	18.787526	3.13125	29.2907
Error	47	5.024422	0.10690	Prob > F
C. Total	53	23.811948		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ST	2	2	1.449166	6.7780	0.0026
ΔTM	1	1	1.013378	9.4795	0.0035
PS	1	1	17.058957	159.5748	<.0001
ST*PS	2	2	0.696977	3.2599	0.0472

Expanded Estimates
Nominal factors expanded to all levels

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	6.2046812	0.102891	60.30	<.0001
ST_A	0.137427	0.063528	2.16	0.0356
ST_B	-0.235689	0.064309	-3.66	0.0006
ST_C	0.0982618	0.06345	1.55	0.1282
ΔTM	-0.838889	0.272466	-3.08	0.0035
PS	-0.11304	0.008948	-12.63	<.0001
ST_A *(PS-8.55556)	0.0012377	0.01193	0.10	0.9178
ST_B *(PS-8.55556)	-0.029921	0.013805	-2.17	0.0353
ST_C *PS	0.0286837	0.012148	2.36	0.0224

Table 5.6 shows the multiple regression results for yarn hairiness. *Size Type, ΔTM, % Solids and Size Type * %Solids* show significant effects on the response. The R-Square value of 78.90% indicates a relatively good fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for yarn hairiness is:

$$Y = 6.2046812 + 0.137427\boldsymbol{ST_A} - 0.235689\boldsymbol{ST_B} + 0.0982618\boldsymbol{ST_C} - 0.838889\boldsymbol{\Delta TM} +$$

$$[0.0012377\boldsymbol{ST_A}(\boldsymbol{PS} - 8.55556)] - [0.\ 0.029921\boldsymbol{ST_B}(\boldsymbol{PS} - 8.55556)] + 0.0286837(\boldsymbol{ST_C PS})$$

where, $ST_A = \begin{Bmatrix} 1 \\ 0, \ otherwise \end{Bmatrix}$, $ST_B = \begin{Bmatrix} 1 \\ 0, \ otherwise \end{Bmatrix}$,

$$ST_C = \begin{Bmatrix} 1 \\ 0, \ otherwise \end{Bmatrix}.$$

5.6.2 Observation and Prediction Analyses for Yarn Hairiness

Figures 5.30-5.32 depict an overall decreasing trend for the actual observations for hairiness as solid concentration increases for yarns treated with all sizes. The hairiness of Size C for the 4.0 TM yarn appears unusually high at 14% solids and does not concur with the overall trend. Size B lowers hairiness the most for the 4.0 TM yarn at 13% solids. This is noteworthy since Sizes A and C contain higher solid concentrations for this comparison. For the 4.2 and 4.4 TM yarn, Size B performs the best at the highest comparison level of 11%.

The predicted regressions for yarn hairiness are shown in Figures 5.33-5.35. The twist level, size type, solid content, and interaction between size type and % solids all show significant effects on the yarns based on the model. Close comparison of the three graphs shows that yarn hairiness decreases slightly as the twist multiple increases. Yarns treated with Size B have less hairiness than those treated with Sizes A and C as % solids increase.

Figure 5.30 Hairiness vs. Solid Content (Twist Multiple = 4.0)

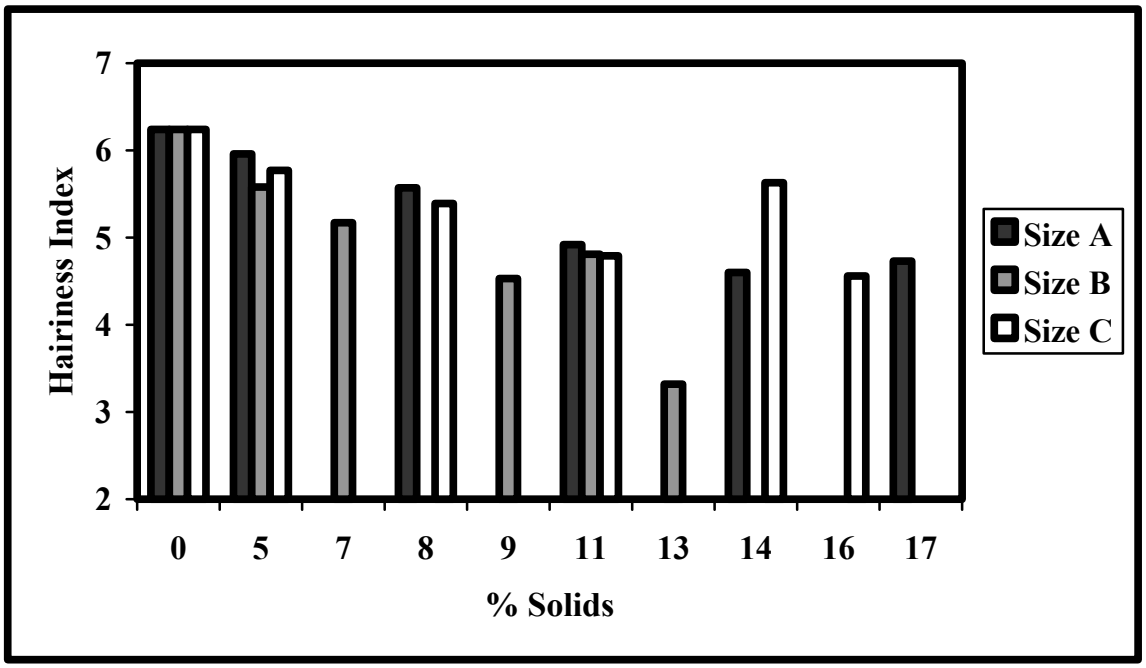


Figure 5.31 Hairiness vs. Solid Content (Twist Multiple = 4.2)

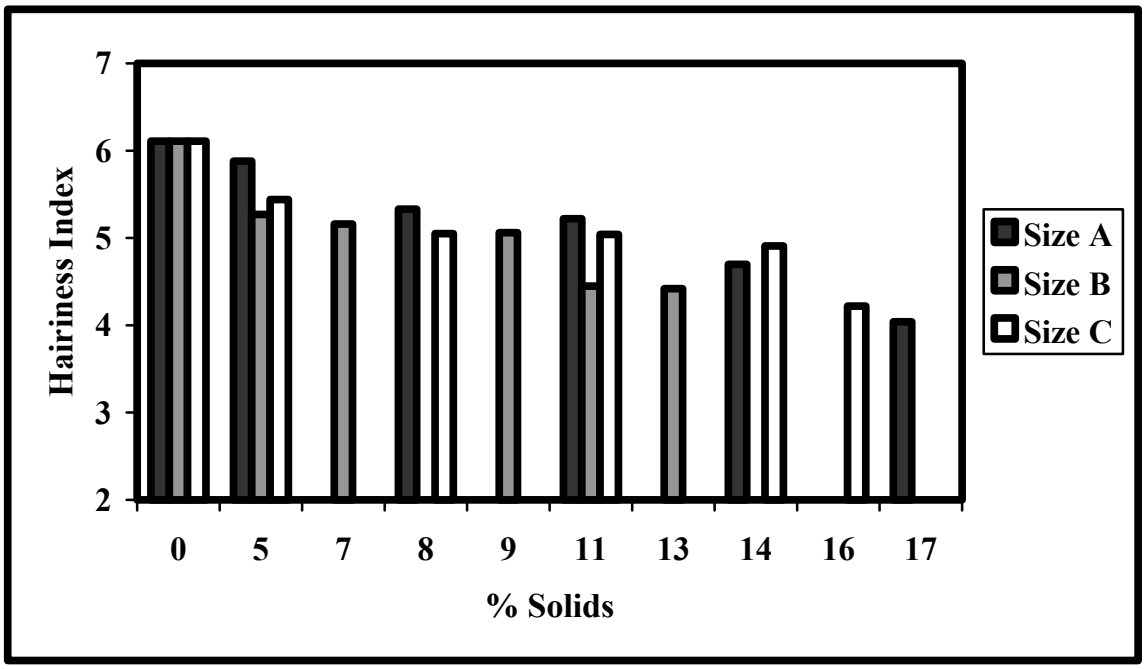


Figure 5.32 Hairiness vs. Solid Content (Twist Multiple = 4.4)

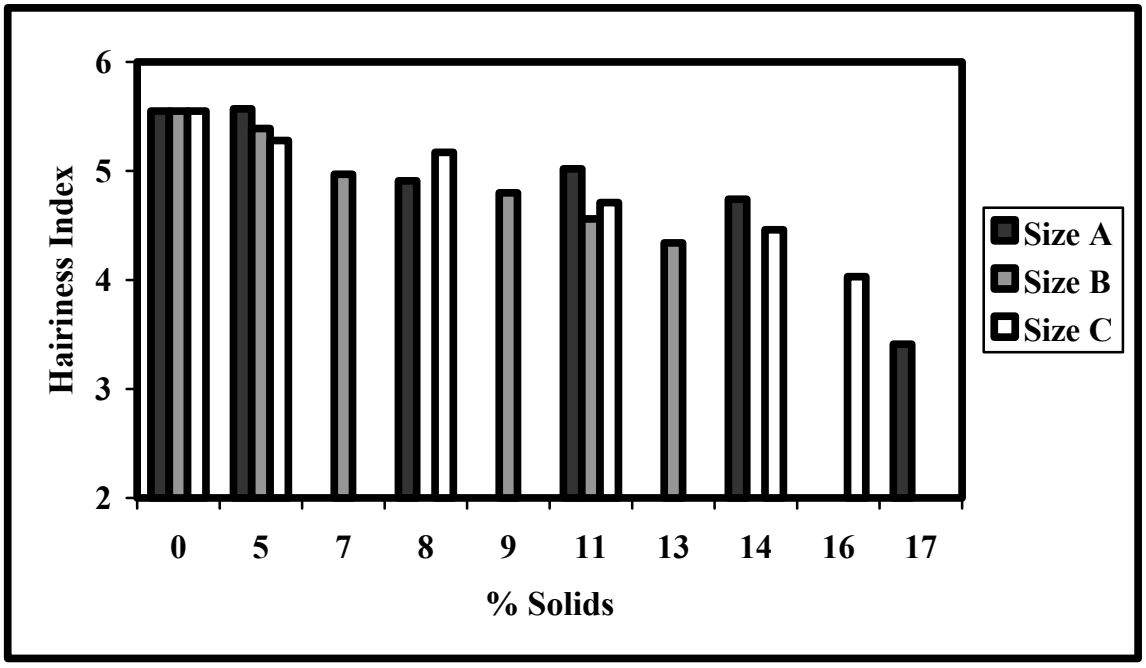


Figure 5.33 Predicted Hairiness vs. Solid Content (Twist Multiple = 4.0)

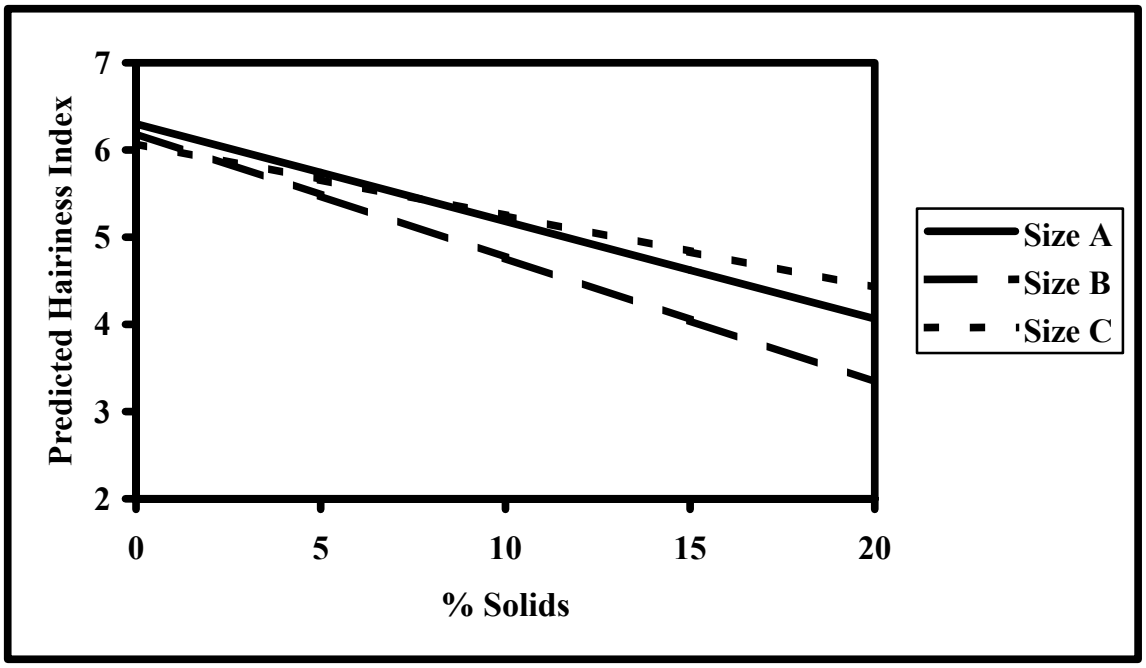


Figure 5.34 Predicted Hairiness vs. Solid Content (Twist Multiple = 4.2)

