

ABSTRACT

CRUTCHFIELD, JOHN MATTHEW. Durable Antistatic Finishes via Plasma Aided Electroless Deposition. (Under the direction of Dr. Peter Hauser and Dr. Abdel-Fattah Seyam.)

The objective of this research is to develop durable an antistatic treatment using a green approach to combat numerous problems associated with static electricity, including gas pump fires, increased fabric soiling, startling shocks, and microelectronic damage. Fabrics studied in this research include: woven staple nylon, woven staple polyester, woven staple polypropylene, woven staple acrylic, woven filament nylon, and woven filament polyester. Electroless deposition is common antistatic finish for textile materials. Currently, nitric acid is the accepted pretreatment for electroless deposition systems. Despite the effectiveness of nitric acid pretreatment with electroless deposition in industry, acid pretreatment produces harmful effluent. Replacing acid pretreatment with plasma pretreatment, in electroless deposition systems, will create a more “green” process. Since plasma pretreatment and electroless deposition have been used successfully in other textile end uses using electroless deposition, it is anticipated that this technology can be applied to the creation of antistatic finishes.

Research showed that plasma pretreatment imparted hydrophilic properties to samples, without negatively impacting tensile strength values. Scanning electron microscopy images showed that plasma pretreatment had superior etching abilities, compared to nitric acid pretreatment. Samples pretreated with plasma and electroless deposition had significantly more copper present on the surface, evidenced by scanning electron microscopy and inductively coupled plasma elemental analysis. Nitric acid pretreated samples with electroless deposition had better resistivity values than plasma pretreated samples with

electroless deposition. Superior static charge and dissipation results flip-flopped between nitric acid pretreatment with electroless deposition and plasma pretreatment with electroless deposition. Plasma pretreatment with electroless deposition was a superior dissipation treatment for spun nylon 6,6 and filament polyester. Contrarily, nitric acid pretreatment with electroless deposition was a superior dissipation treatment for filament nylon 6,6 and spun acrylic. Spun polyester and spun polypropylene had similar dissipation results for both treatments.

This research solidifies that plasma pretreatment increases copper uptake on textile materials. Increased copper uptake will allow manufacturers to cut back on chemicals used in electroless deposition baths. Though increased copper uptake was associated with plasma pretreatment used in this research, uniformity of the finish was visible through the human eye and scanning electron microscopy. Since samples with more copper didn't always have superior antistatic properties, it is apparent that the uniformity of the finish plays a significant part with regard to its performance. Thus, future research should focus on improving the uniformity of the finish through plasma pretreatment application and electroless bath application.

Durable Antistatic Finishes via Plasma Aided Electroless Deposition

by
John Matthew Crutchfield

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APPROVED BY:

Dr. Peter Hauser
Co-Committee Chair

Dr. Abdel-Fattah Seyam
Co-Committee Chair

Dr. Ahmed El-Shafei
Committee Member

Dr. Henry Boyter, Jr.
ITT Committee Member

BIOGRAPHY

Matt Crutchfield was born in Raleigh, North Carolina on May 21, 1987 to John Urquhart Crutchfield Jr. and Pamela Fitch Crutchfield. He grew up in Cary, North Carolina, attending West Lake Elementary School, West Lake Middle School, and Athens Drive High School. At Athens Drive High School, Matt was part of the National Honors Society and graduated as a Honors Scholar in May 2005. In the fall of 2005, Matt enrolled in the First Year College program at North Carolina State University. After taking general education classes, Matt matriculated into the College of Textiles in the fall of 2006. After enrolling in the College of Textiles, Matt became involved with the student association of the American Association of Textile Chemists and Colorists, Phi Psi National Textile Fraternity, and Textile Entrepreneurs Club. Matt worked on undergraduate research projects with Dr. Martin King and Professor Nancy Powell. For his senior design project, Matt worked with the Institute of Textile Technology and Raleigh Denim on denim redesign. In May 2009, Matt graduated with Magna Cum Laude honors from the College of Textiles. Matt was accepted into the Textile Technology Management Master's program at the North Carolina State University College of Textiles in the fall of 2009 and was a recipient of the Institute of Textile Technology fellowship.

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CHAPTER 1: INTRODUCTION

Static electricity occurs on the surface of insulators when there is an imbalance of positive and negative charges in a material. Disparity in charges stems from interaction with dissimilar materials. Problems with static electricity include gas pump fires, increased fabric soiling, and startling shocks. Chemical finishing has commonly been used to eliminate static electricity in textiles. One common type antistatic chemical finishing uses an acid pretreatment to etch the fiber surface, followed by electroless deposition of a metal to dissipate static electricity. A problem with this acid pretreatment is the creation of a hazardous effluent.

Previous research using plasma as a pretreatment etching mechanism in electroless deposition has utilized a number of variables, including different gasses used, substrates, and metals deposited. Oxygen and nitrogen gas has been used to etch the surface of polymeric materials (Zhu et al, 2006; Tao et al, 2008). Substrates examined in previous research include polyester, carbon nanofibers, nylon, and polypropylene. Plasma etching has been used as replacement of acid treatment to achieve water repellency, electrical conductivity, ultra-violet protection, and electromagnetic shielding (Tao, et al 2008; Stefecka et al, 2004; Kan and Yuen, 2007; and Zhu et al, 2006). However, no research has been conducted on its viability in creating antistatic finishes on synthetic textiles.

Electroless deposition of copper with pretreatment has been used with carbon nanofibers to impart antistatic properties (Zhu et al, 2008). Synthetic substrates, such as nylon and polypropylene, have used electroless deposition of nickel with acid pretreatment (Yuen et al, 2007; Gan et al, 2008; Jiang et al, 2008). Since acid pretreatment and electroless deposition

has been used as a successful antistatic finish, and plasma pretreated electroless deposition has been used effectively for other textile applications, it can be argued that plasma aided electroless deposition has the potential to be a viable antistatic finish.

The goal of this research is to compare the performance of plasma pretreated electroless deposition finishes to acid pretreated electroless deposition finishes. Antistatic finishes, imparted by acid aided electroless deposition and plasma aided electroless deposition, will be used on woven spun nylon, woven filament nylon, woven spun polyester, woven filament polyester, woven spun acrylic, and woven spun polypropylene fabrics. Control samples, without any treatment, will also be used to quantify the improvement in antistatic properties, imparted via acid or plasma pretreated electroless deposition. Fabrics treated with plasma pretreated electroless deposition will be compared against acid pretreated electroless deposition samples, to see if plasma pretreatment improves antistatic properties.

Acid pretreated samples will be treated according to a standard commercial pretreatment procedure used in conjunction with electroless deposition. Samples pretreated with plasma will be exposed to helium and oxygen based atmospheric plasma to etch the fabric surface. It is anticipated that plasma pretreated electroless deposition will increase the deposition rate of metal on the surface of fabric samples. It is surmised that fabrics with more metal present on their surface, will yield superior antistatic properties. Metal on the surface of fabrics allows for charge dissipation and decreased surface resistivity. Antistatic testing, including surface resistivity and dissipation will be conducted on untreated, acid pretreated, and plasma pretreated samples with electroless deposition. Therefore, antistatic properties can be quantified and conclusions can be drawn on the performance of the finish.

CHAPTER 2: LITERATURE REVIEW

2.1 Causes of static

Static electricity is not a recent phenomenon. Over 25 centuries ago, the Greek scientist Thales of Meletus, discovered that amber attracts particles of dust when rubbed with animal fur. Nearly 2000 years later a London physician, William Gilbert, discovered that other materials, in addition to amber, could be rubbed together to create charge which attracts other objects. It wasn't until 1757, by J.C. Wilcke that any charged body had an electrical sphere of action. Based on this the triboelectric series was developed (Pionteck and Wypych, 2007). A simplified triboelectric series is shown in **Table 2.1**.

**Table 2.1 Triboelectric Series
(Schindler and Hauser, 2004)**

	Fiber Type
Positive end of series	Glass
	Wool
	Nylon 6
	Nylon 6.6
	Rayon
Neutral	Cotton
	Acetate
	Hemp
	Silk
	Polyester
	Acrylic, modacrylic
	Polyethylene, polypropylene
Negative end of series	Polytetrafluoroethylene

When two materials interact with each other, electrons are exchanged. One material becomes positively charged, and the other negatively charged (Pionteck and Wypych, 2007). Rubbing increases charge generation, though rubbing isn't necessarily needed for charge production (Schindler and Hauser, 2004). Uneven rubbing of materials causes a temperature gradient. A temperature gradient contributes to electron unbalance across the surface of a material. The surface of a material will have both positive and negative areas in this situation (Pionteck and Wypych, 2007). Polymeric materials, including textiles, are classified as insulators.

Insulators hold the imparted charge for an elongated period of time. Contrarily, conductive materials, such as metals, dissipate electrostatic buildup quickly. A fiber becomes positively charged when it is rubbed with a fiber below it in the triboelectric series. For instance, if polyester and nylon 6,6 are rubbed together, polyester becomes negatively charged and nylon becomes positively charged. The greater the difference in the triboelectric series of two materials, the greater the charge produced (Schindler and Hauser, 2004). The rate of electrostatic discharge depends on the relative humidity, material type, and the environment (Pionteck and Wypych, 2007).

2.2 Problems with static buildup

Static buildup in textile materials plagues manufacturers and consumers, alike. In manufacturing, fibers and fabrics move over the surface of processing equipment, causing a buildup of electrons on the surface of the textile material. This is commonly known as electrostatic charging. Electrostatic charge causes ballooning, where yarns repel each other during manufacturing processes. To prevent ballooning, synthetic fibers are treated with a finish after exiting extrusion spinnerets. The finish lubricates the fiber surface, reducing the friction coefficient.

Consumers are also affected by electrostatic charging in fabrics and nonwoven materials. Electrostatic buildup causes electric shocks in areas with low humidity, particularly in winter months. Electronic equipment, including computers, can be damaged by electrostatic discharge (Schindler and Hauser, 2004). The most dangerous problem associated with electrostatic buildup is fires at gas stations and dust explosions (Seyam et al, 2009, Glor, 2008). Gas pump fires and dust explosion lead to loss of personal belongings and human life. According to Pionteck and Wypych (2007), factors that lead to electrostatic ignition are:

1. When static electricity becomes a source of ignition. This occurs when the charge generation rate exceeds the dissipation.
2. When discharge of static electricity occurs in a flammable area.
3. When energy of static discharge exceeds the ignition energy of the flammable area.
4. When a flame generated enters into the surrounding atmosphere.

Based on significant problems associated with electrostatic discharge, research on antistatic finishes is needed.

2.3 Antistatic textiles

Antistatic finishes are used for a variety of synthetic textiles, including carpets, protective clothing, airbags, upholstery, and parachutes (Schindler and Hauser, 2004). Two main methods exist for application of antistatic finishes on polymeric materials. One method occurs when an antistatic finish is applied to the outer surface of a polymer. The other method incorporates an antistatic finish within the molten polymer matrix. Surface finishes are used when it is crucial to avoid surface charging. Meanwhile, antistatic chemistry is mixed into the polymer matrix when electrostatic charge needs to flow through the polymer (Pionteck and Wypych, 2007).

Most antistatic agents form an intermediate layer on the surface, which absorbs moisture. Increased surface moisture increases the conductivity of the material. However, problems arise with these finishes when relative humidity is low. Other antistatic finishes act as surfactants, where hydrophobic parts of the finish reduce charge build up. These finishes result in better ion mobility and dissipation. Examples of these finishes include cationic surfactants. In a cationic surfactant, hydrophilic parts point toward the air, promoting the absorption of moisture, as seen in **Figure 2.1**. These finishes create a hydrated layer on the surface of the textile (Schindler and Hauser, 2004). Antistatic finishes are grouped into three groups: durable finishes, non-durable finishes, and conductive finishes.

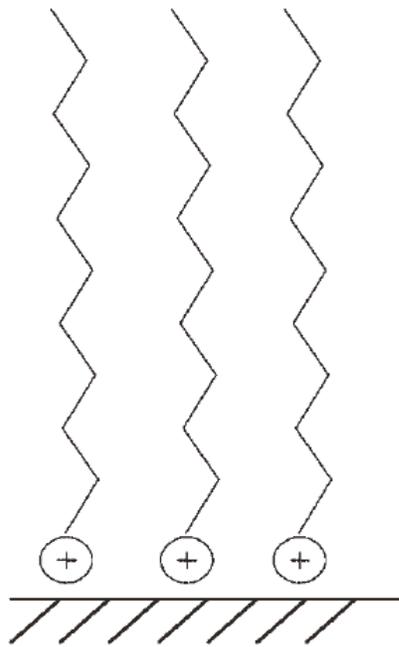


Figure 2.1 Cationic hydrophilic group (Schindler and Hauser, 2004)

2.3.1 Durable antistatic finishes

An antistatic finish, that can withstand multiple washings, is classified as a durable finish.

Antistatic finishes can be created prior to application or directly on the fabric surface.

Durable antistatic finishes are commonly synthesized by combining polyglycols and

polyamines as seen in **Figure 2.2**. These finishes are composed of crosslinked polymers that

contain hydrophilic groups. Increased numbers of hydrophilic groups improve antistatic

properties. As hydrophilic groups increase, softening increases, weakening the finish. This

leads to abrasion problems. Other variables in durable antistatic finishes can be manipulated,

including crosslinking. Increased crosslinking reduces moisture absorption, improving

durability. However, too much crosslinking decreases antistatic effectiveness.

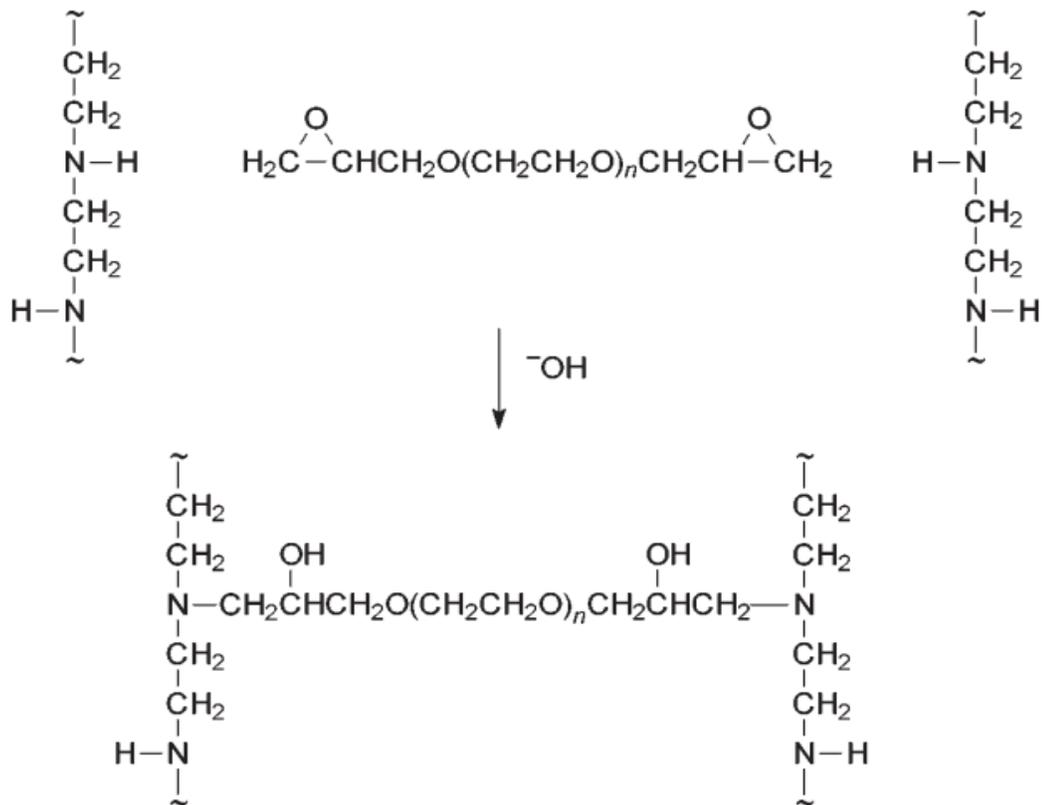


Figure 2.2 Crosslinking of polyamines to form a durable antistatic finish (Schindler and Hauser, 2004)

2.3.2 Non-durable antistatic finishes

Non-durable antistatic finishes are commonly used in manufacturing processes, where ease of removal is crucial. These finishes serve as a lubricant between fibers and machinery. Non-durable finishes must be heat resistant, non-yellowing, non-corrosive, and have low volatility. Common types of non-durable finishes include polyethylene glycol, polyethylene oxide, quaternary ammonium salts containing fatty alkyl chains, and esters of salts of alkylphosphonium acid. The largest group of non-durable antistatic finishes is esters of phosphoric acid. As molecular size increases, so does the durability of a phosphoric ester finish (Schindler and Hauser, 2004). **Figure 2.3** shows examples of phosphoric ester antistatic finishes.

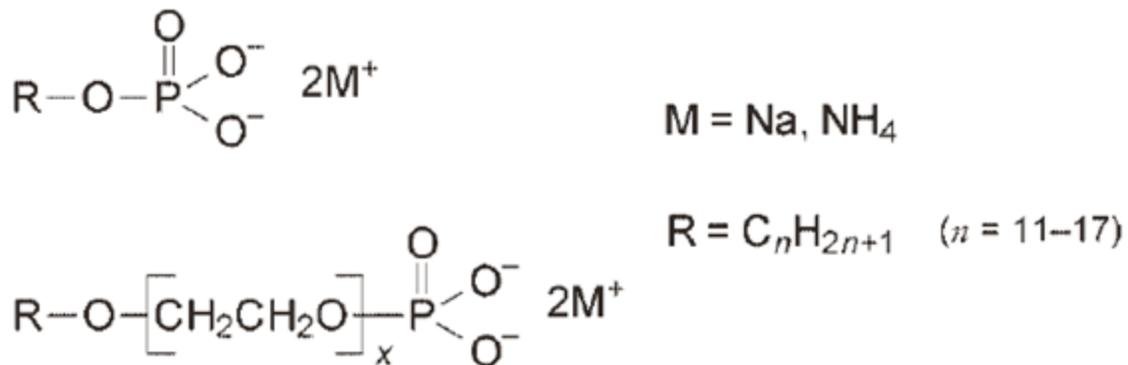


Figure 2.3 Chemical structure of a generic phosphoric ester antistatic finish (Schindler and Hauser, 2004)

Another common type of non-durable antistatic finishes is quaternary ammonium compounds. Quaternary ammonium compounds, such as ditallowdimethylammonium chloride and dehydrogenated tallowdimethylammonium chloride, are commonly used in household laundry softener (Schindler and Hauser, 2004). Quaternary ammonium compounds are depicted in **Figure 2.4**.

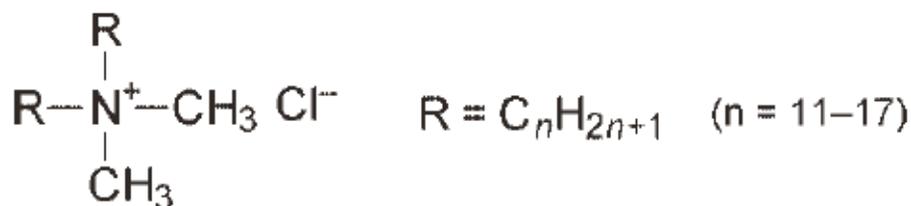


Figure 2.4 Chemical structure of generic quaternary ammonium antistatic finish (Schindler and Hauser, 2004)

Non-ionic antistatic finishes contain compounds such as ethoxylated fatty esters, alcohols, and alkyamines. When non-ionic compounds are mixed with surfactants, antistatic properties are increased. Non-ionic surfactants increase moisture absorption and cationic surfactants contribute mobile counterions (Schindler and Hauser, 2004). **Figure 2.5** illustrates a non-ionic antistatic compound.

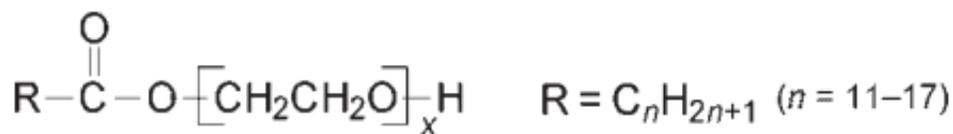


Figure 2.5 Chemical structure of a generic non-ionic antistatic finish (Schindler and Hauser, 2004)

2.3.3 Conductive modification of polymers

Conductive coatings obstruct and dissipate electrostatic charge quickly. The main challenge regarding conductive coatings is adhesion. Without adhesion, finish durability will be an issue (Pionteck and Wypych, 2007). Conductive coatings are applied to textiles by means of polymer melt additives, including carbon particles and other antistatic agents. Conductive coatings, such as nickel or copper, are used to coat fiber surfaces. A popular means of achieving conductive coatings is electroless deposition (Guo, 2009). Finally, cloth fabrication from metallic fibers has been used as an antistatic treatment. This technique affects the aesthetics of the final product, though metallic fibers can be used to create aesthetic appeal on textiles (Schindler and Hauser, 2004).

2.4 Use of plasma in textiles

2.4.1 Plasma chemistry

Plasma was discovered in the early 20th century by American chemist and physicist, Irving Langmuir. It is referred to as the 4th state of matter, after solids, liquids, and gases. When a significant voltage is applied across a gas filled space, gas breaks down and conducts electricity. This occurs when one electron is not attached to an atom or molecule, thus the atom or molecule has a positive charge. This is known as ionized gas. As temperature increases, molecules and atoms gain energy. Changes occur in the following sequence: solid, liquid, gas, and plasma. An ionized gas can be classified as plasma gas when it becomes electrically neutral (Fridman, 2008). Plasma is a suitable surface modification technique for polymeric materials because it doesn't alter bulk properties of the treated material (Costa et al, 2006).

Plasma has become popular across a number of disciplines due to several advantages:

1. Plasma can produce high concentrations of vigorous, chemically active species. This can include atoms, ions, electrons, and radicals. The temperature during this process can be as low as room temperature.
2. Temperatures of traditional chemical technologies can be exceeded by certain plasma components.
3. The plasma process is environmentally friendly and can eliminate wet processing in certain industries (Coburn, 1982; Zhu et al, 2006).
4. Use of plasma treatments can reduce the use of dye and chemicals used in subsequent stages of textile dyeing and finishing. (Shishoo, 2007).

Table 2.2 depicts the perceived advantages of plasma treatments, compared to traditional wet processing.

Table 2.2 Perceived advantages of plasma processing over conventional wet processing (Shishoo, 2007)

Manufacturing Operation	Conventional Wet/Heat Processing	Plasma Processing
Handling and storage of bulk chemicals	Yes (-)	No (+)
Mixing of chemicals, formulation of baths	Yes (-)	No (+)
Use of water	Heavy (-)	None or very low (+)
Raw materials consumption	High (-)	Low (+)
Drying ovens and curing operations	Yes (-)	No (+)
Need for solvents, surfactants, and acids	Yes (-)	No (+)
Number of processing steps	Multiple (-)	Single (+)
Energy consumption	High (-)	Very low (+)
Waste disposal/recycling needs	High (-)	Negligible (+)
Environmentally costly	Yes (-)	No (+)
Equipment footprint	Large (-)	Small (+)
Manufacturing versatility from single kit	Few process options (-)	Depending on the kit, can be highly flexible with wide range of available processes (+)
Innovation potential	Moderate (-)	Very high (+)

Despite its advantages, plasma treatments have several disadvantages:

1. Plasma treatments require high initial investment costs. Additionally, skilled labor is needed to run the process.
2. Machines currently on the market cannot be used with a wide variety of products.
3. Correct application of plasma requires a significant amount of knowledge in plasma chemistry (Shishoo, 2007).

2.4.2 Plasma systems in textiles

Both low-pressure plasma and atmospheric plasma can be used in textile applications. Low-pressure plasma is difficult to apply on a production scale, as it requires a vacuum, an extensive amount of energy, and can only be carried out in batch production. Thus, low-pressure plasma is not feasible for mass production of textile products. Contrarily, atmospheric plasma can be applied on an industrial scale and does not require a vacuum. Atmospheric plasma systems are usually between 1.5 and 10 meters in width. Atmospheric plasma systems can be used to etch the surface of textiles, increasing wettability. This can improve a number of properties associated with fabrics including improved interactions between textile materials and liquids, durability after treatment, adhesion of coatings, and absorption by capillary action (Shishoo, 2007).

2.4.3 Effect of plasma on textiles

Atmospheric plasma treatments change characteristics associated with synthetic fibers.

Plasma has been used to increase the surface area of textile fibers through a process known as etching. Etching is a process that increases the nano-roughness of a textile material and typically is a slow process. On average, etching removes several hundred nanometers of material (Shishoo, 2007). Gases such as nitrogen, oxygen, and argon, have etched textile fibers such as, acrylic, nylon nanofibers, polyester, and carbon nanofibers (Liu et al, 2006; Tao et al, 2008; Yuen et al, 2007; Zhu et al, 2006). Etched fibers differ from unetched fibers in a number of ways. Etching has been shown to improve cohesion in electroless nickel deposition on polyester fibers (Yuen et al, 2007). Research by Liu et al (2006), depicted by the scanning electron microscope photograph in **Figure 2.6**, proves that as treatment time increases, so does etching on acrylic fibers. Conditions used for nitrogen plasma pretreatment in research by Liu et al (2006) were a frequency of 13.56 megahertz, 0.2 Torr of pressure, and 300 watts of power. Samples were treated for 0, 1, 3, and 5 minutes in plasma.

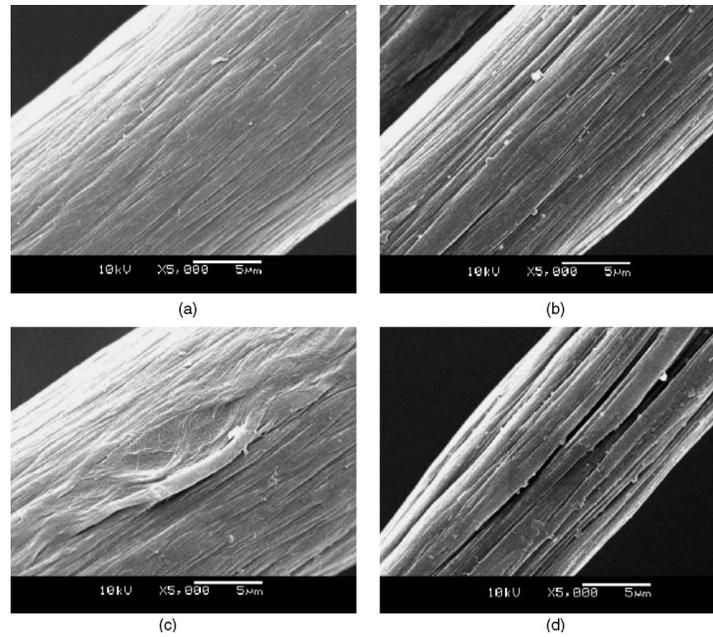


Figure 2.6 SEM images of untreated and plasma-treated acrylic samples: (a) untreated, (b) plasma for 1 minute, (c) plasma for 3 minutes, (d) plasma for 5 minutes (Liu et al, 2006)

Plasma treatments also increase hydrophilicity of textiles. Etching contributes to increased moisture adsorption and transportation, therefore wettability and antistatic properties are increased (Liu et al, 2006; Shishoo, 2007). Hydrophilicity is measured by water contact angle. Research by Höcker (2002) demonstrated that the contact angle could be decreased on polypropylene, using oxygen plasma treatment, from 90° to 55° . Increased hydrophilicity remained nearly constant 2 weeks later, with the contact angle increasing only marginally, to 60° (Höcker 2002). Similar results were seen with polyester, where the surface contact angle decreased from 80° - 40° (Leroux et al, 2006).

Additionally, plasma has been shown to increase adhesion between fibers and the accompanying matrix. Dyeing and printing are improved by plasma treatments. This occurs because of capillarity improvement, enhancement of surface area, lessening of external crystallinity, and creation of reactive sites (Shishoo, 2007).

2.4.4 Gases used in plasma treatments

Different gasses influence etching. Yuen et al (2007) compare the use of oxygen and argon gas on polyester fibers. Both oxygen and argon gas etch the surface of polyester. However, oxygen gas is more effective at etching than argon gas. Nitrogen gas has also been shown as a viable etching agent in plasma systems. Costa et al (2006) compared a variety of plasma gas treatments on polyester fabric to a control sample using a vertical drag test. Research by Costa et al (2006) investigated the differences between different concentrations of oxygen, hydrogen, nitrogen, and methane gas. This research concluded that gas mixtures made of predominately oxygen (90% oxygen, 9% nitrogen, 1% hydrogen) and nitrogen (92% nitrogen and 8% hydrogen) are the most efficient at etching a 100% polyester substrate. Zhu et al (2006) and Tao, et al (2008) both confirm that nitrogen and oxygen are efficient at etching the surface of carbon nanofibers and nylon nanofibers, respectively. Nitrogen, hydrogen, oxygen, argon, and methane gases were used at varying concentrations in the plasma treatment. Based on the vertical drag test, plasma treatments increase the wettability of polyester fabric as shown in **Figure 2.7** (Costa et al, 2006).

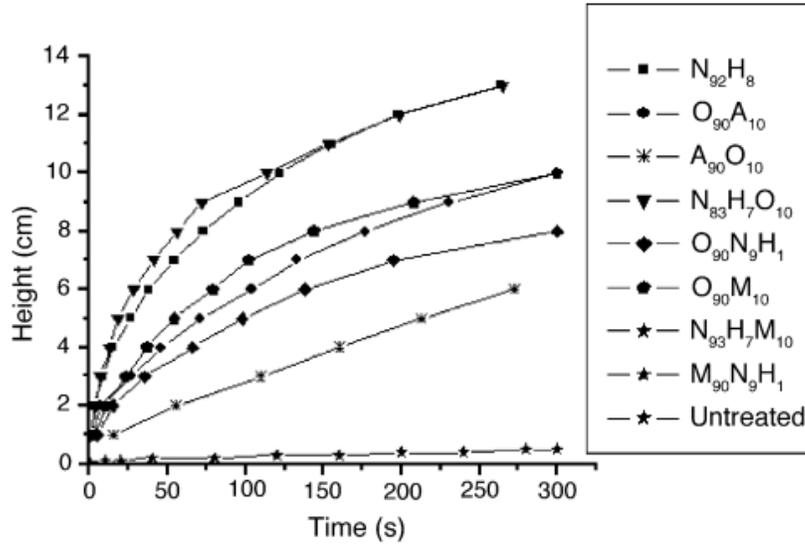


Figure 2.7 Plot of height of vertical advance vs. time for plasma treated fabrics
N=Nitrogen, H=Hydrogen, O=Oxygen, A=Argon, M=Methane
(Costa et al, 2006)

2.5 Electroless deposition process

Electroless deposition coats non-metallic surfaces with metal without using an electrical current (Cohen et al, 1977). This is not to be confused with electroplating, where an electrical current reduces cations of a material from a solution to coat an object with metal (Abu-Isa, 1973). Electroless deposition is preferred over other methods of producing metal-coated textiles, including chemical vapor deposition, electrochemical deposition, and heat spray (Guo et al, 2009; Zhu et al, 2006). Electroless deposition produces a low cost, continuous, uniform coating of metal. Additionally, electroless deposition can be applied at any step in the production process, including yarn, stock, fabric, or clothing (Guo et al, 2009).

Electroless deposition is a technique used to impart antistatic, electromagnetic shielding, ultra-violet properties, and electrical conductivity properties to textile surfaces (Tao, et al 2008; Stefecka et al, 2004; Kan and Yuen, 2007; and Zhu et al, 2006). These fabrics can be used in a variety of products including biomedical, battery electrode substrates, cable

manufacturing, and fabric conductors (Šimor et al, 2003). Electroless deposition is an extensively used process due to its easily controlled thickness, low cost, and low operating temperature. The process requires three steps to achieve desired qualities: pretreatment, sensitization/activation, and plating. Pretreatment can be achieved by using an acid or plasma treatment to etch a fibers surface (Zhu et al, 2006). Sensitization and activation refer to processes used to make the fiber surface catalytic. The final process, plating, refers to the process of reducing metal ions to metal particles on the textile surface (Cohen et al, 1977). A commonly used electroless plating process can be summarized by the **Figure 2.8**.

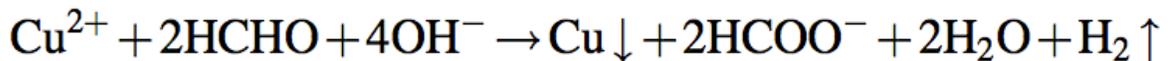


Figure 2.8 Commonly used electroless plating equation (Guo et al, 2009)

2.5.1 Pretreatment

Prior to sensitization and activation, polymers must be activated by acid etching or plasma treatment (Stefecka et al, 2004). Traditionally, pretreatment has been achieved by acid etching. However, recently problems stemming from neutralization of acid effluent and contamination of the environment have initiated research on electroless deposition pretreatment options. The primary method investigated has involved the use of plasma (Zhu et al, 2006).

2.5.1.1 Acid Pretreatment

Acid etching provides excellent adhesion of metal to substrate materials due to several factors, including micro-roughening and chemical modification through oxidation (Zhu et al, 2006). This step is required in order to change the smooth hydrophobic surface of polymers

to a rough, hydrophilic surface. Roughening of the surface is required in order to ensure sufficient bonding and uniform deposition of metal (Abu-Isa, 1973). However, acid etching has been questioned as environmental issues have become increasingly more important. Furthermore, problems with acid pretreatments include environmental contamination stemming from effluent. Often, acid pretreatments involve rinsing before and after treatment. These steps are needed to clean the fiber before to pretreatment and neutralize acid after pretreatment (Han et al, 2001). This, in combination with the pretreatment process, yields excessive amounts of effluent. Traditionally acids used in acid plasma pretreatment have included sulfuric acid and chromic acid (Zhu et al, 2006). Concentrations of acid solutions used in etching typically vary depending on the polymer being treated (Abu-Isa, 1973). As restrictions have been tightened on effluent release and the environment, other alternative pretreatment techniques have been examined, such as plasma (Zhu et al, 2006).

2.5.1.2 Plasma pretreatment

Plasma pretreatment is a novel technique that is being examined to replace acid pretreatment. Plasma treatments are an environmentally friendly alternative, which use less energy and consume no water. Thus, plasma pretreatment is a “greener” alternative to acid pretreatment (Zhu et al, 2006). Two types of plasma systems are currently used: vacuum and atmospheric. Vacuum systems can only be used for batch production and use extensive amounts of energy. Thus, this isn't a viable option for mass production of textiles. The type of plasma system is known as an atmospheric pressure system. This system can be used for mass production of textiles, as it doesn't require a vacuum (Shishoo, 2007). Pretreatment, using plasma is used to achieve hydrophilic properties by etching. Plasma pretreatment improves hydrophilic properties and adhesion during electroless deposition (Zhu et al, 2006). Activator sites are an

important factor influencing the electroless deposition process. Typically, more activator sites are found on substrates that are etched and have hydrophilic properties. It is important to note, the density of activator sites varies, depending on the type of substrate. (Abu Isa, 1973). In addition, plasma pretreatment increases hydrophilic and absorption properties, while leaving bulk properties unaltered (Kan and Yuen, 2007).

Due to interest in plasma pretreatment as a replacement for acid pretreatment, several studies have been conducted. Research has focused on a number of substrates including polypropylene nonwovens and carbon and polyamide nanofibers (Stefecka et al 2004; Zhu et al, 2006; Tao et al, 2008; Tao et al, 2010). These studies have focused on a variety of end uses, including electromagnetic shielding and ultra-violet protection (Han et al, 2001; Stefecka et al, 2004; Kan and Yuen, 2007). The viability of antistatic finishes imparted through plasma pretreatment has yet to be investigated. **Figure 2.9** depicts an electroless deposition process. Steps colored grey can be eliminated if plasma pretreatments are used in lieu of acid pretreatments. Grey colored steps use large quantities of water.