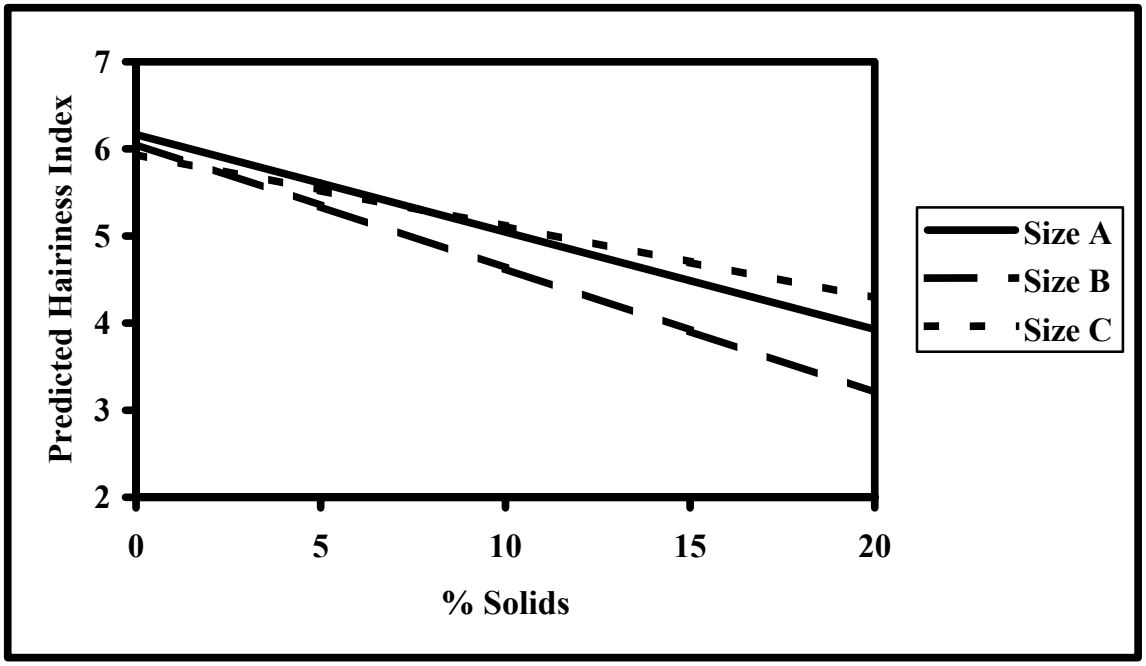
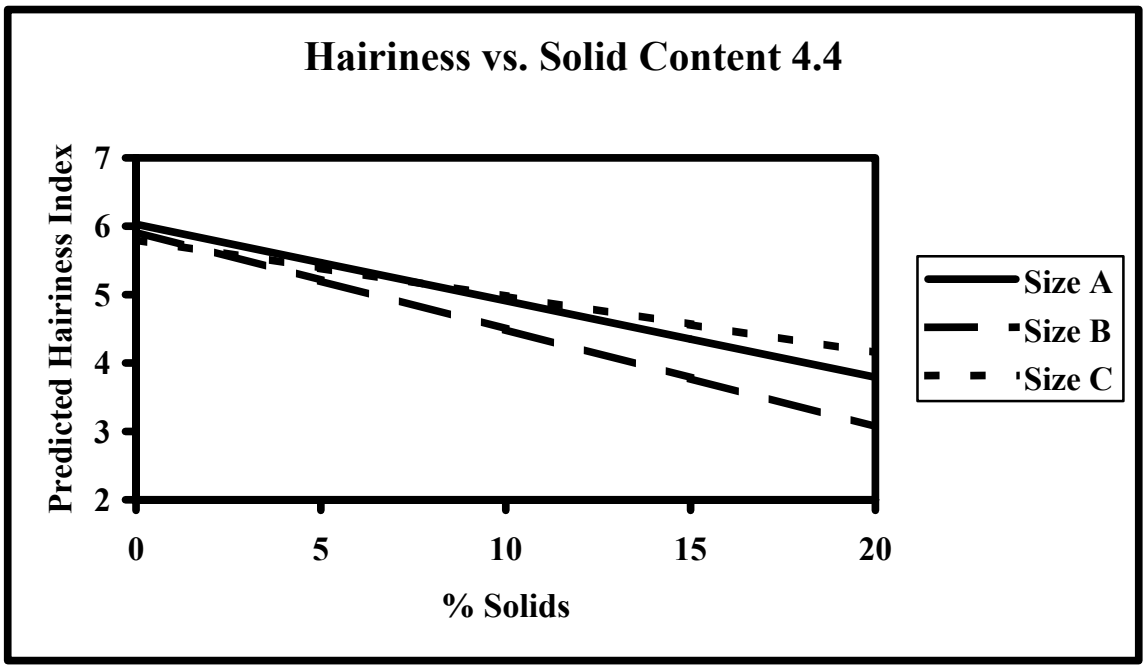


Figure 5.35 Predicted Hairiness vs. Solid Content (Twist Multiple = 4.4)



5.7 Coefficient of Friction

5.7.1 Statistical Results for Coefficient of Friction

Table 5.7 Model for Coefficient of Friction

Response Coefficient of Friction
Whole Model
Summary of Fit

RSquare	0.793688
RSquare Adj	0.788982
Root Mean Square Error	0.010955
Mean of Response	0.182926
Observations (or Sum Wgts)	270

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	0.12142522	0.020238	168.6285
Error	263	0.03156330	0.000120	Prob > F
C. Total	269	0.15298852		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ST	2	2	0.00248893	10.3695	<.0001
PS	1	1	0.03326061	277.1428	<.0001
ST*PS	2	2	0.00099446	4.1432	0.0169
PS*PS	1	1	0.04742311	395.1513	<.0001

Expanded Estimates
Nominal factors expanded to all levels

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1892105	0.001692	111.82	<.0001
ST_A	0.0000549	0.000968	0.06	0.9549
ST_B	0.0038574	0.001007	3.83	0.0002
ST_C	-0.003912	0.000958	-4.08	<.0001
PS	-0.00236	0.000142	-16.65	<.0001
ST_A*(PS-8.55556)	-0.000344	0.000184	-1.87	0.0633
ST_B*(PS-8.55556)	0.0006234	0.000217	2.87	0.0044
ST_C*PS	-0.00028	0.000183	-1.53	0.1279
(PS-8.55556)*(PS-8.55556)	0.0005289	0.000027	19.88	<.0001

Table 5.7 shows the multiple regression results for yarn-to-metal coefficient of friction. *Size Type*, % Solids, and the interactions of *% Solids* with itself and with *Size Type* show significant effects on the response. The R-Square value of 79.37% indicates a good fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for yarn hairiness is:

$$Y = 0.1892105 + 0.0000549\mathbf{ST_A} + 0.0038574\mathbf{ST_B} - 0.003912\mathbf{ST_C} - 0.00236\mathbf{PS} - [0.000344\mathbf{ST_A}(\mathbf{PS} - 8.55556)] + [0.0006234\mathbf{ST_B}(\mathbf{PS} - 8.55556)] - 0.00028(\mathbf{ST_C PS}) + [0.0005289(\mathbf{PS}-8.55556)(\mathbf{PS}-8.55556)]$$

where, $ST_A = \begin{cases} 1 \\ 0, \quad otherwise \end{cases}$, $ST_B = \begin{cases} 1 \\ 0, \quad otherwise \end{cases}$,

$ST_C = \begin{cases} 1 \\ 0, \quad otherwise \end{cases}$.

5.7.2 Observation and Prediction Analyses for Coefficient of Friction

Figures 5.36-5.38 show the results for the average coefficient of friction. It can be seen that the coefficient of friction for yarns treated with all three sizes at each twist level is considerably less than the untreated yarns. Among the size types, little difference in coefficient of friction occurs as solid concentration increases except for the 4.4 TM yarn. Here, Size A promotes the least amount of friction at 7%, 11% and 14% solids.

The regression model shows that twist multiple does not have a significant effect on coefficient of friction. The prediction equation produces equal values for all three twist levels at any solid content depicted in Figure 3.39. The regression lines show that the predicted coefficient of friction for all yarns initially decreases at a decreasing rate as % solids increase. At 10% solids, the predicted coefficient of friction for all yarns increases at an increasing rate with an increase in solid content. This is due to excessive stickiness of the sizing agents as more solids are added. Overall, yarns treated with Size C show less yarn-to-metal friction than Sizes A and B.

Figure 5.36 Coefficient of Friction vs. Solid Content (Twist Multiple = 4.0)

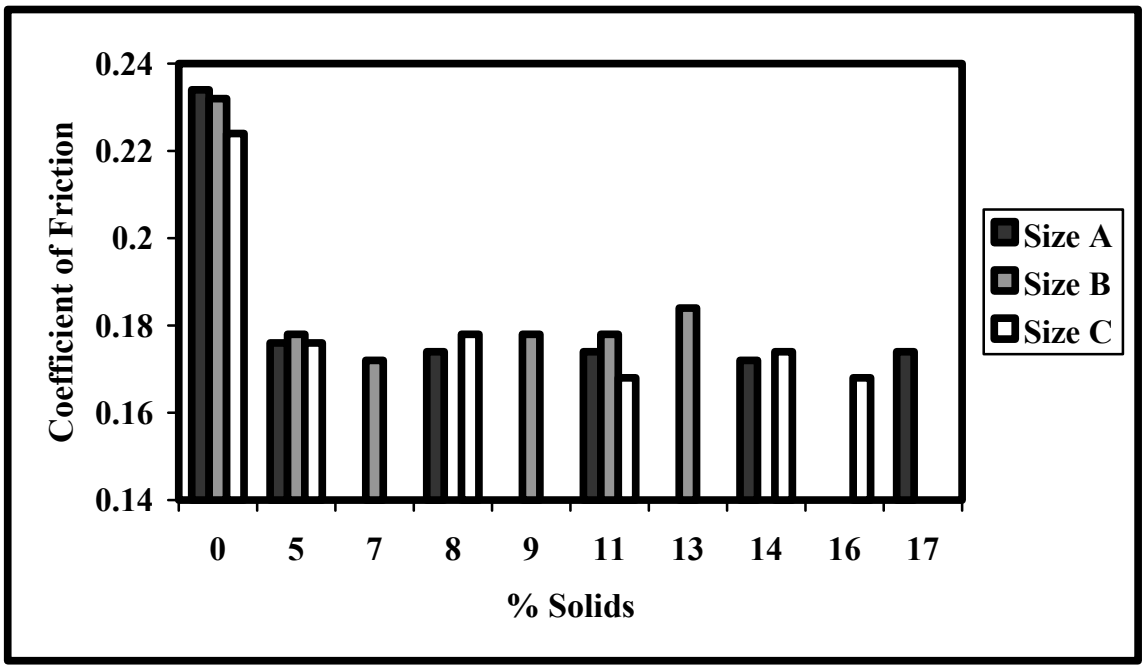


Figure 5.37 Coefficient of Friction vs. Solid Content (Twist Multiple = 4.2)

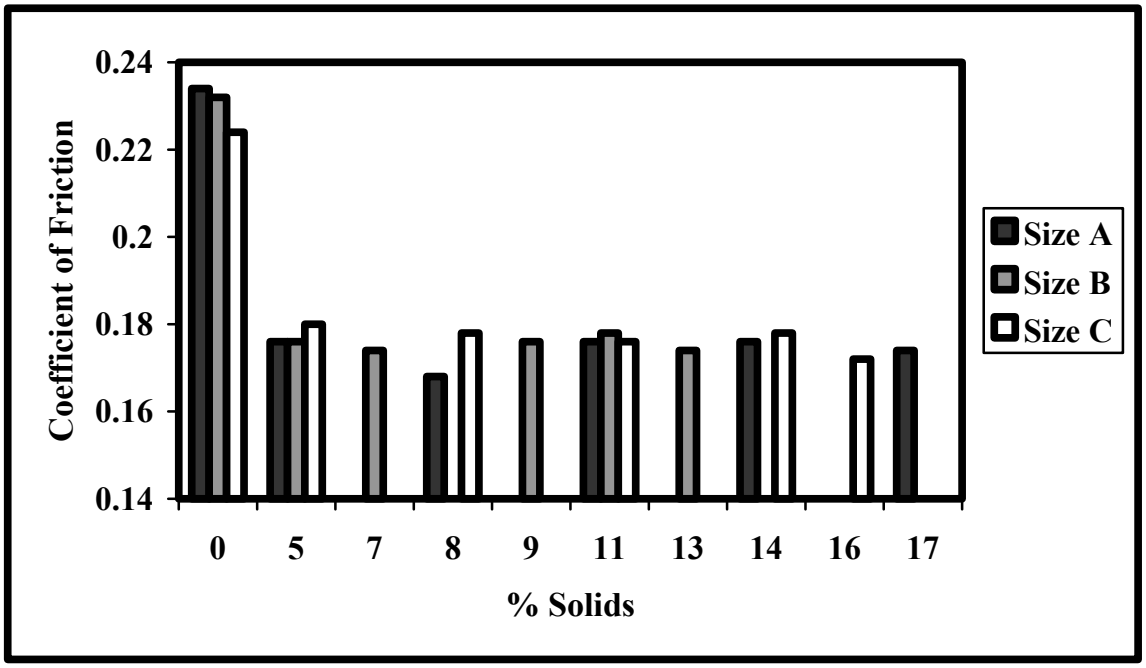


Figure 5.38 Coefficient of Friction vs. Solid Content (Twist Multiple = 4.4)

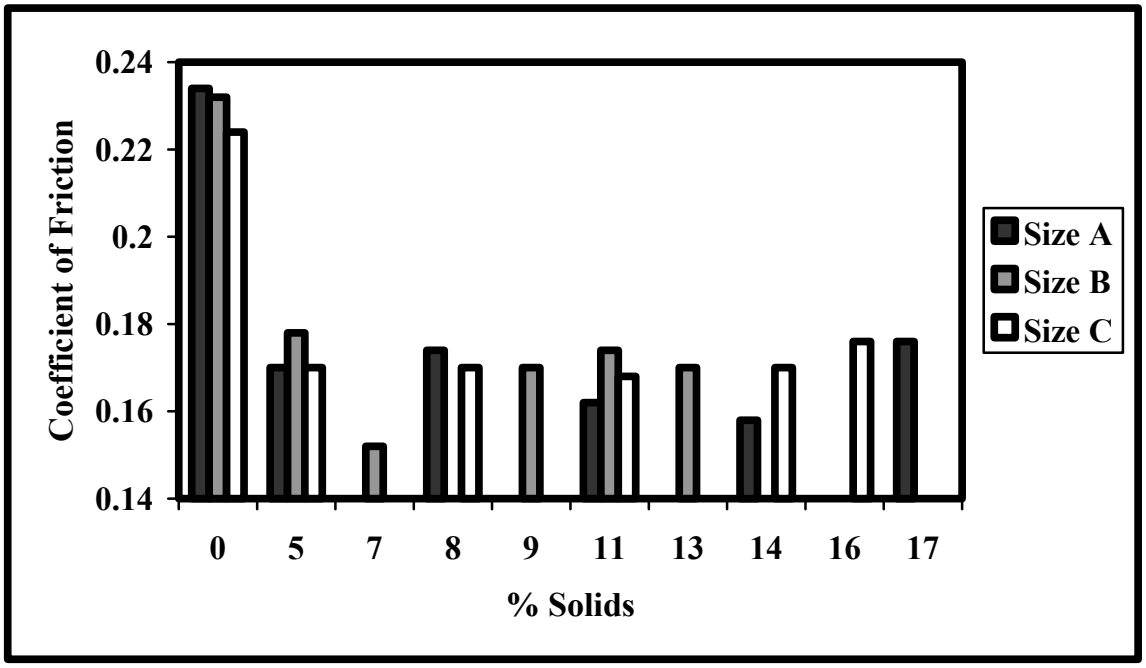
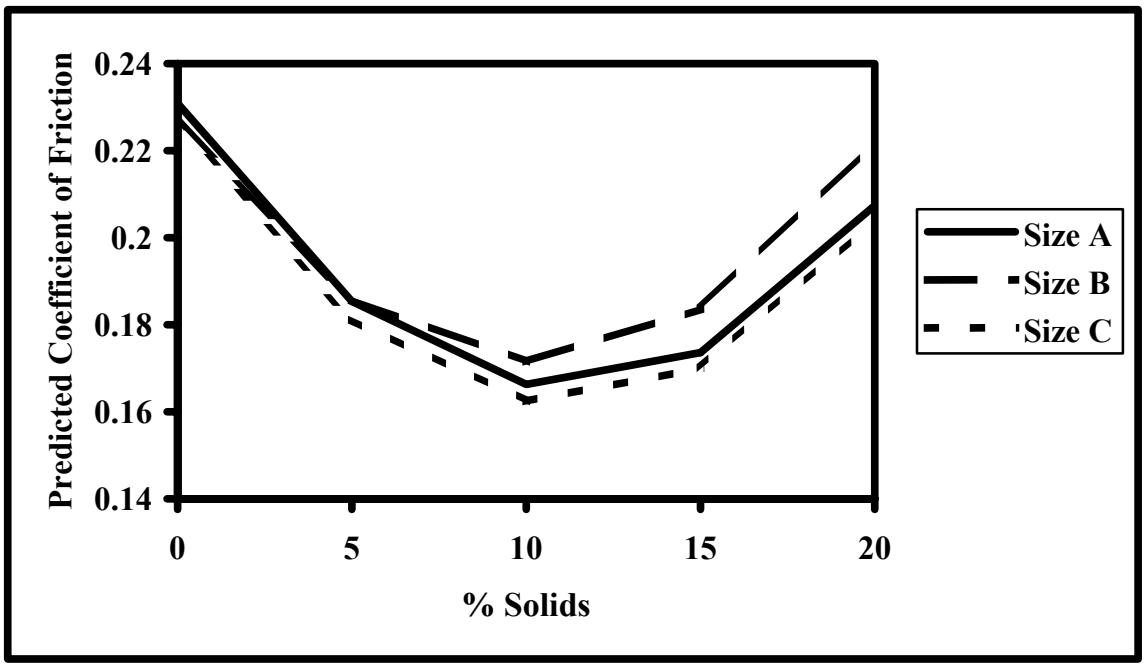


Figure 5.39 Predicted Coefficient of Friction vs. Solid Content (Twist Multiple Insignificant)



5.8 Abrasion Resistance

5.8.1 Statistical Results for Abrasion Resistance

Table 5.8 Model for Abrasion Resistance

Response Abrasion Resistance
Whole Model
Summary of Fit

RSquare	0.38039
RSquare Adj	0.351458
Root Mean Square Error	9.289864
Mean of Response	42.70741
Observations (or Sum Wgts)	270

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	12	13616.382	1134.70	13.1481
Error	257	22179.503	86.30	Prob > F
C. Total	269	35795.885		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ST	2	2	2249.5821	13.0333	<.0001
^TM	1	1	4888.8999	56.6490	<.0001
PS	1	1	614.2489	7.1175	0.0081
ST*^TM	2	2	1614.3057	9.3527	0.0001
ST*PS	2	2	813.3206	4.7121	0.0098
^TM*^TM	1	1	933.5185	10.8169	0.0011
PS*PS	1	1	2147.2638	24.8809	<.0001
ST*^TM*PS	2	2	1240.6584	7.1879	0.0009

Expanded Estimates

Nominal factors expanded to all levels

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	29.320999	1.785977	16.42	<.0001
ST_A	-4.133188	0.820982	-5.03	<.0001
ST_B	1.6650936	0.85394	1.95	0.0523
ST_C	2.4680942	0.812206	3.04	0.0026
ΔTM	26.384598	3.505535	7.53	<.0001
PS	0.3206639	0.120195	2.67	0.0081
ST_A *(ΔTM-0.2)	11.744917	4.920697	2.39	0.0177
ST[B]*(ΔTM-0.2)	-21.27149	4.927238	-4.32	<.0001
ST[C]* ΔTM	9.5265705	4.920803	1.94	0.0540
ST_A *(PS-8.55556)	-0.381247	0.156189	-2.44	0.0153
ST_B *(PS-8.55556)	0.542547	0.1841	2.95	0.0035
ST_C *PS	-0.1613	0.15543	-1.04	0.3004
(ΔTM-0.2)*(ΔTM-0.2)	98.611111	29.98291	3.29	0.0011
(PS-8.55556)*(PS-8.55556)	0.11254	0.022562	4.99	<.0001
ST_A *(ΔTM-0.2)*(PS-8.55556)	2.5153397	0.915232	2.75	0.0064
ST_B *(ΔTM-0.2)*(PS-8.55556)	-3.840211	1.041092	-3.69	0.0003
ST_C * ΔTM*PS	1.3248712	0.93887	1.41	0.1594

Table 5.8 shows the multiple regression results for abrasion resistance. *Size Type, ΔTM, % Solids, Size Type * ΔTM, Size Type * % Solids, ΔTM * ΔTM*, *% Solids * % Solids*, and *Size Type * ΔTM * % Solids* show significant effects on the response. The R-Square value of 38.04% indicates a relatively moderate fitting for the observed response data. This may due to the extreme variability in measuring the resistance of yarns to abrasion. According to the expanded parameter estimates, the prediction equation for abrasion resistance is:

$$Y = 29.320999 - 4.133188\mathbf{ST_A} + 1.6650936\mathbf{ST_B} + 2.4680942\mathbf{ST_C} + 26.384598\mathbf{\Delta TM} + 0.3206639PS + [11.744917\mathbf{ST_A}(\mathbf{\Delta TM} - 0.2)] - [21.27149\mathbf{ST_B}(\mathbf{\Delta TM} - 0.2)] + 9.5265705\mathbf{ST_C}(\mathbf{\Delta TM}) - [0.381247\mathbf{ST_A}(\mathbf{PS}-8.55556)] + [0.542547\mathbf{ST_B}(\mathbf{PS}-8.55556)] - 0.1613(\mathbf{ST_C PS}) + [98.611111(\mathbf{\Delta TM}-0.2)(\mathbf{\Delta TM}-0.2)] + [0.11254(\mathbf{PS}-8.55556)(\mathbf{PS}-8.55556)] + [2.5153397\mathbf{ST_A}(\mathbf{\Delta TM}-0.2)(\mathbf{PS}-8.55556)] - [3.840211\mathbf{ST_B}(\mathbf{\Delta TM}-0.2)(\mathbf{PS}-8.55556)] + 1.3248712(\mathbf{ST_C \Delta TM PS})$$

where, $ST_A = \begin{Bmatrix} 1 \\ 0, \quad otherwise \end{Bmatrix}$, $ST_B = \begin{Bmatrix} 1 \\ 0, \quad otherwise \end{Bmatrix}$,

$ST_C = \begin{Bmatrix} 1 \\ 0, \quad otherwise \end{Bmatrix}$.

5.8.2 Observation and Prediction Analyses for Abrasion Resistance

Figures 5.40-5.42 illustrate the results of the actual observations for abrasion resistance. Theoretically, the abrasion resistance should increase as the solid concentration increases. The inclination of the abrasion resistance to move up and down as solid concentration increases for all yarns creates difficulty in determining a constant

trend for the average number of strokes per yarn break for any of the three twist levels. In fact, the sized yarns actually perform worse compared to the un-sized yarns at several solid concentration levels. Inconsistency in the encapsulation of sizing material around the diameter of the yarns may be the cause of the sporadic trends. Overall, yarns treated with Size C seem to have the highest resistance to abrasion compared to Sizes A and B for all twist multiples, especially at the comparable solid concentrations of 5% and 11%. Again, Size B shows an unexpected result at 13% solids.

The regression lines in Figure 5.43-5.45 show that the predicted abrasion resistance for yarns treated with Size C is greater than Sizes A and B as the twist level increases. Size B appears to show the best abrasion resistance as solid content increases for the 4.0 TM and 4.2 TM yarns. Size C shows the best abrasion resistance for the 4.4 TM yarns as % solids increases. The initial decreasing trend in abrasion resistance as solid content increases is unusual. This may be caused by inconsistencies in size encapsulation for the suspected levels of concentration.

Figure 5.40 Abrasion Resistance vs. Solid Content (Twist Multiple = 4.0)

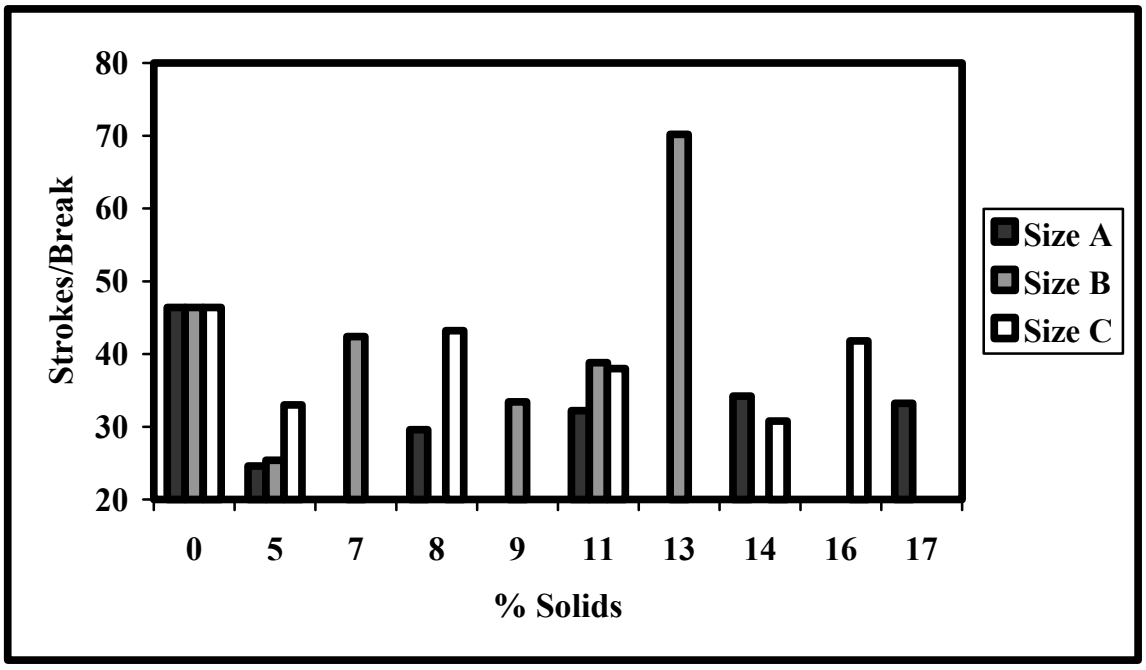


Figure 5.41 Abrasion Resistance vs. Solid Content (Twist Multiple = 4.2)

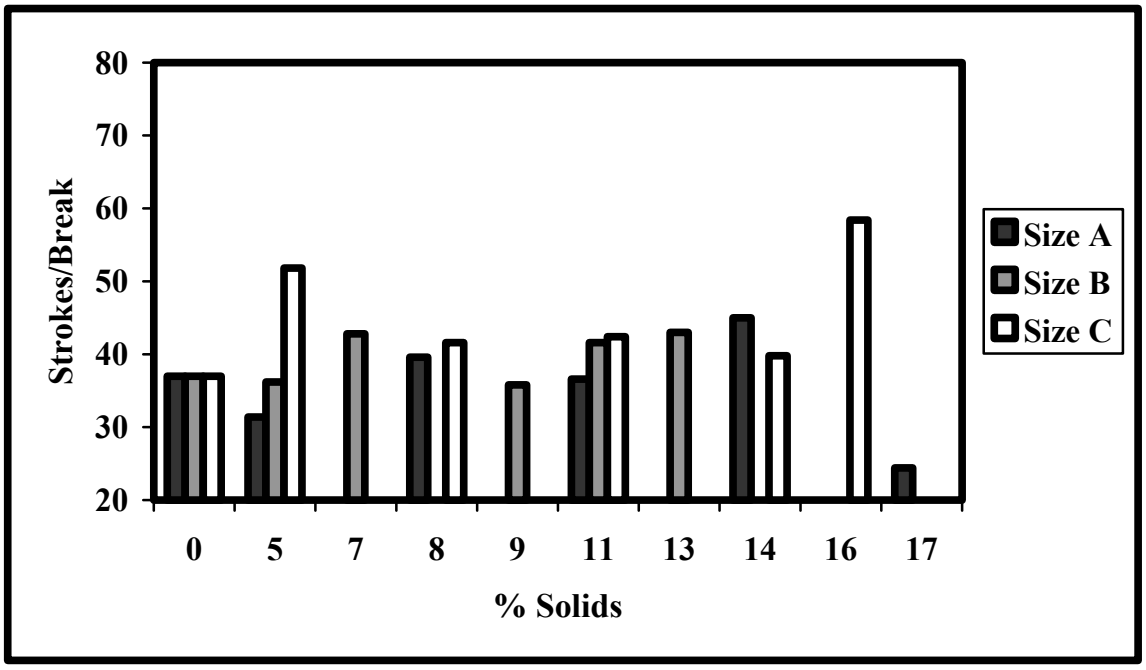


Figure 5.42 Abrasion Resistance vs. Solid Content (Twist Multiple = 4.4)