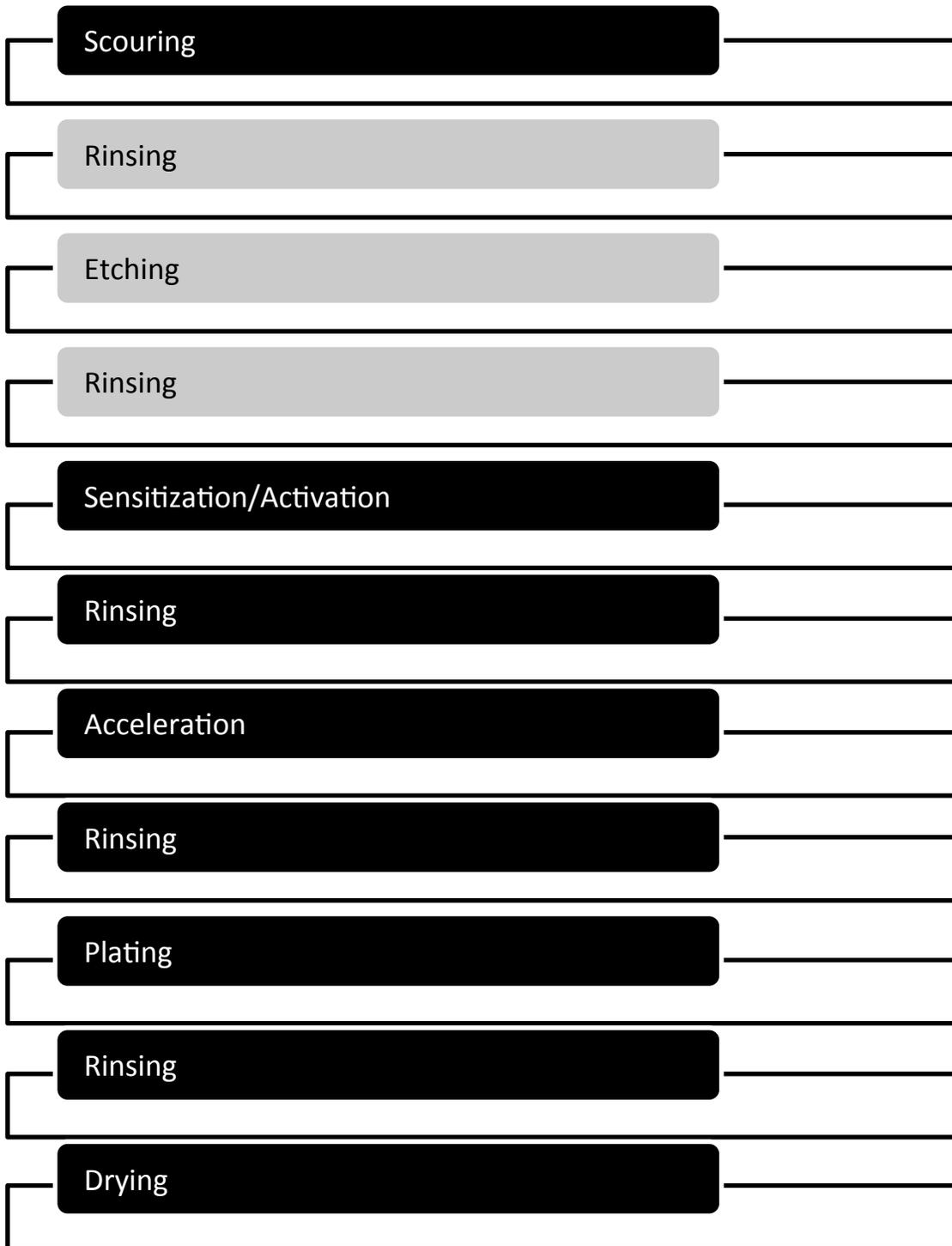
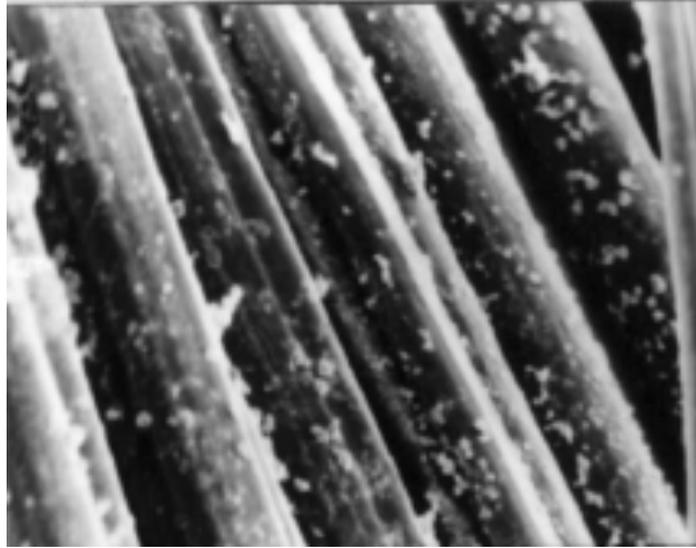


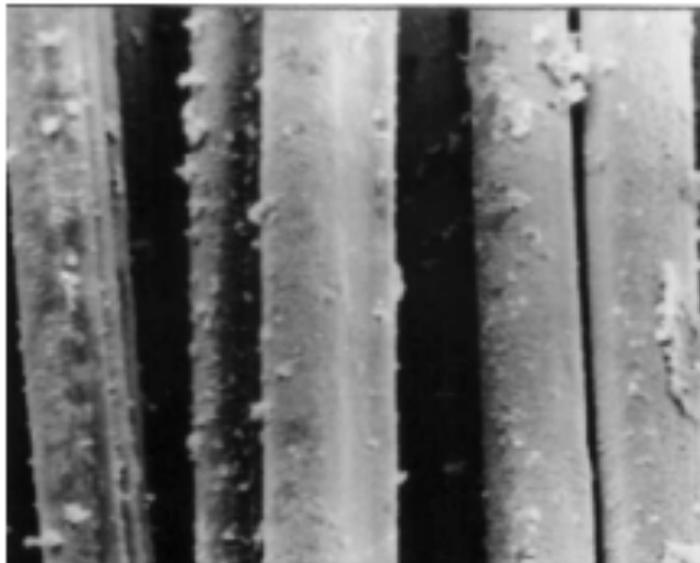
Figure 2.9 Standard electroless deposition process with acid pretreatment steps highlighted in grey (Adapted from Han et al, 2001)

2.5.1.3 Effect of plasma pretreatment parameters on electroless deposition

Zhu et al (2006), Stefecka (2004), and Tao et al (2010) researched plasma pretreatment parameters, including discharge power, pressure, time, and gas type. After plasma pretreatment, electroless deposition was completed. Percent uptake, resistivity, and adhesion values were measured. Work by Zhu et al (2006) studied the effect of discharge power, pressure, time, and gas type on the percent copper uptake by PAN based carbon fibers. It was concluded that the ideal combination of variables to maximize copper uptake was 25 watts of power, 40 pascals of pressure, and 10 minutes of exposure to air plasma. Zhu et al (2006) determined that power and time have more influence on percent copper uptake than discharge pressure and type of gas. Copper uptake for fibers without plasma pretreatment was 18.65%. Meanwhile, under ideal plasma conditions, copper uptake was 239%. These results can be attributed to plasma pretreatment incorporating increased polar oxygen groups and etching. Carbon nanofibers without plasma pretreatment had large, separate copper grains on their surface. This is in contrast to air plasma treated samples, which were covered in finer copper grains. Etching with air plasma produced a rougher surface allowing carbon nanofibers to hold more silver catalyst during the activation stage. Increased silver catalyst deposited on the fiber surface during activation, allowed for the formation of finer grains of copper during the subsequent plating stage. The differences in crystal size and uniformity, between carbon nanofibers without plasma pretreatment and with pretreatment, are shown in **Figures 2.10 and 2.11.**



**Figure 2.10 Copper coated carbon nanofibers without plasma pretreatment (18.65% copper uptake)
(Zhu et al, 2006)**



**Figure 2.11 Copper coated carbon nanofibers with plasma treatment (239% copper uptake)
(Zhu et al, 2006)**

Stefecka et al (2004) demonstrate that spunbonded polypropylene nonwovens treated with nitrogen atmospheric plasma exhibit increased copper adhesion, after electroless deposition, compared to those with no pretreatment. Polypropylene nonwoven samples were exposed to nitrogen plasma for 2 seconds, on both sides of the fabric. The estimated depth of plating on samples was 1 μm . After electroless deposition, a standard industrial test, known as the scotch tape test was performed. In samples without plasma pretreatment, copper showed little adhesion to the substrate. Meanwhile, copper on plasma pretreated samples showed excellent adhesion. **Figure 2.12** depicts results of the scotch tape test. Dark areas represent copper peeled off the fabric (Stefecka et al, 2004).

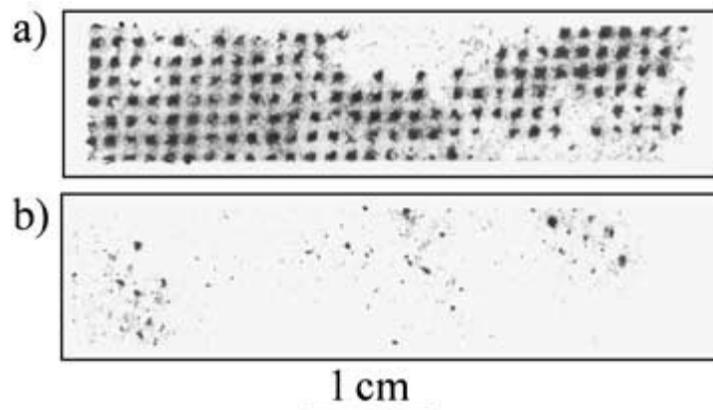


Figure 2.12 Scotch tape tests on copper coated spunbonded polypropylene nonwoven with a) No pretreatment b) 2-second nitrogen plasma pretreatment (Stefecka et al, 2004)

Experiments performed on polypropylene melt-blown nonwovens by Tao et al (2010) examined varying plasma treatment power, before subsequent electroless deposition steps were conducted. Tao et al (2010) determined that increasing plasma power (watts), improved the adhesion strength of copper deposited in electroless deposition. Adhesion was tested using a LRXPlus material mechanical testing machine by Lloyd Instruments. The test method used utilized a 2.54 cm wide x 7 cm long piece of tape, attached to a 400 gram weight, for duration of 60 minutes. **Figure 2.13** depicts the effect of plasma power (watts) on adhesion strength. It is apparent that as plasma power (watts) increases, so does adhesion strength (newtons). The largest increase in adhesion strength (newtons) was observed as plasma power (watts) increased from 50w to 60w. Increased adhesion can be attributed to more surface roughening and adhesion sites. Surface roughening and more adhesion sites are linked to increased plasma power (watts). Additionally, oxygen plasma introduces polar groups to the fiber surface creating van der Waals bonds. Therefore, increased adhesion of copper to the surface of the polypropylene nonwoven material is observed (Tao et al, 2010).

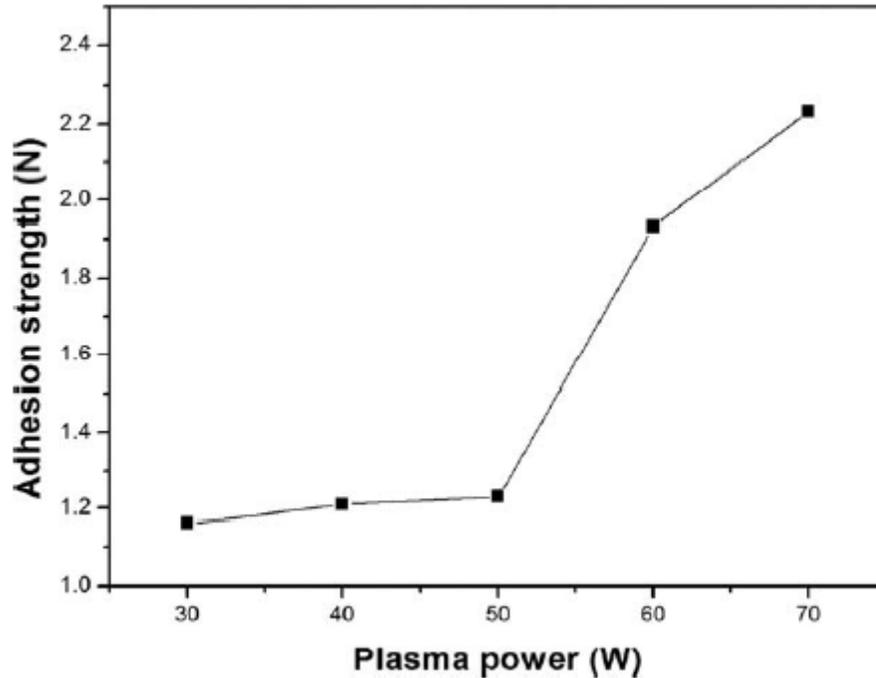


Figure 2.13 Effect of plasma power (watts) on adhesion strength after electroless deposition of copper (Tao et al, 2010)

2.5.2 Sensitization and activation

Sensitization and activation are processes that make the substrate catalytic for electroless deposition. First, the substrate is subjected to the sensitizer, followed by activation. This two step process yields faster electroless plating, and less loss of palladium and tin in the electroless copper bath (Cohen et al, 1977). The main chemical used in sensitization is SnCl_2 (Cohen et al, 1977; Abu Isa, 1973). Hydrochloric acid is used at 10 -60 mL/L (Cohen et al, 1977). Electron microscopy techniques have been used to study the size and distribution of tin particles. Typically, particles of tin have been approximately 30 Å in diameter. Some tin aggregates have been observed forming clumps ranging from 100-250 Å in diameter (Abu Isa, 1973).

In activation, PdCl₂ is prevalently used. However, other noble metals including gold, platinum, and silver can be used. Hydrochloric acid is used at concentrations ranging from 0.02 mL/L-20mL/L. Activation is carried out at room temperature, with bath exposure ranging from 30 seconds to 1 minute. Higher temperatures have been shown to be more efficient, when dilute solutions are utilized. The substrate should be rinsed with deionized water, after both sensitization and activation, to eliminate contamination between baths (Cohen et al, 1977).

Studies have been conducted to streamline the electroless deposition process. Stefecka et al (2004) combined sensitization and activation in one bath, using 50-200 mg/L of PdCl₂, 5-20 g/L of SnCl₂, and 100-200 mL/L of 37% HCl at 25-30°C for 1-3 minutes. Research by Guo et al (2009) suggests that AgNO₃ can be used, instead of PdCl₂, to produce a lower cost fabric, without compromising properties of copper plating.

2.5.3 Plating

The most commonly metals deposited in electroless plating are nickel and copper. A typical electroless plating solution consists of a metal ion and a reducing agent used to reduce the metal ion to metal (Abu-Isa, 1973). Typically, chemistry used in plating bath is made of a copper salt such as CuSO₄, a complexing agent such as ETDA or sodium potassium tartrate, and a reducing agent such as formaldehyde. Additionally, an alkaline metal hydroxide must be used to raise the pH to 11. Reduction of the metal doesn't occur a catalyst is deposited on the fiber surface. This occurs during sensitization and activation. Catalysts used in electroless deposition are noble metals such as gold, platinum, palladium, or silver. Conditions such as pH, temperature, and complexing agents must be controlled throughout the process (Cohen et al, 1977).

Electron microscopy and X-ray analysis techniques have been used to analyze the copper electroless deposition process. These techniques yielded results depicting copper nuclei initially 25Å in diameter, forming aggregates approximately 300Å wide. As the deposition process continues, aggregate size increases until it becomes actively unstable and recrystallizes. The process produces a crystalline heterogeneous nanolayer of copper (Abu-Isa, 1973). Plating occurs as long as the substrate is submerged in the plating. Based on this concept, it is possible to plate an unlimited amount of metal on the substrate surface, if chemicals are replaced throughout the process (Kan and Yuen, 2007). Stefecka (2004) quantified the rate of plating as 1 µm/20 min. The thickness of metal plating is controlled by the amount of activator sites on the surface of the polymer. Activator sites are produced through pretreatment etching (Abu Isa, 1973).

2.5.3.1 Factors affecting the rate of deposition during plating

Gan et al (2008) and Jiang et al (2008) discuss factors influencing the rate of deposition during electroless plating of nickel. Factors discussed include bath composition, pH, and temperature. When the concentration of nickel ions in the bath was low, deposition of nickel plating decreased, before completely stopping because of a lack of catalytic replenishment. For this reason, concentration of metal ions must remain above a critical point. **Figure 2.14** depicts the effect of nickel ion concentrations and different temperatures on the deposition rate. Deposition rates increase as the nickel ion concentration and bath temperature increases. At concentrations below 0.0030 M, only a thin layer of plating was achieved. Hence, 0.0030 M is the critical point of nickel ions in the plating bath. Also, as temperature increased, more deposition of nickel plating occurred. Higher temperatures, combined with increased levels of nickel ions, yielded the highest deposition rate (Gan et al, 2008).

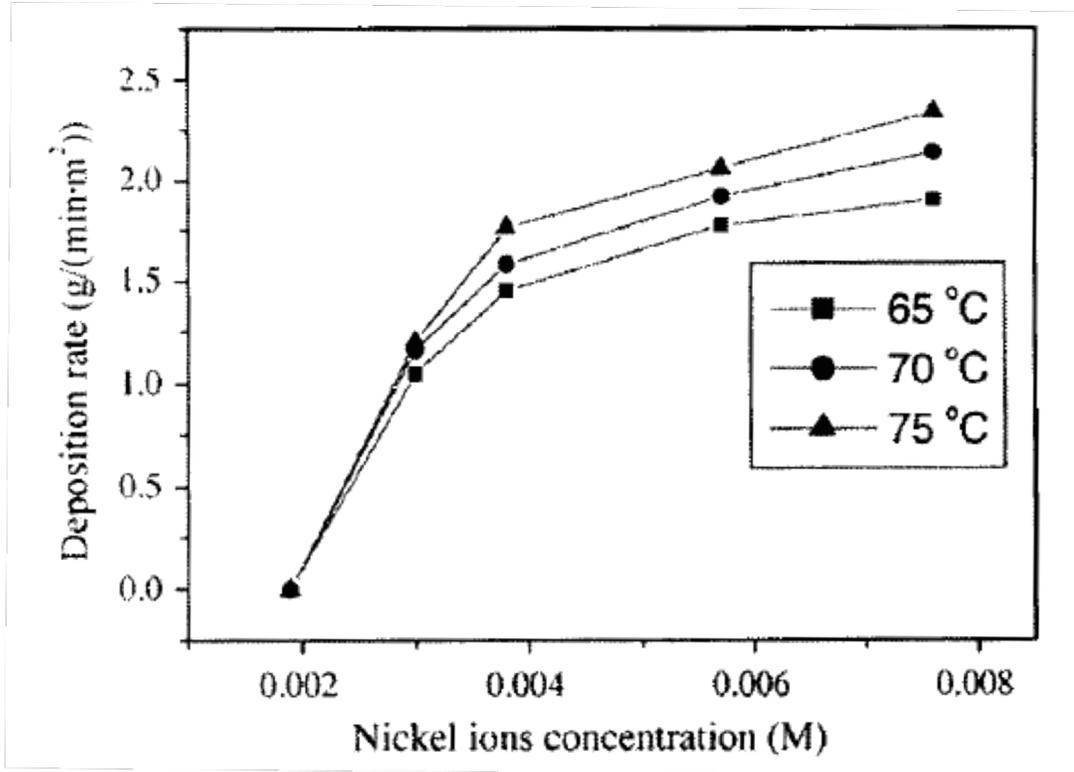


Figure 2.14 The effect of nickel ions concentration on the deposition rate at different temperatures (pH =10.0) (Gan et al, 2008)

A correlation was established between surface resistivity and CuSO_4 concentration by Tao et al (2008) on polyamide 6 nanofibers. Polyamide 6 nanofibers were pretreated with plasma for 90 seconds, under 90 watts of power, and 30 pa of pressure. As the concentration of CuSO_4 increased from 10 g/L to 25 g/L, the surface resistivity decreased from 831.35 Ω/sq to 24.41 Ω/sq . The decrease in surface resistivity is linked to an increase in copper deposited on the fiber surface (Tao et al, 2008). This relationship is shown in **Figure 2.15**. Other research by Tao et al (2010) confirmed a similar relationship between surface resistance (Ω/sq) and CuSO_4 (g/L) on polypropylene nonwovens.

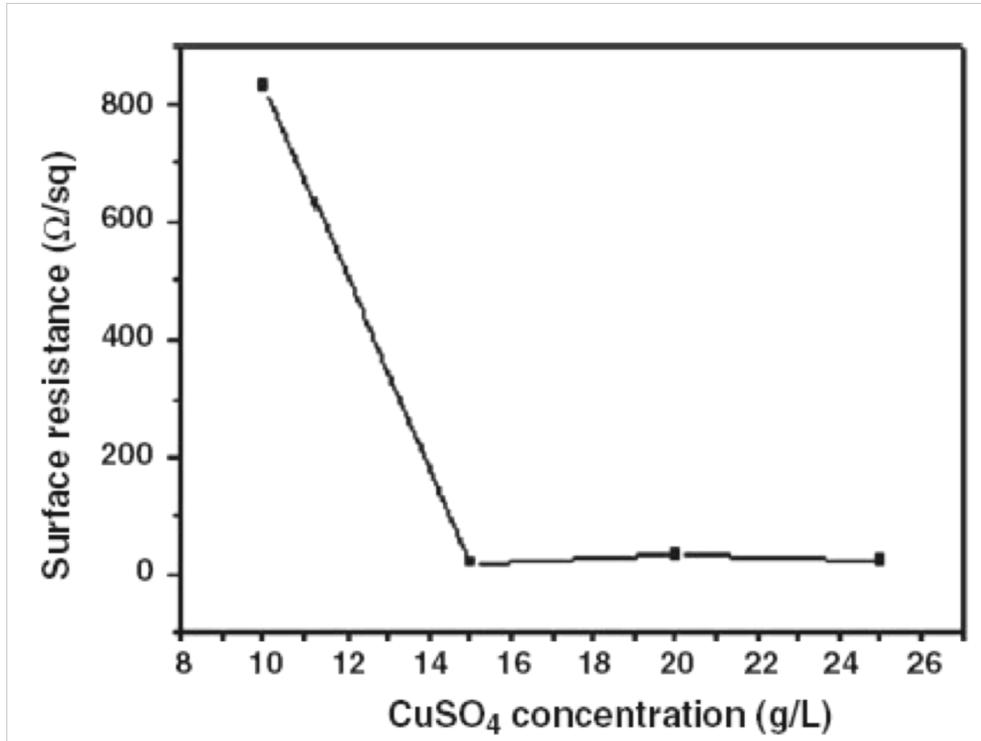


Figure 2.15 Effect of CuSO₄ concentration in the treatment bath on surface resistivity (Ω/sq) (Tao et al, 2008)

pH has an impact on the rate of deposition during electroless plating as shown in **Figure 2.16**. At 60°C pH had little influence on the deposition rate. However, the opposite was seen at 65°C, 70°C, and 75°C. At these temperatures, higher pH values led to increased deposition rates. The highest deposition rate was observed using a 10.5 pH at 75°C (Gan et al, 2008).

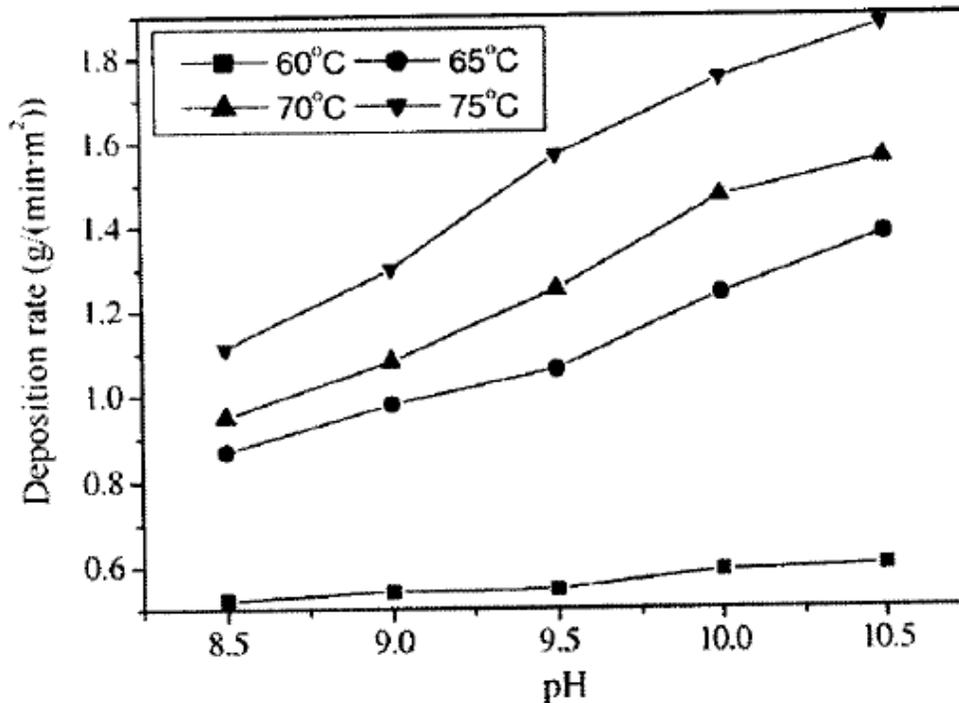


Figure 2.16 The effect of pH on the deposition rate at different temperatures (Nickel ions=0.0038 M) (Gan et al, 2008)

Jiang et al (2008) also studied the relationship between pH and weight gain (%) using electroless nickel plating on woven polyester fabrics. Results were similar to those gathered by Gan et al (2008). While Gan et al (2008) examined the deposition rate (g/(min·m²)); Jiang et al (2008) examined the weight gain (%). Higher deposition rates (g/(min·m²)) yield higher weight gain (%). Jiang et al (2008) determined that the highest weight gain (35%) was observed at a pH of 10 as shown in **Figure 2.17**. Thus, both studies confirm that higher pH levels in a plating bath increase the amount nickel being deposited on the fabric surface (Gan et al, 2008; Jiang et al, 2008). These results are similar to the optimal pH of 10.5 observed in the study performed by Gan et al (2008).

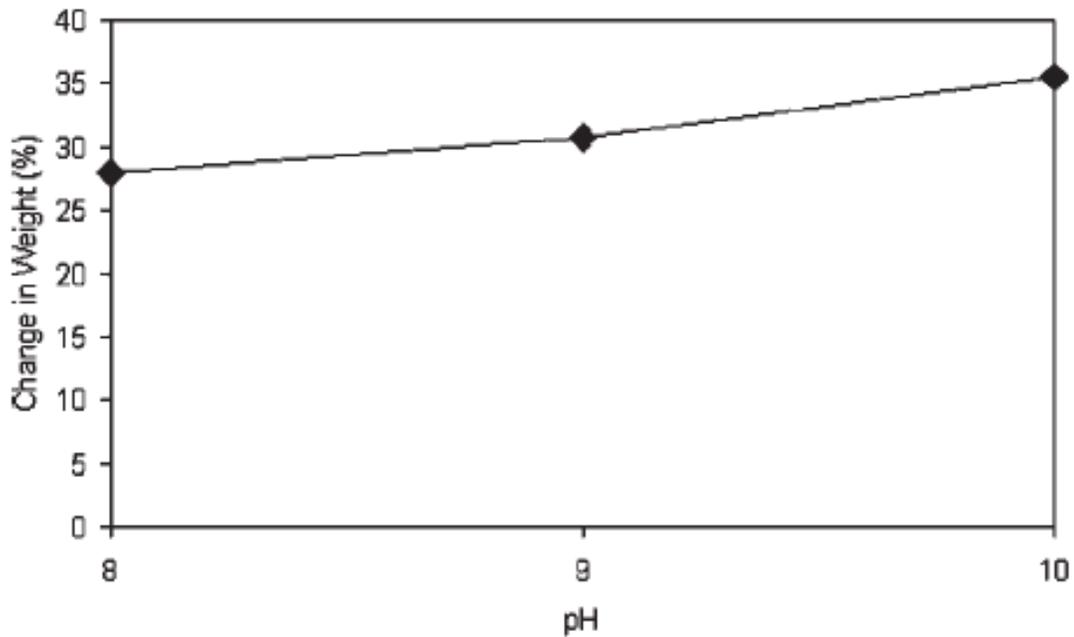


Figure 2.17 Effect of different pH levels during the electroless deposition plating process on weight gain (%) (Jiang et al 2008)

Jiang et al (2008) also showed that higher temperature levels increase the percent weight gain as depicted in **Figure 2.18**. There is a considerable increase in percent weight change when the plating bath temperature increased from 25°C to 40°C. However, when the plating bath temperature increased from 40°C to 55°C there was a negligible percent weight change (Jiang et al, 2008).

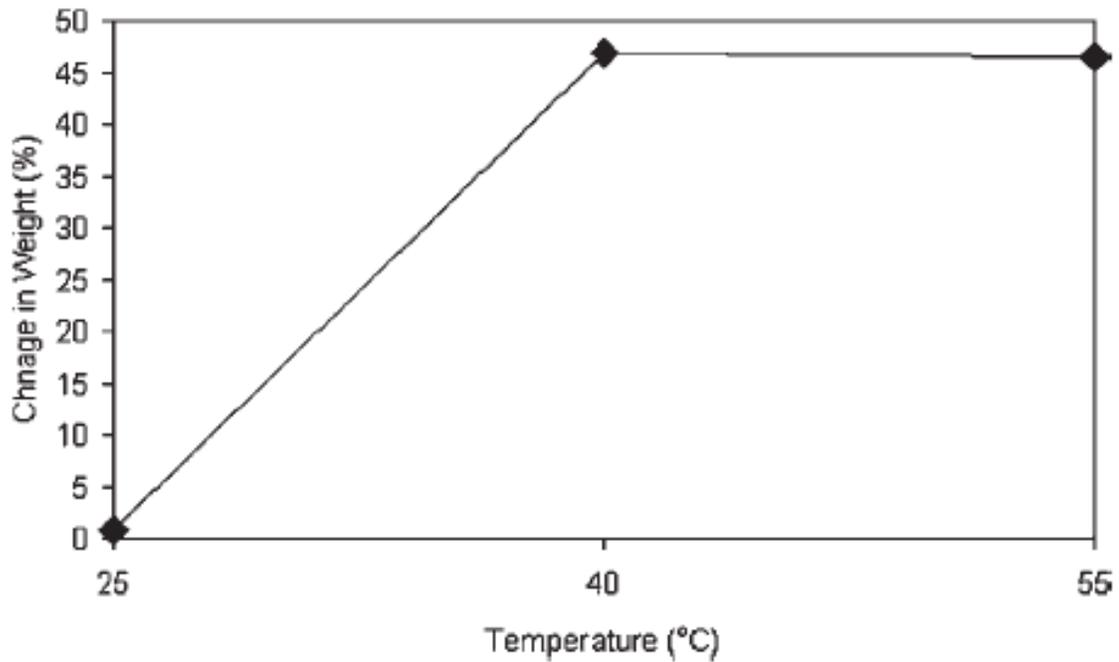


Figure 2.18 Effect of different electroless plating temperatures on the percent weight change (Jiang et al, 2008)

Jiang et al (2008) examined the effect of time on the change in weight. This variable was not studied in the experiment performed by Gan et al (2008). It was determined that the effect of time on percent change in weight isn't significant as shown in **Figure 2.19**. Jiang et al (2008) discovered the optimal treatment time in electroless plating was 20 minutes.

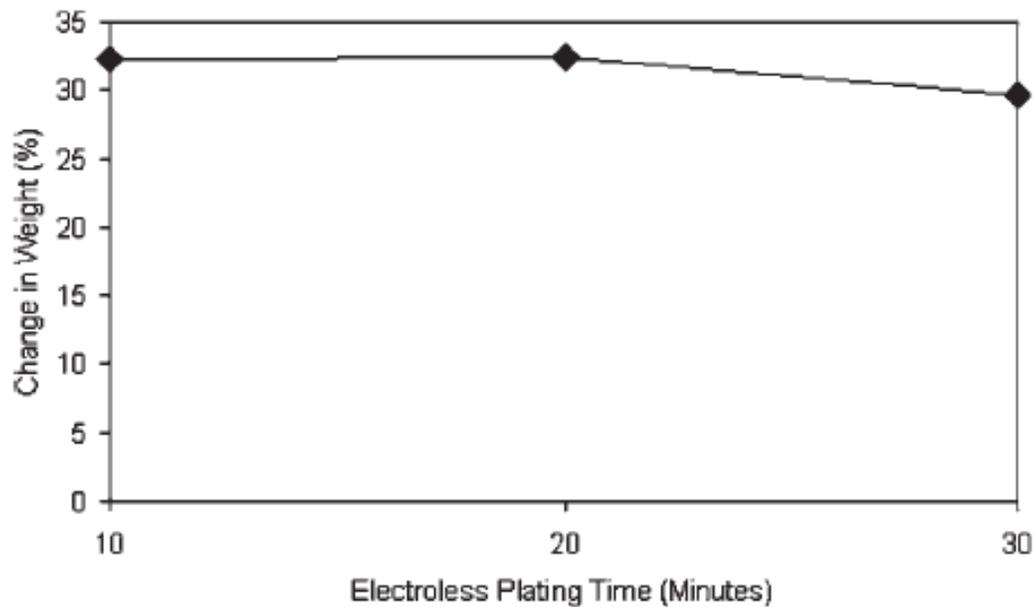


Figure 2.19 Effect of different electroless plating times on percent weight change (Jiang et al, 2008)

2.5.4 Impact of electroless deposition on fabric characteristics

Kan and Yuen (2007) studied a number of fabric characteristics that are altered during a plasma aided electroless deposition process, using nickel plating on 100% polyester fabric. These include weight change, thickness change, tensile strength, color fastness to crocking, colorfastness to light, color fastness to laundering, and water-repellency. Overall, plasma pretreatment, combined with electroless deposition, increased fabric weight, fabric thickness, tensile strength, and color fastness to crocking. Properties such as color fastness to light and color fastness to laundering weren't affected. Untreated and plasma pretreated samples, with electroless deposition, were more water repellent (Kan and Yuen, 2007). This is in contrast to hydrophilic properties observed on plasma pretreated samples before electroless deposition (Costa et al, 2006).

2.5.5 Industrial equipment design for electroless deposition

It is important to be able to produce an electroless deposition process on an industrial scale.

This is critical to ensure technology will be successful commercially. Industrial scale electroless deposition systems have been created and used for industrial applications including: printed circuit boards, hard memory disks, and electroplated plastics. In these industries, a typical electroless deposition manufacturing line consists of a storage tank and a heating system (Mallory and Hajdu, 1991). The number of storage tanks depends on how the process is broken up. Commonly, etching, deposition, and rinsing are separated into different tanks. Goods being produced can be submerged in a full immersion bath or a spray immersion bath. In a full immersion bath, the product is completely submerged in the tank. A spray immersion system consists of a dispersing apparatus that showers the product until the tank is full (Shacham-Diamand et al, 1996).

The construction of electroless deposition storage tanks varies. Tanks used in industrial applications have been made of polypropylene, chlorinated polyvinyl chloride (CPVC), stainless steel, or coated materials. Advantages and disadvantages are present, depending on the construction of the tank. Polypropylene tanks are most widely used in industry because of the low cost and versatility of tank constructions. However, these tanks have a finite lifespan, as metal builds up on the inside of the tank. CPVC tanks are occasionally used in industry. These tanks are usually not recommended as they are costly and are hard to fabricate and weld together. Stainless steel tanks have an infinite lifespan, but become expensive when tank stripping and clean up is factored in. Teflon and ceramics are used to create coated tanks. These tanks are practical for short-term use, but are easily damaged over time.

Heating systems viable for an electroless deposition system include steam, electric immersion, panel coils, and Teflon coils. The heating system used depends on the product and manufacturing line setup. Electric immersion heating systems have a low installation cost, but can create a fire hazard if solution levels drop too low. Steam heating is commonly used, however a high installation cost associated with this system. A steam based heating system saves money over the long run, as running costs are inexpensive. Panel coils are inexpensive to install. However, panel coils cause hot spots between the coil and tank wall and must be stripped often. Teflon coated coils are inert to plating solution, thus do not require cleaning. Despite this advantage, Teflon coils can be ruined if nicked. The proper heating system must be picked based on the product being made and company resources available (Mallory and Hajdu, 1991).

2.6 Methods for testing antistatic behavior

Several methods exist to test antistatic behavior of textile materials. These methods can be classified in three groups: direct methods, indirect methods, and simulation. In direct measurement methods, charge is imparted on the textile material by a standard technique and then measured (Holme et al, 1998). Measurements derived from a direct system include electrical field (E), potential (V), charge amount (Q), or electrostatic discharge rate (Gayler et al, 1965). Conversely, in indirect measurement methods a property that is linked to electrostatic charge is measured. Measurements include electrical resistance or conductance of the textile material. Finally, in simulation systems, a test method is used to charge the textile. Thereafter, a test is run simulating the desired end use of the product. In all systems mentioned, environment control is of utmost importance. Samples must be conditioned in

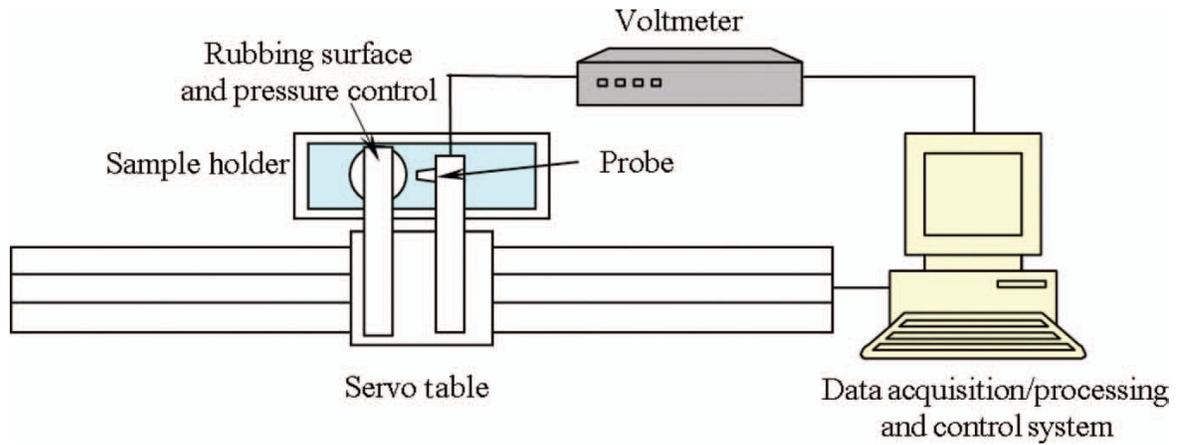
the environment they are being tested in and relative humidity in the room must be carefully controlled (Holme et al, 1998).

2.6.1 Static dissipation methods for measuring electrostatic behavior

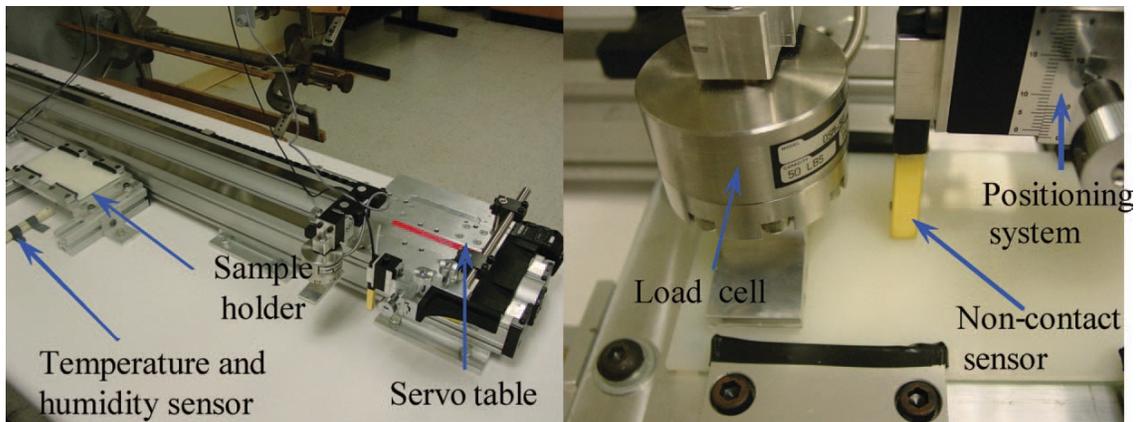
A common direct measurement system that assesses electrostatic behavior is static dissipation. Static dissipation is important to consider, as resistance measurements are only accurate when materials are completely homogeneous and have linear current/voltage characteristics (Chubb, 2004). Many materials are not homogenous, as they contain conducting fibers or matrices. Standard static dissipation methods involve charging a material by means of corona charging or direct current charging. An example of a direct current charging system is the triboelectric system (Holme et al, 1998; Chubb, 1990). Several testing systems have been developed, based on the concept of static dissipation. Systems include the National Aeronautics and Space Administration (NASA) Triboelectric Tester, Shirley Method, John Chubb Instrumentation (JCI) Tribo-charging Tester, Linear Tester, and Rubbing Tester (Suh, 2008).