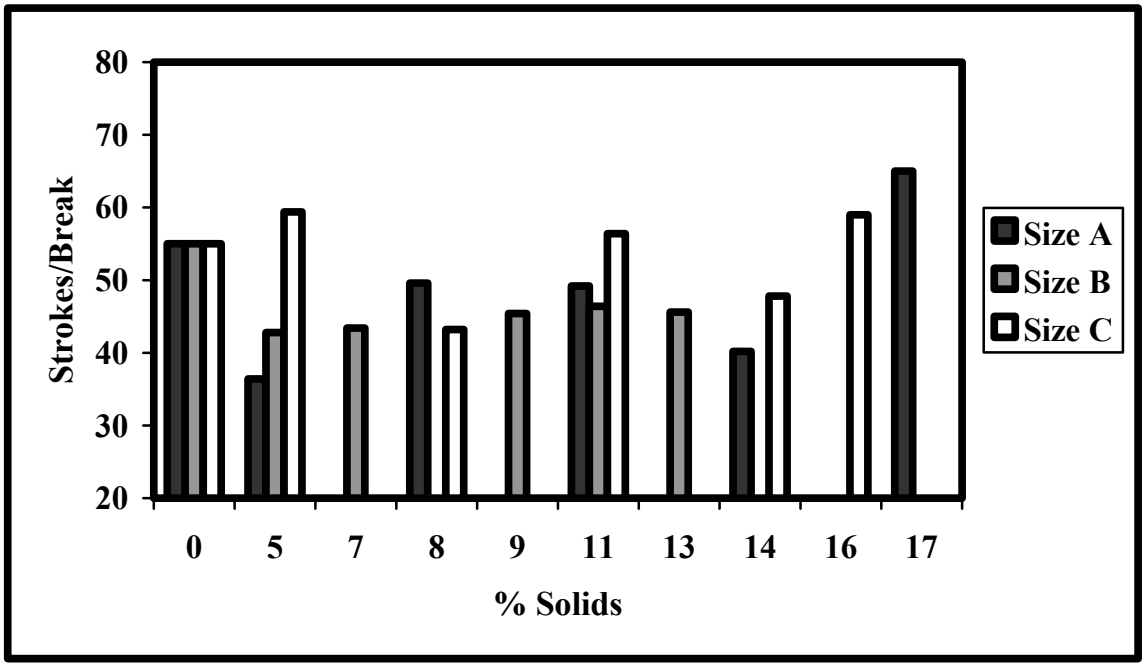


Figure 5.43 Predicted Abrasion Resistance vs. Twist Multiple (TM = 4.0)

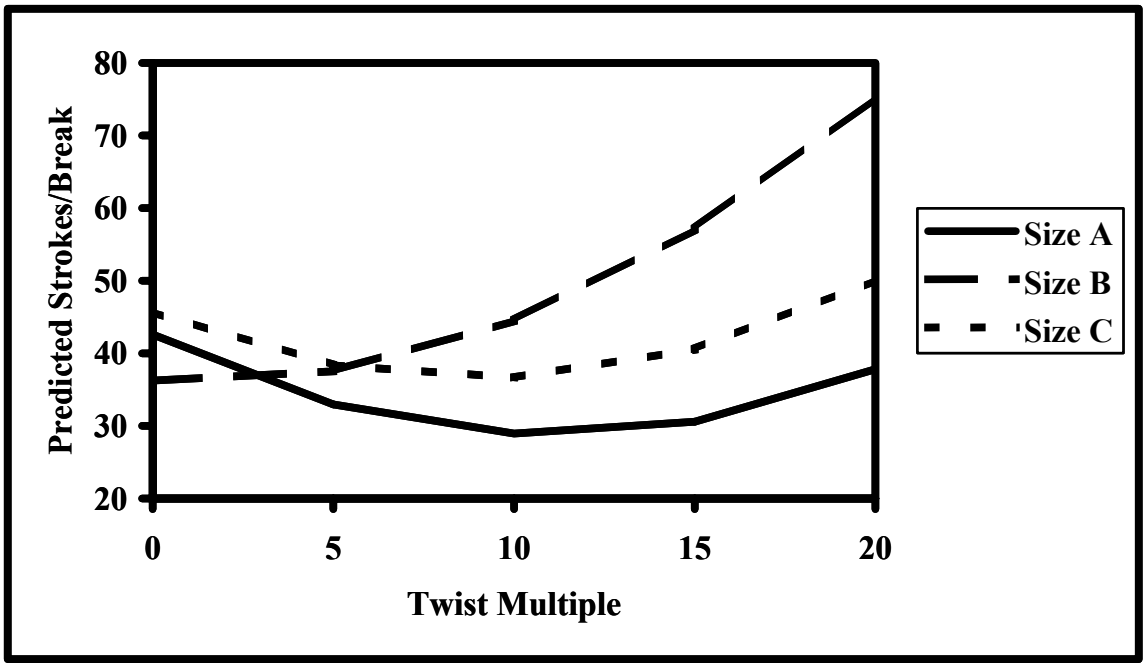


Figure 5.44 Predicted Abrasion Resistance vs. Twist Multiple (TM = 4.2)

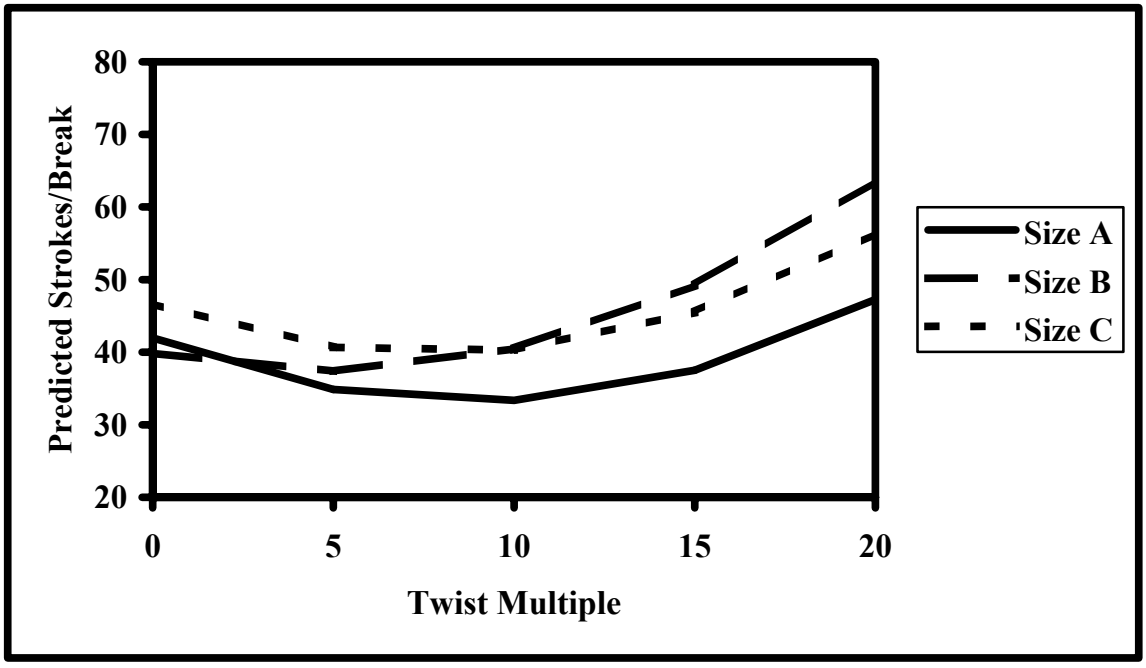
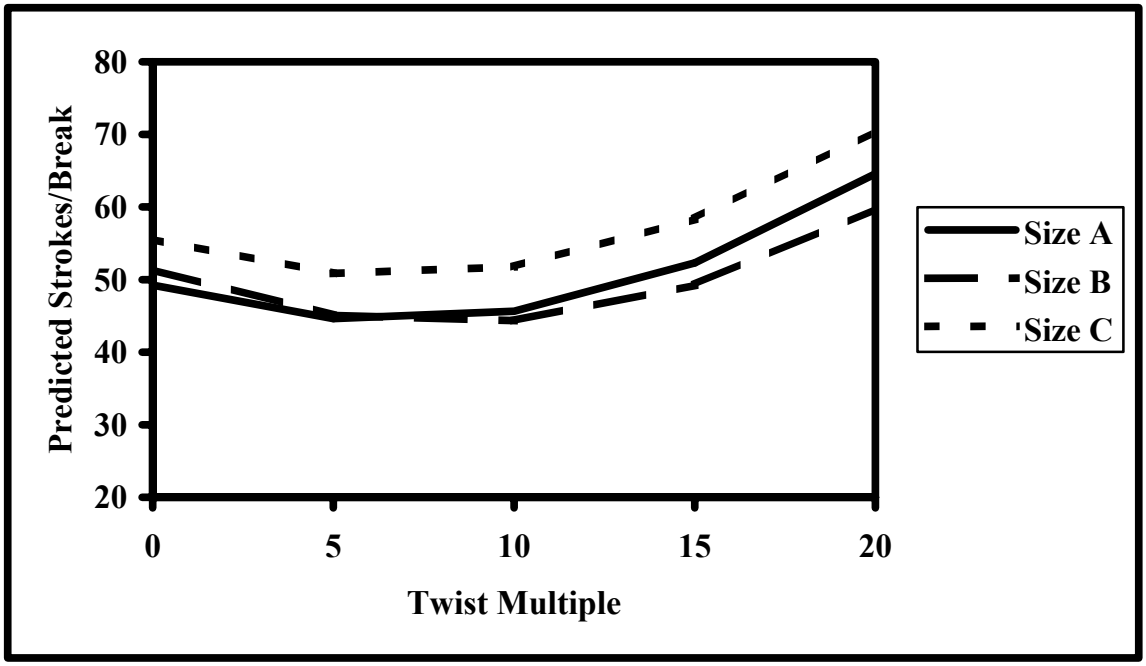


Figure 5.45 Predicted Abrasion Resistance vs. Twist Multiple (TM = 4.4)



5.9 Fabric Weight Loss

5.9.1 Statistical Results for Fabric Weight Loss

Table 5.9 Model for Fabric Weight Loss

Response % Weight Loss
Whole Model
Summary of Fit

RSquare	0.903774
RSquare Adj	0.894152
Root Mean Square Error	0.80703
Mean of Response	6.345333
Observations (or Sum Wgts)	45

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	244.68561	61.1714	93.9223
Error	40	26.05191	0.6513	Prob > F
C. Total	44	270.73752		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ST	2	2	39.76709	30.5291	<.0001
ΔW	1	1	202.95603	311.6179	<.0001
ΔW*ΔW	1	1	1.96249	3.0132	0.0903

Expanded Estimates
Nominal factors expanded to all levels

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	4.0396667	0.255205	15.83	<.0001
ST_A	-0.374667	0.170137	-2.20	0.0335
ST_B	1.292	0.170137	7.59	<.0001
ST_C	-0.917333	0.170137	-5.39	<.0001
ΔW	0.1734	0.009823	17.65	<.0001
(ΔW-15)*(ΔW-15)	-0.001969	0.001134	-1.74	0.0903

Table 5.9 shows the multiple regression results for sample fabric weight loss. *Size Type, ΔWashings,* and the interaction of *ΔWashings* with itself *s*how significant effects on the response. The R-Square value of 90.37 indicates a good fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for size add-on is:

$$Y = 4.0396667 - 0.374667\boldsymbol{ST_A} + 1.292\boldsymbol{ST_B} - 0.917333\boldsymbol{ST_C} + 0.1734\boldsymbol{\Delta W} - [0.001969(\boldsymbol{\Delta W}\text{-}15)(\boldsymbol{\Delta W}\text{-}15)]$$

where, $ST_A = \begin{Bmatrix} 1 \\ 0, otherwise \end{Bmatrix}$, $ST_B = \begin{Bmatrix} 1 \\ 0, otherwise \end{Bmatrix}$,

$ST_C = \begin{Bmatrix} 1 \\ 0, otherwise \end{Bmatrix}$.

5.9.2 Observation and Prediction Analyses for Fabric Weight Loss

Figures 5.46-5.50 illustrate the results of the actual observations fabric weight loss at different heat set temperatures. The overall trend suggests that % weight loss increases with the number of washings. Fabric samples woven with yarns treated with Size B appear to have the most weight loss compared to Sizes A and C at all heat-set temperatures. Size C seems to promote the least amount of weight loss for fabric samples at each temperature level. Although the samples not treated with heat setting have the lowest amount of weight loss, little difference is seen when comparing each temperature level.

The predicted regressions shown in Figure 5.51 depict the % weight loss of the fabrics as a function of washes based on the statistical insignificance of heat setting. It is clear that size type and the number of washings are significant. The fabric sample treated with Size C shows less weight loss than Sizes A and B as the number of washings increases.

Figure 5.46 Fabric Sample Weight Loss vs. Washing (Heat Set Temp. = 160°F)

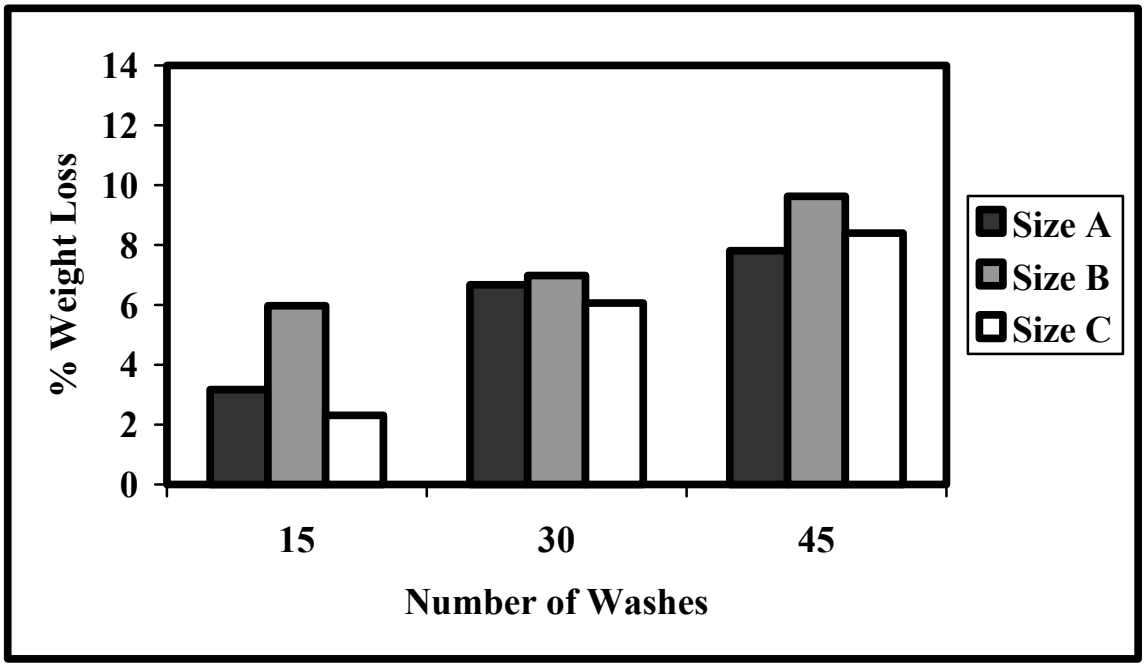


Figure 5.47 Fabric Sample Weight Loss vs. Washing (Heat Set Temp. = 150°F)