2.6.1.1 Rubbing tester system

A device for measuring electrostatic generation and dissipation, known as the rubbing tester system, was developed by Seyam et al (2009) at North Carolina State University. The rubbing tester system proves especially useful when studying surface modification on different textile substrate surfaces. This device is used to investigate electrostatic generation and dissipation between two flat polymer surfaces, which rub together. The tester measures charge potential on a stationary fabric. The rubbing device has a variable rubbing speed, pressure, and number of cycles. **Figure 2.20** shows a schematic of the system, while **Figure 2.21** shows the main parts of the rubbing tester system.



**Figure 2.20 Schematic of a rubbing tester system
(Seyam et al, 2009)**



**Figure 2.21 Main components of the rubbing tester system
(Seyam et al, 2009)**

2.6.1.2 Federal Test Method 4046

Federal Test Method (FTM) 4046 is an example of a test method used to measure electrostatic decay. In order to use this test method, the material being tested must be homogeneous and free from holes or tears. The size of the sample used in testing is 5x3 inches. The sample was mounted between electrodes connected to a fieldmeter. The sample is then charged to 5000 volts and the time taken for complete electrostatic decay is measured. This process should be repeated three times. The testing area must be 73 +/- 3.5°F and 15% relative humidity (FTM 4046, 1980).

2.6.2 Surface resistance and resistivity measurements on textile substrates

Electrical resistance and resistivity are the most popular forms of indirect measurement of electrostatic evaluation in textiles. The American Association of Textile Chemists and Colorists (AATCC) defines resistance (R) as the physical property of a material which is a measurement of the ability of electrons to flow when voltage is applied across two points on the textile substrate (AATCC Test Method 84, 2000). Resistivity (ρ) is defined as the material property equal to the ratio of the voltage gradient to the current density. More simply, known as the degree to which the textile substrate opposes the flow of current. Surface resistivity, measured by a parallel electrode, is the measured resistance multiplied by the ratio of the width of the specimen to the distance between parallel electrodes. For this set-up 2 sets of 3 samples are needed. In one set, the direction of testing is parallel to the yarns of the length direction of the fabric. Meanwhile, the direction of testing of the other set is parallel to the width direction of the fabric. This can be calculated as shown in **Equation 2.1**.

$$R = O * W / D$$

Equation 2.1 Surface resistivity for a parallel electrode

Where variables are defined as:

- R=resistivity in ohms per square
- O=Measured resistance in ohms
- W=Width of the specimen
- D=Distance between electrodes

(AATCC Test Method 76, 2005)

2.6.2.1 AATCC Test Method 76

Surface resistivity can also be measured with concentric ring electrodes. This type of set-up measures both fabric length and width directions simultaneously. Only one set of three samples is required for concentric ring electrode testing. Results can be calculated as shown in **Equation 2.2**.

$$R = \frac{2.73(O)}{\log \frac{r_0}{r_i}}$$

Equation 2.2 Surface resistivity for a concentric ring case

Where variables are defined as:

- R=resistivity in ohms per square
- O=measured resistance in ohms
- r_0 =outer electrodes radius
- r_i =inner electrodes radius

(AATCC Test Method 76, 2005)

2.6.2.2 Meaning of resistivity values

A textile product must meet certain antistatic values for it to be safe for its intended end use.

For example a value of 10^{11} ohm/square indicates the fabric has negligible static propensity.

Associations, such as the National Fire Protection Association (NFPA), have certain set

resistivity values. NFPA requires textiles to have a resistivity of 10^{11} ohm/square for

applications in operating rooms with flammable anesthetic gases. **Table 2.3** shows standard

surface resistivity values of antistatic textiles with regard to static propensity at 65% relative

humidity (Schindler & Hauser, 2004)

**Table 2.3 Ratings for surface resistivity of antistatic finished textiles
(Schindler & Hauser, 2004)**

Surface resistivity range (Ω)	Assessment
$1 \times 10^6 - 1 \times 10^8$	Very good
$1 \times 10^8 - 1 \times 10^9$	Good
$1 \times 10^9 - 1 \times 10^{10}$	Satisfactory
$1 \times 10^{10} - 5 \times 10^{10}$	Limit of sufficiency
$> 5 \times 10^{10}$	Insufficient

2.6.3 Simulation methods for measuring electrostatic behavior

Both the American Association of Textile Colorists and Chemists (AATCC) and the International Standard Organization (ISO) have developed simulation methods. AATCC titles their test, Electrostatic Propensity of Carpets, also known as Test Method 134 (AATCC Test Method 134, 2006). Similarly, ISO labeled their test, Textile Floor Coverings- Assessment of Static Electrical Propensity-Walking Test (International Standards Organization Test Method 6356, 2000). Simulation methods are used to test products in their intended end use. In the case of these tests, laboratory simulations are conducted to focus on the simulation of static accumulation in carpets. Both tests are similar in nature. Comparisons of these tests are described in **Table 2.4**.

**Table 2.4 Comparison of AATCC and ISO simulation methods of static charge accumulation on carpets
(Suh, 2008)**

	AATCC Standard	ISO Standard
Temperature	21 ± 1°C	23 ± 1°C
Humidity	20 ± 2 %	20 ± 1% or 25 ± 1%
Specimen Size	70 cm × 90 cm	200 cm × 100 cm
Underlay	Jute / Hair Cushion	Rubber Mat
Sandal Soles	Neolite / Suede leather	Neolite
Walking Manner	Stepping and Scuffing	Stepping
Walking Speed	120 ± 10 steps/min	120 steps/min
Sandal Lifting During Walk	8 cm	5 ~ 8 cm
Measurement	Potential in voltage	Potential in voltage

The main differences between the two standards are with respect to sample size, underlay, and walking manner. The AATCC standard allows for jute or hair underlay cushion. In contrast, the ISO standard requires a rubber matt. Both standards allow for the use of neolite in the sandal soles used to walk on the carpet; however, the AATCC standard also allows suede leather to be utilized. Finally, the AATCC method allows scuffing to occur during testing, whereas the ISO standard does not. Both standards state that stepping must occur during testing (AATCC Test Method 134, 2006; International Standards Organization Test Method 6356, 2000).

CHAPTER 3 METHODOLOGY AND EXPERIMENTAL PROCEDURES

3.1 The approach of research and its advantages

This research will focus on treating a number of synthetics woven fabrics. Fabrics used in this research are listed in **Table 3.1**.

Table 3.1 Fabric weight and pick/end densities

Fabric	Weight (g/m²)	Ends	Picks
Woven Spun Nylon 6,6 DuPont Type 200 (ISO 105/F03): Style 361	124	51	51
Woven Filament Nylon 6,6 Semi-Dull Taffeta, Scoured, Heat Set, 25 filaments in warp and fill direction: Style 306A	59	85	85
Woven Spun Polyester 100% (Disperse Dyeable): Style 777	126	50	50
Woven Filament Taffeta Polyester (Disperse Dyeable), 70 filaments in warp and fill direction: Style 738	147	50	50
Woven Spun Acrylic, Orlon Type 75, Plain Weave: Style 864	135	53	30
Woven Spun Polypropylene: Style 983	170	31	31

This research aims to create an antistatic finish that will have advantages such as:

1. Excellent antistatic properties
2. Durability
3. Low cost
4. Simple processing

An electroless deposition process will be employed to create a durable antistatic finish. As a part of this process conventionally used acid pretreatment will be replaced with plasma pretreatment. Pretreatment is needed in an electroless deposition to achieve etching. Plasma pretreatment has been shown to be a superior method to acid pretreatment in carbon nanotubes, increasing copper uptake by 239% (Zhu et al, 2006). Other advantages of plasma pretreatments include monetary savings and elimination of potentially hazardous effluent. Since plasma treatments are a dry-processing technique, it can be argued that plasma is more environmentally friendly than conventional wet-processing (Shishoo, 2007).

After plasma pretreatment is completed, a standard electroless deposition process will be conducted using copper. Through this process, copper will be deposited on the surface of the fabric. Deposition of copper will decrease the resistivity of the fabric, allowing electrostatic charge to dissipate quickly.

3.2 Experimental design

Research conducted will focus on how nitric acid pretreatment and NCAPS pretreatment influence fabric samples and static testing after electroless deposition. Thus, one experimental design exists for samples pretreated with nitric acid and another experimental design exists for samples treated with the NCAPS.

3.2.1 Nitric acid pretreated samples

Table 3.2 depicts the experimental design for nitric acid pretreatment. Since acid pretreatment has been used commercially with success, this will serve as a baseline for plasma pretreated samples. Properties can be measured on plasma pretreated samples and compared against acid pretreated samples. Thus, antistatic and durability properties can be analyzed. The nitric acid bath and rinse bath were both 3,000 mL. All fabric samples were treated according to **Table 3.2** for nitric acid pretreatment.

Table 3.2 Nitric acid pretreatment experimental design for woven spun nylon 6,6, filament nylon 6,6, spun polyester, filament polyester, spun acrylic, and spun polypropylene

Nitric Acid Pretreatment	Nitric acid (%)	6.7
	Time (minutes)	10
	Bath temperature (°C)	23
	Rinse temperature (°C)	23
	Drying temperature (°C)	110
	Drying time (minutes)	10

3.2.2 NCAPS pretreated samples

All samples were pretreated with plasma on the NCAPS. **Table 3.3** depicts the experimental design for all fabric samples pretreated on the NCAPS.

Table 3.3 NCAPS pretreatment experimental design for woven spun nylon 6,6, filament nylon 6,6, spun polyester, filament polyester, spun acrylic, and spun polypropylene

NCAPS Pretreatment	Time (seconds)	60
	System power (Hz)	1380
	Helium (%)	99
	Oxygen (%)	1

3.2.3 Control samples

Control samples were made for each fabric in the experiment. These samples were not treated with any chemicals. This allows for the classification of electrostatic behavior of untreated fabric samples. Thus, properties of nitric acid pretreatment with electroless deposition and NCAPS pretreated samples with electroless deposition can be compared to an untreated fabric.

3.3 Experimental procedure

3.3.1 Fabric preparation

Fabric used in this experiment was obtained from Test Fabrics, Inc. of West Pittiston, PA.

All fabric samples were desized and scoured. The experiment was carried out on fabric samples that measured 8 inches by 8 inches. The face of all samples was marked and all testing and plasma treatment was conducted on this surface. Weight (g/m^2), end and pick densities, and other pertinent information of samples are shown in **Table 3.1**.

3.3.2 Experimental procedures

3.3.2.1 Plasma pretreatment

The plasma machine used in this research is known as the North Carolina Atmospheric Plasma System (NCAPS). The NCAPS utilizes atmospheric plasma technology. The NCAPS is located in Dr. Marian McCord's lab, room 3230, at North Carolina State University's College of Textiles. The NCAPS was customly built for the university. The NCAPS is depicted in **Figure 3.1**. A compressed air system is used to raise and lower the electrode during loading and unloading of the cell. Gas enters through one side of the NCAPS and escapes through the exhaust canopy. The system contains an upper electrode and a lower electrode. Both electrodes are directly linked to production of plasma. The power supply for the NCAPS is located opposite of the gas flow. **Figure 3.1** depicts a schematic of the NCAPS. The system can be made into a continuous system through use of fabric rollers; however, a continuous system was not utilized in this research.

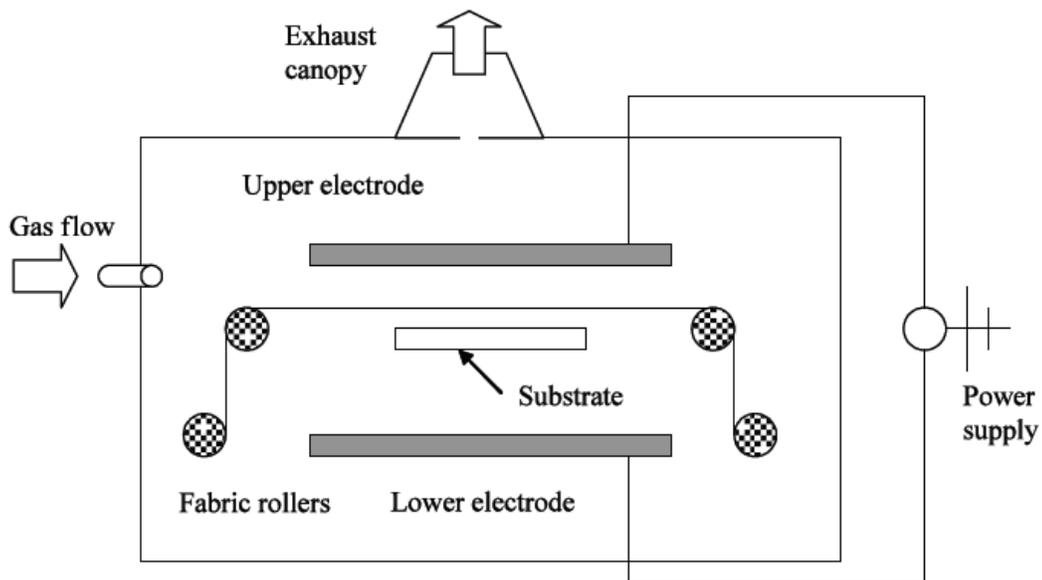


Figure 3.1 Schematic of the North Carolina Atmospheric Plasma System (NCAPS) (Cornelius, 2009)

Figure 3.2 provides a better view of where the substrate is placed in **Figure 3.1**. The area is known as the batch treatment cell. The frame used to house both the upper and lower electrode is made of quasi-opaque polycarbonate and measures 11.5" x 11.5". The width of the area where samples are placed is 1.5". The mesh used to hold samples is made of nylon.

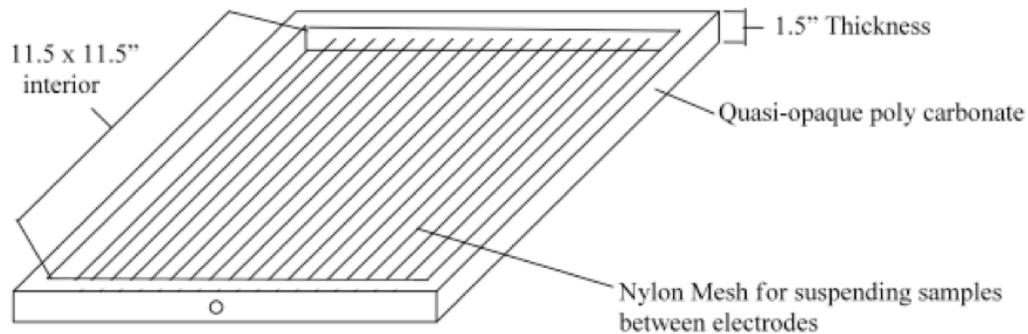


Figure 3.2 Schematic of the NCAPS batch treatment cell (Cornelius, 2009)

Two gases were used in plasma pretreatment on the NCAPS, helium and oxygen. The helium-oxygen mixture was adjusted so helium made up 99% of the total volume of gas, and oxygen, 1%. Oxygen was needed in this experiment to provide surface etching and roughening. Surface etching and roughening has been shown to increase the cohesion and deposition occurring during electroless plating. Before running samples, gas was allowed to fill the chamber for 5 minutes before turning on the power supply. Samples were treated for 60 seconds with 1380 Hz of power.

3.3.2.2 Nitric acid pretreatment

Samples were submersed in a 6.7% nitric acid solution for 10 minutes. The total bath volume was 3000 mL. Based on a 6.7% concentration of nitric acid, 201 mL of nitric acid were added to 2,799 mL of deionized water. The nitric acid pretreatment took place at room temperature, 23°C. After etching occurred, samples were rinsed 3x, each time in 3000 mL of deionized water. Samples were then centrifuged for 1 minute and dried at 110°C for 10 minutes. Nitric acid used was supplied by Fisher Scientific.

3.3.2.3 Electroless deposition treatment

After samples were treated with nitric acid or plasma pretreatment, an electroless deposition process was performed. This process remained constant for all samples and followed a commercial process. The electroless deposition process used is outlined below. Deionized water was added to specified chemicals to make bath solutions. The total bath volume for each step was 2,500 mL. A magnetic stirring rod was used to ensure each bath was mixed thoroughly and a hot plate was used to heat baths to the specified temperature. Samples were rinsed 3x with 3000 mL of deionized water after each step. After step 3, fabric samples were centrifuged for 1 minute and dried at 80°C for 10 minutes.

1. **Sensitization/Activation:** PdCl₂ (200 mg/L), SnCl₂ (20 g/L), and 37% HCl (200 mL/L) at 25°C for 1-3 minutes.
2. **Acceleration:** 37% HCl (120 mL/L) at 35°C for 1-3 minutes.
3. **Electroless copper plating:** CuSO₄ (10 g/L), EDTA (30 g/L, NaOH (8 g/L), Formaldehyde (7.5 mL/L), Polyethylene Glycol (7.5 mL/L) at 45°C for 20 minutes.

Chemicals in the electroless deposition bath were from a number of manufacturers.

Manufacturers and information on chemicals used in electroless deposition treatment are as seen in **Table 3.4**.

Table 3.4 Chemical information for the electroless deposition process

37% Hydrochloric acid (HCl), ACS certified, Fisher Scientific
Tin chloride (SnCl ₂), Fisher Scientific
Palladium chloride (PdCl ₂), 5% weight/volume, Acros
Copper (II) sulfate (CuSO ₄), 98%, Aldrich Chemical Company
Ethylenediaminetetraacetic acid (EDTA), Fisher Scientific
Polyethylene glycol: Molecular Weight 600, Alfa Aesar
Formaldehyde: 37% weight solution, stabilized, Acros

3.4 Testing

Water absorption testing was performed on untreated, nitric acid, and plasma pretreated samples in order to quantify changes in the surface of the fabric. Tensile strength testing was performed to observe any changes in nitric acid and plasma pretreatment, compared to the untreated samples. SEM images were taken in order to observe changes in the surface of fabric samples. SEM images were taken of untreated, nitric acid pretreated, plasma pretreated, nitric acid pretreatment with electroless deposition, and plasma pretreatment with electroless deposition samples. Surface resistivity values were measured in order to analyze antistatic properties. Resistivity testing was completed on untreated, nitric acid pretreated with electroless deposition, and plasma pretreated with electroless deposition samples.

Dissipation values were also recorded for untreated, nitric acid pretreated with electroless deposition, and plasma pretreated with electroless deposition samples. Dissipation testing depicted the rate that fabric samples were able to rid themselves of electrostatic charge.

3.4.1 Absorption

Absorption rates of fabric samples were completed in conditions of 70°F and 65% relative humidity. 1 mL of deionized water was dropped onto the surface of untreated, nitric acid pretreated, and plasma pretreated samples. The rate, in seconds, needed for 1 mL of water to absorb was recorded for each sample. If a sample didn't absorb water, the phrase no absorption was recorded. For each sample, 3 absorption values were recorded. These values were averaged and the standard deviation was calculated.

3.4.2 Tensile strength grab test

All samples were testing for changes in tensile strength according to the American Society for Testing and Materials (ASTM) Test Method D5034. In accordance with the test method, samples which measured 4 inches x 6 inches were prepared. Samples were mounted in a MTS Q-TestTM/5 EliteTM Controller in the Physical Testing Laboratory (room 1414A) at the North Carolina State University College of Textiles. The jaw face size was 1 inch x 1 inch and 80 PSI of pressure was used to hold samples. The gage length used during testing was 3 inches and the crosshead speed was 12 inches/minute. A 250 pound load cell was used in testing and standard conditions of 70°F and 65% relative humidity were present during testing. 3 warp and 3 fill samples were prepared for all untreated, nitric acid pretreated, and plasma pretreated fabric types.

3.4.3 Yarn tensile strength test

Though fabric tensile strength testing gave an accurate view changes in fabric strength (lbf), it was important to study tensile strength change on individual yarns. Since spun nylon 6,6 experienced a significant change in warp and fill fabric tensile strength, its yarns were selected for yarn tensile strength testing. Yarn tensile strength testing was conducted to ASTM D2256, with one adaption. Instead of using a yarn length of 10 inches as ASTM D2256 calls for, a yarn length of 6 inches was used. This was because of limitations of treated sample sizes. Other parameters used in association with ASTM D2256 were 60 PSI of grip pressure and a crosshead speed of 300 millimeters/minute. Yarn tensile strength testing took place on the MTS Q-Test™/5 Elite™ Controller in the Physical Testing Laboratory (room 1414A) at the North Carolina State University College of Textiles. Standard conditions of 70°F and 65% relative humidity were present during testing.

3.4.4 Scanning electron microscopy

Scanning electron microscopy (SEM) imaging was performed to analyze the surface morphology of fabric samples. SEM images were taken for untreated, nitric acid pretreated, NCAPS pretreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition samples. The SEM used for analysis was the field-emission scanning electron microscope (FESEM), JEOL model JSM-6400F. The FESEM is located in room 2117 at the North Carolina State University College of Textiles.

3.4.5 Elemental analysis

Elemental analysis of fabric samples, using inductively coupled plasma atomic emission spectrometry (ICP-AES) emission spectroscopy, was conducted by Pace Analytical Labs in Huntersville, North Carolina. ICP was used to quantify the amount of copper present on

fabric samples. Two United States Environmental Protection Agency (EPA) standards were used for elemental analysis via inductively coupled plasma: EPA 3050B and EPA 6010B. EPA 3050B was followed to digest all samples before elemental analysis via inductively coupled plasma was performed. EPA 3050 utilized 1 gram of fabric. Samples were digested in nitric acid and hydrogen peroxide and diluted to a volume of 100 mL (EPA Test Method 3050B, 1989). Once samples were digested using EPA 3050B, EPA 6010B was used to determine the amount of copper present on the sample. Samples were nebulized and the resulting aerosol is transported to the plasma torch. Since EPA 6010B can be used to identify other trace metals besides copper, each element has a unique emission spectra that is used for element identification. Based on the emission spectra, the concentration of the corresponding element can be determined. EPA 6010B can be used for a number of items including soil, wastewater, and textiles (EPA 6010B, 1996).

3.4.6 Antistatic testing

Electrical surface resistivity and charge generation/dissipation testing was performed in an environmental chamber. The environmental room is housed in room 1311 at the North Carolina State University College of Textiles. The environmental room measures 3.25 m (width) x 2.64 m (length) x 2.64 m (height). The temperature and relative humidity of the environmental room can be controlled between 7°C-60°C and 10-95% relative humidity, respectively (Suh, 2008). In this research, conditions of 21°C and 43% relative humidity were used. All samples were conditioned for 48 hours.

3.4.6.1 Antistatic testing preparation

Before testing, untreated samples were cleaned to ensure antistatic testing was accurate.

Cleaning was conducted to remove all oil, sizing, or other impurities on the fabric that may be present. The cleaning procedure used is listed below:

1. Place fabric in a deionized water bath at a temperature of 60° C for 20 minutes.
2. Place fabric in a 2-propanol (isopropyl alcohol) for 20 minutes and remove.
3. Dry fabric for 30-40 minutes (or until it gets dried completely) at 80° C.

3.4.6.2 Electrical surface resistivity

Electrical resistivity, defined by AATCC, is a material property of a substance whose numerical value is equal to the ratio of the voltage gradient to the current density. Testing for electrical surface resistivity was conducted according to AATCC Test Method 76. The resistivity device used for this research was manufactured by Trek, Inc. The device consists of a meter, which gives the resistivity measurement, and two electrodes arranged in a concentric manner. The inner electrode is a solid disc with a diameter of 30.48 +/- 0.64 mm. The outer electrode is a ring measuring 57.15 +/- 0.64 mm in diameter with a thickness of 3.18 +/- 0.30 mm. **Figure 3.3** depicts the concentric ring used in resistivity testing.

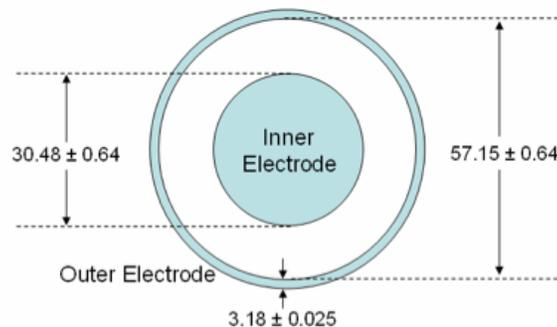


Figure 3.3 Schematic of the concentric ring electrode (Suh, 2008)

Four readings were taken across an 8 inch x 8 inch sample as shown in **Figure 3.4**. Each black circle in **Figure 3.4** represents a reading taken by a concentric ring electrode.

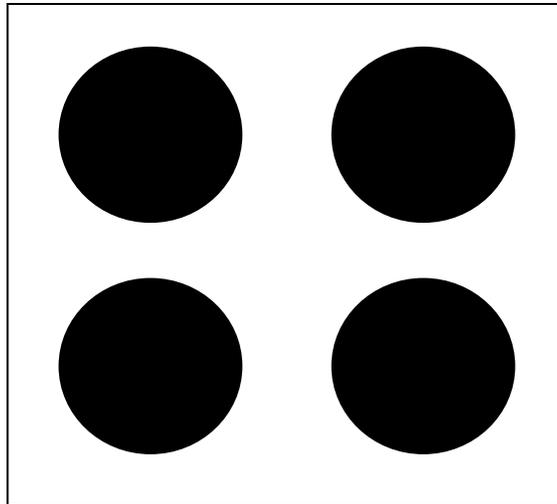


Figure 3.4 Schematic depicting locations tested for surface resistivity on each sample

Fabrics were placed on a non-conductive surface. Samples were subjected to 100 Volts of electricity for 60 seconds, before a reading was taken. Electrical resistivity was calculated according to **Equation 3.1** (AATCC Test Method 76, 2005).

$$\rho_s = \frac{2\pi R_s}{\ln \frac{D_2}{D_1}}$$

Equation 3.1 Electrical resistivity calculation

Where variables are defined as:

- ρ_s = surface resistivity (Ω/cm^2)
- R_s =measured surface resistance (Ω)
- D_1, D_2 =electrode diameter (cm)

3.4.6.3 Rubbing charge generation and dissipation

In addition to electrical resistivity, electrostatic dissipation testing was conducted. Each fabric sample was rubbed 50 times with a polytetrafluoroethylene (PTFE) rubbing head. Rubbing with PTFE built up a charge on the surface of the fabric. After 50 rubbings, the fabric was allowed to dissipate static charge. The time needed to reduce static charge on the fabric sample was recorded. Ideally, the fabric sample would reduce its charge to 0 volts. If the charge didn't dissipate to 0 volts, the lowest voltage reached was recorded. A 6 mm sample in diameter was used in this research. 3 locations were tested across each fabric sample. A schematic of the contact charge and dissipation testing machine is shown earlier in **Figure 2.21**.

The device depicted in **Figure 2.21**, is known as the rubbing tester system. This device was developed by Seyam et al (2009) at North Carolina State University. The rubbing tester system proves especially useful when studying surface modification on different textile substrate surfaces. This device is used to investigate electrostatic generation and dissipation between two flat polymer surfaces, which rub together. The sample is placed on a stationary surface and rubbed with a PTFE rubbing head. The parameters used were a track length of 50.4 mm and 1 newton of force. The tester measures charge potential on a stationary fabric. The rubbing device has a variable rubbing speed, pressure, and number of cycles (Seyam et al, 2009).

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Absorption rates

A technique used to quantify the effectiveness of etching, is the rate of absorption. 1 mL of water was dropped on each fabric sample. The time needed for the droplet to absorb was recorded in seconds. The adsorption rates for untreated, nitric acid, and NCAPS samples are shown in **Tables 4.1-4.4**. For NCAPS samples, associated treatment parameters are listed. Absorption rates were measured 3 times across the surface each sample and the mean and standard deviation were calculated.

Table 4.1 Untreated absorption values (n=3)

Fiber	Mean Absorption Rate (sec)	Absorption Std. Dev. (sec)
Spun Nylon 6,6	26	4
Filament Nylon 6,6	No absorption	No absorption
Spun Polyester	163	15
Filament Polyester	270	21
Spun Acrylic	0	0
Spun Polypropylene	No absorption	No absorption

Initial absorption values were recorded for fabric samples, prior to pretreatment. Both filament nylon 6,6 and spun polypropylene did not absorb water. Spun nylon 6,6 absorbed water after 26 seconds, spun polyester absorbed water after 163 seconds, and filament polyester absorbed water after 270 seconds. Spun acrylic fabric absorbed water instantaneously.

Table 4.2 Nitric Acid adsorption rates, standard deviations, and associated treatment parameters (n=3)

Fiber	Nitric Acid (%)	Nitric Acid Temp (°C)	Time (min)	Drying Temp (°C)	Drying Time (min)	Absorption Rate (sec)	Adsorption Std. Dev. (sec)
Spun Nylon 6,6	6.7	25	10	110	10	155	74
Filament Nylon 6,6	6.7	25	10	110	10	23	5
Spun Polyester	6.7	25	10	110	10	No absorption	No absorption
Filament Polyester	6.7	25	10	110	10	217	76
Spun Acrylic	6.7	25	10	110	10	0	0
Spun Polypropylene	6.7	25	10	110	10	No absorption	No absorption

After nitric acid pretreatment, absorption values associated with spun nylon 6,6 rose from 26 seconds to 155 seconds. Filament nylon 6,6 absorption values decreased from no absorption, to absorption in 23 seconds. Thus, water was absorbed faster. Spun polyester fabric samples saw hydrophobicity increase, as fabric wouldn't absorb any water after nitric acid pretreatment. Absorption of filament polyester samples decreased slightly from 270 seconds to 217 seconds. Absorption values for spun acrylic and spun polypropylene remained the same after nitric acid pretreatment, with instantaneous absorption and no absorption, respectively.

Table 4.3 NCAPS adsorption rates, standard deviations, and associated plasma parameters (n=3)

Fiber	Power (Hz)	He (%)	O₂ (%)	Time (sec)	Absorption Rate (sec)	Adsorption Std. Dev. (sec)
Spun Nylon 6,6	1380	99	1	60	6	1
Filament Nylon 6,6	1380	99	1	60	7	4
Spun Polyester	1380	99	1	60	79	11
Filament Polyester	1380	99	1	60	28	17
Spun Acrylic	1380	99	1	60	0	0
Spun Polypropylene	1380	99	1	60	No absorption	No absorption

The absorption rates for the majority of NCAPS pretreated fabric increased significantly, compared to untreated and nitric acid pretreated fabric as depicted in **Table 4.3**. Less time, in seconds, taken for fabric to absorb 1 mL of water indicated water is being absorbed quicker. Spun nylon 6,6 adsorption decreased to 6 seconds, in comparison to 26 seconds for untreated samples and 155 seconds for nitric acid pretreated samples. The relationship observed with filament nylon 6,6 was similar to spun nylon 6,6. The absorption time of filament nylon 6,6 decreased from no absorption, to 23 seconds, to 7 seconds, for untreated, nitric acid pretreated, and NCAPS pretreated samples, respectively. NCAPS pretreated spun polyester absorbed water in 79 seconds. In comparison, untreated spun polyester took 163 seconds to absorb water and nitric acid pretreated spun polyester didn't absorb water. NCAPS pretreated spun acrylic and spun polypropylene retained similar absorption properties to untreated and nitric acid pretreated samples. Spun acrylic absorbed water instantaneously and spun polypropylene remained hydrophobic, with no absorption.

4.2 Fabric tensile strength values

Tensile strength values were recorded for untreated, nitric acid pretreated, and NCAPS pretreated samples. Untreated tensile strength values served as a baseline. Nitric acid and NCAPS pretreated tensile strength values were compared to untreated tensile strength values. Tensile strength values of nitric acid and NCAPS pretreatment samples were also compared to see if one pretreatment option was superior in retention of tensile strength. Three repetitions were performed in the warp and fill direction for each sample and the mean and standard deviation were calculated.

Table 4.4 Fabric tensile strength for original, nitric acid pretreated, and NCAPS pretreated warp samples (n=3)

	Untreated		Nitric Acid Pretreated		NCAPS Pretreated	
	Mean (lbf)	Std. Dev. (lbf)	Mean (lbf)	Std. Dev. (lbf)	Mean (lbf)	Std. Dev. (lbf)
Spun Nylon 6,6	245.19	6.57	206.40	9.02	237.32	9.37
Filament Nylon 6,6	134.06	2.95	111.05	4.71	131.54	2.68
Spun Polyester	153.67	3.58	152.42	8.27	153.69	3.70
Filament Polyester	233.96	7.61	234.13	4.82	234.74	7.63
Spun Acrylic	136.60	3.74	129.99	10.30	128.54	7.02
Spun Polypropylene	235.11	10.45	163.86	8.63	233.01	3.93

Several trends are evident when comparing warp tensile strength (lbf) values of untreated fabric samples to values associated with nitric acid and NCAPS pretreated samples as displayed in **Table 4.4**. After nitric acid pretreatment, spun and filament nylon 6,6 experienced a reduction in tensile strength. The tensile strength of spun nylon 6,6 decreased from 245.19 lbf to 206.40 lbf. NCAPS pretreated spun nylon 6,6 fared slightly better, decreasing to 237.32 lbf. A similar trend to spun nylon 6,6 was observed in untreated filament nylon 6,6 and nitric and NCAPS pretreated filament nylon 6,6 samples. The tensile strength of filament nylon 6,6 dropped from 134.06 lbf to 111.05 lbf. NCAPS pretreated filament nylon 6,6's tensile strength decreased from 134.06 lbf to 131.54 lbf. Nitric acid and NCAPS treatment didn't significantly affect the tensile strength of spun or filament polyester. Untreated spun polyester's tensile strength measured 153.67 lbf, compared to 152.42 lbf and 153.69 lbf, observed with nitric acid and NCAPS pretreated samples. The tensile strength of untreated filament polyester was 233.96 lbf. Nitric acid and NCAPS pretreated filament polyester samples measured 234.13 lbf and 234.74 lbf, respectively. Nitric acid and NCAPS pretreated spun acrylic samples sustained a small decrease in tensile strength, compared to untreated spun acrylic. The tensile strength of nitric acid pretreated spun acrylic dropped to 129.99 lbf, compared to 136.60 lbf observed with the untreated sample. Similarly, after NCAPS treatment, the tensile strength of spun acrylic decreased to 128.54 lbf. The greatest loss in tensile strength was observed in spun polypropylene. Spun polypropylene's tensile strength decreased from 235.11 lbf, to 163.86 lbf, after nitric acid treatment. Spun polypropylene pretreated with the NCAPS fared better. NCAPS pretreated spun polypropylene only decreased to 233.01 lbf.

Table 4.5 Fabric tensile strength for original, nitric acid pretreated, and NCAPS pretreated fill samples (n=3)

	Untreated		Nitric Acid Pretreated		NCAPS Pretreated	
	Mean (lbf)	Std. Dev. (lbf)	Mean (lbf)	Std. Dev. (lbf)	Mean (lbf)	Std. Dev. (lbf)
Spun Nylon 6,6	123.34	7.14	108.37	7.10	121.23	3.24
Filament Nylon 6,6	115.63	4.29	95.16	5.99	107.60	5.95
Spun Polyester	75.22	1.52	77.04	4.38	77.33	2.99
Filament Polyester	229.90	20.55	236.20	7.72	230.51	2.51
Spun Acrylic	79.99	1.60	76.28	9.71	77.04	1.93
Spun Polypropylene	161.77	7.47	164.72	1.83	165.96	7.18

Several observations can be made regarding the fill direction of untreated, nitric acid pretreated, and NCAPS pretreated samples displayed in **Table 4.5**. Spun nylon 6,6 lost strength in the fill direction after both nitric and NCAPS pretreatment. Nitric acid pretreated nylon 6,6 had the greatest decrease in tensile strength, compared to the untreated nylon 6,6 sample, weakening from 123.34 lbf to 108.37 lbf. NCAPS pretreated nylon 6,6 decreased marginally, falling to 121.23 lbf. Filament nylon 6,6 samples followed a similar trend to spun nylon 6,6 samples. Nitric acid pretreated filament nylon 6,6 declined from 115.63 lbf to 95.16 lbf. NCAPS pretreated filament nylon 6,6 weakened less than nitric acid pretreated filament nylon 6,6, declining to 107.60 lbf. Nitric acid pretreatment interacted with the chemical structure in a way that weakened spun and filament nylon 6,6, since minimal etching was observed on the fiber surface in SEM images. This is apparent as NCAPS pretreated spun and filament nylon 6,6 decreased by a significantly smaller amount.

Untreated spun filament polyester measured 75.22 lbf. Nitric acid and NCAPS pretreated samples rose an insignificant amount, to 77.04 lbf and 77.33 lbf, respectively. A similar trend was observed with nitric and NCAPS pretreated filament polyester samples. Nitric acid and NCAPS samples rose from 229.90 lbf, to 236.20 lbf and 230.51 lbf, correspondingly. Spun acrylic samples lost a marginal amount of strength after nitric acid and NCAPS pretreatment. Untreated acrylic had a tensile strength of 79.99 lbf. Nitric acid pretreated samples declined slightly to 76.28 lbf. The tensile strength of NCAPS pretreated samples fell to 77.04 lbf.

After both nitric acid and NCAPS pretreatment, spun polypropylene samples gained minimal strength, compared to untreated spun polypropylene. Nitric acid and NCAPS pretreated samples increased from 161.77 lbf, to 164.72 lbf and 165.96 lbf, respectively.

4.3 Yarn tensile strength values

After analyzing fabric tensile strength values, it was questioned whether consistent fabric tensile strength values for NCAPS pretreated samples, compared to untreated tensile strength values, were a result of increased friction between yarns. It was anticipated that increased friction between yarns would increase tensile strength, even if individual yarns had been weakened by NCAPS Pretreatment. Also in question was chemical degradation of nitric acid pretreated spun nylon 6,6. It was surmised that nitric acid pretreatment penetrated fibers, leading to chemical degradation. By picking spun nylon 6,6, a sample that had similar warp and fill fabric tensile strength values for NCAPS pretreated samples and lower values for nitric acid pretreated samples, it was expected that information regarding changes in yarn strength could be obtained. **Table 4.6** depicts changes observed in tensile strength between untreated, nitric acid pretreated, and NCAPS pretreated samples.

Table 4.6 Yarn tensile strength for original, nitric acid pretreated, and NCAPS pretreated spun nylon 6,6 warp and fill samples (n=10)

	Untreated		Nitric Acid Pretreated		NCAPS Pretreated	
	Mean (gf)	Std. Dev. (gf)	Mean (gf)	Std. Dev. (gf)	Mean (gf)	Std. Dev. (gf)
Spun Nylon 6,6 Warp Yarns	1,685.24	126.15	1,385.80	144.72	1,549.31	121.80
Spun Nylon 6,6 Fill Yarns	663.34	96.20	512.32	58.18	574.48	50.40

Table 4.5 indicates nitric acid and NCAPS pretreatment weakened yarn tensile strength, compared to untreated yarn tensile strength, for spun nylon 6,6. However, NCAPS pretreated samples had retained more tensile strength than nitric acid pretreated samples. Untreated samples had warp and fill tensile strength values of 1,685.24 gf and 663.34 gf. In comparison, NCAPS pretreated samples had a tensile strength of 1,549.31 gf and 574.48 gf in the warp and fill directions, respectively. Nitric acid pretreated samples had the poorest yarn tensile strength performance with 1,385.80 gf and 512.32 gf for warp and fill yarns. Based on yarn tensile strength values for spun nylon 6,6, it is apparent that chemical degradation is responsible for fabric and yarn tensile strength loss. While NCAPS pretreated samples had similar warp and fill fabric tensile strength values, NCAPS pretreated yarn tensile values declined. Etching from NCAPS pretreatment causes physical changes, such as etching, which weakens yarn strength. However, increased etching on the surface increases the coefficient of friction, helping to preserve fabric tensile strength as seen in Table 4.3 and 4.4.

4.4 Digital photography comparison of samples

Digital photographs were taken of all samples to depict the changes seen visually pertaining to the pretreatment type.



Figure 4.1 Digital photography comparison between untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun nylon 6,6 (from left to right)



Figure 4.2 Digital photography comparison between untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition filament nylon 6,6 (from left to right)



Figure 4.3 Digital photography comparison between untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun polyester (from left to right)



Figure 4.4 Digital photography comparison between untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition filament polyester (from left to right)



Figure 4.5 Digital photography comparison between untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun acrylic (from left to right)

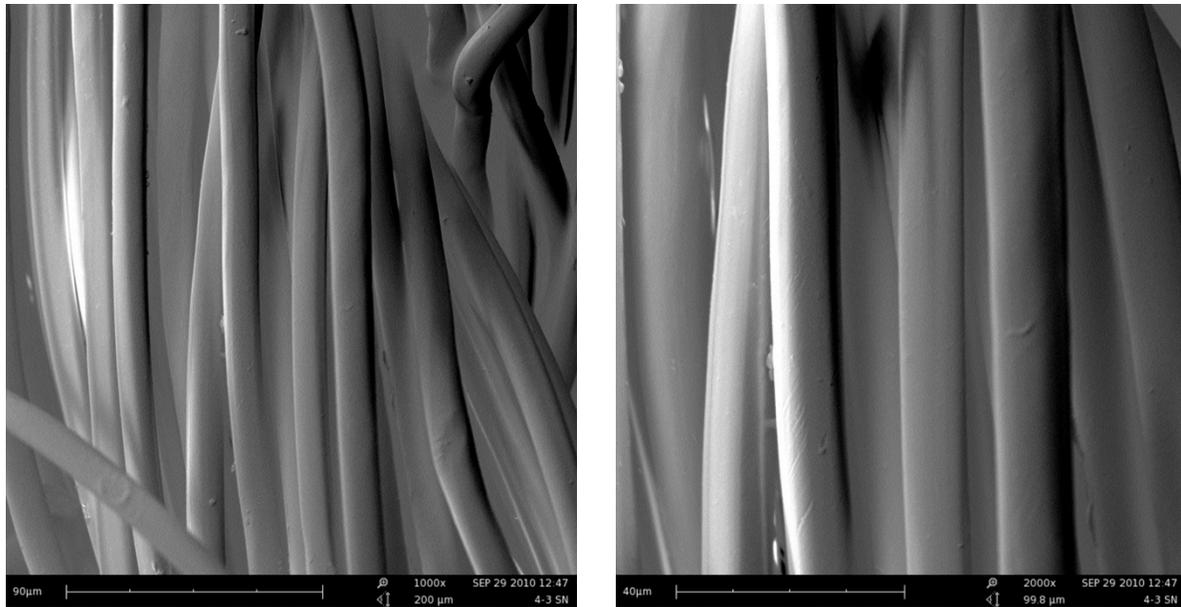


Figure 4.6 Digital photography comparison between untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun polypropylene (from left to right)

Spun nylon 6,6, filament nylon 6,6, spun polyester, filament polyester, spun acrylic, and spun polypropylene samples all exhibit results similar to each other, as depicted in **Figures 4.1-4.6**. Nitric acid pretreated samples with electroless deposition are marginally darker than untreated samples. NCAPS pretreated samples with electroless deposition were much darker than untreated and nitric acid pretreated samples with electroless deposition. NCAPS pretreated samples with electroless deposition appeared reddish brown in color from copper deposition. Based on visual inspection, NCAPS pretreated samples with electroless deposition have significantly more copper than nitric acid pretreated samples with electroless deposition.

4.5 Scanning electron microscopy images

SEM images were taken of untreated, nitric acid pretreated, NCAPS pretreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition samples at 1000x and 2000x. Physical differences exist on the fiber surface depending on the treatment type. Untreated, nitric acid pretreated, NCAPS pretreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun nylon 6,6 samples are seen in **Figures 4.7-4.11**, filament nylon 6,6 samples in **Figures 4.12-4.17**, spun polyester samples in **Figures 4.17-4.21**, filament polyester in **Figures 4.22-4.26**, spun acrylic in **Figures 4.27-4.31**, and spun polypropylene in **Figures 4.32-4.36**.

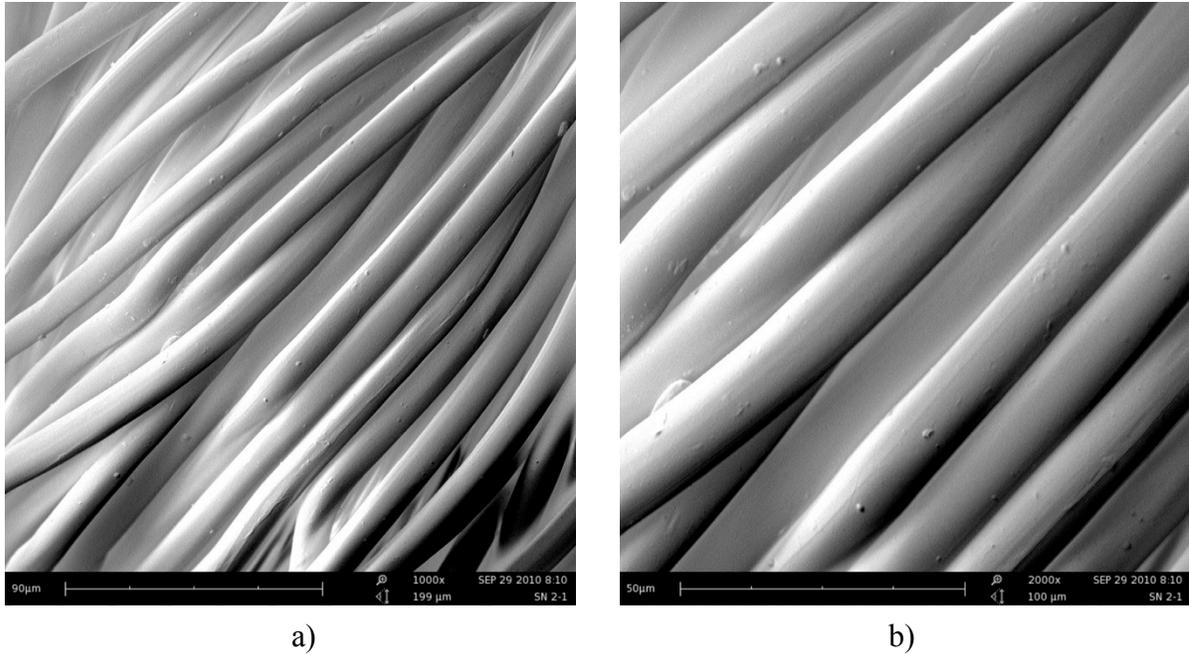


a)

b)

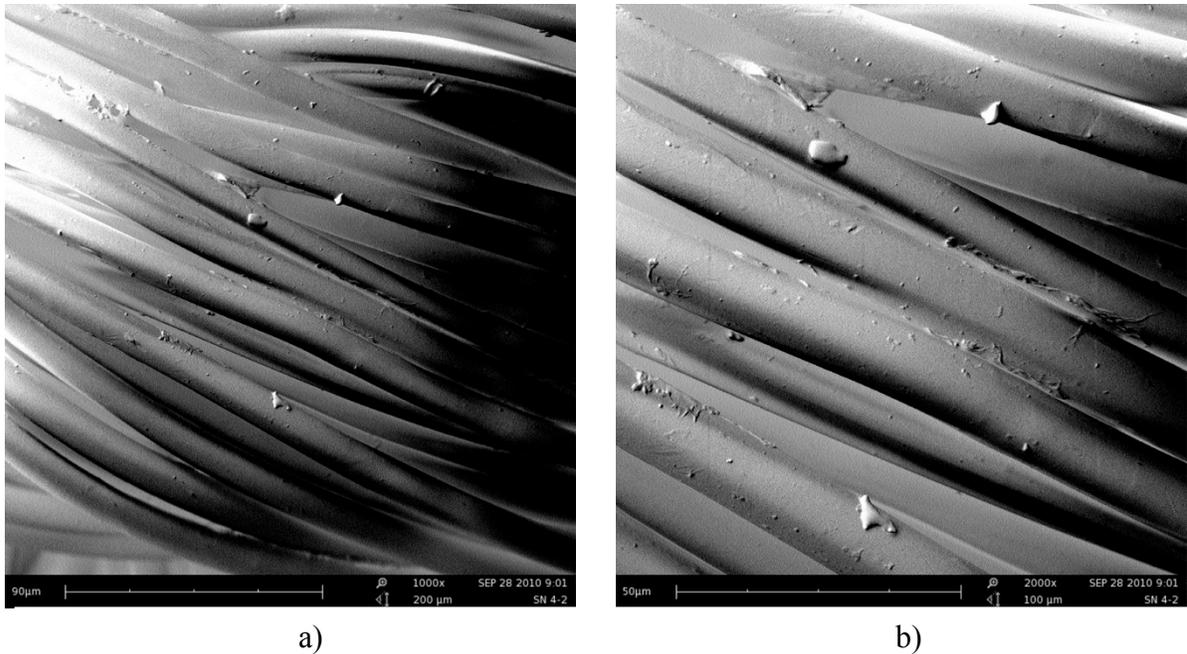
**Figure 4.7 SEM of Untreated woven spun nylon 6,6
a) 1000x b) 2000x**

The surfaces of untreated woven spun nylon 6,6 fibers appear round and smooth. A few small bumps are present, that were created during manufacturing. The fiber surface appears flawless.



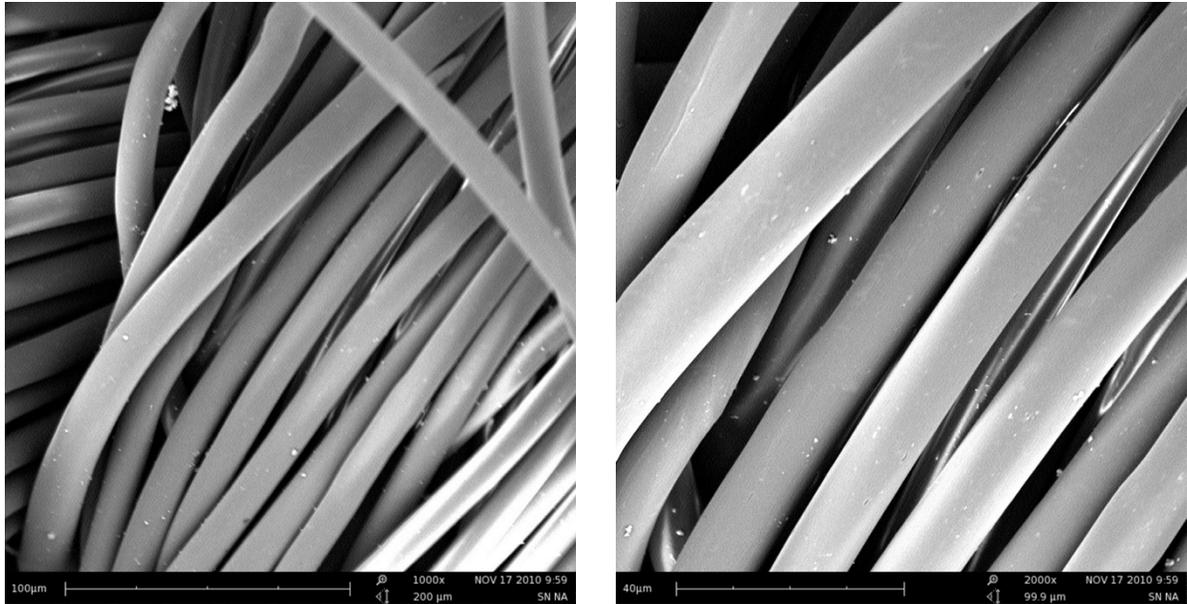
**Figure 4.8 SEM of Nitric acid pretreated woven spun nylon 6,6
a) 1000x b) 2000x**

The surface morphology of nitric acid pretreated woven spun nylon 6,6 appears to be the same as the untreated spun nylon 6,6 sample. Though there are no visible striations, the overall surface appears to have several pits and bumps. Similar bumps were present on the untreated sample. The nitric acid treatment didn't affect the surface morphology of woven spun nylon 6,6 fibers as seen through SEM images.



**Figure 4.9 SEM of NCAPS pretreated woven spun nylon 6,6
a) 1000x b) 2000x**

The surface of NCAPS pretreated woven spun nylon 6,6 seconds is much rougher than untreated or nitric acid pretreated samples. The surface of NCAPS pretreated woven spun nylon 6,6 has pits scattered across the surface. Additionally, several raised areas are present on the surface of the fibers. The temperature of the NCAPS is between 20-25°C (Cornelius, 2009). Therefore, it is highly unlikely these raised areas are due to polymer bubbling as the melting point of nylon 6,6 is 265°C (Mutz, 1974). The raised areas are likely pieces of polymer that were etched off of other fibers and landed in their current position.

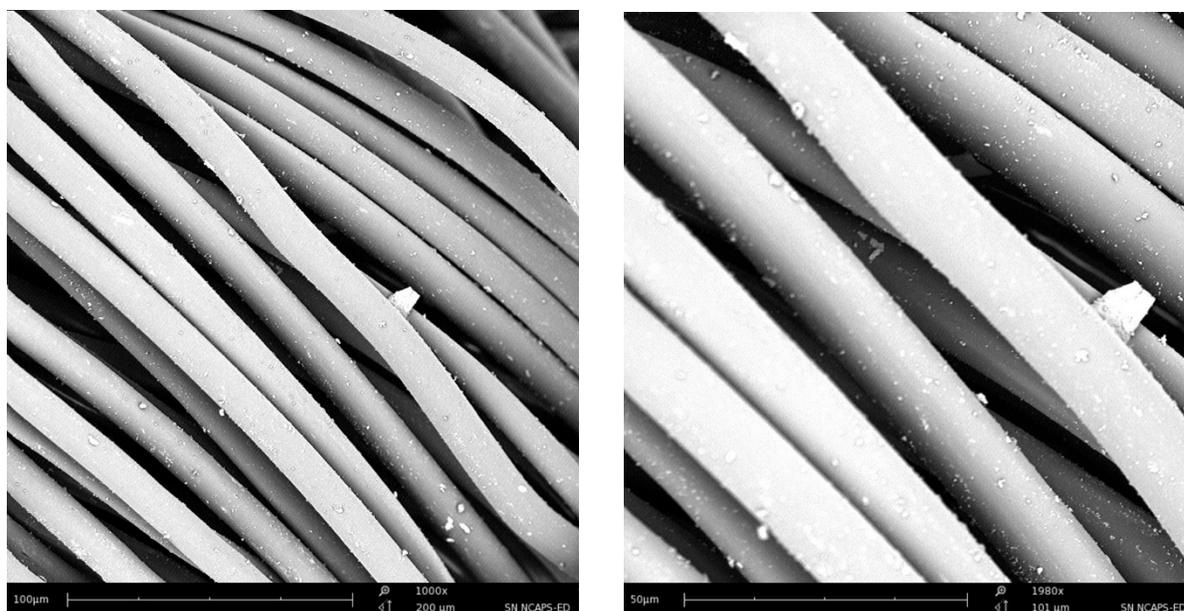


a)

b)

**Figure 4.10 SEM of Nitric acid pretreated woven spun nylon 6,6 with electroless deposition
a) 1000x b) 2000x**

A thin copper layer is visible on the surface of the woven spun nylon 6,6 sample with electroless deposition. Small copper aggregates are visible under 2000x magnification. The surface of the nitric acid pretreated sample with electroless deposition is a different color than its counterpart without electroless deposition.

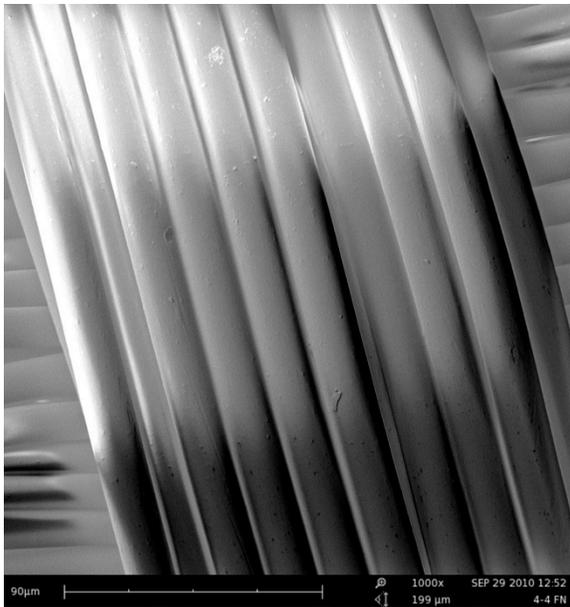


a)

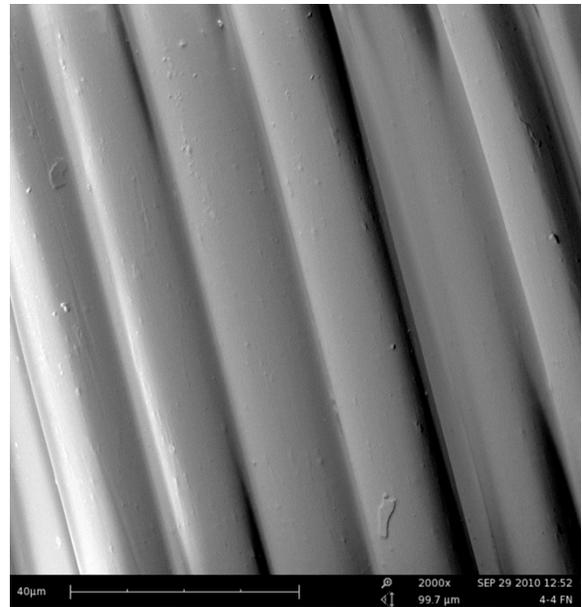
b)

**Figure 4.11 SEM of NCAPS pretreated woven spun nylon 6,6 with electroless deposition
a) 1000x b) 2000x**

Particles of copper are visible on the surface of NCAPS pretreated woven spun nylon 6,6 with electroless deposition. These aggregates are visible in both 1000x and 2000x magnification. Aggregate clusters of copper, visible on the surface of NCAPS pretreated woven spun nylon 6,6 with electroless deposition, indicate that a layer of copper was deposited on the fabric surface. The roughened surface present on NCAPS pretreated spun nylon 6,6 gave increased area for palladium catalyst to attach. More palladium catalyst on the fabric gives copper more areas to attach, thus increasing copper uptake.



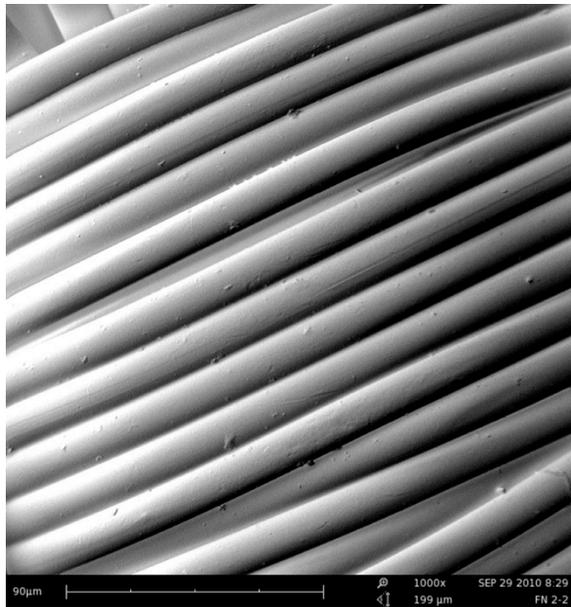
a)



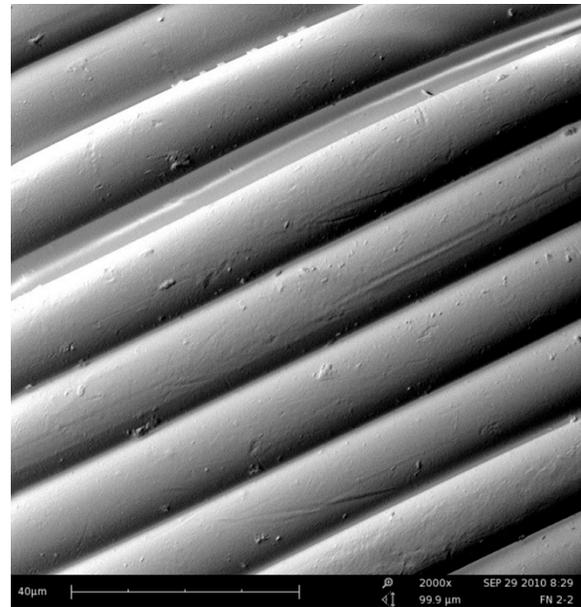
b)

**Figure 4.12 SEM of Untreated woven filament nylon 6,6
a) 1000x b) 2000x**

Untreated woven filament nylon 6,6 fibers appear smooth and round. Under 2000x magnification a few minor blemishes are visible on the surface of the fibers. Imperfections noted include several bumps present on the surface of the fibers. These bumps are attributed to the manufacturing and packaging process.



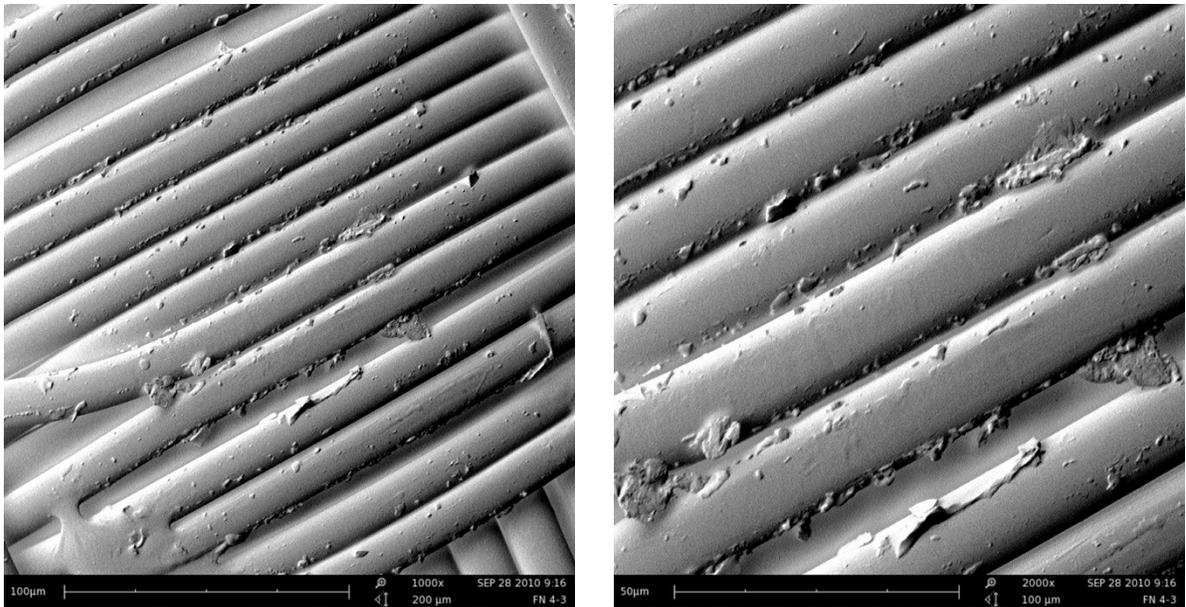
a)



b)

**Figure 4.13 SEM of Nitric acid pretreated woven filament nylon 6,6
a) 1000x b) 2000x**

After being pretreated with nitric acid, the surface of the woven filament nylon 6,6 fabric is smooth. A few bumps are present on the surface of individual fibers. Bumps observed are similar to those seen on the untreated sample. Nitric acid pretreatment didn't etch the surface of woven filament nylon 6,6 fabric.

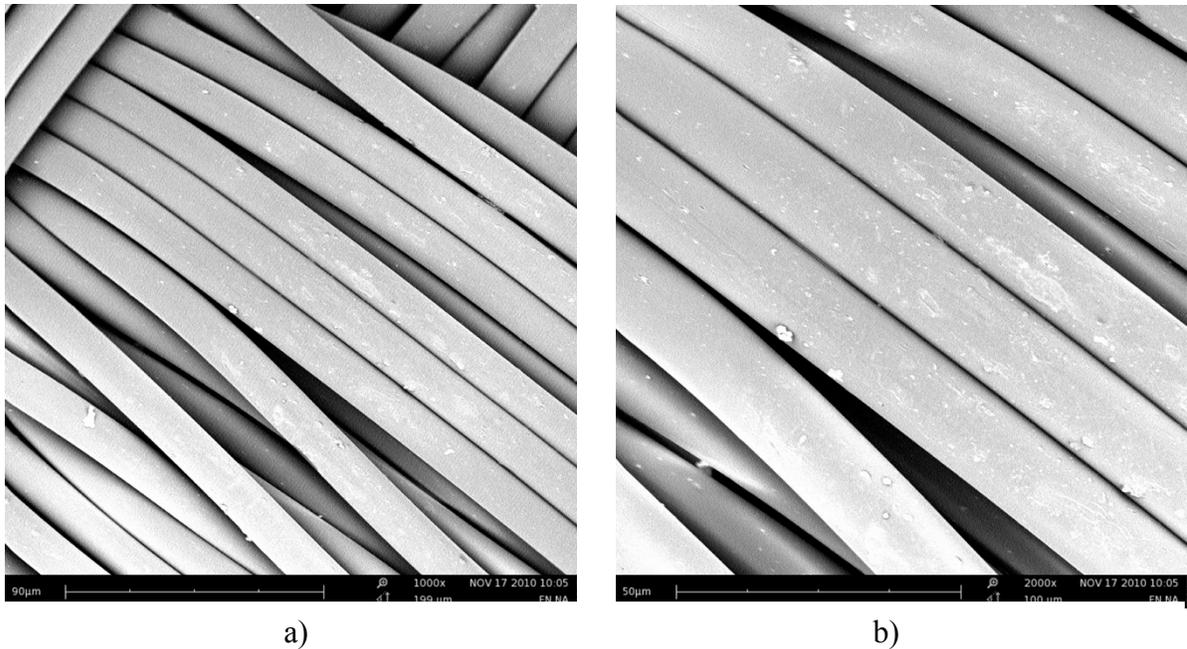


a)

b)

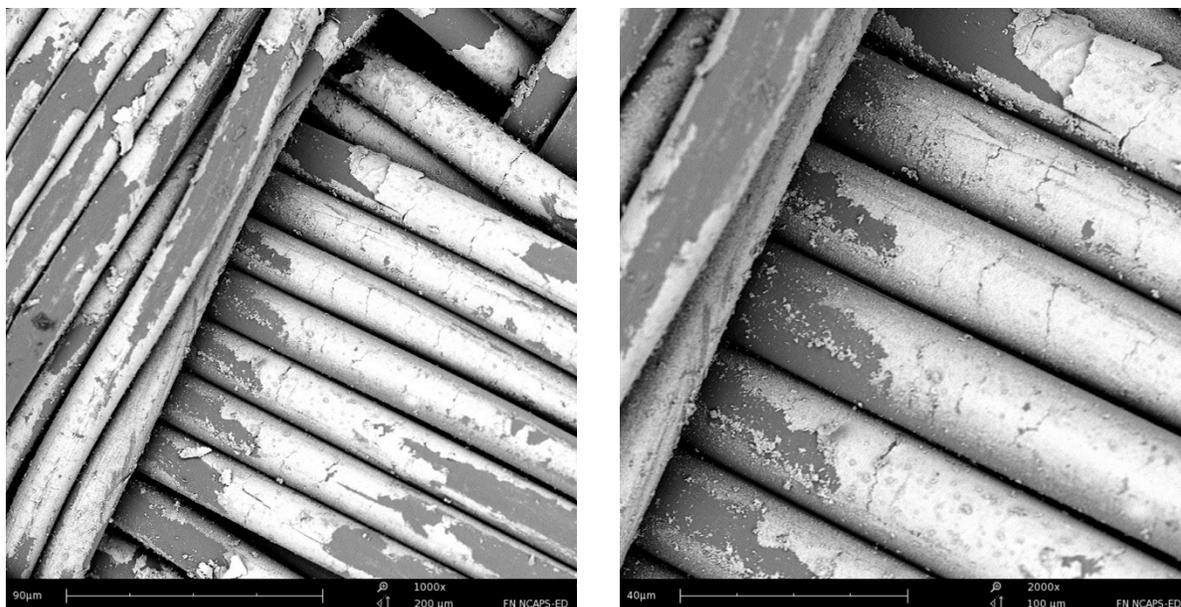
**Figure 4.14 SEM of NCAPS pretreated woven filament nylon 6,6
a) 1000x b) 2000x**

The NCAPS pretreated woven filament nylon 6,6 sample has a rough surface, in comparison to untreated and nitric pretreated samples. Pieces of nylon 6,6 polymer were distorted during NCAPS treatment. This can be seen in both 1000x and 2000x magnification. The NCAPS runs at 20-25°C (Cornelius, 2009). Therefore, nylon 6,6 polymer didn't melt, as the melting point of nylon 6,6 is 265°C (Mutz, 1974). The surface of NCAPS pretreated woven filament nylon 6,6 was distorted through plasma energy.



**Figure 4.15 SEM of Nitric acid pretreated woven filament nylon 6,6 with electroless deposition
a) 1000x b) 2000x**

Nitric acid pretreated woven filament nylon 6,6 fabric, with electroless deposition, has a smooth surface, like nitric acid pretreated woven nylon 6,6 fabric without electroless deposition. However, there is discoloration present on the fiber surface, particularly noticeable in 2000x magnification, that wasn't visible in the nitric acid pretreated sample without electroless deposition. Discoloration present is attributed to a thin layer copper present on the fiber surface.

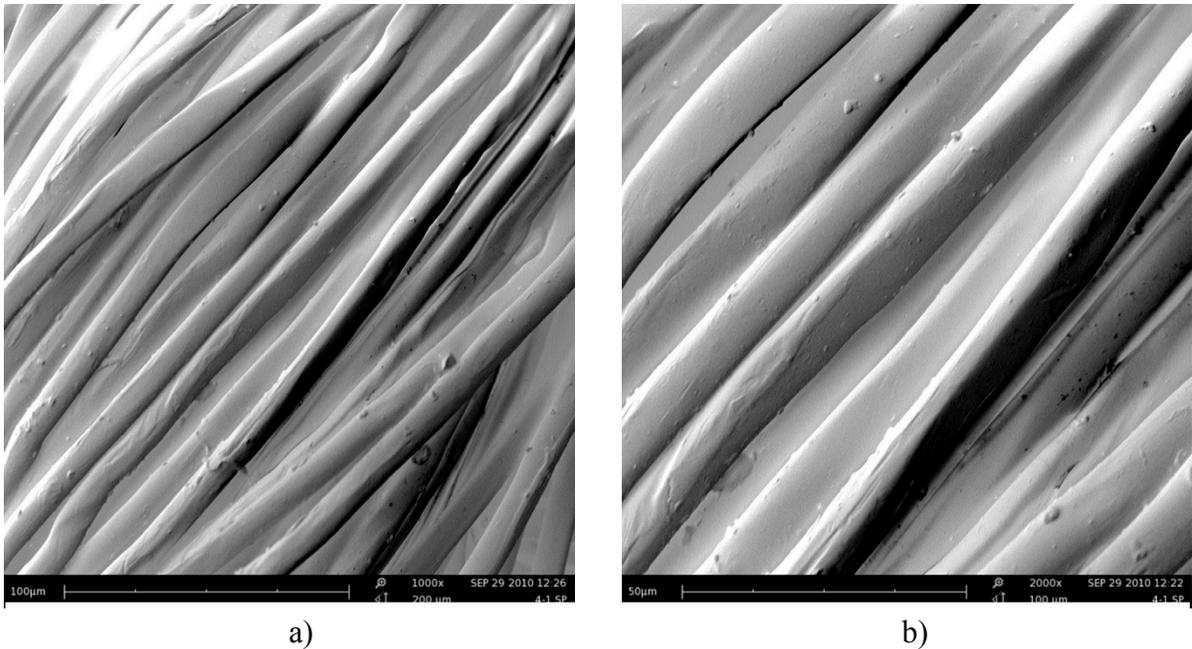


a)

b)

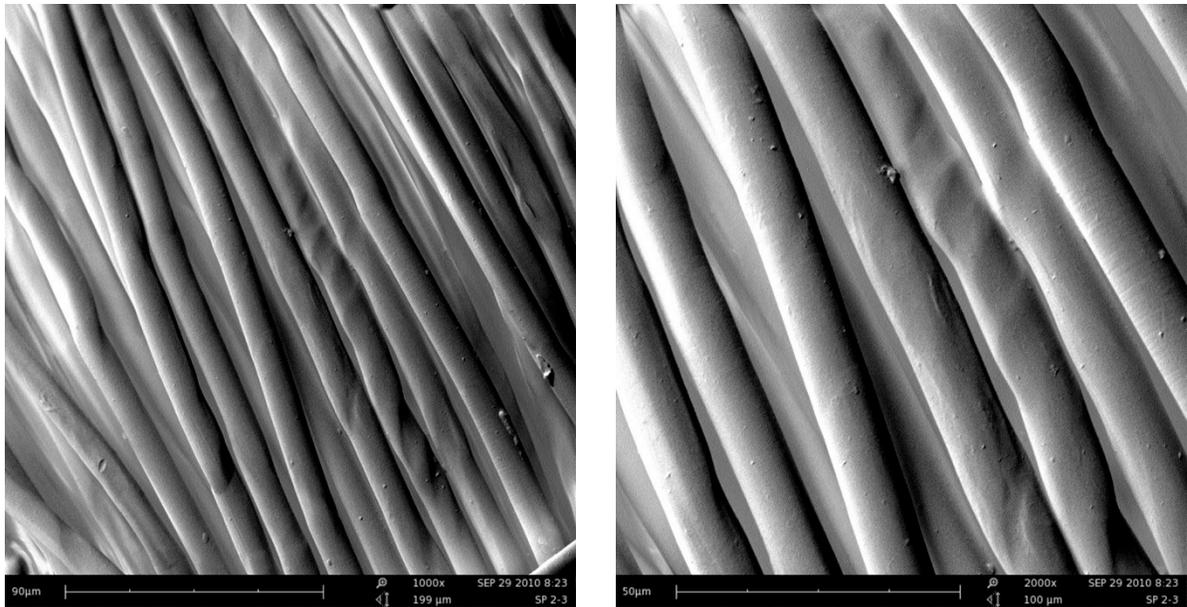
**Figure 4.16 SEM of NCAPS pretreated woven filament nylon 6,6 with electroless deposition
a) 1000x b) 2000x**

A copper layer is visible on the surface of NCAPS pretreated woven filament nylon 6,6. The coating of copper isn't uniform, as depicted under 1000x and 2000x magnification. Some areas of the fiber surface appear to have more copper than other areas. More copper is visible on the surface of NCAPS pretreated woven filament nylon 6,6 with electroless deposition, than the nitric acid pretreated nylon 6,6 with electroless deposition. Increased etching gives more area for palladium catalyst to attach. More palladium catalyst is linked to improved copper uptake on NCAPS pretreated woven filament nylon 6,6.



**Figure 4.17 SEM of Untreated woven spun polyester
a) 1000x b) 2000x**

While the surface of untreated woven spun polyester is mostly smooth, it contains several imperfections, including bumps and striations. These are particularly visible at 2000x magnification. Since these imperfections are present on the untreated sample, they occurred during the manufacturing process.