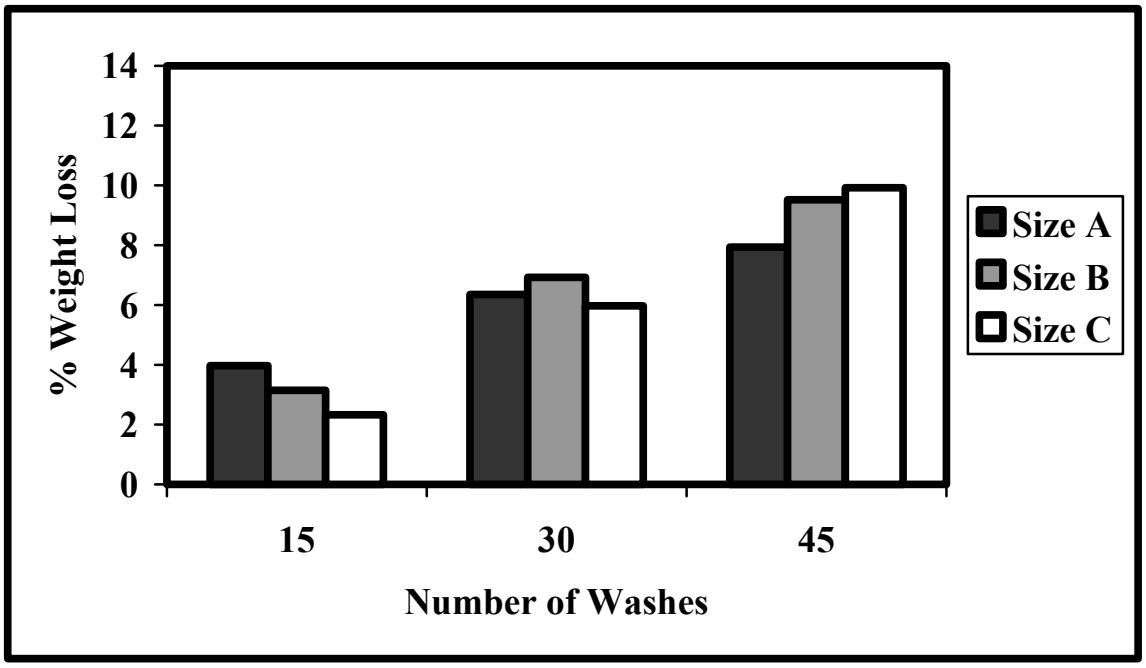


Figure 5.48 Fabric Sample Weight Loss vs. Washing (Heat Set Temp. = 140°F)

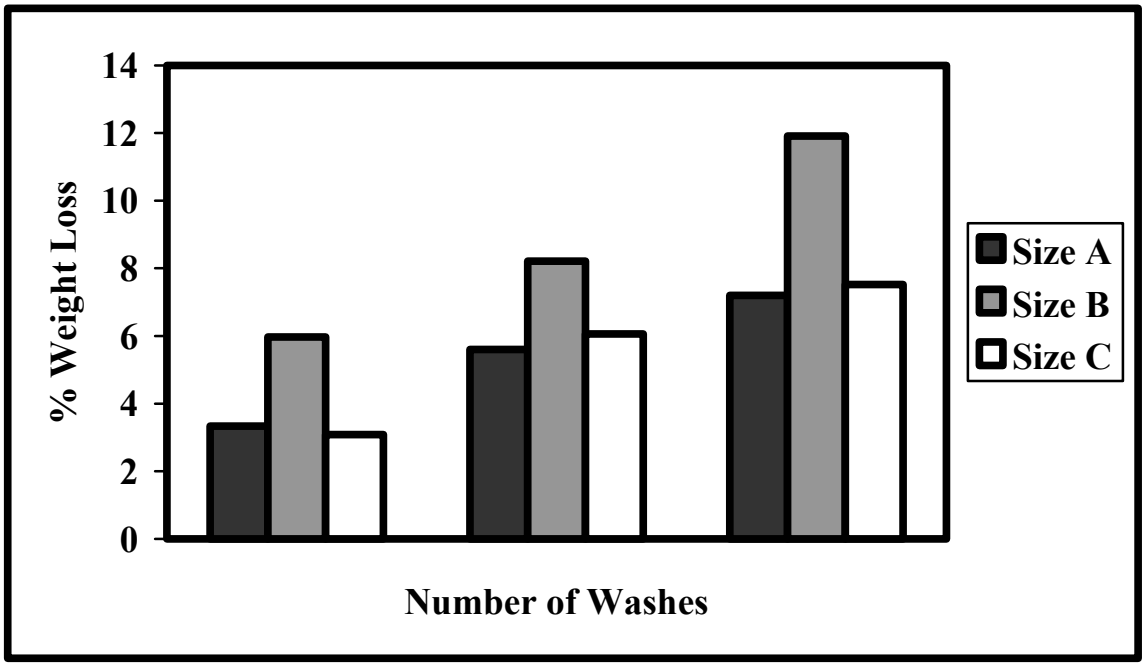


Figure 5.49 Fabric Sample Weight Loss vs. Washing (Heat Set Temp. = 130°F)

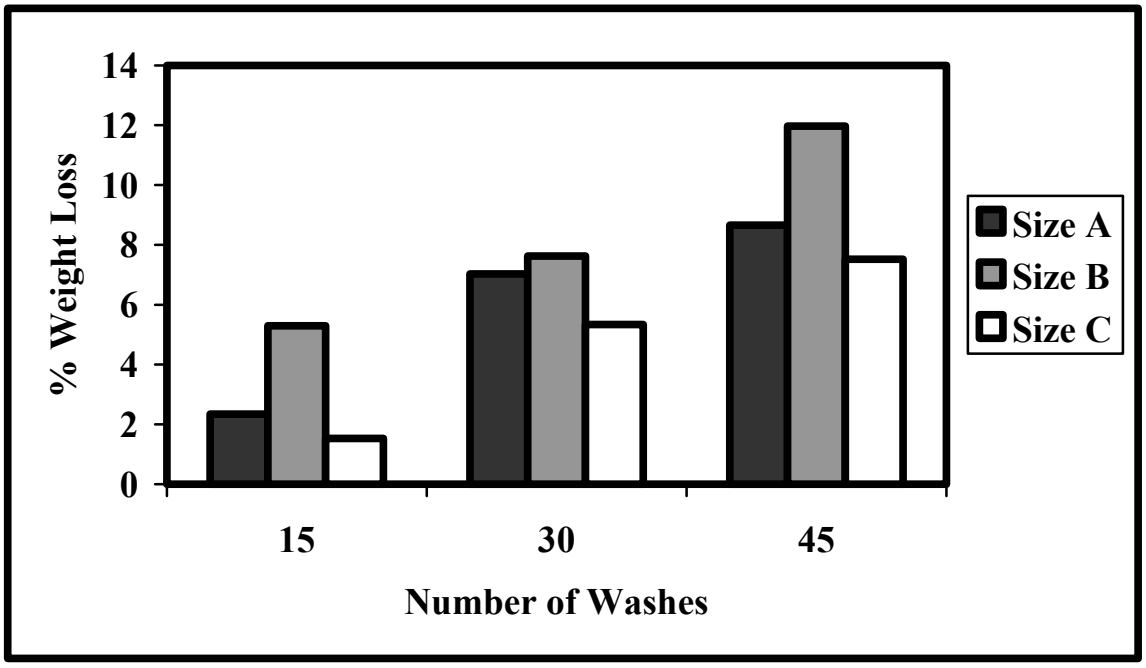


Figure 5.50 Fabric Sample Weight Loss vs. Washing (No Heat Set)

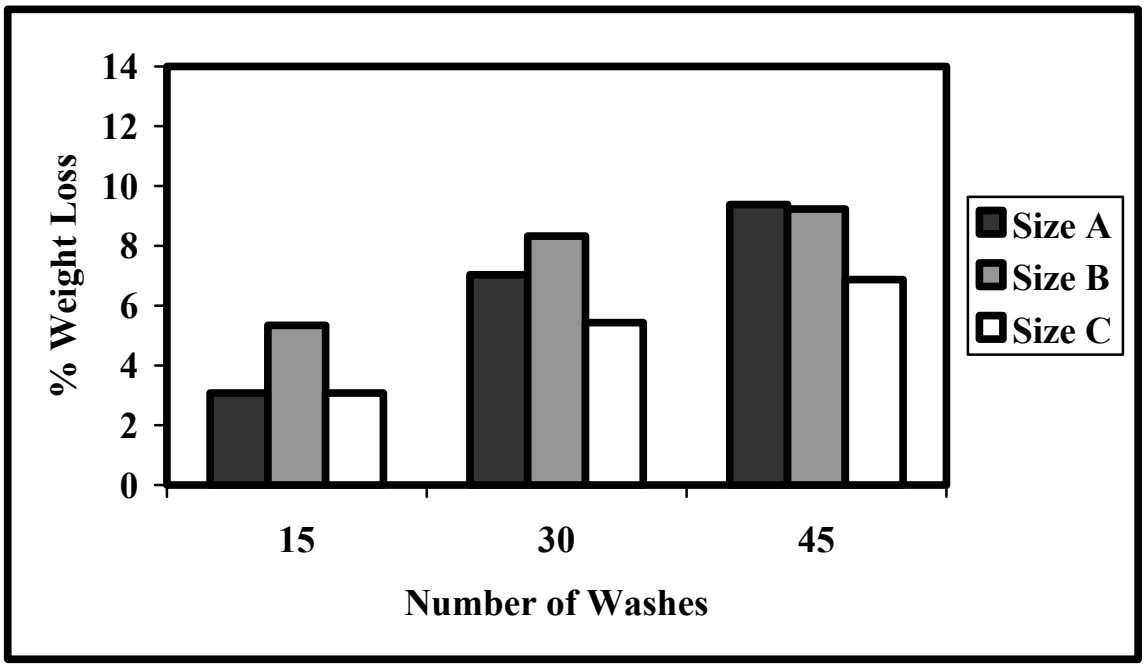
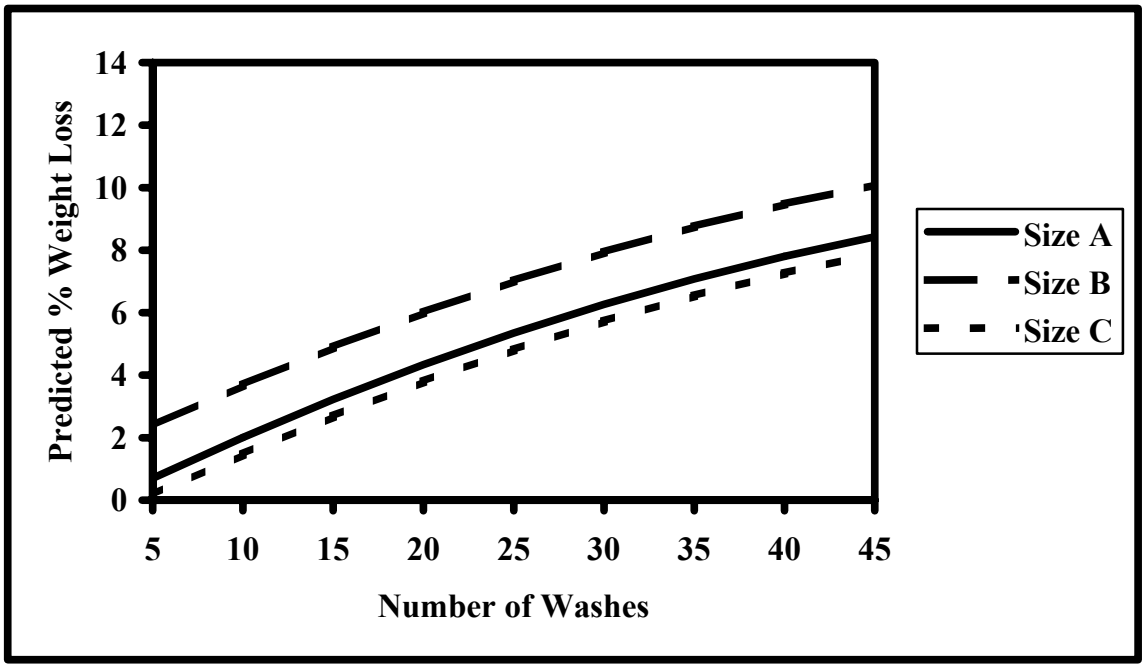


Figure 5.51 Predicted Fabric Sample Weight Loss vs. Washing (Twist Multiple = 4.4, Solid Content = 15%)



5.10 Iodine Color Intensity

5.10.1 Statistical Results for Iodine Color Intensity

Table 5.10 Model For Color Intensity

Response Color Intensity (K/S)
Whole Model
Summary of Fit

RSquare	0.82952
RSquare Adj	0.817121
Root Mean Square Error	0.040114
Mean of Response	0.123333
Observations (or Sum Wgts)	60

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	0.43063133	0.107658	66.9045
Error	55	0.08850200	0.001609	Prob > F
C. Total	59	0.51913333		<.0001

Effect Tests

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
ST	2	2	0.02652333	8.2415	0.0007
ΔW	1	1	0.33200133	206.3238	<.0001
ΔW*ΔW	1	1	0.07210667	44.8110	<.0001

Expanded Estimates
Nominal factors expanded to all levels

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.1132667	0.008607	13.16	<.0001
ST_A	0.0001667	0.007324	0.02	0.9819
ST_B	0.0256667	0.007324	3.50	0.0009
ST_C	-0.025833	0.007324	-3.53	0.0009
ΔW	-0.004436	0.000309	-14.36	<.0001
(ΔW-7.5)*(ΔW-7.5)	0.0001541	0.000023	6.69	<.0001

Table 5.10 shows the multiple regression results for iodine color intensity. *Size Type, ΔWashings,* and the interaction of *ΔWashings* with itself *s*how significant effects on the response. The R-Square value of 82.95% indicates a good fitting for the observed response data. According to the expanded parameter estimates, the prediction equation for size color intensity is:

$$Y = 0.1132667 + 0.0001667\mathbf{ST_A} + 0.0256667\mathbf{ST_B} - 0.025833\mathbf{ST_C} - 0.004436\,\mathbf{\Delta W} + [0.0001541(\mathbf{\Delta W}-7.5)(\mathbf{\Delta W}-7.5)]$$

where, $ST_A = \begin{Bmatrix} 1 \\ 0, otherwise \end{Bmatrix}$, $ST_B = \begin{Bmatrix} 1 \\ 0, otherwise \end{Bmatrix}$,

$ST_C = \begin{Bmatrix} 1 \\ 0, otherwise \end{Bmatrix}$.

5.10.2 Observation and Prediction Analyses for Iodine Color Intensity

Figures 5.52-5.56 depict the results of the actual observations for iodine color intensity at different levels of heat setting. The overall trend implies that the color intensity of the iodine decreases after the first 15 washes and continues to descend towards zero with further washes. This suggests some of the starch in each size is removed after the first 15 washes. The color intensity of Size B is noticeably high before washing occurs. This is because the fabric contains yarns sized at the 13% level (which had an unusually high % size add-on).

According to the regression model, heat setting does not have a significant effect on color intensity level of the iodine drops placed on the fabric samples. The predicted regressions shown in Figure 5.57 illustrate that as the number of washings increases, fabric samples treated with Size B have higher levels of iodine spot color intensity than Sizes A and C. Overall, the color intensity level for all size types decreases at a decreasing rate as the number of washes increases to about 35. At this point, the color intensity level begins to plateau.

Figure 5.52 Iodine Sample Color Intensity vs. Washing (Heat Set Temp. = 160°F)

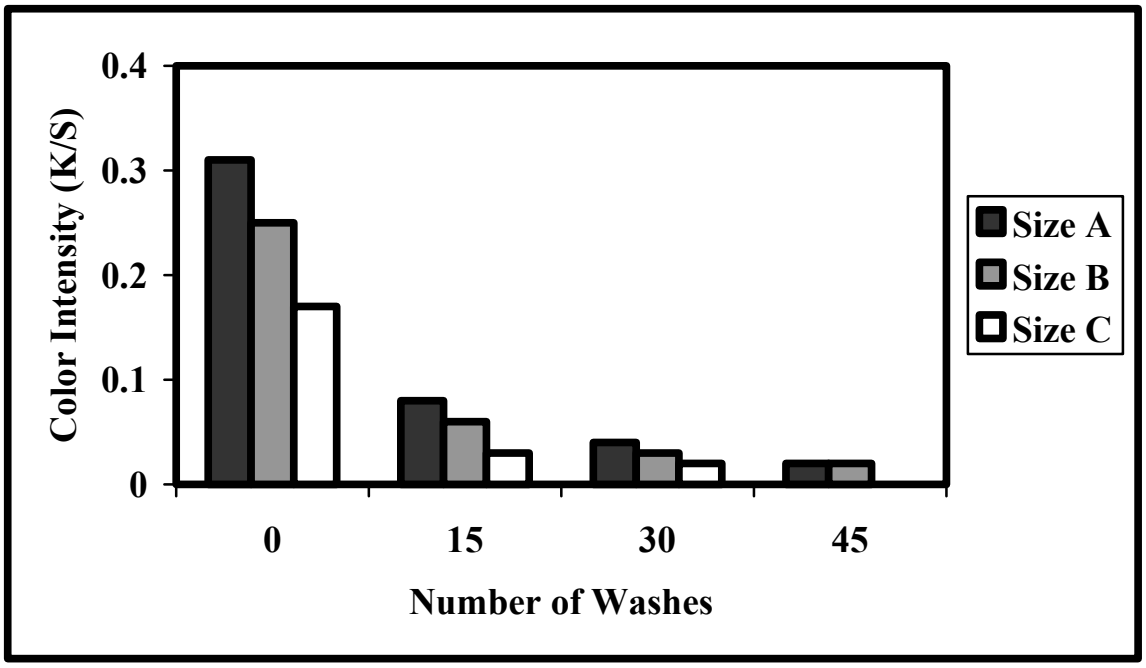


Figure 5.53 Iodine Sample Color Intensity vs. Washing (Heat Set Temp. = 150°F)

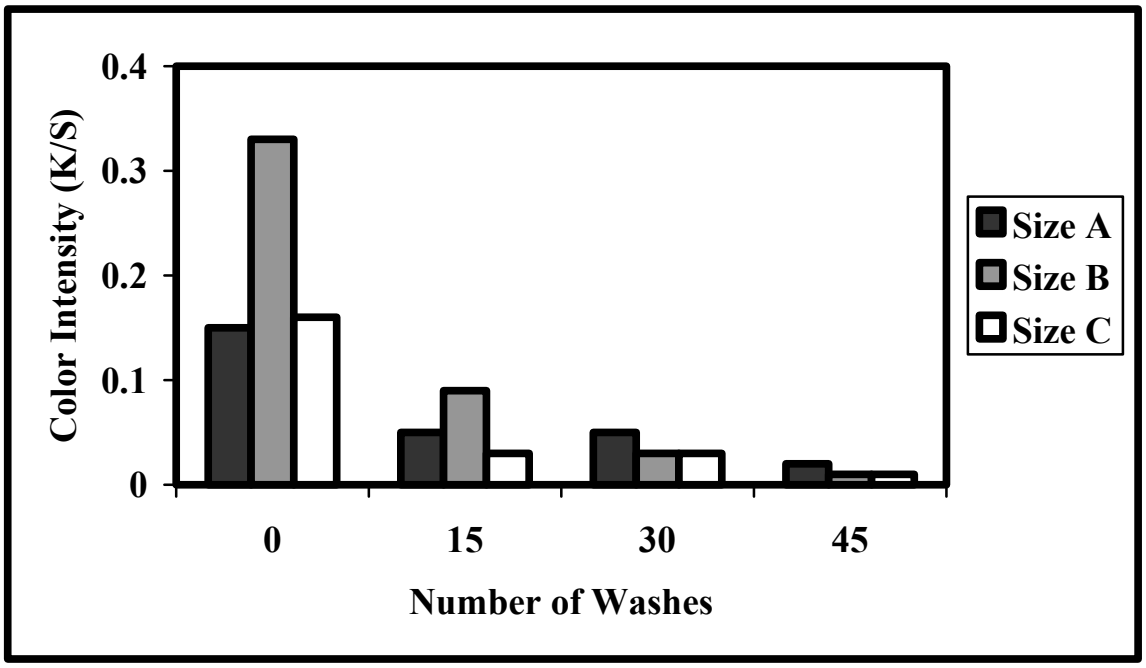


Figure 5.54 Iodine Sample Color Intensity vs. Washing (Heat Set Temp. = 140°F)

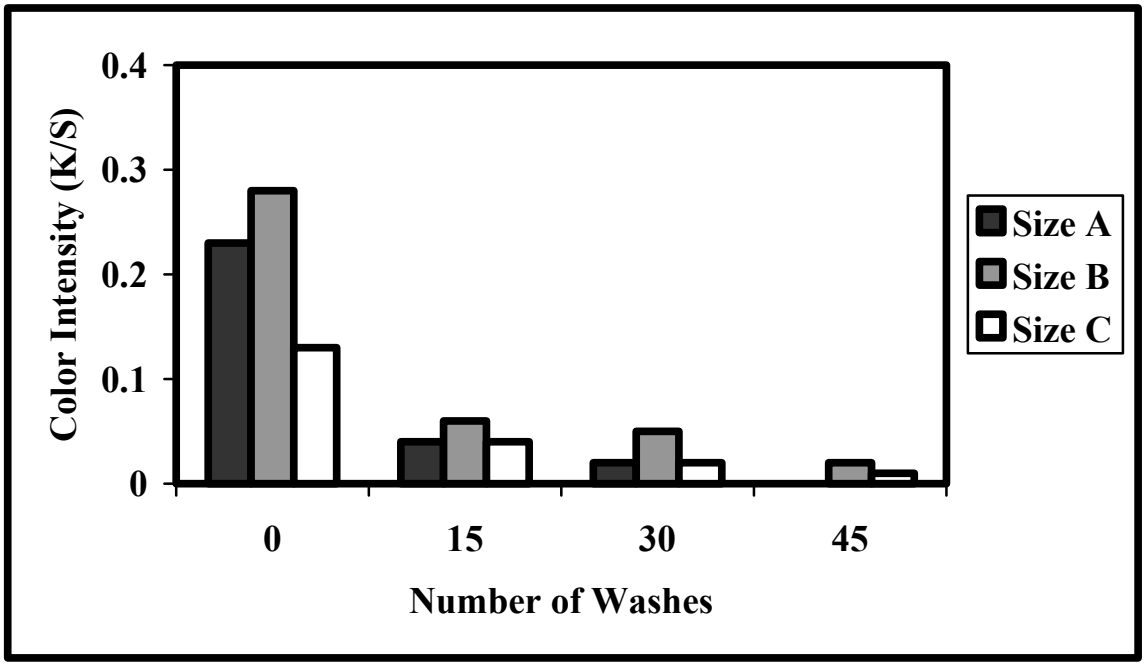


Figure 5.55 Iodine Sample Color Intensity vs. Washing (Heat Set Temp. = 130°F)

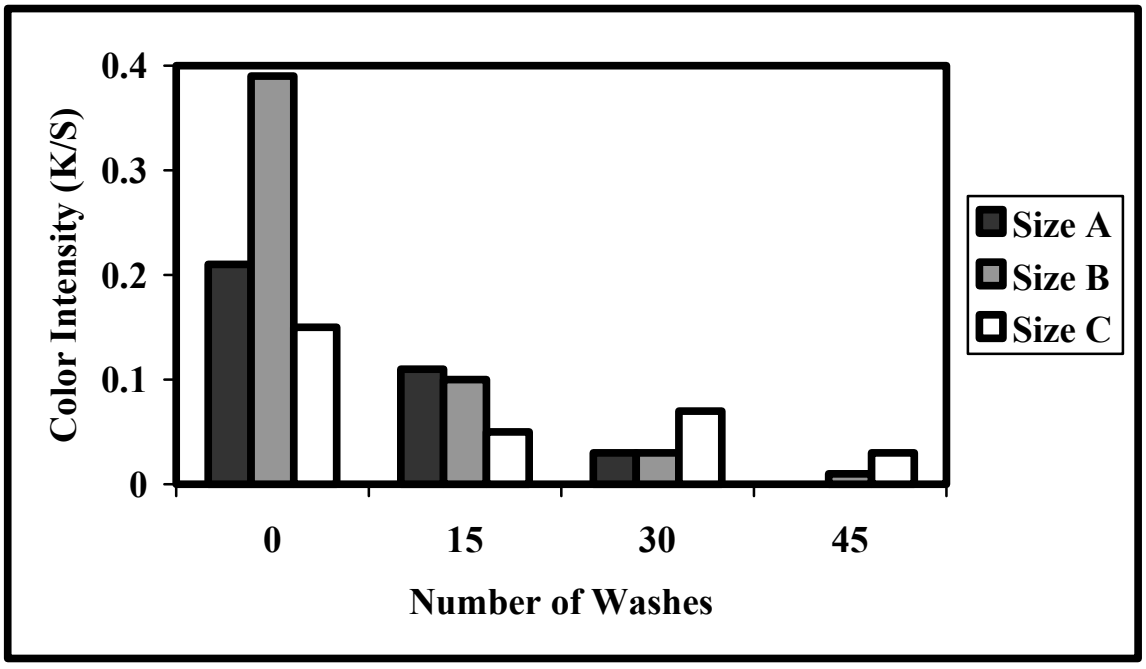


Figure 5.56 Iodine Sample Color Intensity vs. Washing (No Heat Set)

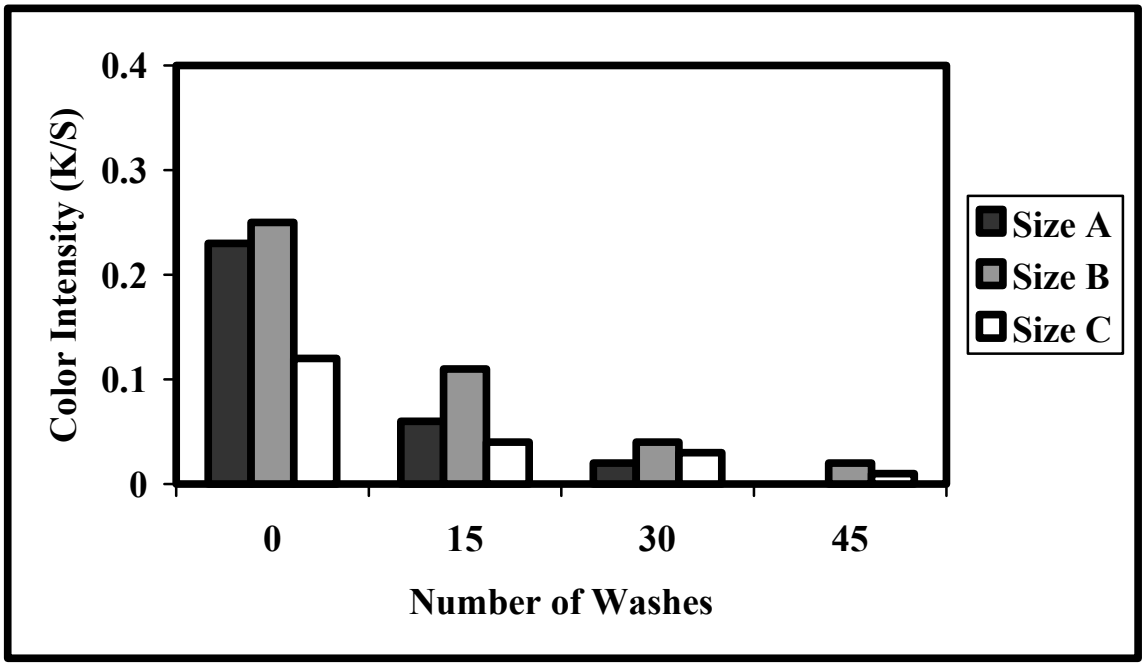
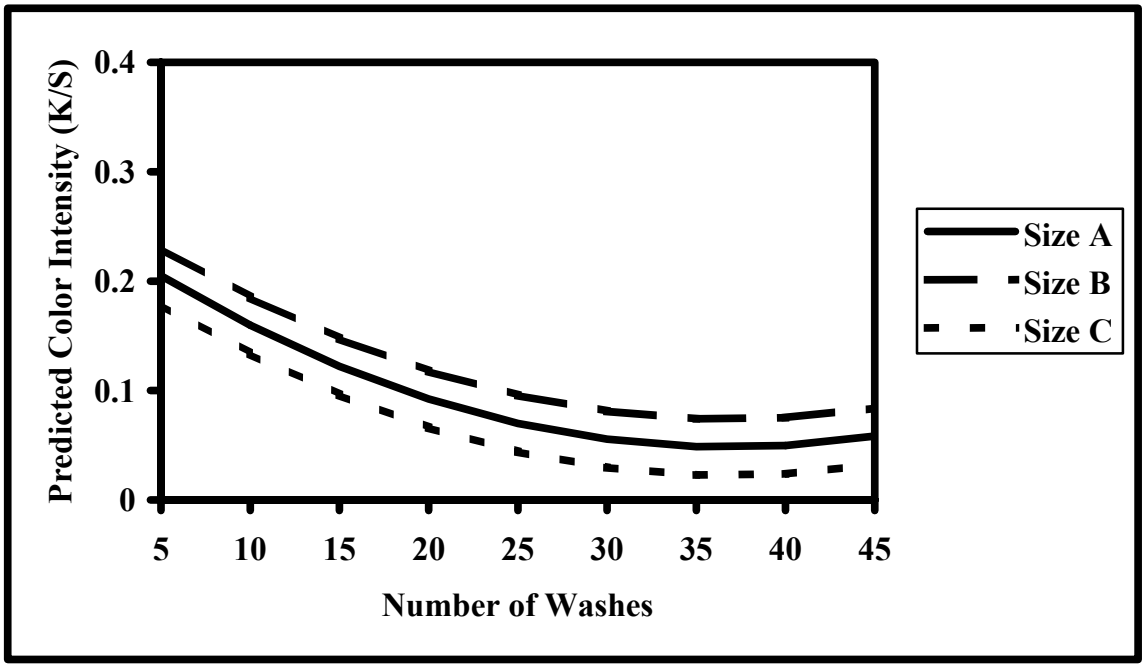


Figure 5.57 Predicted Iodine Sample Color Intensity vs. Washing (TM = 4.4, Solid Content = 15%)



5.11 Experimental Sizes Versus Commercial Sizes

Figures 5.58 and 5.59 compare the three experimental reactive sizing agents to two sizes presently used in commercial slashing operations at 12% solids using 4.2 TM cotton yarns. The first commercial size is a starch styrene/butadiene copolymer, and the second commercial size comes from liquid PVA. Figure 5.58 shows that both commercial sizes have less yarn hairiness than the reactive sizes. Figure 5.59 depicts that the reactive sizes promote less yarn-to-metal friction than the commercial sizes.