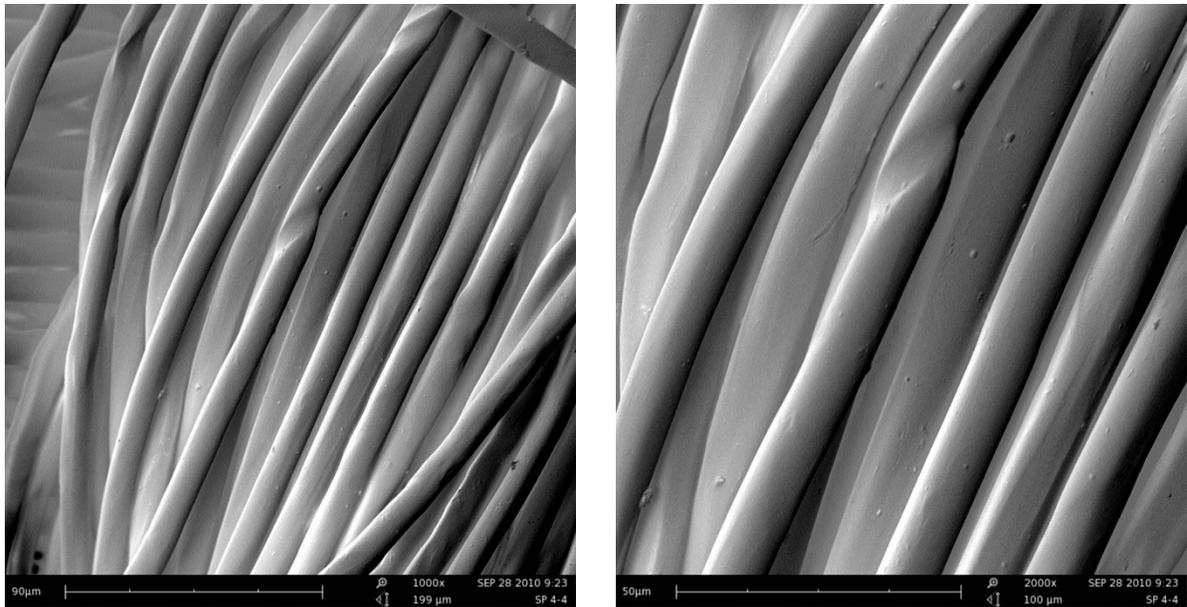


a)

b)

**Figure 4.18 SEM of Nitric acid pretreated woven spun polyester
a) 1000x b) 2000x**

Etching woven spun polyester with nitric acid did not affect the surface morphology. When compared to untreated woven spun polyester fibers, nitric acid pretreated woven spun polyester has the same surface morphology. Several bumps and rough spots are present on the surface of the fibers. These are identical to those observed on the untreated sample and are attributed to manufacturing. Nitric acid pretreatment didn't etch the surface of woven spun polyester fabric.

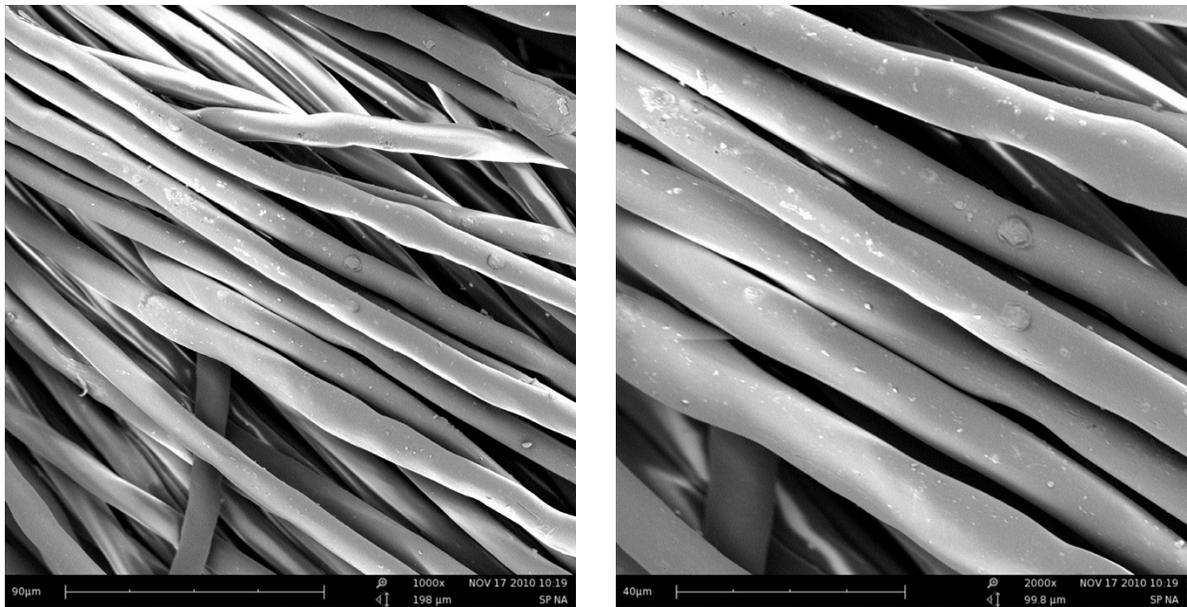


a)

b)

**Figure 4.19 SEM of NCAPS pretreated woven spun polyester
a) 1000x b) 2000x**

As observed with the nitric acid pretreated woven spun polyester, NCAPS pretreated woven polyester appears the same as the untreated sample. There are several imperfections across the surface of the NCAPS woven spun polyester, mostly bumps. These are apparent at 2000x magnification. Overall, the surface of the NCAPS pretreated woven spun polyester is smooth and nearly identical to the surface of untreated, and nitric acid pretreated samples. The surface of woven spun polyester wasn't etched by NCAPS treatment.

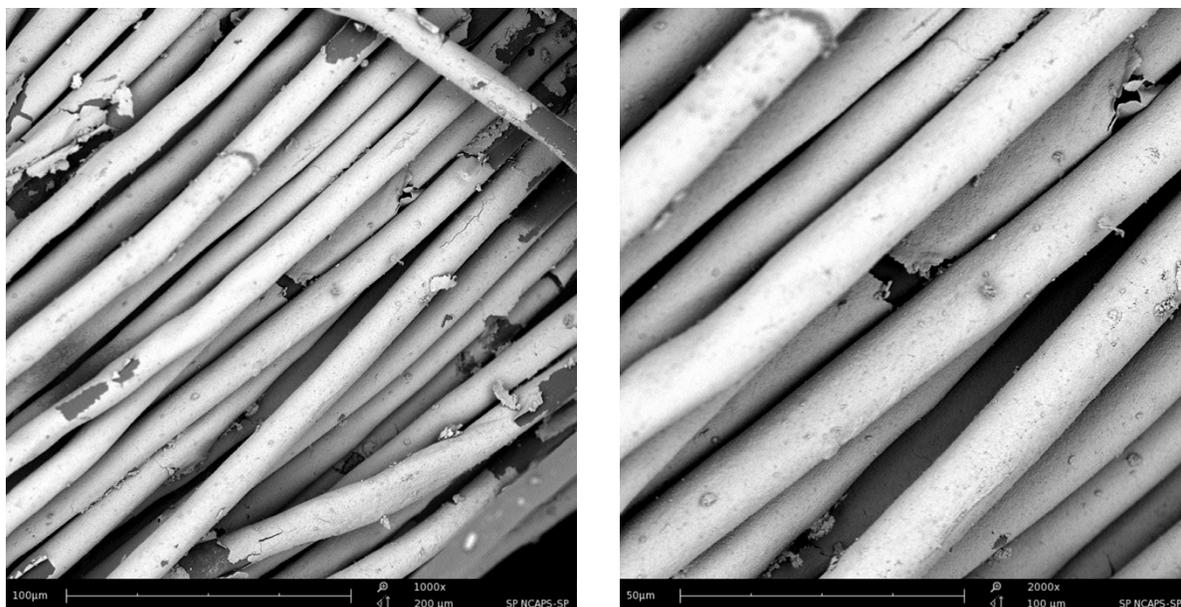


a)

b)

**Figure 4.20 SEM of Nitric acid pretreated woven spun polyester with electroless deposition
a) 1000x b) 2000x**

Though the nitric acid pretreated woven spun polyester with electroless deposition has a smooth surface, the color of the surface isn't uniform. This shift in color, evidenced by visible spots on the surface of the fabric under 2000x magnification, indicates that copper is present on the surface of the sample.

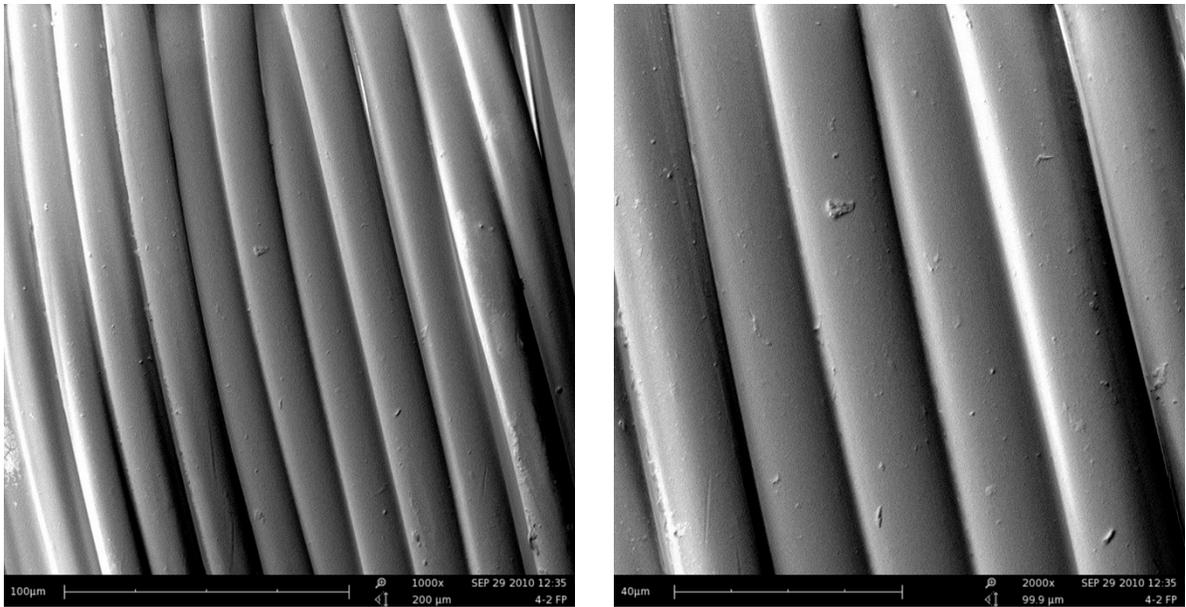


a)

b)

**Figure 4.21 SEM of NCAPS pretreated woven spun polyester with electroless deposition
a) 1000x b) 2000x**

A visible layer of copper is present on the surface of NCAPS pretreated woven spun polyester with electroless deposition. The finish is especially apparent, as several portions have peeled off, exposing the polyester fiber beneath. Compared to nitric acid pretreated woven spun polyester with electroless deposition, NCAPS pretreated fabric with electroless deposition has more copper present on the surface sample, as evidenced by SEM images. Increased copper deposition on the surface of NCAPS pretreated woven spun polyester with electroless deposition can be attributed to etching. Etching adds surface area for palladium catalyst to attach during the activation stage. Thus, improved copper uptake is observed.

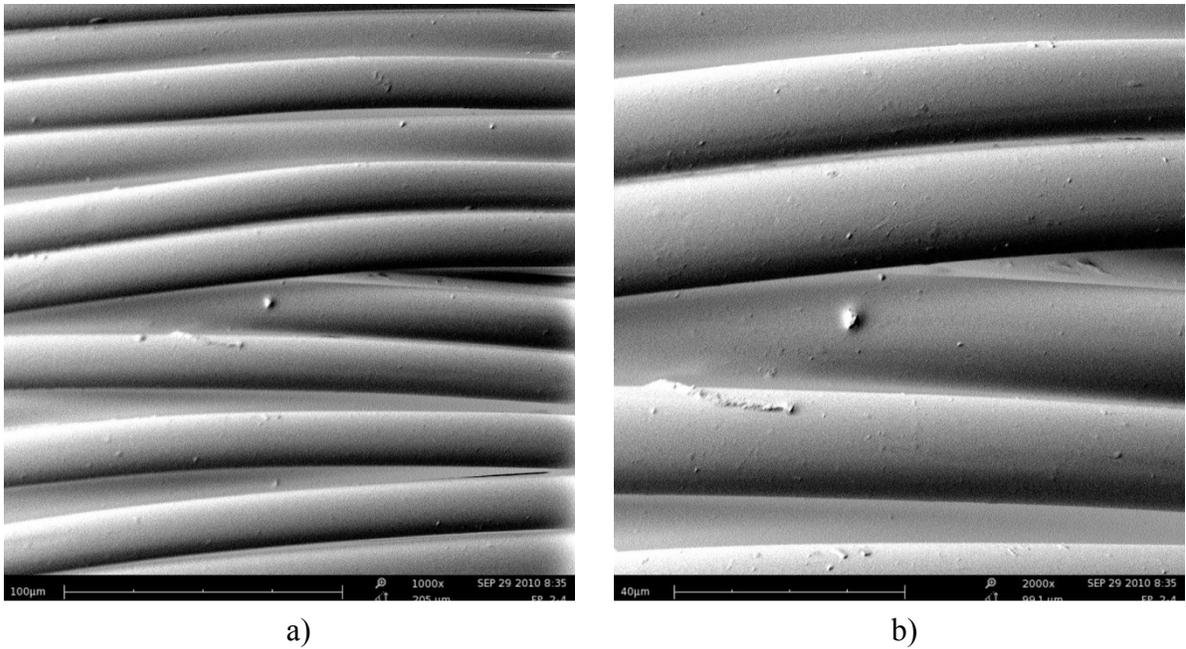


a)

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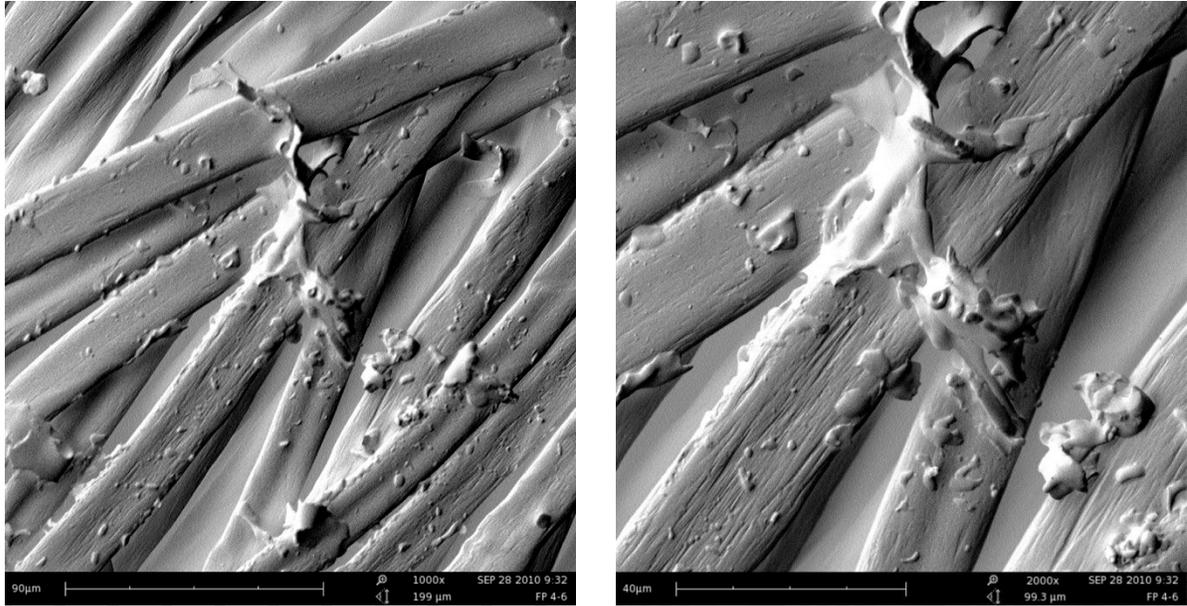
**Figure 4.22 SEM of Untreated woven filament polyester
a) 1000x b) 2000x**

Untreated woven filament polyester fibers are smooth and oriented. There are small bumps and nicks evident under 2000x magnification. These imperfections occurred during the manufacturing process, as the sample is untreated.



**Figure 4.23 SEM of Nitric acid pretreated woven filament polyester
a) 1000x b) 2000x**

Nitric acid pretreated woven filament polyester appears identical to the untreated sample. Overall, the surface is smooth. However, small imperfections, such as bumps, are present on the surface of the nitric acid pretreated sample. Similar blemishes were present on the untreated sample. Nitric acid pretreatment didn't affect the surface of woven filament polyester.

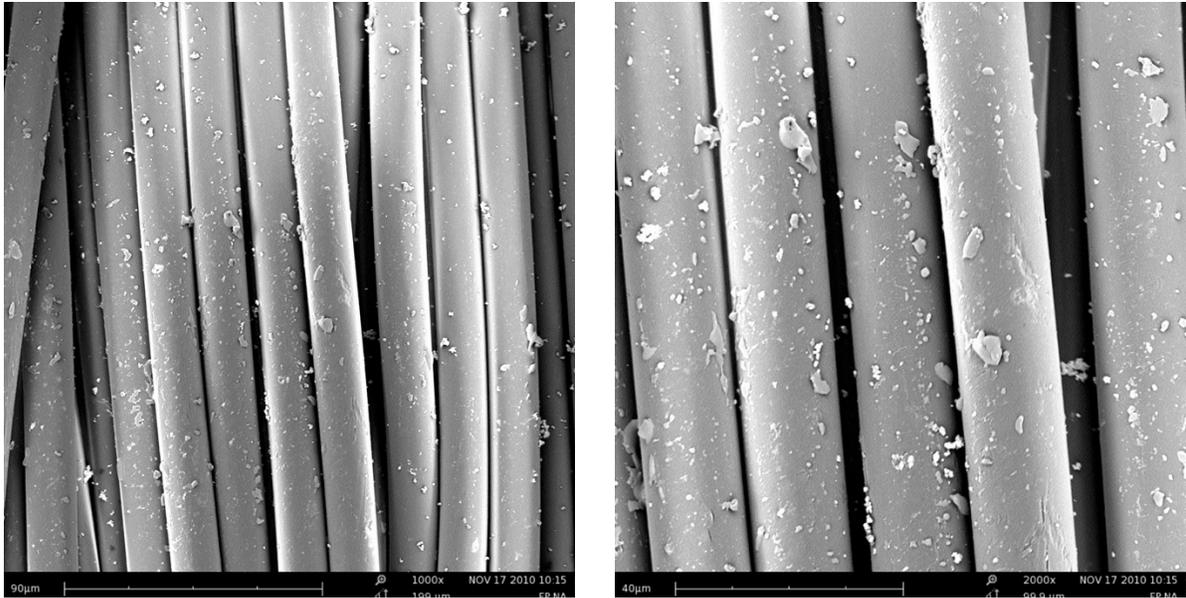


a)

b)

**Figure 4.24 SEM of NCAPS pretreated woven filament polyester
a) 1000x b) 2000x**

NCAPS pretreated woven filament polyester is severely etched and pitted, in comparison to the untreated sample. Many striations are present on the surface of the fibers. It is evident in the 1000x and 2000x SEM image that polymer has been peeled back from the surface of the fiber. As with nylon 6,6 fabrics, the woven filament polyester fabric wasn't melted by plasma pretreatment, because the melting point of polyester is between 245 and 264°C (Ludewig and Roth, 1971). The NCAPS operates between 25-30°C (Cornelius, 2009).

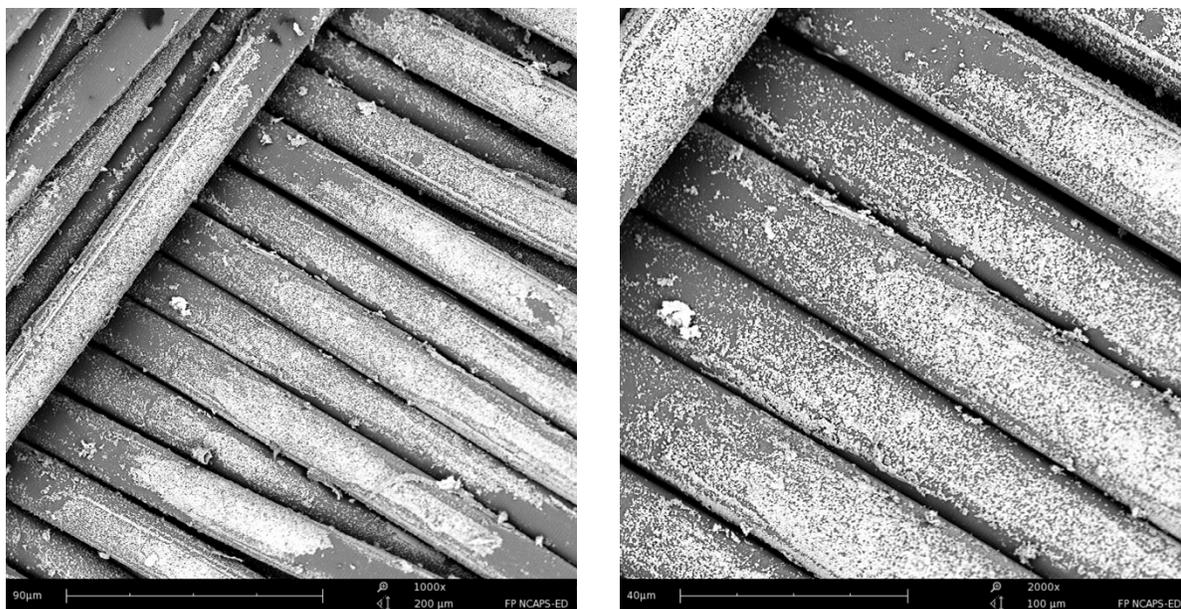


a)

b)

**Figure 4.25 SEM of Nitric acid pretreated woven filament polyester with electroless deposition
a) 1000x b) 2000x**

Nitric acid pretreated woven filament polyester with electroless deposition has a different surface morphology, compared to the sample with nitric acid pretreatment. Nitric acid pretreated woven filament polyester with electroless deposition has small flecks of copper visible on the fiber surface, especially under 2000x magnification. This indicates the presence of a copper layer on the fabric surface.

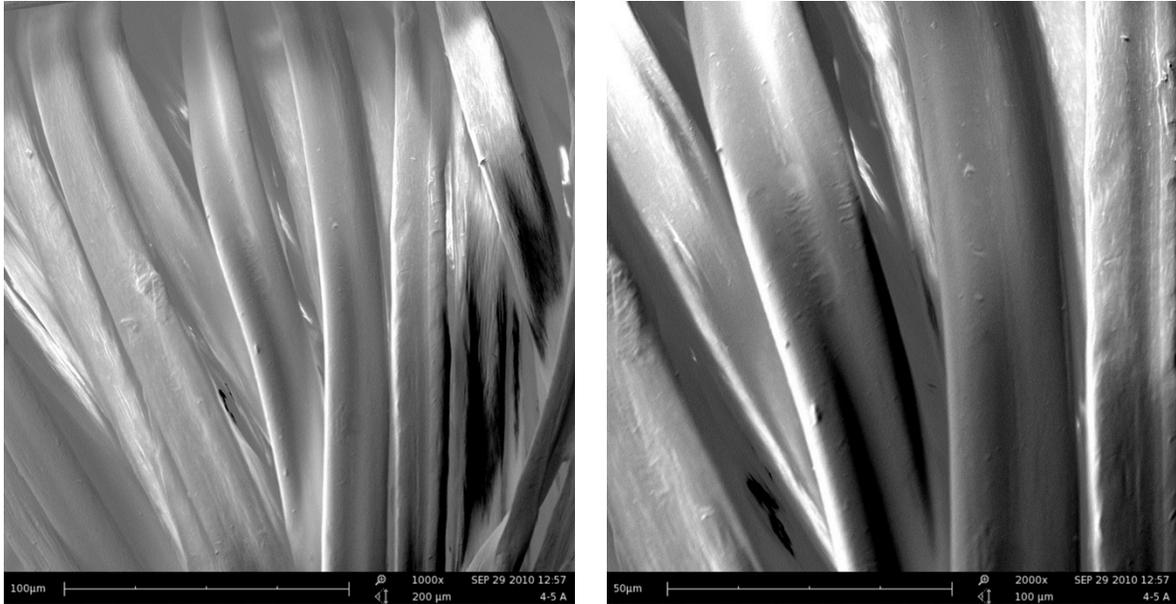


a)

b)

**Figure 4.26 SEM of NCAPS pretreated woven filament polyester with electroless deposition
a) 1000x b) 2000x**

Copper is present on the surface of NCAPS pretreated woven filament polyester with electroless deposition. The copper layer on NCAPS pretreated woven filament polyester isn't uniform and is granular in nature. The copper coating isn't uniform, as some areas of the fabric contain more copper than others. NCAPS pretreatment etched the fiber surface, which gave palladium catalyst increased surface area to attach. Thus, increased copper uptake is present on NCAPS pretreated woven filament polyester with electroless deposition compared to nitric acid pretreated woven filament polyester.

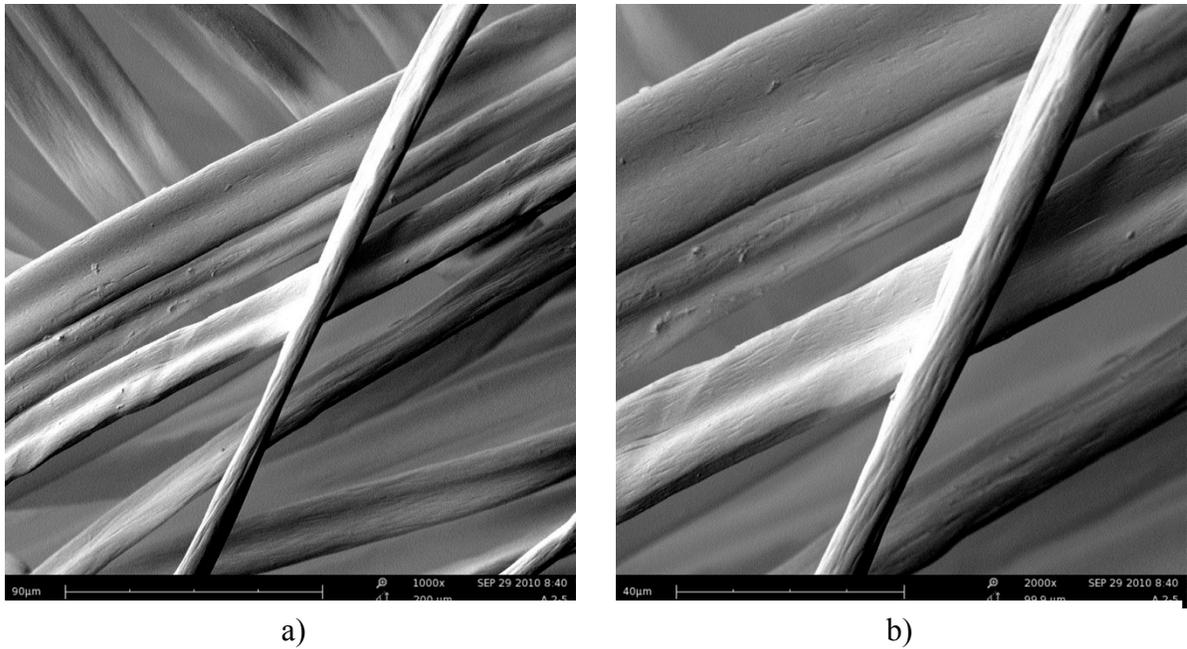


a)

b)

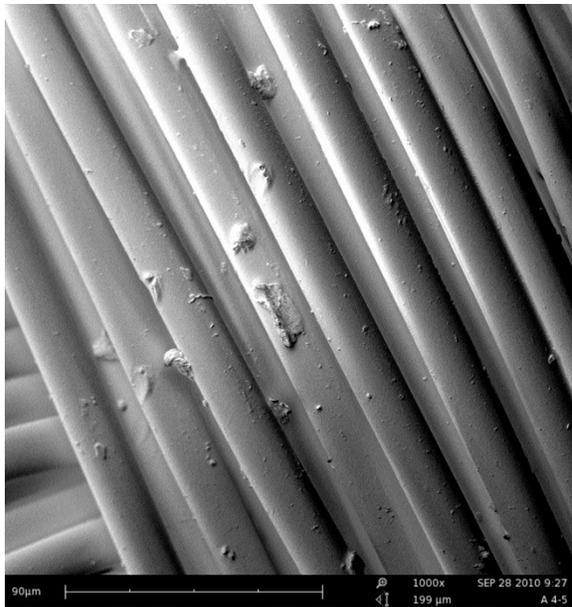
**Figure 4.27 SEM of Untreated woven spun acrylic
a) 1000x b) 2000x**

Untreated woven spun acrylic samples are “ribbon like” in structure. The surface of untreated woven spun acrylic is relatively smooth. Small ridges can be seen at 2000x magnification, running horizontal across the fiber surface.

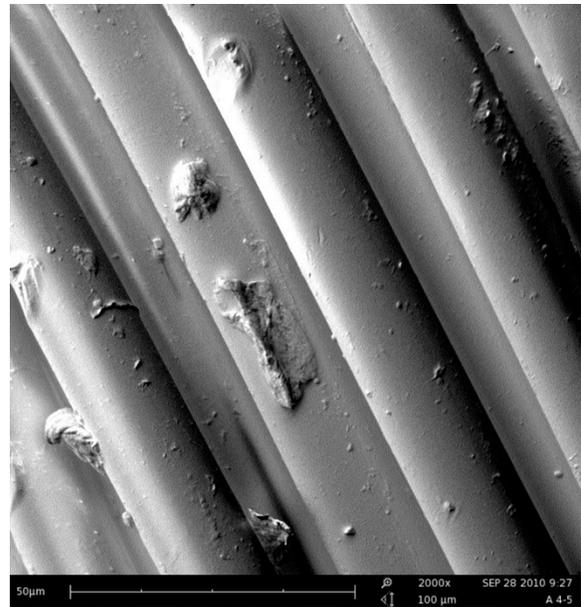


**Figure 4.28 SEM of Nitric acid pretreated woven spun acrylic
a) 1000x b) 2000x**

Nitric acid pretreated woven acrylic has striations on the surface, that weren't present on the untreated sample. Striations run vertically, with respect to the fiber orientation. This is evident in 2000x magnification.



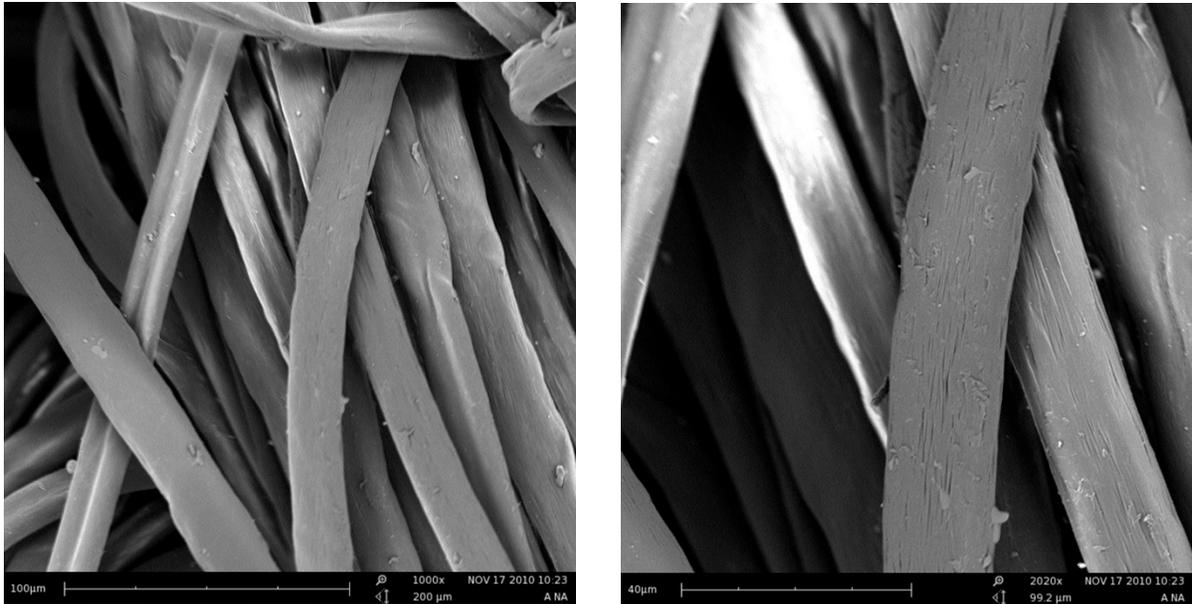
a)



b)

**Figure 4.29 SEM of NCAPS pretreated woven spun acrylic
a) 1000x b) 2000x**

The surface of the NCAPS pretreated woven spun acrylic sample is rougher than untreated woven spun acrylic. NCAPS pretreated woven spun acrylic has raised spots present on the fiber surface. Raised spots aren't present on untreated fibers. Thus, raised spots can be attributed to NCAPS pretreatment.

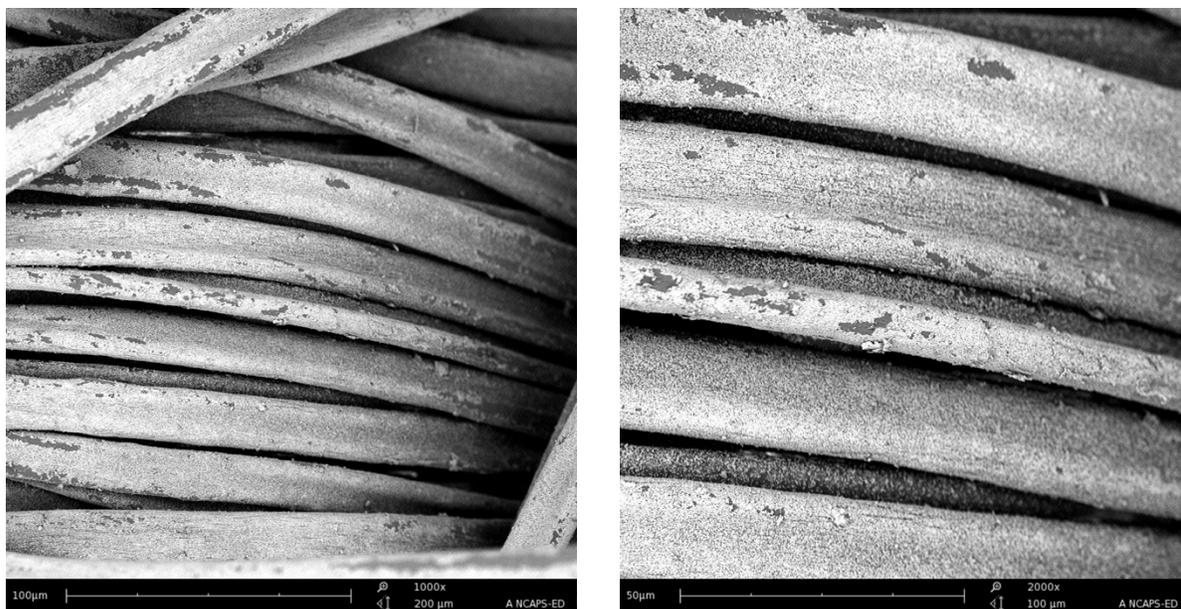


a)

b)

**Figure 4.30 SEM of Nitric acid pretreated woven spun acrylic with electroless deposition
a) 1000x b) 2000x**

Nitric acid pretreated woven spun acrylic with electroless deposition has different surface morphology, in comparison to the nitric acid pretreated sample and no electroless deposition. The striations present in the nitric acid pretreated sample with electroless deposition are less defined, than in the sample without electroless deposition. Striations are less apparent because a thin layer of copper is present on the fabric surface.

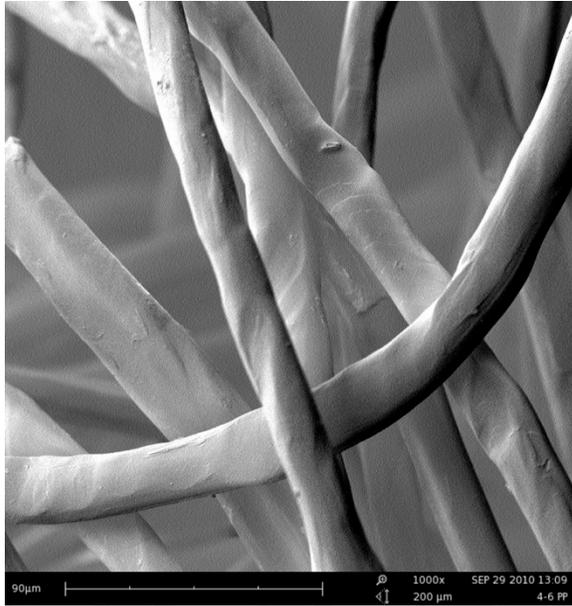


a)

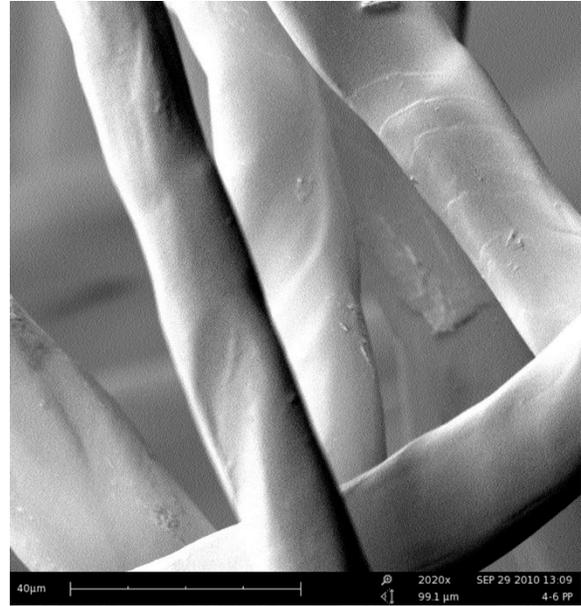
b)

**Figure 4.31 SEM of NCAPS pretreated woven spun acrylic with electroless deposition
a) 1000x b) 2000x**

Copper is visible on the surface of NCAPS pretreated spun acrylic. Copper present on the fabric is mostly uniform. A few areas on the fabric surface are not uniform, as a result of un-level deposition or peeling. Copper uptake on NCAPS pretreated woven spun acrylic with electroless deposition is greater than copper uptake observed on the nitric acid sample with electroless deposition. Increased copper uptake can be attributed to additional surface area due to etching from NCAPS pretreatment.