Figure 5.58 Yarn Hairiness Comparison
Between Experimental Sizes and Commercial Sizes
(30/1 100% Cotton, 4.2 TM, 12% Solids)

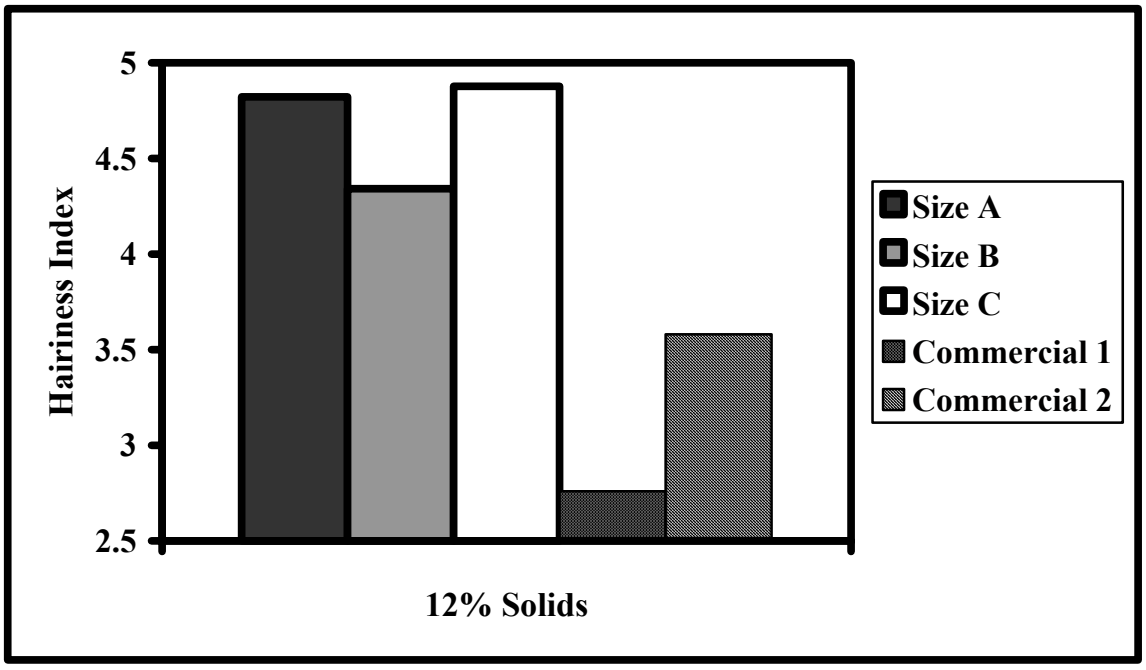
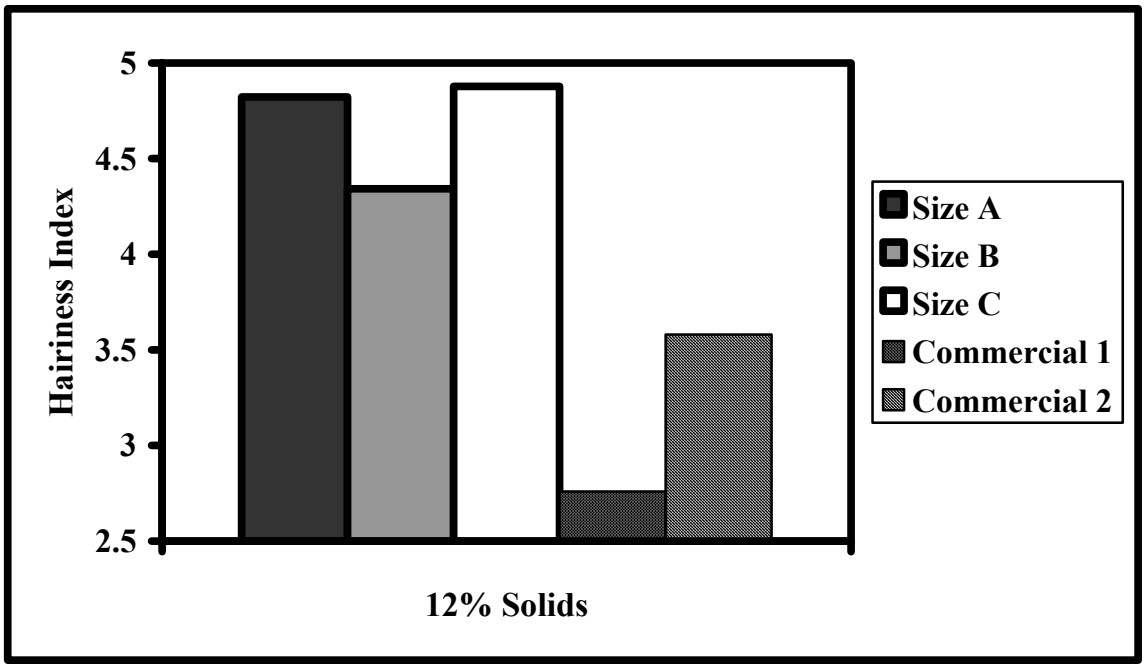


Figure 5.59 Coefficient of Friction Comparison Between Experimental Sizes and Commercial Sizes (30/1 100% Cotton, 4.2 TM, 12% Solids)



6. CONCLUSIONS

Since this is the first investigation performed on the newly developed reactive sizing agents: N-methylolcarbamoylethyl starch (Size A), N-methylolcarbamoylethyl starch + PVA (Size B), and N-methylolcarbamoylethyl starch + PVA + melamine formaldehyde (Size C), the results of this work cannot be benchmarked. According to the research, yarns treated with the three experimental reactive sizing agents show promising results. Compared to the untreated samples, the sized yarns have higher strength and tenacity, lower coefficient of friction, and lower hairiness. Although more research needs to be conducted concerning the overall stability of the reactive sizes, the results show that the sizing agents are forming some permanent bonds with cotton cellulose.

The following conclusions can be made regarding the tensile and stability properties of 30/1, 100% cotton, ring-spun yarns based on statistical analyses:

- Size add-on increases as the twist multiple increases to 4.2 TM, then decreases toward 4.4 TM. Yarns treated with Size B have the highest % size add-on.
- The yarn breaking force is not significantly different among the three size types, and increases at an increasing rate as the twist multiple and solid concentration increases.
- The weakest point in terms of breaking force for all untreated yarns is improved by all three size types
- The yarn tenacity is not significantly different among the three size types, and increases at an increasing rate as the twist multiple and solid concentration increases.

- The yarn elongation is not significantly different among the three size types. The % elongation initially decreases then increases at an increasing rate after 10% solids. The % elongation also becomes higher with an increase in twist multiple.
- The yarn breaking work is not significantly different among the three size types. The breaking work initially decreases until reaching 5% solids, then increases at an increasing rate as % solids and twist multiple increase.
- Size C reduces yarn hairiness more effectively than Sizes A and B as solid content increases.
- The coefficient of friction for all yarns is not significantly different among the three twist multiples. The coefficient of friction initially decreases at a decreasing rate with an increase in solid content. At 10% solids, the coefficient of friction for all yarns increases at an increasing rate as solid content increases. Overall, yarns treated with Size C show less yarn-to-metal friction than Sizes A and B.
- The abrasion resistance for yarns treated with Size C is greater than Sizes A and B as the twist level increases. Size B appears to show the best abrasion resistance as solid content increases for the 4.0 TM and 4.2 TM yarns. Size C shows the best abrasion resistance for the 4.4 TM yarns as % solids increases.
- The weight loss for fabric samples containing Sizes C is lower than samples containing Sizes A and B.
- The color intensity level for all size types decreases at a decreasing rate as the number of washes increases to about 35. At this point, the color intensity level begins to plateau. Overall, the color intensity of Size B remains higher than the other two sizes.

- The increase in % fabric weight loss suggests that the reactive sizes may not be stable during laundering. However, definite conclusions of this cannot be made due to the fact that it is impossible to distinguish which ingredients in the sizes were removed the most after washing. In other words, the wax and lubricant additives may have been removed while the reactive material remained intact with the cellulose molecules in the cotton. Furthermore, fiber loss during washing and drying may be a large contributor to the reduced fabric sample weights.
- The decrease in the color intensity values from the applied iodine spots also implies that the reactive functional groups are not forming complete bonds with the cotton cellulose. However, close examination of the predicted regression shows promising results. Although the color intensity values for the three sizes decrease as the number of washings increases, 50% of the color intensity values at five washes remains for all three sizes at 30 washes.
- Yarns treated with the experimental sizing agents had more hairiness than the two commercial sizes, but had less coefficient of friction values than the two commercial sizes.

7. RECOMMENDATIONS

7.1 Replication of Observations

Due to the limited amount of sizing material and yarn available, the replication of observations for the performed tests was limited. This can cause bias and variability in the statistical analyses. This is especially the case for the tests run on the Uster Tensojet and the Uster Tester 3. Although these testing devices compute the average of many observations per yarn sample, they do not provide data for each individual observation. The "average" value represents only one observation when put into the statistical model. Therefore, it is recommended that more replicate observations be taken for each response in order to reduce the variation of the results and provide a better fit for the regression models.

7.2 Size Encapsulation Study

The results for the abrasion resistance tests seem very skeptical considering not much difference can be seen between yarns treated with the three sizing agents and the untreated yarns according to the actual observations. A reason for this may be that the sizes are penetrating to the core of the yarn and not remaining on the outside surface where initial abrasion occurs. It would be worthwhile to look under the microscope in order to study the amount of sizing that encapsulates the treated yarn samples and determine if this is the case.

7.3 Permanent Bonding Analysis

Due to limited funding, further analysis of the stability of the reactive sizes could not be performed (e.g. nitrogen content analysis, size depth penetration, etc.) Therefore, further investigation is needed to determine the causes for the instability of the three sizes after laundering. Chemical changes or preparation processes may be needed in order to achieve the goal of producing durable and non-removable sizing agents.

7.4 Comparison to Industrial Sizes

Further comparison of the tensile property results for the sized yarns in the experiment should be made to similar yarns treated with selected sizes currently used in slashing operations for cotton warp yarns. This will determine if the currently researched reactive sizing agents are suitable for industrial use.

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9. APPENDIX

Table A1 Data Set for Experimental Design I
(Add-on, B-Force, B-Work, Elongation, Tenacity, Hairiness)

Size	*TM*	*% Solids*	*% Add-on*	*B-Force (cN)*	*B-Work (cN.cm)*	*% Elong.*	*Tenacity (cN/tex)*	*Hairiness (H)*
A	4	17	7.81	302	440.5	5.29	15.1	4.73
A	4	14	7.44	283	398.5	5.14	14.15	4.6
A	4	11	3.3	282.8	410.9	5.27	14.14	4.92
A	4	8	4.43	275.2	387	5.05	13.76	5.57
A	4	5	1.82	251.7	339.8	4.81	12.59	5.96
A	4.2	17	15	324.8	521.2	5.95	16.24	4.04
A	4.2	14	7.71	301.8	463.3	5.7	15.09	4.7
A	4.2	11	7.12	292.9	406.1	4.99	14.64	5.22
A	4.2	8	4.94	282.3	419.5	5.41	14.11	5.33
A	4.2	5	5.54	270.7	383.2	5.08	13.53	5.88
A	4.4	17	8.43	352.2	566.3	6.03	17.61	3.41
A	4.4	14	7.16	312	453.3	5.28	15.6	4.74
A	4.4	11	4.02	301	449.4	5.46	14.05	5.02
A	4.4	8	2.52	286.1	410.6	5.2	14.97	4.91
A	4.4	5	1.5	283.7	414.8	5.31	14.18	5.57
B	4	13	10.21	332.4	447.8	4.91	16.62	3.32
B	4	11	8.52	283	357.1	4.42	14.15	4.81
B	4	9	6.67	278.9	393.5	5.11	13.94	4.53
B	4	7	4.57	276.4	379.2	5	13.82	5.17
B	4	5	3.09	266.8	366.4	4.82	13.34	5.58
B	4.2	13	9.09	297.5	401.2	4.84	14.88	4.42
B	4.2	11	8.08	302.3	411.9	4.81	15.12	4.45
B	4.2	9	8.64	291.9	417	5.16	14.6	5.06
B	4.2	7	5.29	286.6	399	5.03	14.33	5.16
B	4.2	5	4.28	283.4	404.5	5.19	14.17	5.27
B	4.4	13	7.84	312.3	481.4	5.67	15.62	4.34
B	4.4	11	6.64	310.1	463	5.41	15.5	4.56
B	4.4	9	3.28	304.4	460	5.58	15.22	4.8
B	4.4	7	4.97	297.5	427.1	5.19	14.88	4.97
B	4.4	5	1.06	289	429.2	5.44	14.45	5.39
C	4	16	8.86	301.1	454.5	5.47	15.06	5.63
C	4	14	10.82	295.8	407.2	4.91	14.79	4.56
C	4	11	9.81	280.4	407.3	5.19	14.02	4.79
C	4	8	7.26	275	375.6	4.87	13.75	5.39
C	4	5	1.49	259.7	364.7	5.02	12.99	5.77
C	4.2	16	11.41	306.8	439.7	5.14	14.34	4.91
C	4.2	14	8.9	300.4	444.6	5.35	15.02	4.22
C	4.2	11	8.08	297.4	410.7	4.9	14.87	5.04
C	4.2	8	8.56	291.7	415.2	5.1	14.58	5.05
C	4.2	5	1.62	290.1	410.5	5.07	14.51	5.44
C	4.4	16	7.8	318.5	479.6	5.52	15.92	4.46
C	4.4	14	9.97	323.8	488.5	5.53	16.19	4.03
C	4.4	11	5.64	309.5	464.9	5.53	15.47	4.71
C	4.4	8	1.97	296.2	402.8	4.86	14.81	5.17
C	4.4	5	1.28	287.8	427.5	5.34	14.39	5.28
Untreated	4	0	.	262.7	373.9	5.15	13.14	6.24
Untreated	4.2	0	.	283.8	416.8	5.33	14.19	6.11
Untreated	4.4	0	.	293.3	458	5.68	14.66	5.55