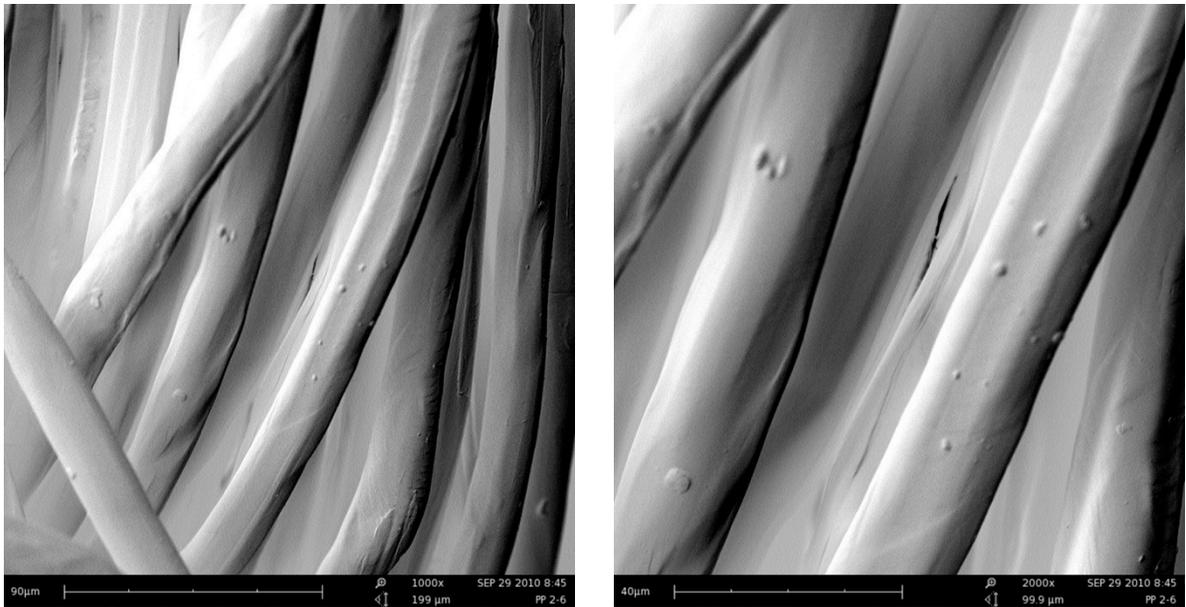
a)



b)

**Figure 4.32 SEM of Untreated woven spun polypropylene
a) 1000x b) 2000x**

Untreated woven spun polypropylene fibers are smooth in nature. Crescent shaped ridges are present on some fiber, oriented in a horizontal direction, with respect to the fiber.

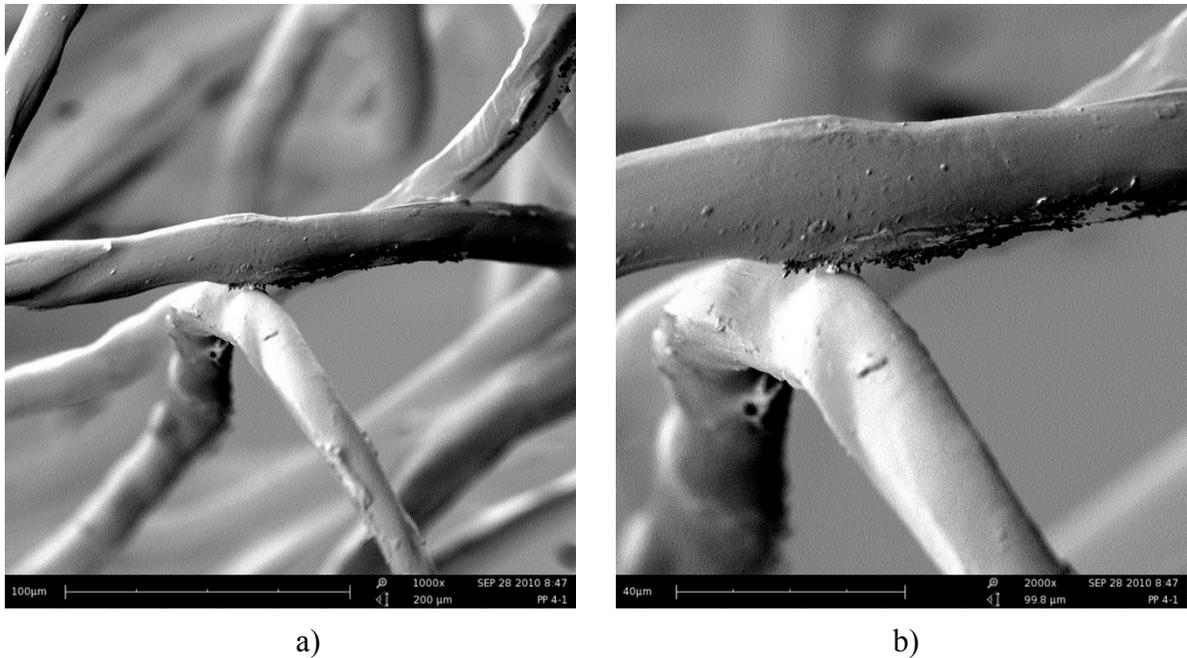


a)

b)

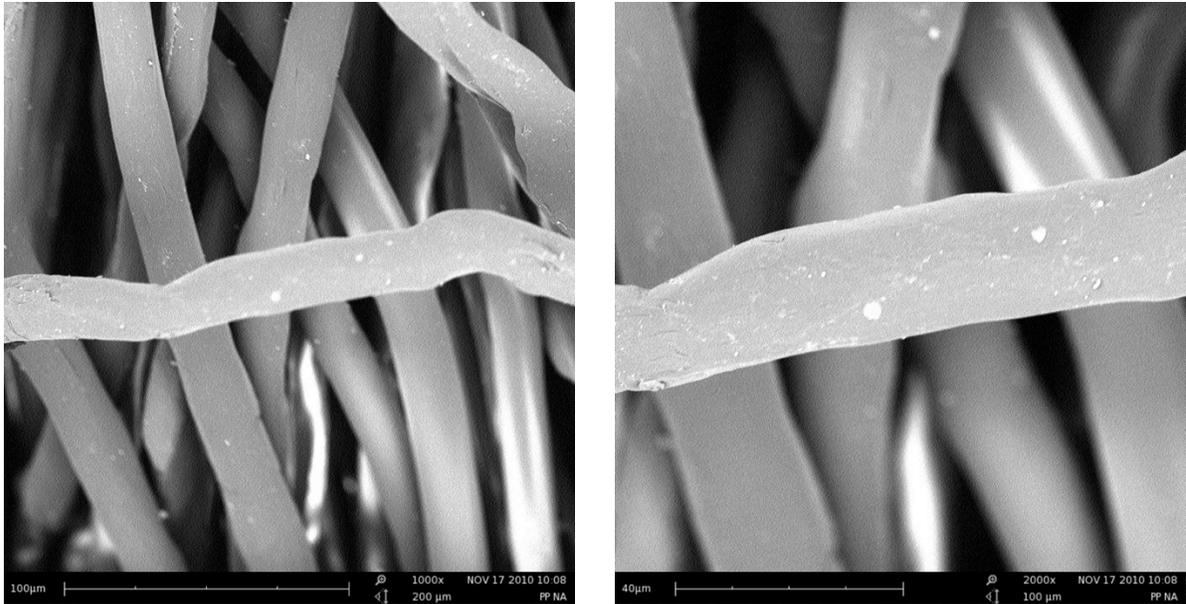
**Figure 4.33 SEM of Nitric acid pretreated woven spun polypropylene
a) 1000x b) 2000x**

The surface of untreated and nitric acid pretreated woven spun polypropylene smooth, with a few raised spots visible. Nitric acid treated spun polypropylene doesn't appear different, compared to the untreated sample.



**Figure 4.34 SEM of NCAPS pretreated woven spun polypropylene
a) 1000x b) 2000x**

NCAPS pretreated woven spun polypropylene is rougher than untreated or nitric acid pretreated samples. This is especially evident on the underside of the fiber running from left-to-right in the 2000X SEM image. Small bumps observed in the untreated and nitric acid pretreated samples are still visible after treatment with the NCAPS. Though some surface etching is present on spun polypropylene, it isn't as apparent as other fibers. This can be attributed to how tough the surface of polypropylene is, compared to other fibers studied in this research.

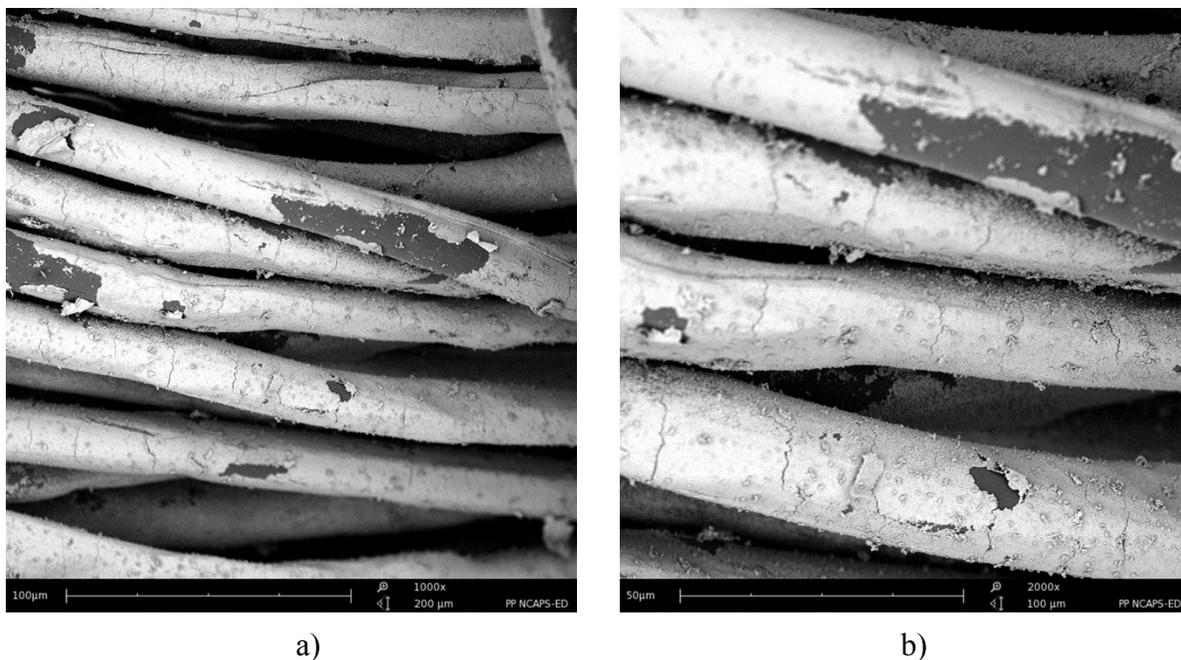


a)

b)

**Figure 4.35 SEM of Nitric acid pretreated woven spun polypropylene with electroless deposition
a) 1000x b) 2000x**

Nitric acid pretreated woven spun polypropylene fibers with electroless deposition changed color, in comparison to the polypropylene without electroless deposition. This can be attributed to nanolayers of copper present on the fiber surface. Small flecks of copper are visible on the surface of nitric acid pretreated woven spun polypropylene with electroless deposition, particularly at 2000x magnification.



**Figure 4.36 SEM of NCAPS pretreated woven spun polypropylene with electroless deposition
a) 1000x b) 2000x**

A layer of copper is present on NCAPS pretreated woven spun polypropylene with electroless deposition. The copper layer is rough, with cracks visible, particularly under 2000x magnification. Portions of the copper layer peeled off the fiber surface, as observed under 1000x and 2000x magnification. Though etching wasn't as apparent with NCAPS pretreated polypropylene, more etching was present on the NCAPS pretreated sample, than with nitric acid sample. This means there are more areas for palladium catalyst to attach. Hence why more copper uptake was associated with NCAPS pretreated woven spun polypropylene with electroless deposition, than with nitric acid pretreated woven spun polypropylene with electroless deposition.

4.6 Elemental analysis using inductively coupled plasma

Elemental analysis was conducted on all fabric samples in order to quantify the amount of copper present on samples. Inductively coupled plasma (ICP) was used for elemental analysis. **Figures 4.37-4.42** depict the amount of copper present on all samples, in mg/kg. Untreated samples served as a baseline, for which nitric acid pretreated samples with electroless deposition and NCAPS pretreated samples with electroless deposition were compared against.

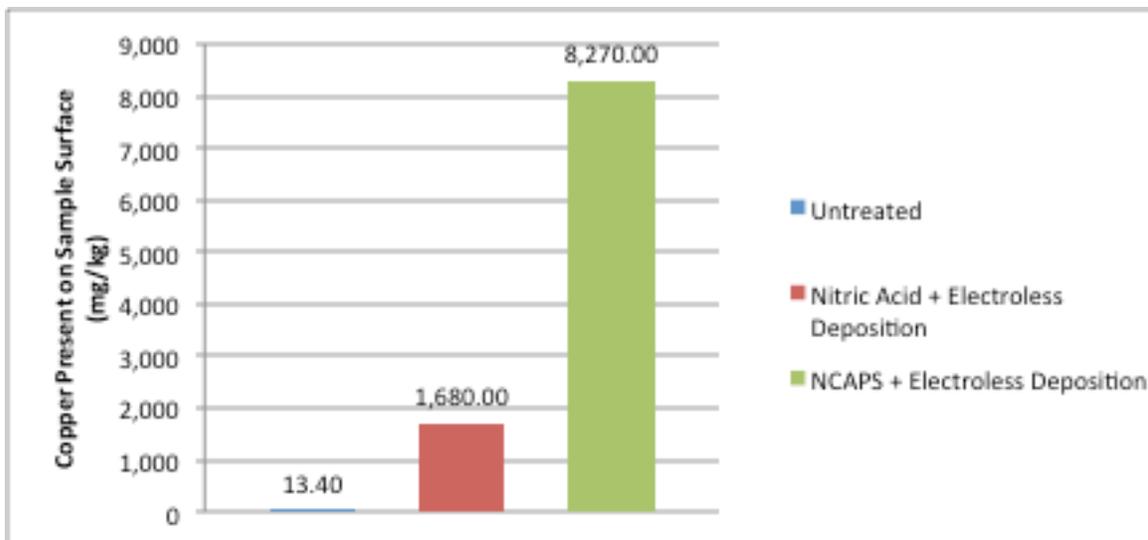


Figure 4.37 Amount of copper (mg/kg) present on untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition woven spun nylon 6,6

Untreated woven spun nylon 6,6 had the least amount of copper present, with 13.40 mg/kg. Nitric acid pretreated woven spun nylon 6,6 had 1,680 mg/kg of copper present on the fabric surface. NCAPS pretreated woven spun nylon 6,6 had 8,270 mg/kg of copper present. Nitric acid pretreated woven spun nylon 6,6 with electroless deposition had 125 times more copper present on the sample surface than untreated woven spun nylon 6,6. Meanwhile, NCAPS pretreated woven spun nylon 6,6 had 617 times more copper present on the surface, compared to the untreated sample. Thus, NCAPS pretreatment yielded higher copper uptake on woven spun nylon 6,6, than nitric acid pretreatment. Overall, untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition fabric samples contained 0.0000134%, 0.00168%, and 0.00827% of copper, respectively.

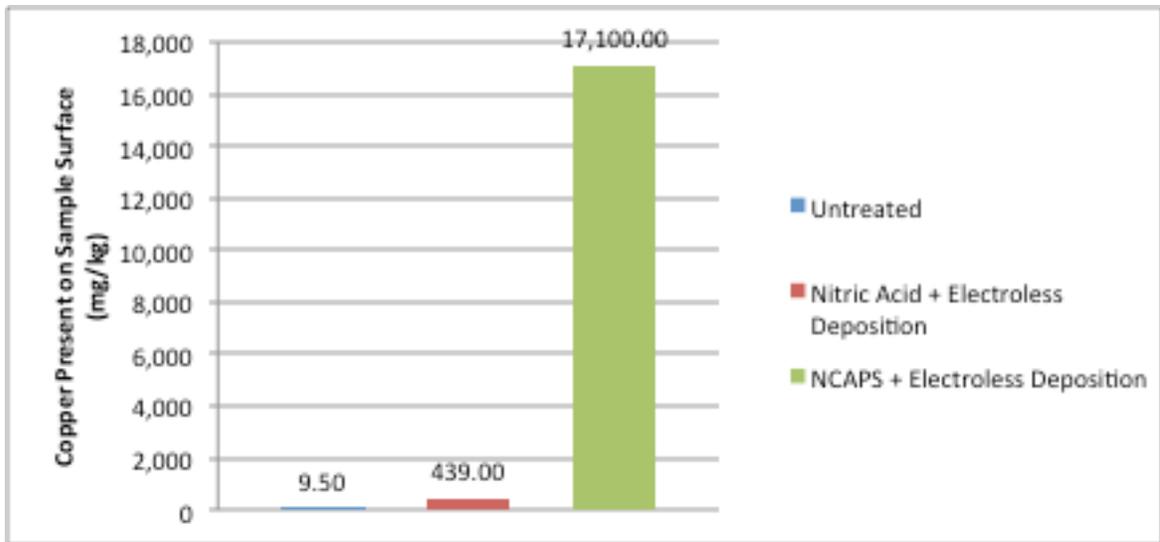


Figure 4.38 Amount of copper (mg/kg) present on untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition woven filament nylon 6,6

Untreated woven filament nylon 6,6, nitric acid pretreated woven filament nylon 6,6, and NCAPS pretreated woven filament nylon 6,6 had 9.50 mg/kg, 439 mg/kg, and 17,100 mg/kg of copper present on the surface, respectively. Compared to the untreated sample, nitric acid pretreated woven spun nylon 6,6 had 46 times more copper present on the sample. In contrast, NCAPS pretreated woven filament nylon 6,6 had 1,800 times more copper present on the sample. NCAPS pretreatment was superior to nitric acid pretreatment, in terms of copper uptake on woven filament nylon 6,6. Overall, untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition fabric samples contained 0.0000095 %, 0.000439%, and 0.0171 % of copper, respectively.

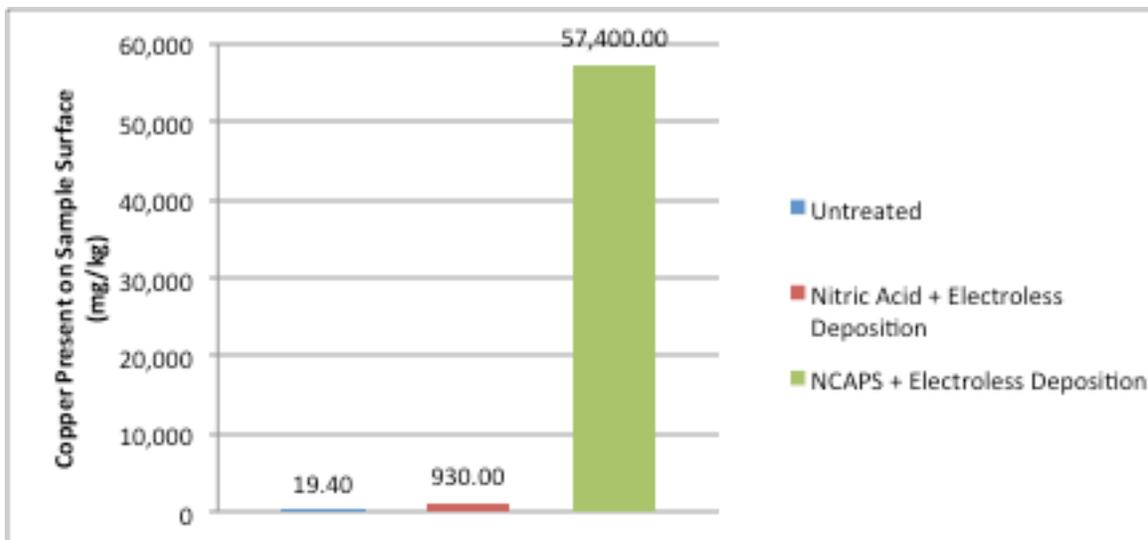


Figure 4.39 Amount of copper (mg/kg) present on untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition woven spun polyester

Untreated woven spun polyester had 19.40 mg/kg of copper present on the sample. Nitric acid pretreated woven spun polyester had 930 mg/kg of copper present on the surface. In contrast, NCAPS pretreated woven polyester had 57,400 mg/kg of copper present on the sample. Nitric acid pretreatment with electroless deposition yielded 48 times more copper uptake than the untreated sample, compared to 2,959 times more copper uptake associated with the NCAPS pretreated sample with electroless deposition. Thus, NCAPS pretreatment with electroless deposition is vastly superior to nitric acid pretreatment, with respect to the amount of copper deposited on the surface of woven spun polyester fabric. Overall, untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition fabric samples contained 0.0000194 %, 0.00093 %, and 0.0574 % of copper, respectively.

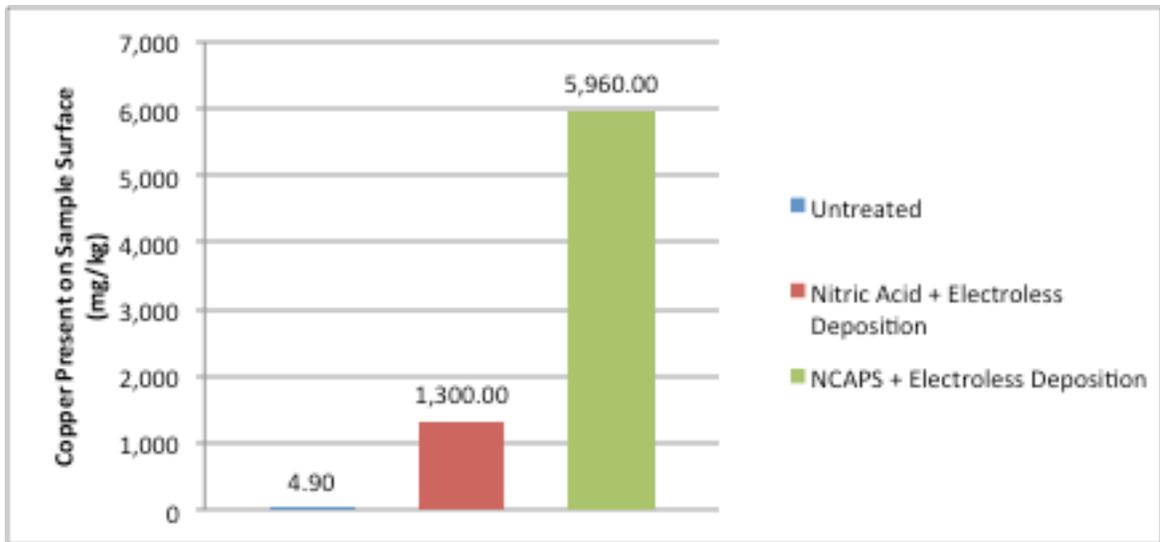


Figure 4.40 Amount of copper (mg/kg) present on untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition woven filament polyester

Untreated woven filament polyester, nitric acid pretreated woven filament polyester, and NCAPS pretreated woven filament polyester had 4.90 mg/kg, 1,300 mg/kg, and 5,960 mg/kg of copper present on the surface, respectively. Nitric acid pretreated woven filament polyester had 265 times more copper present on the sample surface, than untreated woven filament polyester. Meanwhile, NCAPS pretreated woven filament polyester had 1,216 times more copper present, than untreated woven filament polyester. Consequently, it is apparent that NCAPS pretreatment increases copper uptake on woven spun acrylic fabric, compared to nitric acid pretreatment. Overall, untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition fabric samples contained 0.0000049 %, 0.0013%, and 0.00596% of copper, respectively.

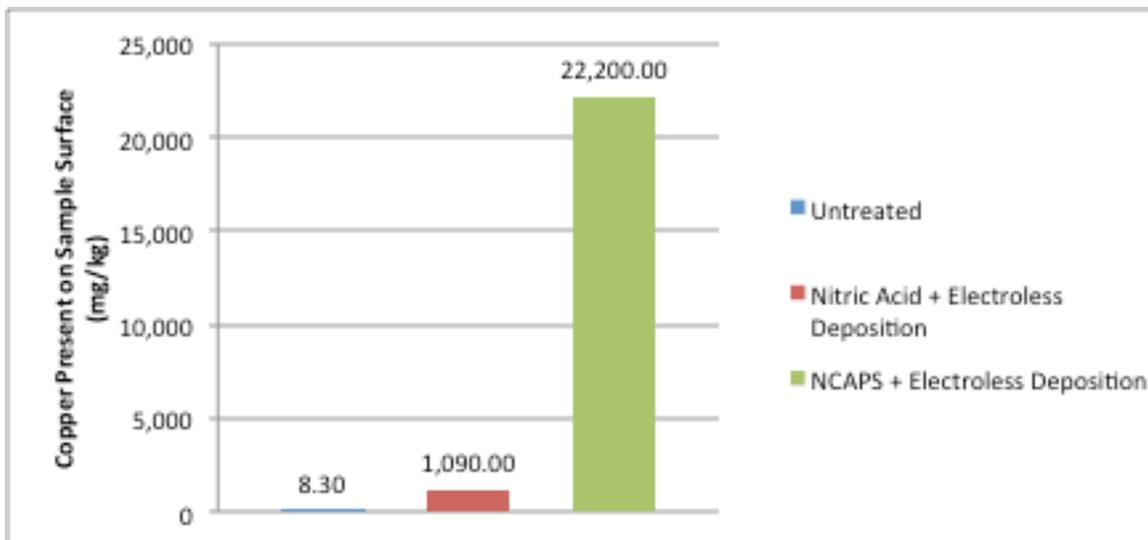


Figure 4.41 Amount of copper (mg/kg) present on untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition woven spun acrylic

Untreated woven spun acrylic had 8.30 mg/kg of copper present on the surface. In comparison, nitric acid pretreated woven spun acrylic and NCAPS pretreated woven spun acrylic had 1,090 mg/kg and 22,200 mg/kg, respectively. Nitric acid pretreated spun acrylic had 131 times more copper present on the fabric surface than the untreated sample. NCAPS pretreated woven spun acrylic had 2,675 times more copper on its fabric surface, compared to the untreated sample. Therefore, increased copper uptake was observed with NCAPS pretreated woven spun acrylic, compared to nitric acid pretreated spun woven acrylic.

Overall, untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition fabric samples contained 0.0000083%, 0.00109%, and 0.0222% of copper, respectively.

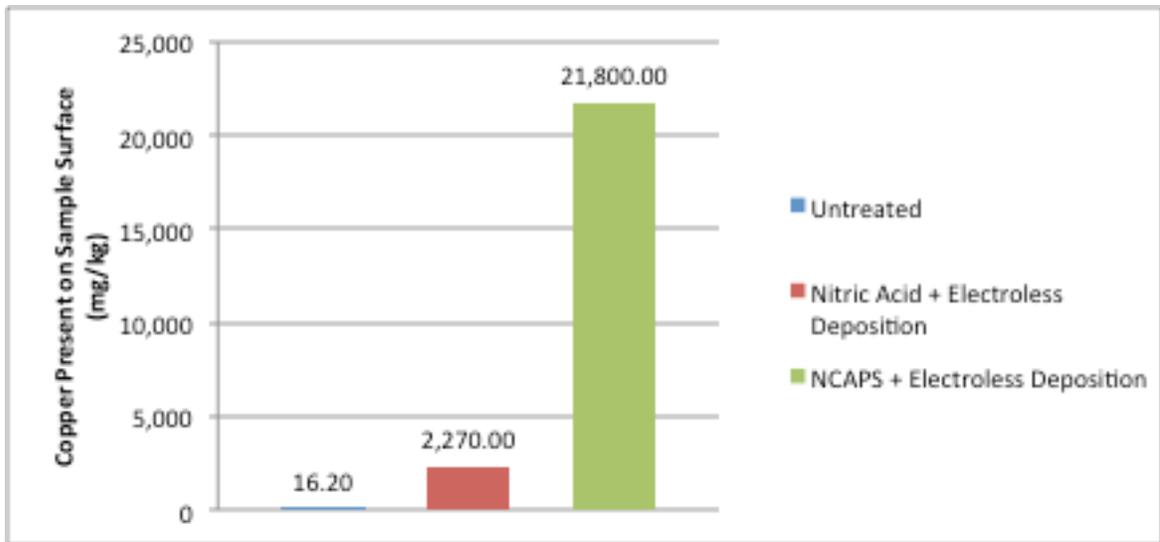


Figure 4.42 Amount of copper (mg/kg) present on untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition woven spun polypropylene

16.20 mg/kg, 2,270 mg/kg, and 21,800 mg/kg of copper were present on untreated woven spun polypropylene, nitric acid pretreated woven spun polypropylene with electroless deposition, and NCAPS pretreated woven spun polypropylene with electroless deposition, respectively. Nitric acid pretreated woven spun polypropylene with electroless deposition had 140 times more copper present on the surface than the untreated sample. In contrast, NCAPS pretreated woven spun polypropylene with electroless deposition had 1,346 times more copper present on the surface, compared to untreated woven spun polypropylene. NCAPS pretreatment provided superior copper uptake on the surface of woven spun polypropylene, compared to nitric acid pretreatment. Overall, untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition fabric samples contained 0.0000162%, 0.00227%, and 0.0218% of copper, respectively.

4.7 Electrical surface resistivity values

Surface resistivity values were taken on untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition samples. Four readings were taken across the surface of one 8 inch x 8 inch sample. Based on these readings, the mean and standard deviation was calculated. All readings greater than $1.00e^{14}$ were labeled as out of range of the measuring instrument.

	Untreated		Nitric Acid + Electroless Deposition		NCAPS + Electroless Deposition	
	Mean (Ω/sq)	Std. Dev. (Ω/sq)	Mean (Ω/sq)	Std. Dev. (Ω/sq)	Mean (Ω/sq)	Std. Dev. (Ω/sq)
Spun Nylon 6,6	Out of range	-	7.30×10^{13}	3.96×10^{13}	9.31×10^{13}	4.01×10^{13}
Filament Nylon 6,6	Out of range	-	2.40×10^{13}	1.59×10^{13}	7.45×10^{13}	3.42×10^{13}
Spun Polyester	Out of range	-	1.40×10^{13}	1.93×10^{13}	9.68×10^{13}	2.96×10^{13}
Filament Polyester	Out of range	-	0.25×10^{13}	0.42×10^{13}	3.56×10^{13}	1.98×10^{13}
Spun Acrylic	Out of range	-	0.16×10^{13}	0.17×10^{13}	2.34×10^{13}	2.45×10^{13}
Spun Polypropylene	Out of range	-	8.56×10^{13}	3.23×10^{13}	9.53×10^{13}	2.42×10^{13}

Table 4.7 Resistivity values for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition samples (n=4)

All untreated samples had resistivity values that were out of range. Once, samples were exposed to nitric acid pretreatment with electroless deposition, surface resistivity decreased for all samples, except spun polypropylene. Spun acrylic had the lowest resistivity value of all nitric acid with electroless deposition samples, followed by filament polyester. While all resistivity values for samples with nitric acid pretreatment with electroless deposition, except spun polypropylene, were better than their untreated counterparts. Samples treated with NCAPS pretreatment and electroless deposition had lower surface resistance than untreated samples. However, when compared to nitric acid pretreated samples with electroless deposition, NCAPS pretreated samples with electroless deposition had higher surface resistivity values. Though visual assessment and SEM images confirm that NCAPS pretreated fabric with electroless deposition has more copper present on the sample surface, samples don't have a uniform coating of copper. The lack of uniformity on fabric samples creates a problem when resistivity testing is conducted. Since copper is a conductive material, more copper on the surface of a fabric should indicate a lower surface resistivity. The lack of uniformity on the surface of NCAPS pretreated samples with electroless deposition served as a "disconnect" when a current was applied to the samples. The current, instead of moving through the sample, hit areas with less copper present and encountered increased resistance. Thus, explaining how samples with more copper, NCAPS pretreated fabric with electroless deposition, had higher surface resistivity than nitric acid pretreated fabrics with electroless deposition.

4.8 Contact charge and dissipation analysis

Figure 4.37 depicts an example of a charge generation and dissipation curve. Three charge generation and dissipation runs were conducted across the surface of a 8 inch by 8 inch sample. Each sample was charged 50 times with a polytetrafluoroethylene (PTFE) block during the charge generation stage. After 50 rubbings with the PTFE block, the peak voltage was recorded. The voltage was recorded again after 60 seconds of charge dissipation and the percent reduction was calculated.

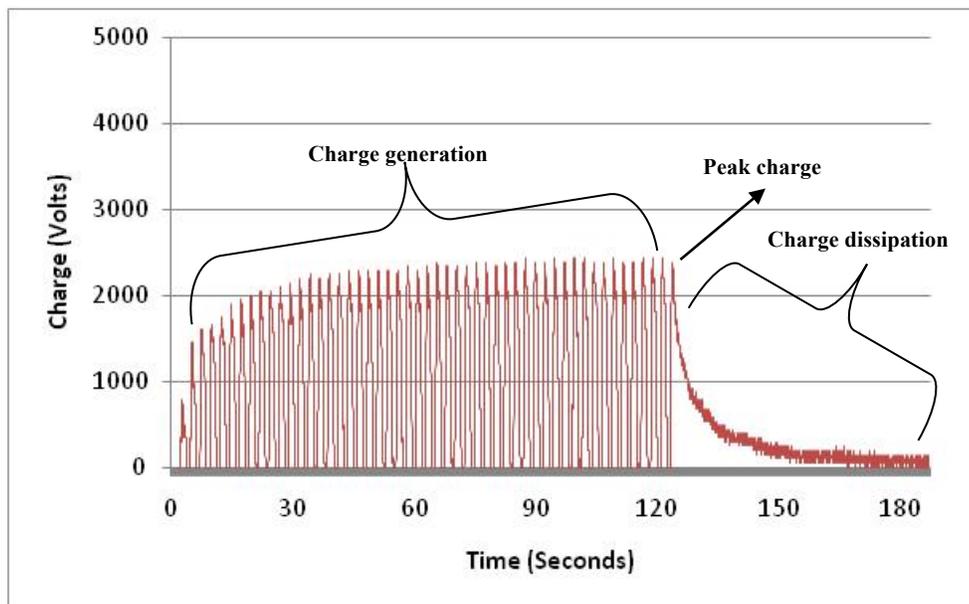


Figure 4.43 Example of a charge generation and dissipation curve

The peak charge and charge after 60 seconds are used to calculate the percent reduction.

Percent reduction is the amount of charge dissipated away from a sample over 60 seconds (in

%). Percent reduction is calculated as shown in **Equation 4.1**.

$$\% \text{ Reduction} = \left(\left(\frac{\text{Charge after 60 seconds dissipation}}{\text{Peak charge}} \right) * 100 \right) - 100$$

Equation 4.1 Percent reduction calculation

Figures 4.44-4.55 depict charge dissipation curve and percent reduction graph comparisons for all fabric samples. Peak charge, charge after 60 seconds, and percent dissipation data is noted in the analysis of **Figures 4.44-4.55**. Raw charge generation and dissipation curves for all repetitions of all samples are included in the appendix.