Table A2 Data Set for Experimental Design I
(Abrasion Resistance, Coefficient of Friction)

Size	*TM*	*% Solids*	*Abras. Resist. (Strokes/Break)*	*Coef. of Friction*
A	4	17	28	0.18
A	4	14	22	0.18
A	4	11	34	0.17
A	4	8	25	0.18
A	4	5	14	0.17
A	4.2	17	36	0.18
A	4.2	14	41	0.17
A	4.2	11	32	0.18
A	4.2	8	34	0.18
A	4.2	5	20	0.18
A	4.4	17	50	0.17
A	4.4	14	36	0.17
A	4.4	11	45	0.17
A	4.4	8	39	0.17
A	4.4	5	31	0.18
B	4	13	55	0.17
B	4	11	29	0.18
B	4	9	26	0.18
B	4	7	36	0.16
B	4	5	12	0.18
B	4.2	13	32	0.17
B	4.2	11	35	0.17
B	4.2	9	30	0.17
B	4.2	7	39	0.18
B	4.2	5	27	0.17
B	4.4	13	32	0.18
B	4.4	11	41	0.18
B	4.4	9	42	0.2
B	4.4	7	36	0.18
B	4.4	5	37	0.19
C	4	16	40	0.18
C	4	14	21	0.14
C	4	11	30	0.17
C	4	8	34	0.19
C	4	5	17	0.16
C	4.2	16	43	0.19
C	4.2	14	37	0.18
C	4.2	11	36	0.17
C	4.2	8	32	0.14
C	4.2	5	45	0.2
C	4.4	16	52	0.19
C	4.4	14	41	0.18
C	4.4	11	45	0.18
C	4.4	8	39	0.18
C	4.4	5	48	0.18

Table A2 Continued

Size	*TM*	*% Solids*	*Abras. Resist. (Strokes/Break)*	*Coef. of Friction*
A	4	17	30	0.17
A	4	14	32	0.17
A	4	11	27	0.17
A	4	8	26	0.18
A	4	5	23	0.18
A	4.2	17	41	0.19
A	4.2	14	42	0.18
A	4.2	11	32	0.17
A	4.2	8	36	0.17
A	4.2	5	29	0.19
A	4.4	17	61	0.16
A	4.4	14	38	0.17
A	4.4	11	48	0.18
A	4.4	8	45	0.18
A	4.4	5	35	0.18
B	4	13	66	0.17
B	4	11	37	0.18
B	4	9	32	0.16
B	4	7	43	0.18
B	4	5	25	0.17
B	4.2	13	34	0.18
B	4.2	11	40	0.18
B	4.2	9	31	0.17
B	4.2	7	42	0.16
B	4.2	5	32	0.17
B	4.4	13	35	0.16
B	4.4	11	42	0.17
B	4.4	9	44	0.17
B	4.4	7	39	0.18
B	4.4	5	39	0.19
C	4	16	40	0.18
C	4	14	28	0.14
C	4	11	36	0.16
C	4	8	40	0.17
C	4	5	35	0.17
C	4.2	16	57	0.16
C	4.2	14	38	0.17
C	4.2	11	37	0.18
C	4.2	8	35	0.14
C	4.2	5	48	0.18
C	4.4	16	53	0.16
C	4.4	14	43	0.14
C	4.4	11	56	0.17
C	4.4	8	41	0.18
C	4.4	5	58	0.17

Table A2 Continued

Size	*TM*	*% Solids*	*Abras. Resist. (Strokes/Break)*	*Coef. of Friction*
A	4	17	32	0.17
A	4	14	35	0.18
A	4	11	29	0.18
A	4	8	28	0.17
A	4	5	26	0.17
A	4.2	17	12	0.2
A	4.2	14	44	0.18
A	4.2	11	36	0.18
A	4.2	8	39	0.17
A	4.2	5	30	0.18
A	4.4	17	64	0.17
A	4.4	14	41	0.17
A	4.4	11	48	0.17
A	4.4	8	49	0.18
A	4.4	5	36	0.18
B	4	13	67	0.18
B	4	11	38	0.17
B	4	9	32	0.18
B	4	7	43	0.19
B	4	5	25	0.17
B	4.2	13	42	0.18
B	4.2	11	41	0.19
B	4.2	9	32	0.18
B	4.2	7	42	0.18
B	4.2	5	36	0.18
B	4.4	13	50	0.17
B	4.4	11	42	0.18
B	4.4	9	45	0.17
B	4.4	7	45	0.18
B	4.4	5	43	0.17
C	4	16	42	0.18
C	4	14	33	0.17
C	4	11	38	0.14
C	4	8	43	0.17
C	4	5	35	0.17
C	4.2	16	60	0.16
C	4.2	14	39	0.18
C	4.2	11	40	0.18
C	4.2	8	42	0.14
C	4.2	5	52	0.17
C	4.4	16	55	0.18
C	4.4	14	46	0.18
C	4.4	11	60	0.16
C	4.4	8	42	0.16
C	4.4	5	60	0.17

Table A2 Continued

Size	*TM*	*% Solids*	*Abras. Resist. (Strokes/Break)*	*Coef. of Friction*
A	4	17	35	0.17
A	4	14	40	0.17
A	4	11	33	0.18
A	4	8	34	0.17
A	4	5	29	0.18
A	4.2	17	15	0.17
A	4.2	14	47	0.18
A	4.2	11	41	0.18
A	4.2	8	42	0.17
A	4.2	5	38	0.17
A	4.4	17	71	0.17
A	4.4	14	43	0.18
A	4.4	11	49	0.16
A	4.4	8	55	0.18
A	4.4	5	39	0.17
B	4	13	76	0.18
B	4	11	40	0.18
B	4	9	36	0.18
B	4	7	45	0.16
B	4	5	32	0.18
B	4.2	13	53	0.17
B	4.2	11	47	0.17
B	4.2	9	38	0.18
B	4.2	7	44	0.17
B	4.2	5	42	0.18
B	4.4	13	55	0.18
B	4.4	11	52	0.18
B	4.4	9	48	0.18
B	4.4	7	46	0.18
B	4.4	5	44	0.18
C	4	16	43	0.17
C	4	14	35	0.17
C	4	11	42	0.17
C	4	8	45	0.18
C	4	5	38	0.17
C	4.2	16	63	0.17
C	4.2	14	42	0.17
C	4.2	11	44	0.16
C	4.2	8	47	0.18
C	4.2	5	54	0.17
C	4.4	16	67	0.18
C	4.4	14	51	0.18
C	4.4	11	60	0.16
C	4.4	8	47	0.17
C	4.4	5	61	0.17

Table A2 Continued

Size	*TM*	*% Solids*	*Abras. Resist. (Strokes/Break)*	*Coef. of Friction*
A	4	17	41	0.18
A	4	14	42	0.17
A	4	11	38	0.17
A	4	8	35	0.17
A	4	5	31	0.18
A	4.2	17	18	0.18
A	4.2	14	51	0.18
A	4.2	11	42	0.18
A	4.2	8	47	0.17
A	4.2	5	40	0.17
A	4.4	17	79	0.17
A	4.4	14	43	0.18
A	4.4	11	56	0.16
A	4.4	8	60	0.18
A	4.4	5	41	0.17
B	4	13	87	0.17
B	4	11	50	0.17
B	4	9	41	0.18
B	4	7	45	0.16
B	4	5	33	0.18
B	4.2	13	54	0.17
B	4.2	11	45	0.18
B	4.2	9	48	0.18
B	4.2	7	47	0.18
B	4.2	5	44	0.17
B	4.4	13	56	0.17
B	4.4	11	55	0.18
B	4.4	9	48	0.16
B	4.4	7	51	0.17
B	4.4	5	51	0.17
C	4	16	44	0.17
C	4	14	37	0.17
C	4	11	44	0.17
C	4	8	54	0.16
C	4	5	40	0.18
C	4.2	16	69	0.17
C	4.2	14	43	0.17
C	4.2	11	55	0.16
C	4.2	8	52	0.16
C	4.2	5	60	0.17
C	4.4	16	68	0.17
C	4.4	14	58	0.17
C	4.4	11	61	0.17
C	4.4	8	47	0.16
C	4.4	5	70	0.16

Table A2 Continued

Size	*TM*	*% Solids*	*Abras. Resist. (Strokes/Break)*	*Coef. of Friction*
Untreated	4	0	41	0.23
Untreated	4.2	0	26	0.22
Untreated	4.4	0	51	0.22
Untreated	4	0	44	0.24
Untreated	4.2	0	37	0.24
Untreated	4.4	0	52	0.24
Untreated	4	0	45	0.25
Untreated	4.2	0	39	0.24
Untreated	4.4	0	52	0.22
Untreated	4	0	46	0.23
Untreated	4.2	0	40	0.22
Untreated	4.4	0	56	0.22
Untreated	4	0	56	0.24
Untreated	4.2	0	43	0.24
Untreated	4.4	0	64	0.22

Table A3 Data Set for Experimental Design II (% Fabric Weight Loss)

Sample #	*Heat Set Temp.*	*# Washes*	*Wt. Before (g)*	*Wt. After (g)*	*% Weight Loss*
A11	160	15	1.26	1.22	3.17
A12	160	30	1.35	1.26	6.67
A13	160	45	1.28	1.18	7.81
A21	150	15	1.26	1.21	3.97
A22	150	30	1.26	1.18	6.35
A23	150	45	1.26	1.16	7.94
A31	140	15	1.20	1.16	3.33
A32	140	30	1.25	1.18	5.60
A33	140	45	1.25	1.16	7.20
A41	130	15	1.28	1.25	2.34
A42	130	30	1.28	1.19	7.03
A43	130	45	1.27	1.16	8.66
A51	0	15	1.30	1.26	3.08
A52	0	30	1.28	1.19	7.03
A53	0	45	1.28	1.16	9.38
B11	160	15	1.34	1.26	5.97
B12	160	30	1.29	1.20	6.98
B13	160	45	1.35	1.22	9.63
B21	150	15	1.27	1.23	3.15
B22	150	30	1.30	1.21	6.92
B23	150	45	1.26	1.14	9.52
B31	140	15	1.34	1.26	5.97
B32	140	30	1.34	1.23	8.21
B33	140	45	1.34	1.19	11.19
B41	130	15	1.32	1.25	5.30
B42	130	30	1.31	1.21	7.63
B43	130	45	1.34	1.19	11.19
B51	0	15	1.31	1.24	5.34
B52	0	30	1.32	1.21	8.33
B53	0	45	1.30	1.18	9.23
C11	160	15	1.30	1.27	2.31
C12	160	30	1.32	1.24	6.06
C13	160	45	1.31	1.20	8.40
C21	150	15	1.29	1.26	2.33
C22	150	30	1.34	1.26	5.97
C23	150	45	1.31	1.18	9.92
C31	140	15	1.30	1.26	3.08
C32	140	30	1.32	1.24	6.06
C33	140	45	1.33	1.23	7.52
C41	130	15	1.31	1.29	1.53
C42	130	30	1.31	1.24	5.34
C43	130	45	1.33	1.23	7.52
C51	0	15	1.30	1.26	3.08
C52	0	30	1.29	1.22	5.43
C53	0	45	1.31	1.22	6.87

Table A4 Data Set for Experimental Design II (Color Intensity)

Sample #	*Heat Set Temp.*	*# Washes*	*Color Intensity (K/S)*
A10	160	0	0.31
A11	160	15	0.08
A12	160	30	0.04
A13	160	45	0.02
A20	150	0	0.15
A21	150	15	0.05
A22	150	30	0.05
A23	150	45	0.02
A30	140	0	0.23
A31	140	15	0.04
A32	140	30	0.02
A33	140	45	0
A40	130	0	0.21
A41	130	15	0.11
A42	130	30	0.03
A43	130	45	0
A50	0	0	0.23
A51	0	15	0.06
A52	0	30	0.02
A53	0	45	0
B10	160	0	0.25
B11	160	15	0.06
B12	160	30	0.03
B13	160	45	0.02
B20	150	0	0.33
B21	150	15	0.09
B22	150	30	0.03
B23	150	45	0.01
B30	140	0	0.28
B31	140	15	0.06
B32	140	30	0.05
B33	140	45	0.02
B40	130	0	0.39
B41	130	15	0.1
B42	130	30	0.03
B43	130	45	0.01
B50	0	0	0.25
B51	0	15	0.11
B52	0	30	0.04
B53	0	45	0.02
C10	160	0	0.17
C11	160	15	0.03
C12	160	30	0.02
C13	160	45	0

Table A4 Continued

Sample #	*Heat Set Temp.*	*# Washes*	*Color Intensity (K/S)*
C20	150	0	0.16
C21	150	15	0.03
C22	150	30	0.03
C23	150	45	0.01
C30	140	0	0.13
C31	140	15	0.04
C32	140	30	0.02
C33	140	45	0.01
C40	130	0	0.15
C41	130	15	0.05
C42	130	30	0.04
C43	130	45	0.03
C50	0	0	0.12
C51	0	15	0.04
C52	0	30	0.03
C53	0	45	0.01