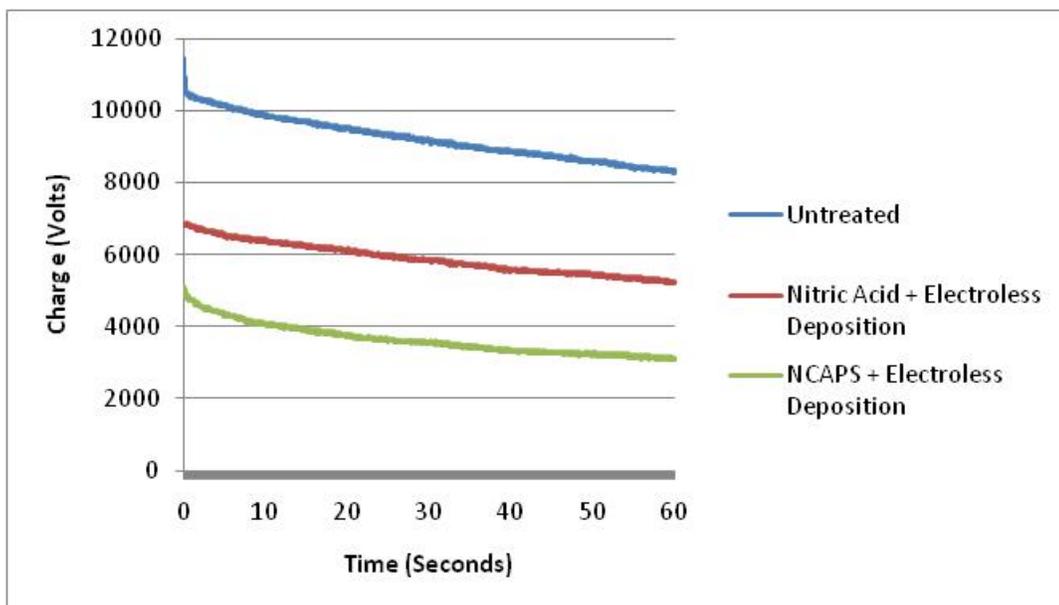


Figure 4.44 Charge decay curves for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun nylon 6,6 samples

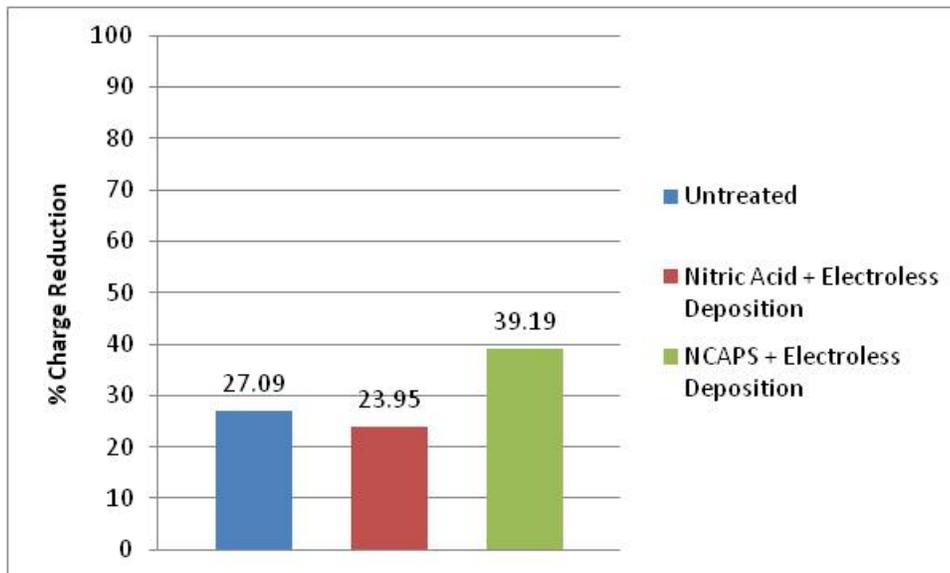


Figure 4.45 Percent charge reduction for nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun nylon 6,6 samples

The peak charge associated with untreated nylon 6,6 was 11,475 volts compared to 6,868 volts for the nitric acid pretreated sample with electroless deposition. NCAPS pretreated nylon 6,6 with electroless deposition had an initial charge of 5,126 volts. After 60 seconds of charge dissipation, untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition samples measured 8,350 volts, 5,241 volts, and 3,109 volts, respectively. Based on the charge lost over 60 seconds, untreated spun nylon 6,6 reduced its charge by 27.09%, nitric acid pretreated spun nylon 6,6 with electroless deposition reduced its charge by 23.95%, and NCAPS pretreated spun nylon 6,6 with electroless deposition reduced its charge by 39.19%. NCAPS pretreated spun nylon 6,6 with electroless deposition was able to reduce its charge by the greatest percentage. The initial peak charge for untreated spun nylon 6,6 was much higher than nitric acid pretreated spun nylon 6,6 with electroless deposition and NCAPS pretreated spun nylon 6,6 with electroless deposition. Thus, NCAPS pretreated spun nylon 6,6 with electroless deposition had a better peak charge value, 60 second charge value, and percent reduction properties than untreated spun nylon 6,6 and nitric acid pretreated spun nylon 6,6 with electroless deposition.

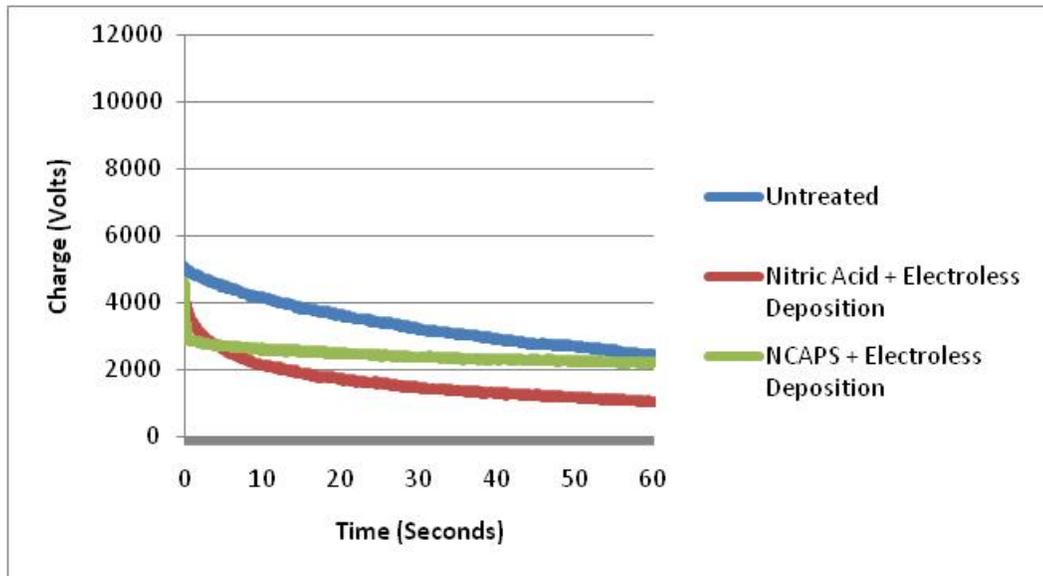


Figure 4.46 Charge decay curves for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition filament nylon 6,6 samples

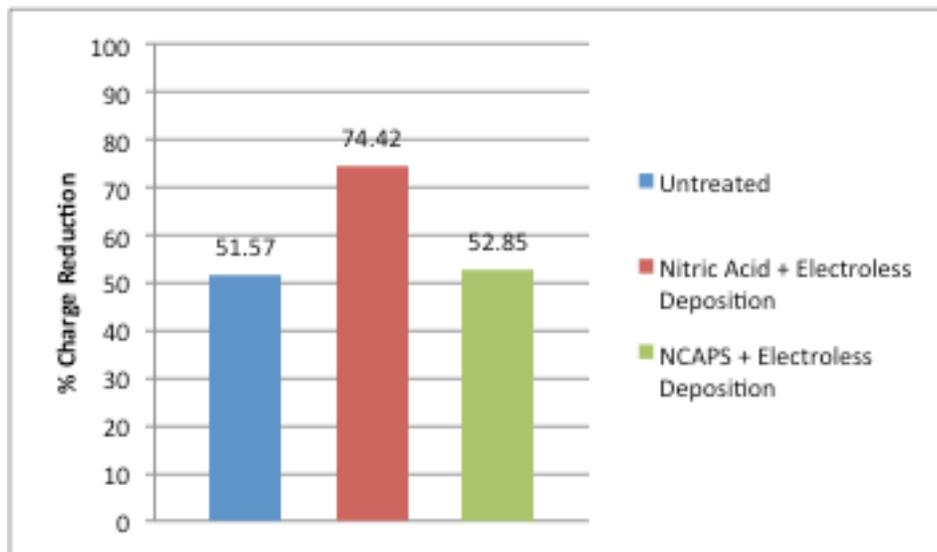


Figure 4.47 Percent charge reduction for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition filament nylon 6,6 samples

After being charged 50 times with the PTFE rubbing head, untreated filament nylon 6,6 had a peak charge of 5,143 volts, filament nitric acid pretreated nylon 6,6 with electroless deposition had a peak charge of 4,199 volts, and NCAPS pretreated filament nylon 6,6 with electroless deposition had a peak charge of 4,638 volts. Based on these results, nitric acid pretreated filament nylon 6,6 had the lowest peak charge. After 60 seconds of charge dissipation, untreated filament nylon 6,6 measured 2,425 volts, nitric acid pretreated filament nylon 6,6 with electroless deposition measured 1,074 volts, and NCAPS pretreated filament nylon 6,6 with electroless deposition measured 2,246 volts. Based on peak voltage and 60 second dissipation values, the untreated sample reduced its charge by 52.85%, the nitric acid pretreated sample with electroless deposition reduced its charge by 74.42%, and the NCAPS pretreated sample with electroless deposition reduced its charge by 51.57%. Nitric acid pretreated filament nylon 6,6 with electroless deposition had the best peak charge value, 60 second charge value, and best % reduction. Since NCAPS pretreated filament nylon 6,6 with electroless deposition had more copper present on the surface, as evidenced in SEM images and elemental analysis, a lack of uniformity of the finish is attributed to the sample not outperforming nitric acid pretreated filament nylon 6,6 in static dissipation.

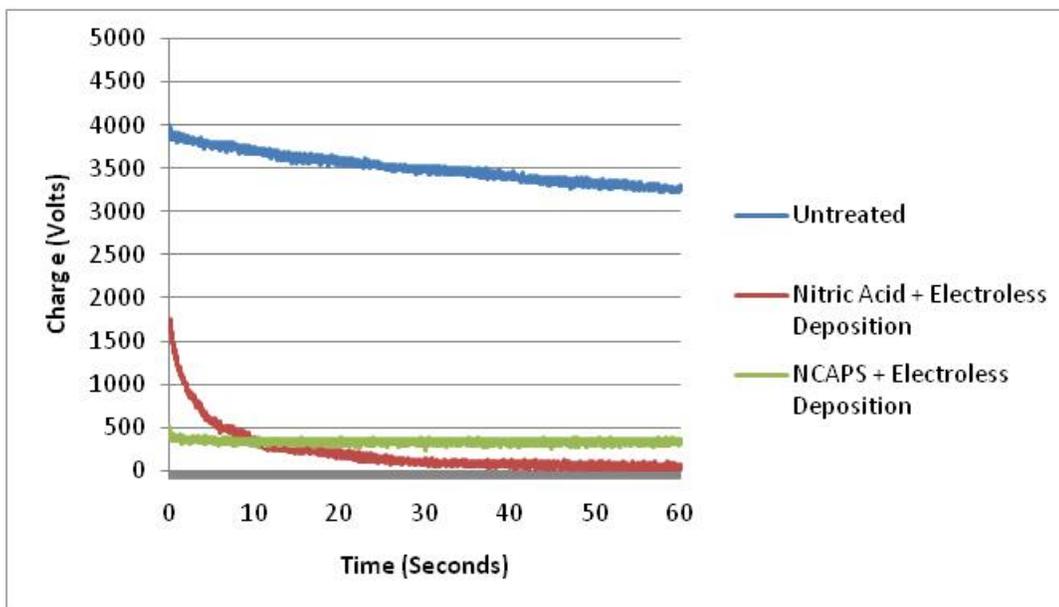


Figure 4.48 Charge decay curves for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun polyester samples

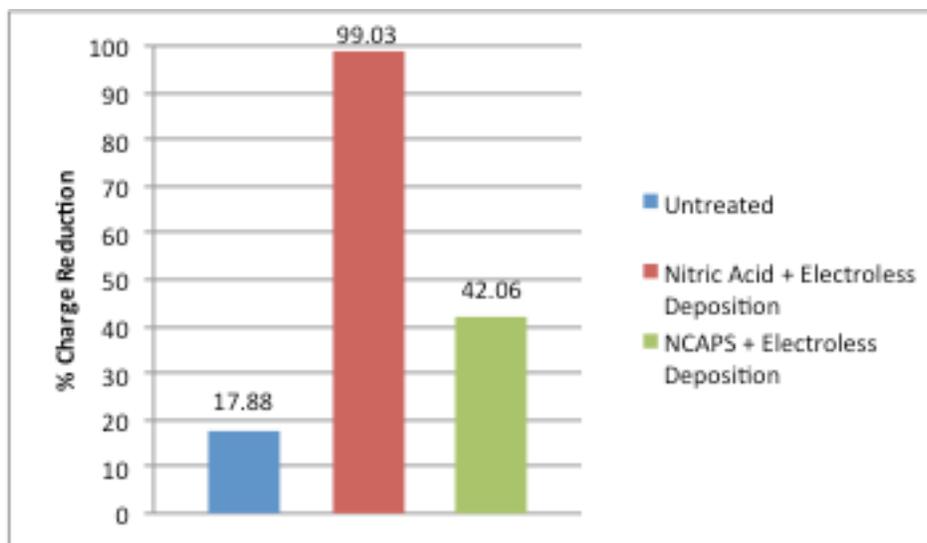


Figure 4.49 Percent charge reduction for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun polyester samples

The highest peak charge observed with untreated spun polyester was 4,004 volts. In comparison, nitric acid pretreated spun polyester and NCAPS pretreated spun polyester had a voltages of 1,758 volts and 504 volts, respectively. NCAPS pretreated spun polyester with electroless deposition had a lower peak charge than nitric acid pretreated samples with electroless deposition. Both nitric acid pretreated samples with electroless deposition and NCAPS pretreated samples with electroless deposition had lower peak charge values, compared to untreated spun polyester. After 60 seconds of charge dissipation, untreated spun polyester had a voltage of 3,288 volts, nitric acid pretreated spun polyester with electroless deposition had a charge of 17 volts, and NCAPS pretreated spun polyester with electroless deposition had a charge of 292 volts. Nitric acid pretreated spun polyester with electroless deposition had a lower charge, than NCAPS pretreated spun polyester with electroless deposition, after 60 seconds of charge dissipation. While, nitric acid pretreated spun polyester with electroless deposition had a lower charge after 60 seconds of dissipation, NCAPS pretreated spun polyester with electroless deposition still showed excellent antistatic properties. Untreated spun polyester, nitric acid pretreated spun polyester with electroless deposition, and NCAPS pretreated spun polyester with electroless deposition had percent reduction values of 17.88%, 99.03%, and 42.06%, respectively. Percent reduction values show that nitric acid pretreated spun polyester with electroless deposition was able to reduce nearly all its charge over a 60 second period. Though this is true, it is important to note that NCAPS pretreated spun polyester with electroless deposition resisted charge better than nitric acid pretreated spun polyester with electroless deposition. Most percent reduction associated with NCAPS pretreated spun polyester with electroless deposition was observed within the first 1-2 seconds of charge dissipation.

Discrepancy exists between resistivity and charge dissipation results for NCAPS woven spun polyester with electroless deposition. Since resistivity for NCAPS pretreated woven spun polyester was higher than nitric acid pretreated woven spun polyester, it was expected that the sample would charge higher when tested for dissipation. However, this was not the case. In dissipation testing, NCAPS pretreated woven spun polyester had a lower voltage than nitric acid pretreated woven spun polypropylene. This can be attributed to the non-uniformity of the finish on NCAPS pretreated woven spun polyester. It seems resistivity testing was conducted on sample with a less uniform finish than the sample used for dissipation testing. Additionally, resistivity testing uses a larger sample for testing than dissipation testing. Thus, there is more room for non-uniformity to occur.

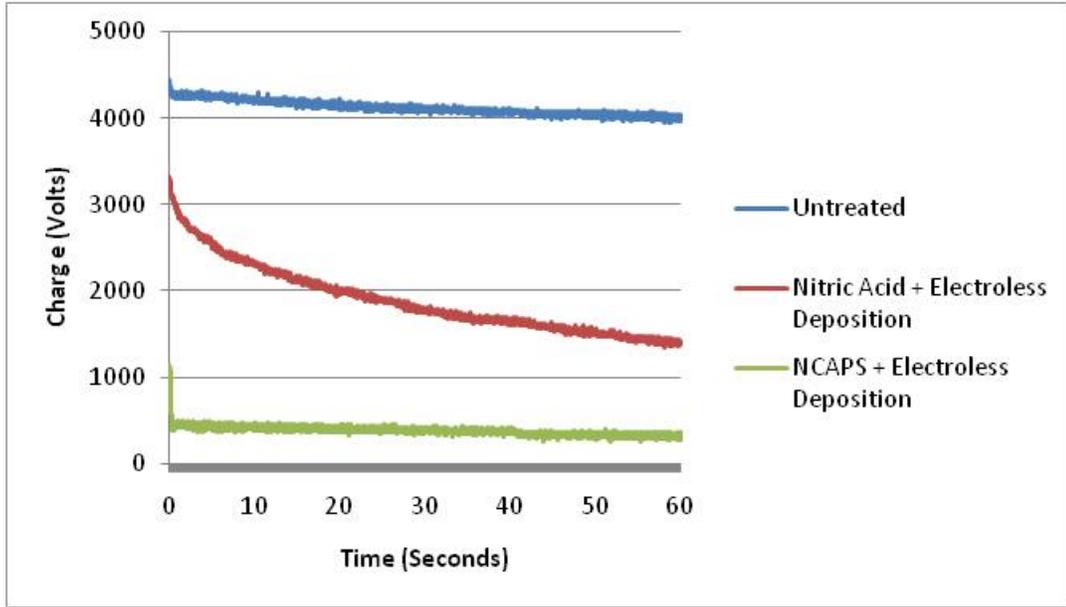


Figure 4.50 Charge decay curves for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition filament polyester samples

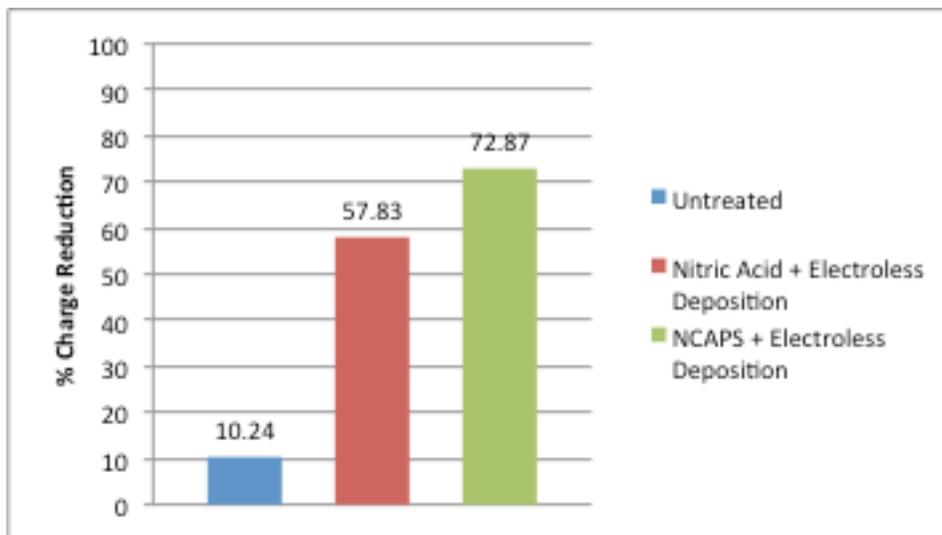


Figure 4.51 Percent charge reduction for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition filament polyester samples

The peak charge observed with untreated filament polyester was 4,443 volts. After nitric acid pretreatment with electroless deposition, the peak charged recorded was 3,320 volts. NCAPS pretreated filament polyester had a peak charge 1,139 volts. Based on peak charge values, NCAPS pretreated filament polyester with electroless deposition has superior performance. After 60 seconds of charge dissipation, untreated filament polyester, nitric acid pretreated filament polyester with electroless deposition, and NCAPS pretreated filament polyester with electroless deposition measured 3,988 volts, 1,400 volts, and 309 volts, respectively. After 60 seconds, NCAPS pretreated filament polyester with electroless deposition had the lowest observed charge. The percent reduction in charge, from peak voltage to voltage after 60 seconds, was 10.24% for untreated samples, 57.83% for nitric acid pretreated with electroless deposition samples, and 72.87% for NCAPS pretreated with electroless deposition. Nitric acid pretreated filament polyester with electroless deposition and NCAPS pretreated filament polyester with electroless deposition both had similar percent reduction values to each other. Both samples were superior to the untreated sample, with regard to percent reduction. Though nitric acid pretreated filament polyester with electroless deposition and NCAPS pretreated filament polyester with electroless deposition had similar percent reduction values, NCAPS pretreated filament polyester with electroless deposition was a superior antistatic finish. NCAPS pretreated filament polyester with electroless deposition had a lower peak charge and was able to reduce its charge to a lower voltage, than nitric acid pretreated spun polyester with electroless deposition, in a 60 second time frame.

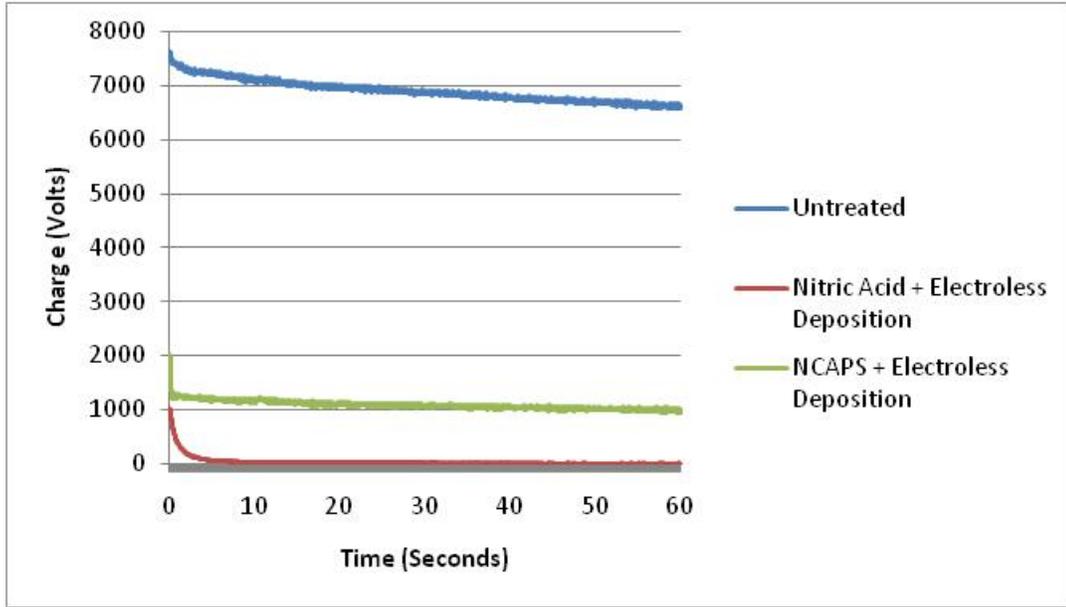


Figure 4.52 Charge decay curves for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun acrylic samples

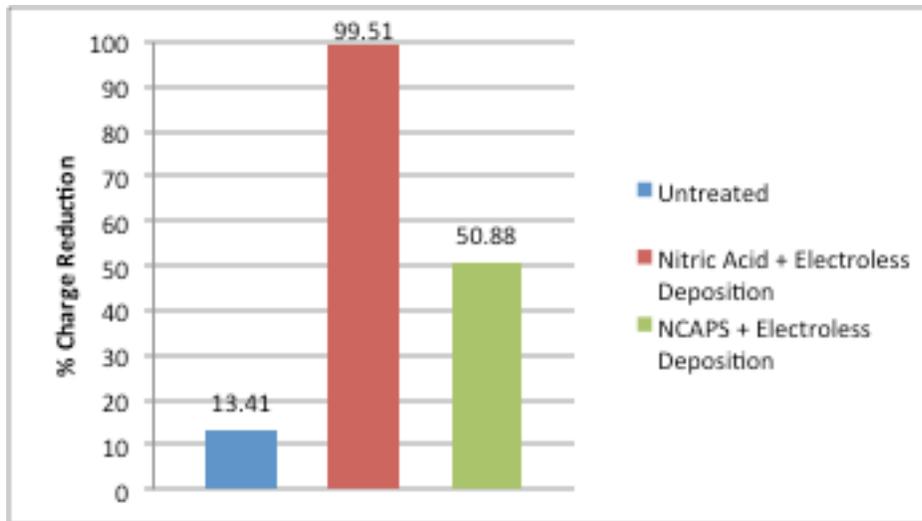


Figure 4.53 Percent charge reduction for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun acrylic samples

The peak charge associated with untreated spun acrylic fabric was 7,650 volts. Nitric acid pretreated spun acrylic with electroless deposition had a peak charge of 1,022 volts and samples pretreated with NCAPS and electroless deposition had a peak charge of 2,031 volts. Nitric acid pretreated samples with electroless deposition had the best antistatic peak charge antistatic properties. After 60 seconds of charge dissipation, untreated spun acrylic had a charge of 6,624 volts. In comparison, nitric acid pretreated spun acrylic with electroless deposition had a charge of 5 volts, and NCAPS pretreated spun acrylic with electroless deposition had a charge of 997 volts. Thus, after 60 seconds of charge dissipation, nitric acid pretreated spun acrylic with electroless deposition had the smallest observed voltage. Percent charge reduction values after 60 seconds were 13.41% for untreated samples, 99.51% for nitric acid pretreated samples with electroless deposition, and 50.88% for NCAPS pretreated samples with electroless deposition. Thus, nitric acid pretreatment with electroless deposition imparted the best antistatic properties, with regard to percent charge reduction. Though nitric acid pretreated spun acrylic with electroless deposition had superior dissipation properties to NCAPS pretreated spun acrylic with electroless deposition, both samples had better dissipation properties, compared to the untreated spun acrylic sample. Uniformity may have hindered the performance of plasma pretreated spun acrylic with electroless deposition. Plasma pretreated spun acrylic with electroless deposition had more copper present on the surface as observed visually, in SEM images and elemental analysis, compared to nitric acid pretreated spun acrylic with electroless deposition.

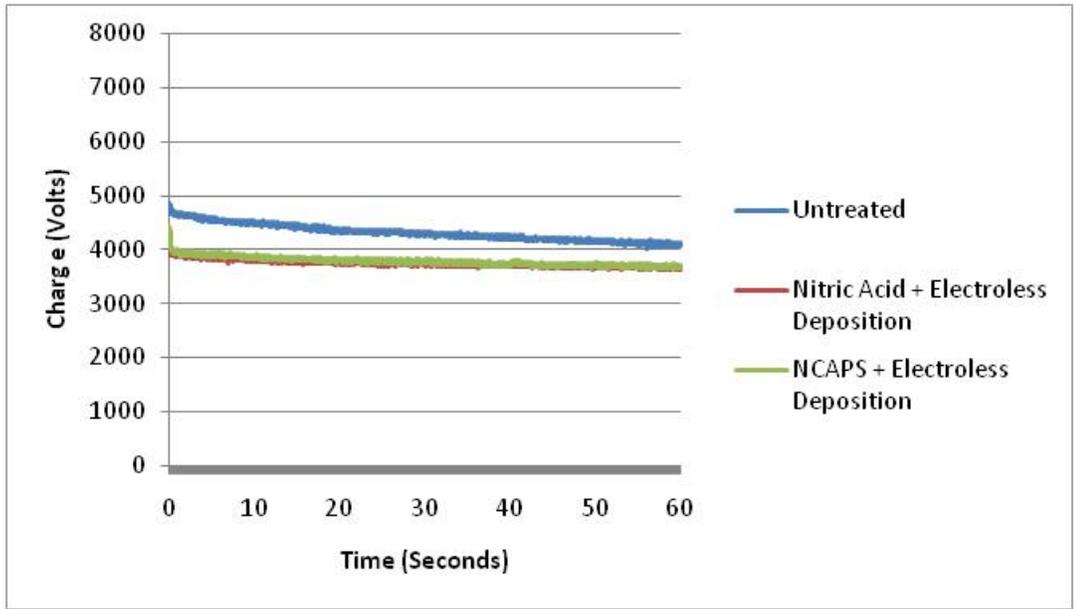


Figure 4.54 Charge decay curves for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun polypropylene samples

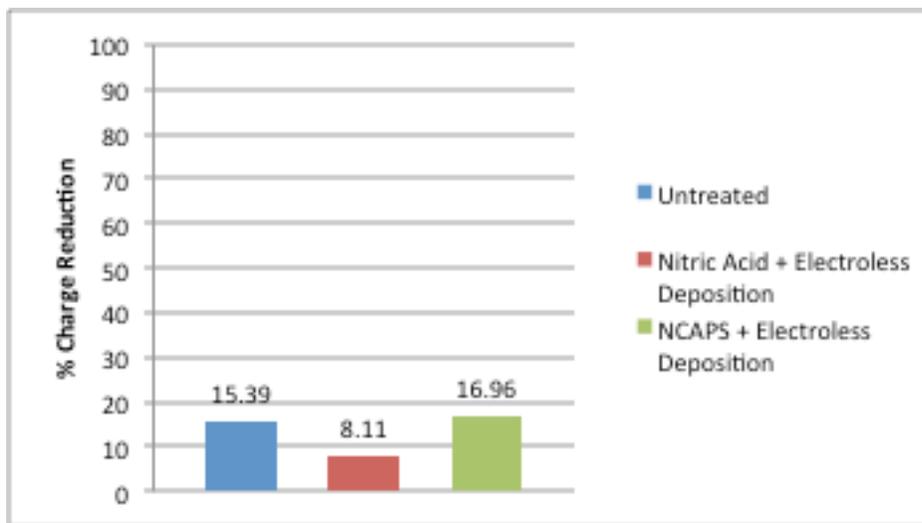


Figure 4.55 Percent charge reduction for untreated, nitric acid pretreated with electroless deposition, and NCAPS pretreated with electroless deposition spun polypropylene samples

Untreated spun polypropylene had a peak charge of 4,867 volts. In comparison, nitric acid pretreated spun polypropylene with electroless deposition had a peak charge of 4,020 volts and NCAPS pretreated spun polypropylene with electroless deposition had a charge of 4,410 volts. Thus, nitric acid pretreated spun polypropylene with electroless deposition had a superior peak charge value, compared to untreated and NCAPS pretreated spun polypropylene with electroless deposition. After 60 seconds of charge dissipation, untreated spun polypropylene had a charge of 4,118 volts, nitric acid pretreated spun polypropylene with electroless deposition had a charge of 3,694 volts, and NCAPS pretreated spun polypropylene with electroless deposition had a charge of 3,662 volts. Thus, nitric acid pretreated spun polypropylene with electroless deposition and NCAPS pretreated spun polypropylene with electroless deposition had nearly identical dissipation values after 60 seconds of charge dissipation. % charge reduction values, after 60 seconds of dissipation were 15.39% for untreated samples, 8.11% for nitric acid pretreated samples with electroless deposition, and 16.96 % for NCAPS pretreated samples with electroless deposition. Though, untreated spun polypropylene and NCAPS pretreated spun polypropylene with electroless deposition had similar percent charge reduction values, NCAPS pretreated spun polypropylene with electroless deposition had a lower peak charge. Therefore, NCAPS pretreated spun polypropylene with electroless deposition had superior charge dissipation values, compared to the untreated sample. Nitric acid pretreated spun polypropylene with electroless deposition had nearly identical charge dissipation values to NCAPS pretreated spun polypropylene with electroless deposition.

CHAPTER 5: CONCLUSIONS

Nitric acid and plasma pretreatment options for electroless deposition were explored through this research and their performance was quantified through tensile strength testing, visual analysis, SEM imaging, charge generation and dissipation testing, elemental analysis, and surface resistivity. Plasma aided finishes represent a potentially environmentally sustainable option for electroless deposition systems, as plasma is a waterless technology. This is becoming increasingly important, as government regulations and consumers are demanding cleaner manufacturing and greener products, respectively.

SEM imagery suggests that use of plasma to etch fiber surfaces prior to electroless deposition produces increased surface roughness, when compared to nitric acid pretreatment. Though increased etching was observed on NCAPS pretreated samples, tensile strength didn't decrease more than 6.94%. In comparison, the worst tensile strength loss associated with nitric acid pretreated samples was 30.30%. Increased surface etching on plasma pretreated samples, combined with the electroless deposition process, yielded increased copper uptake on sample surfaces compared to nitric acid pretreatment and electroless deposition. This was evident through visual analysis, SEM images, and elemental analysis. For all samples, NCAPS pretreatment yielded more uptake on samples, compared to nitric acid pretreatment. Copper uptake for plasma pretreated samples with electroless deposition ranged from 61,716% to 295,876% more than untreated samples.

Since an identical electroless deposition process was used on pretreated samples, the increase in copper pickup is linked to NCAPS pretreatment. An increase in copper deposition means a

possible reduction in the amount of chemicals needed for electroless deposition baths. A reduction in the amount of chemicals translates to monetary savings and a more environmentally friendly process.

On average resistivity values observed for nitric acid pretreated samples with electroless deposition were 3.63×10^{13} greater than values observed with NCAPS pretreated samples with electroless deposition. Both treatment types imparted better antistatic properties, compared to the untreated samples. Since more copper was present on the surface of NCAPS pretreated samples with electroless deposition than nitric acid pretreated samples with electroless deposition, it is thought that uniformity problems impaired resistivity values associated with plasma pretreated samples with electroless deposition. A uniform copper finish should create minimal resistance, while a non-uniform finish should increase the amount of resistivity.

Dissipation values observed on nitric acid pretreated samples with electroless deposition and NCAPS pretreated samples with electroless deposition, depended on the type of substrate utilized. Nitric acid pretreatment and electroless deposition used on filament nylon 6,6, and spun acrylic samples yielded better dissipation results, compared to NCAPS pretreatment with electroless deposition. Contrarily, spun nylon 6,6, filament polyester, and spun polyester treated with NCAPS pretreatment and electroless deposition, yielded superior results compared to samples treated with nitric acid pretreatment and electroless deposition. NCAPS pretreated spun polypropylene samples with electroless deposition yielded similar results to nitric acid pretreated samples with electroless deposition. For all samples, nitric acid pretreatment with electroless deposition and plasma pretreatment with electroless deposition,

imparted significantly better antistatic properties, in contrast to untreated samples.

Since more copper was present on the surface of NCAPS pretreated samples in visual analysis, SEM images, and elemental analysis, the non-uniformity of the copper coating influenced charge dissipation results. When a fabric is trying to rid itself of charge, charge flows takes the easiest path of dissipation. If charge is moving through an area with a uniform layer of copper, it will move quickly. However, if charge encounters an area a non-uniform layer of copper, direct dissipation may not occur.

CHAPTER 6: RECCOMENDATIONS FOR FURURE RESEARCH

Future research on antistatic finishes imparted through plasma pretreatment and electroless deposition involves process optimization. Studies should be conducted on scaling back chemicals used in the electroless deposition bath, improving the uniformity of the finish, optimization of plasma parameters, use of different fabric constructions, and durability testing.

Based SEM and visual assessment data, it appears that after plasma pretreatment, chemicals used during electroless deposition can be scaled back. Research must be conducted on examining the degree to which the electroless deposition bath can be scaled back. Scaling back the amount of chemicals used in the electroless deposition bath will produce a more environmentally friendly process and will save money. Future research should focus on scaling chemicals back by different percentages. For example, chemicals used could be scaled back 25%, 50%, and 75%. Surface resistivity and dissipation values must be measured on all samples and compared.

Additionally, future research should focus on obtaining a uniform coating of copper on the fabric surface. Samples pretreated with plasma, had issues with uniformity. It was apparent from visual assessment and SEM images that copper wasn't evenly distributed on the fabric surface. Even though it is apparent that pretreatment with the NCAPS increased copper present on fabric surfaces, an uneven coating won't yield optimal antistatic properties. In theory, an even coating of copper should dissipate charge quickly and have minimal surface resistance. When the coating of copper isn't uniform, charge isn't able to move efficiently

through fabric samples. This results in higher peak voltage values and resistivity values.

Non-uniformity of copper on plasma pretreated samples with electroless deposition can be attributed to plasma pretreatment, the electroless deposition bath, or a combination of the two. Future studies on uniformity of copper coatings on samples should focus on both plasma pretreatment and the electroless deposition bath. It is possible that in plasma pretreatment, the surface samples aren't being evenly etched. Additionally, it is possible that when samples fold and touch each other in the electroless deposition bath, uneven deposition of copper occurs. Hence, both plasma pretreatment and the electroless deposition bath should be examined for their respective influence on the uniformity of copper deposition.

Additional research can be conducted on manipulating plasma parameters. This research concluded that optimal etching occurs in plasma consisting of 99% helium and 1% oxygen. Thus, additional research isn't needed on this parameter. Parameters such as power and time of exposure should be examined. Three points, (low, middle, and high) should be picked for each variable. The effect of plasma parameters on the rate of copper deposition and antistatic properties must be quantified.

It is critical that future research focus different textile fabric constructions. For example, it is important to compare untreated, nitric acid pretreated, and NCAPS pretreated knit and nonwoven fabrics. This is beneficial in order to understand if knit and nonwoven fabrics will have similar, better, or worse results, compared to woven fabrics.

Finally, future research should examine the durability of the finish. This can be accomplished by using AATCC Test Method 61, known as accelerated laundering. Due to time and budget constraints accelerated laundering was unable to be completed. After accelerated laundering,

the following testing should be completed: SEM images, elemental analysis of copper, surface charge and dissipation, and surface resistivity.

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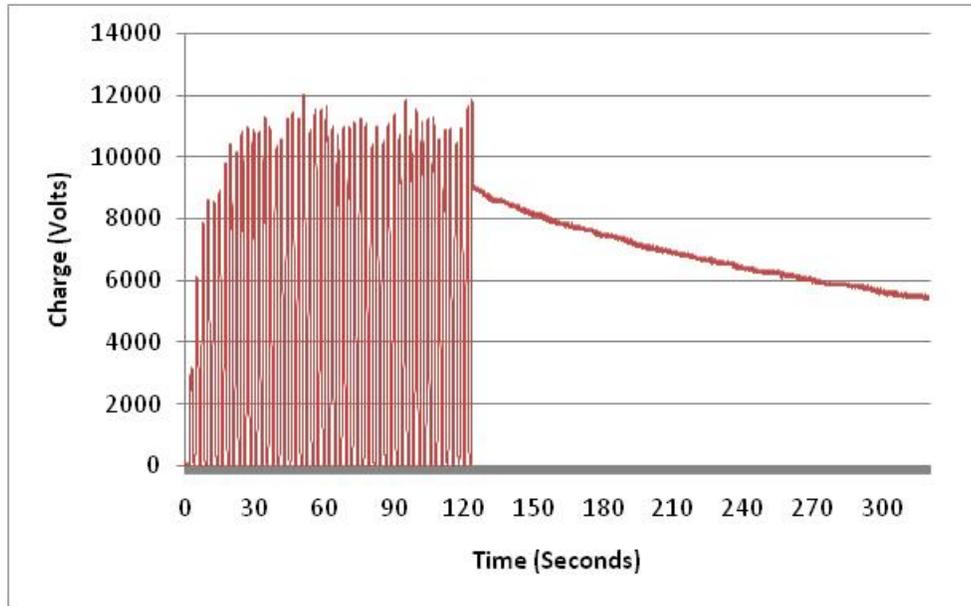
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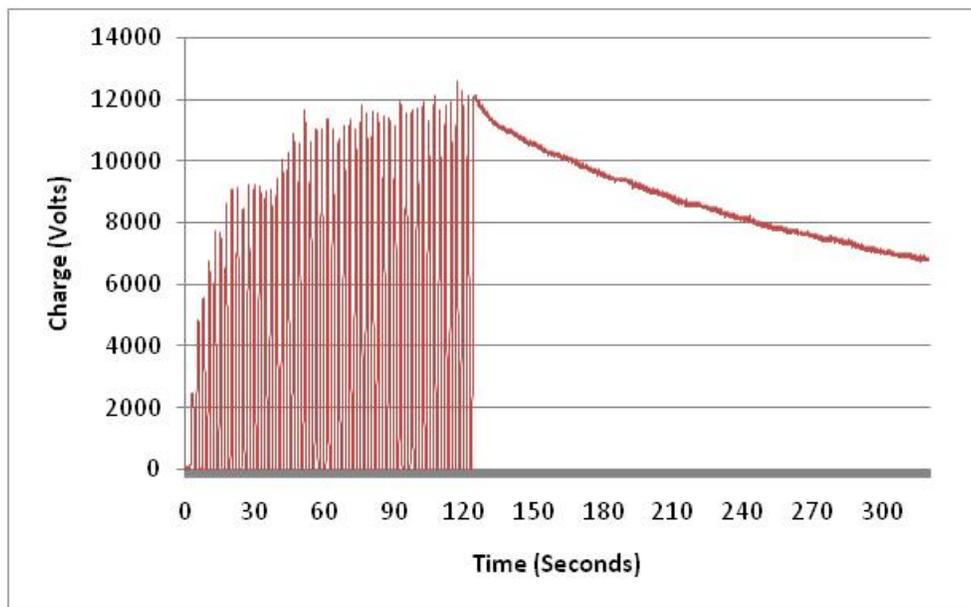
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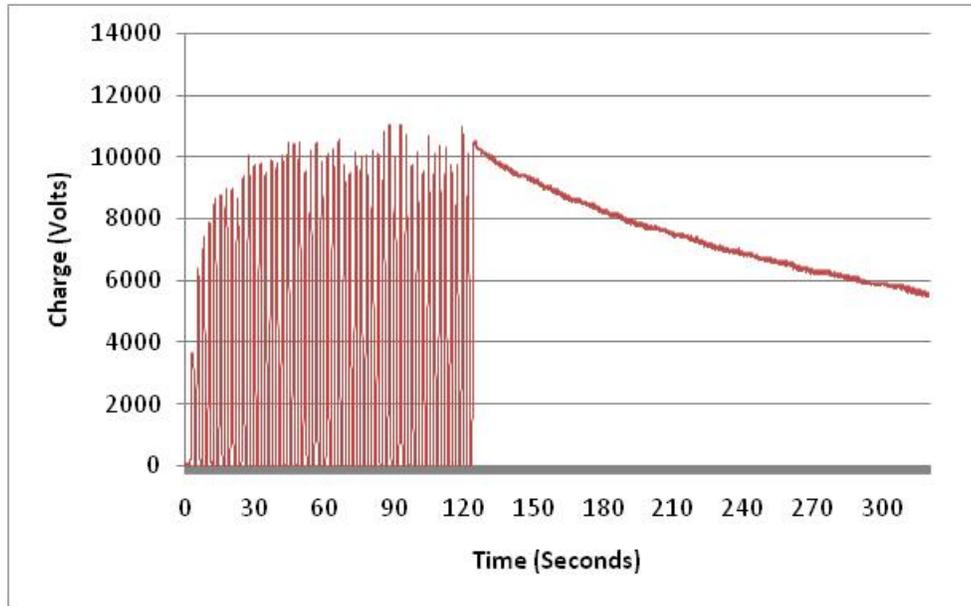
APPENDICES



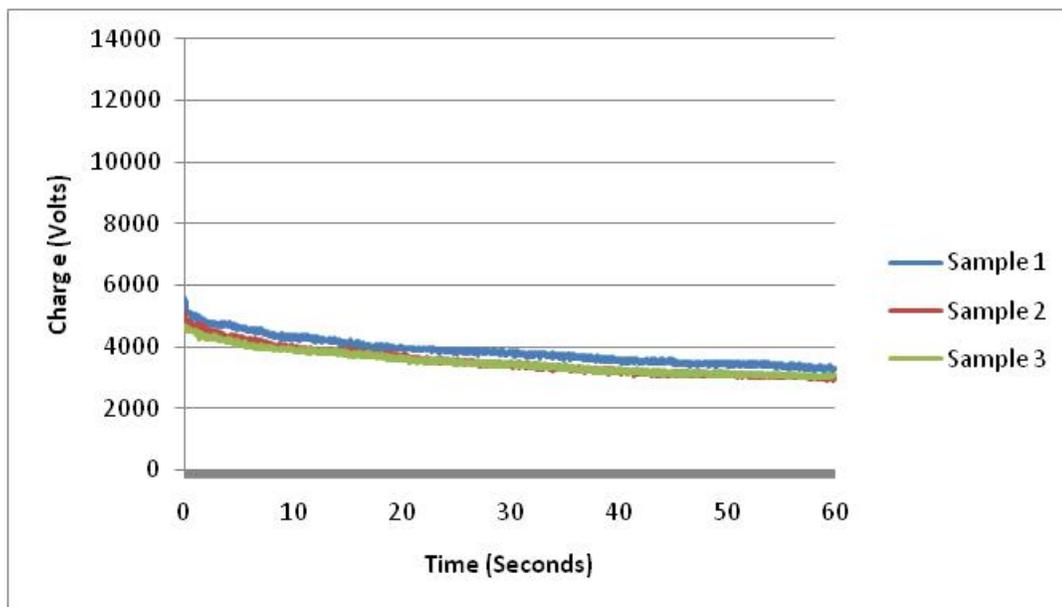
Appendix 1 Charge decay curve for untreated spun nylon 6,6 sample 1



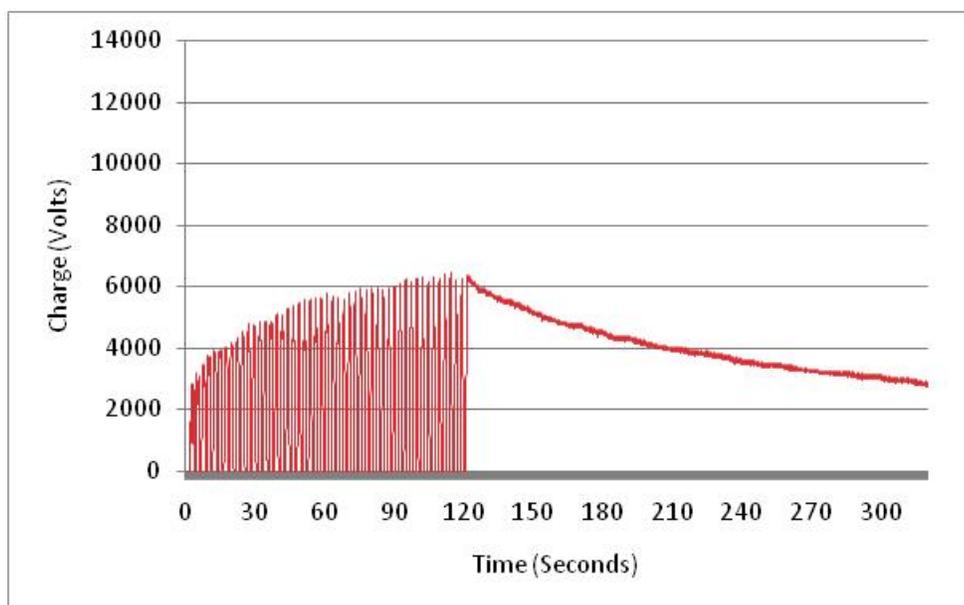
Appendix 2 Charge decay curve for untreated spun nylon 6,6 sample 2



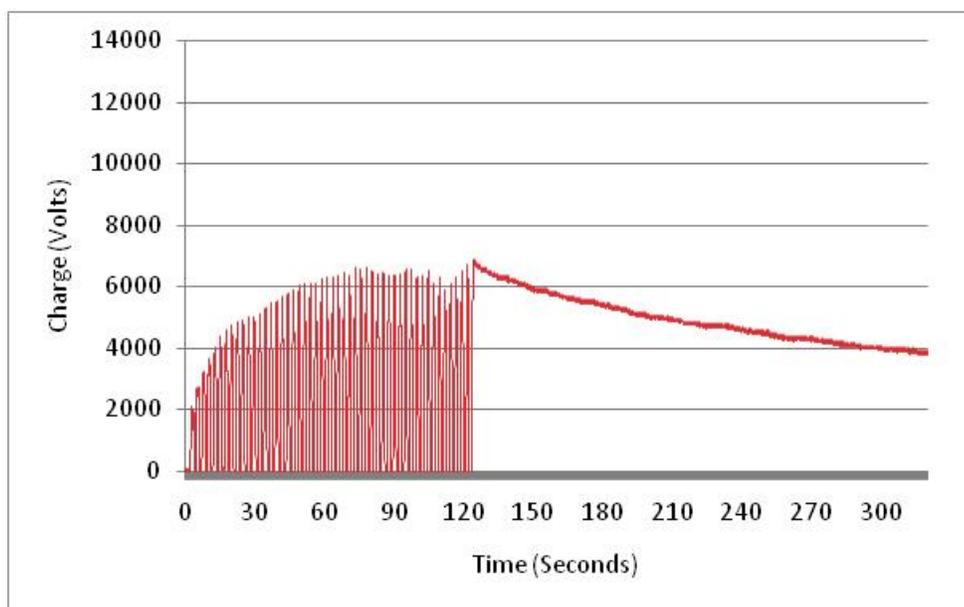
Appendix 3 Charge decay curve for untreated spun nylon 6,6 sample 3



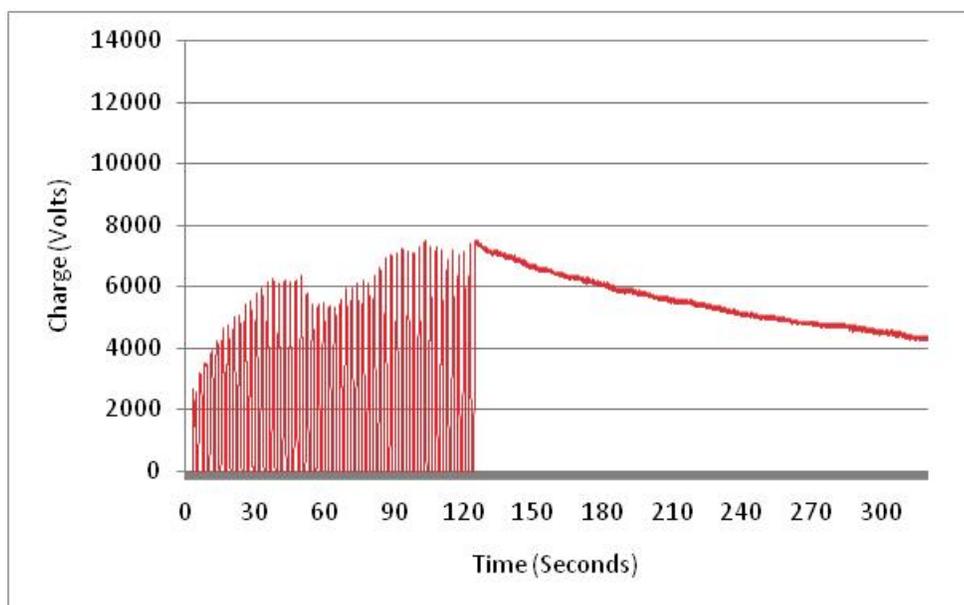
Appendix 4 Decay curves for untreated spun nylon 6,6 samples 1-3



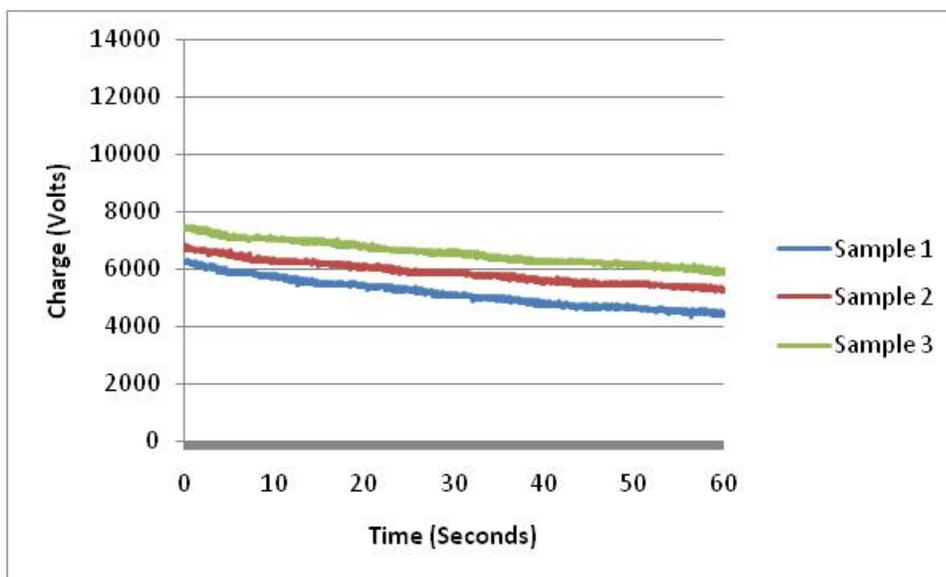
Appendix 5 Charge decay curve for nitric acid pretreated spun nylon 6,6 with electroless deposition sample 1



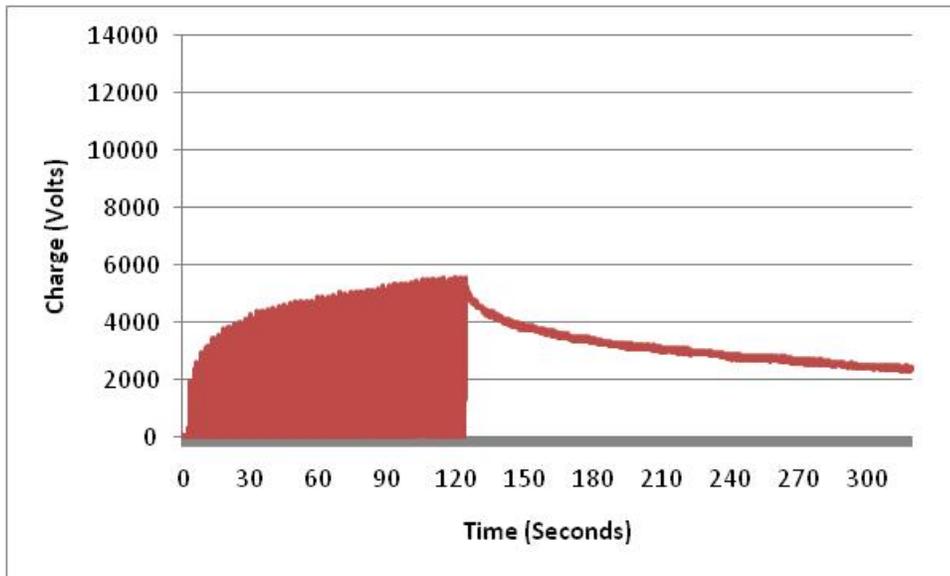
Appendix 6 Charge decay curve for nitric acid pretreated spun nylon 6,6 with electroless deposition sample 2



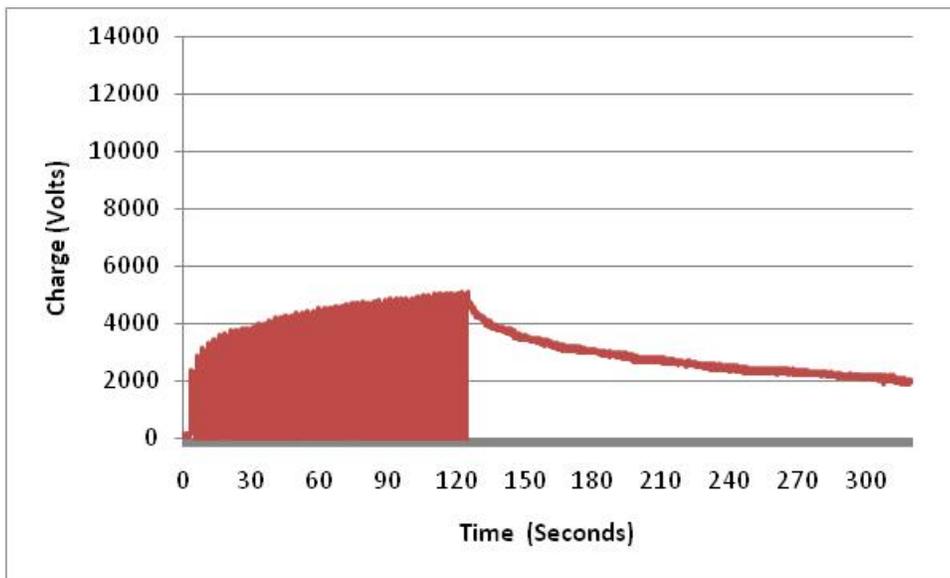
Appendix 7 Charge decay curve for nitric acid pretreated spun nylon 6,6 with electroless deposition sample 3



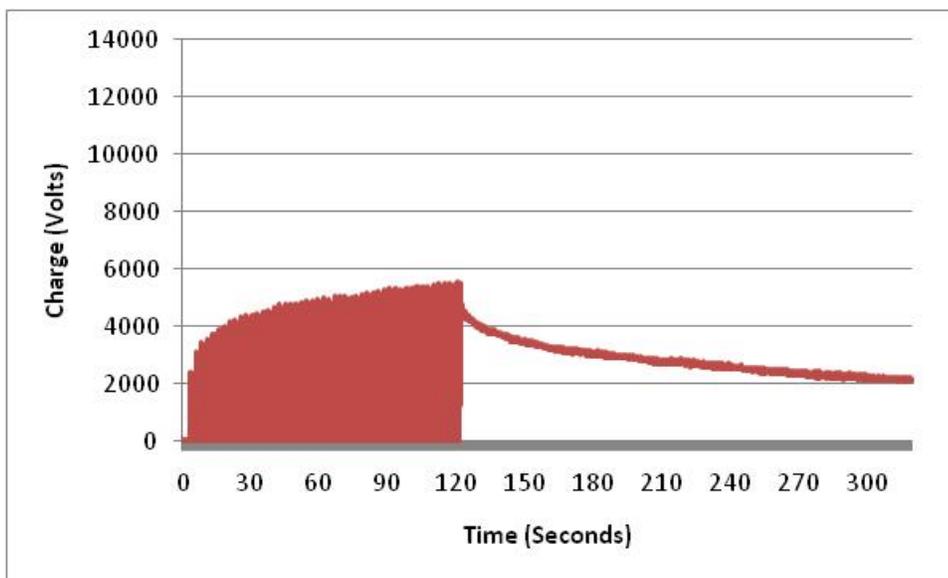
Appendix 8 Decay curves for nitric acid pretreated spun nylon 6,6 with electroless deposition samples 1-3



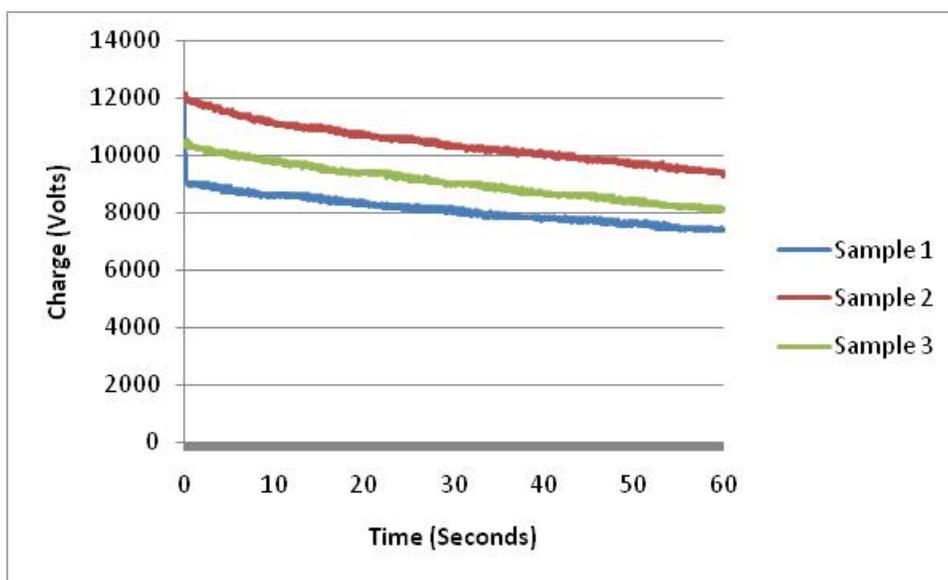
Appendix 9 Charge decay curve for NCAPS pretreated spun nylon 6,6 with electroless deposition sample 1



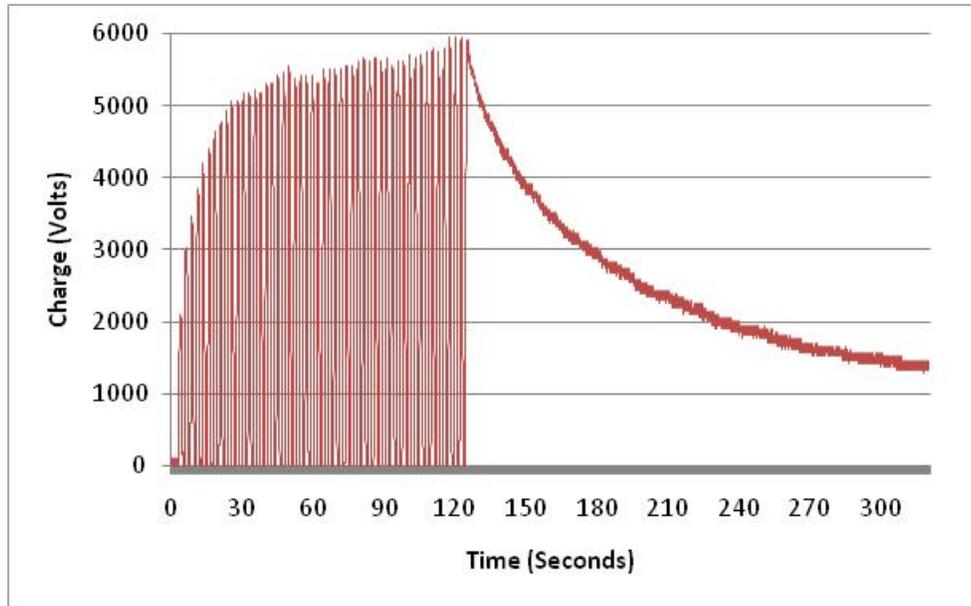
Appendix 10 Charge decay curve for NCAPS pretreated spun nylon 6,6 with electroless deposition sample 2



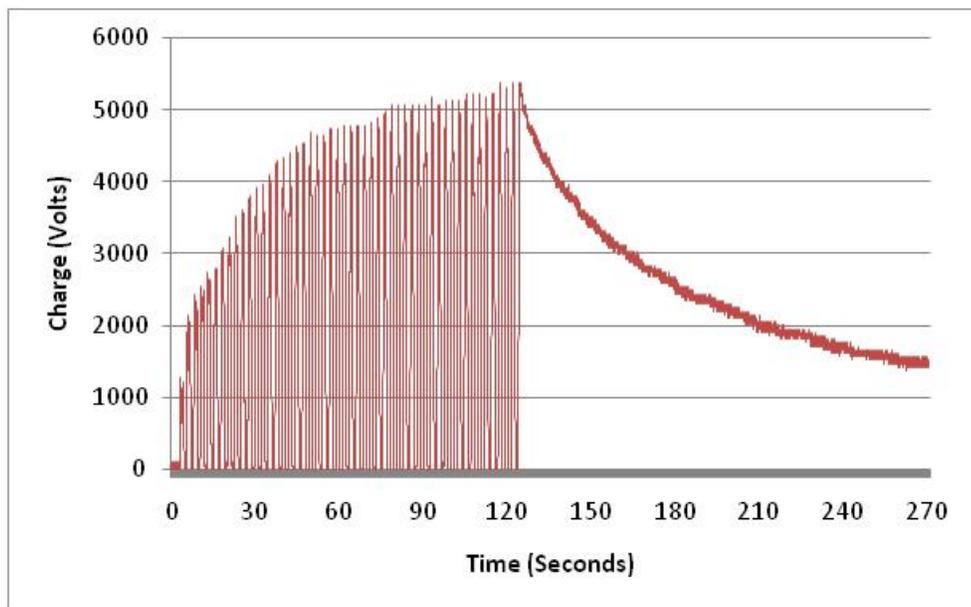
Appendix 11 Charge decay curve for NCAPS pretreated spun nylon 6,6 with electroless deposition sample 3



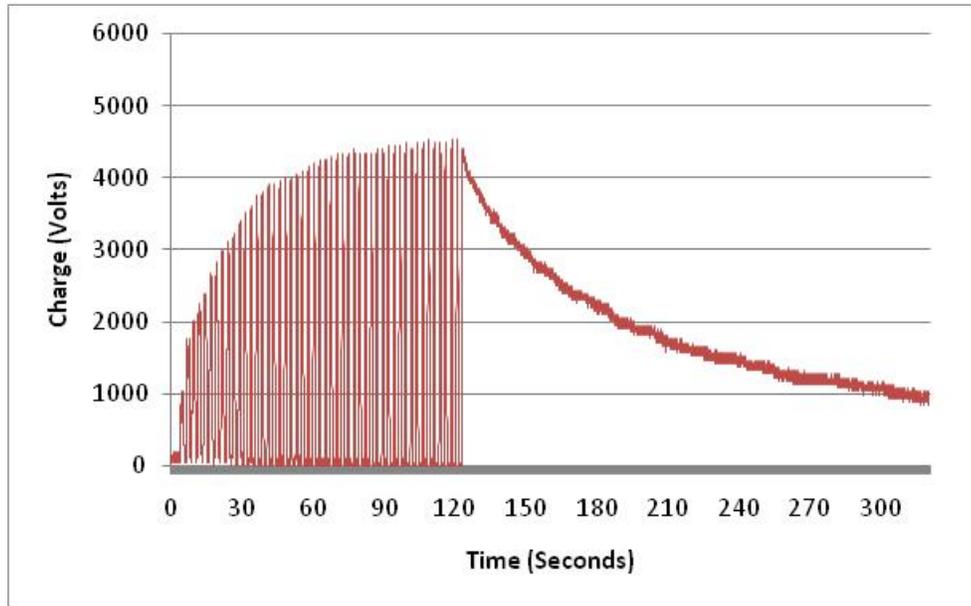
Appendix 12 Decay curves for NCAPS pretreated spun nylon 6,6 with electroless deposition samples 1-3



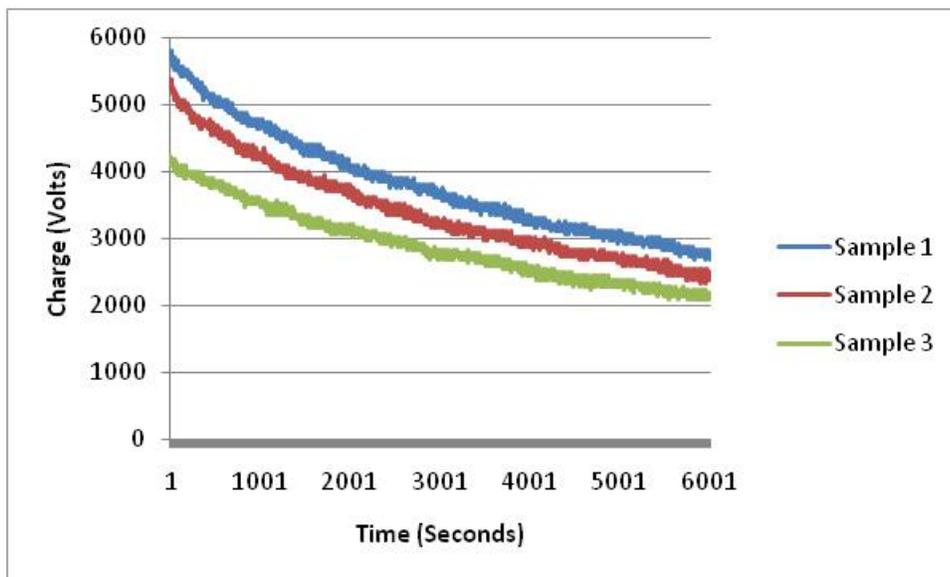
Appendix 13 Charge decay curve for untreated filament nylon 6,6 sample 1



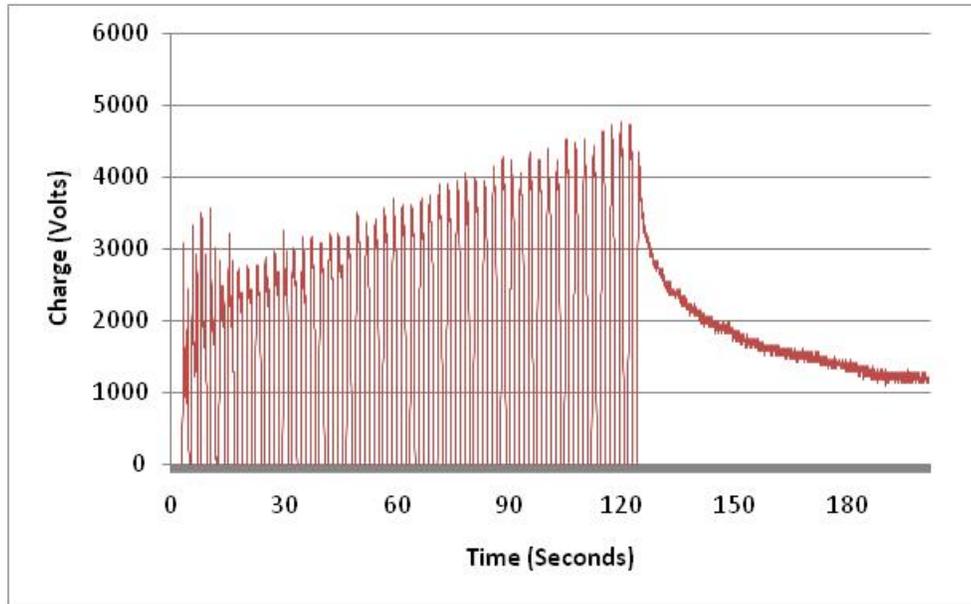
Appendix 14 Charge decay curve for untreated filament nylon 6,6 sample 2



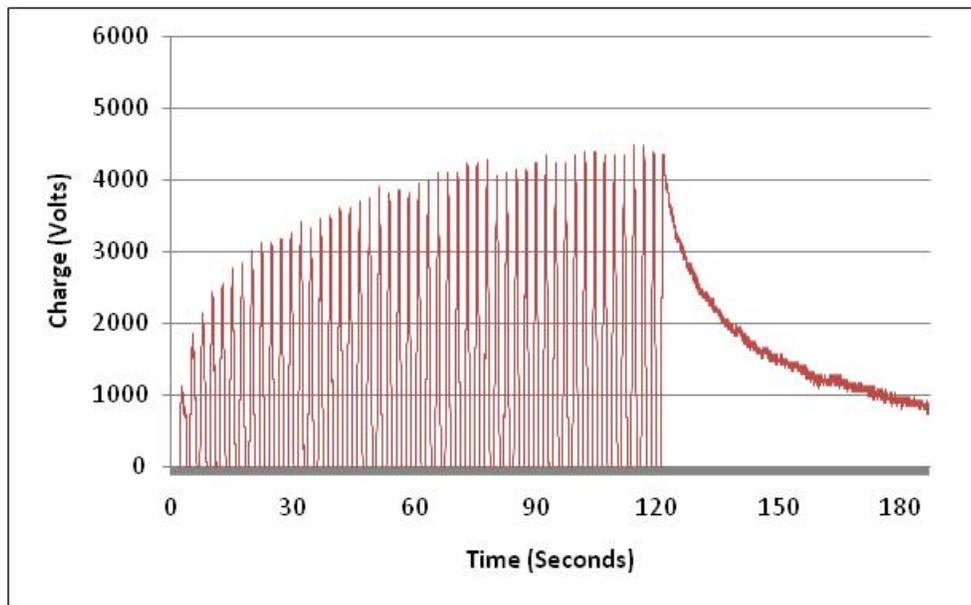
Appendix 15 Charge decay curve for untreated filament nylon 6,6 sample 3



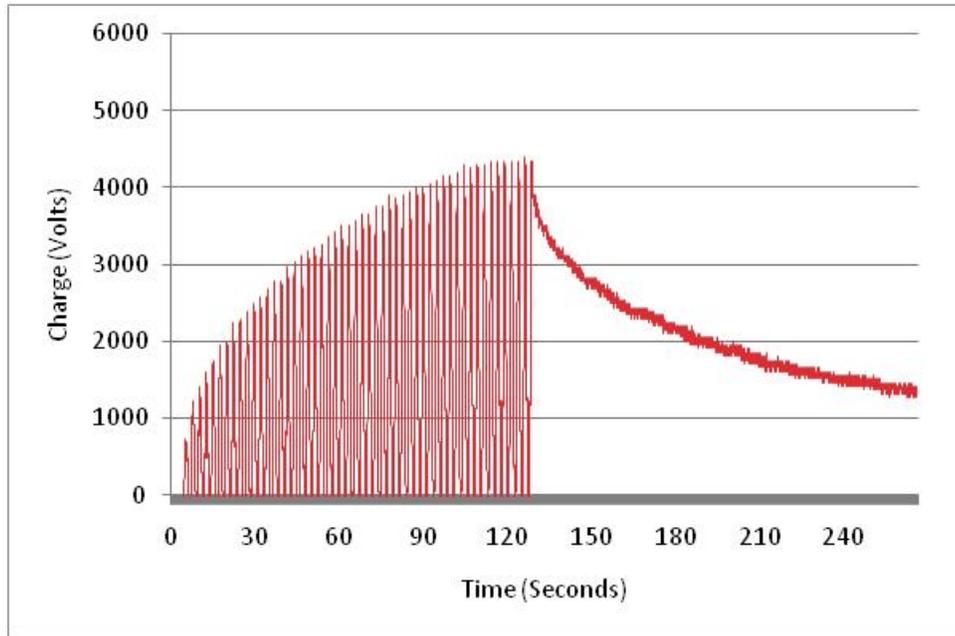
Appendix 16 Decay curves for untreated filament nylon 6,6 samples 1-3



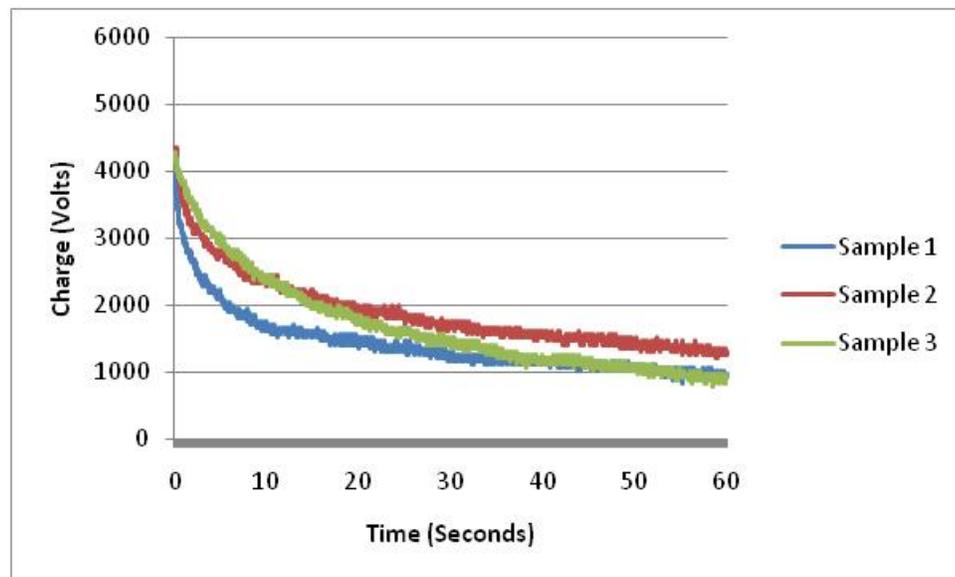
Appendix 17 Charge decay curve for nitric acid pretreated filament nylon 6,6 with electroless deposition sample 1



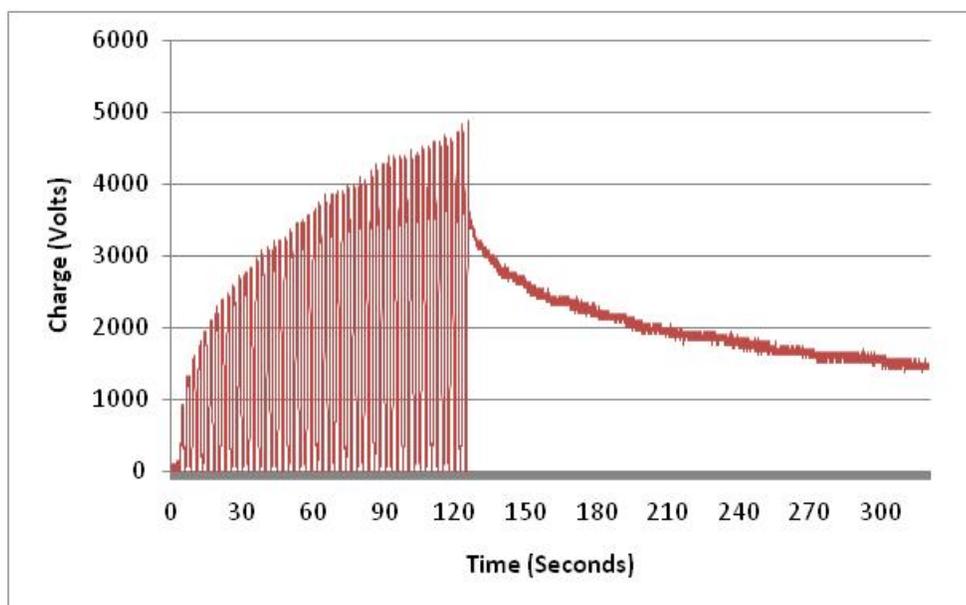
Appendix 18 Charge decay curve for nitric acid pretreated filament nylon 6,6 with electroless deposition sample 2



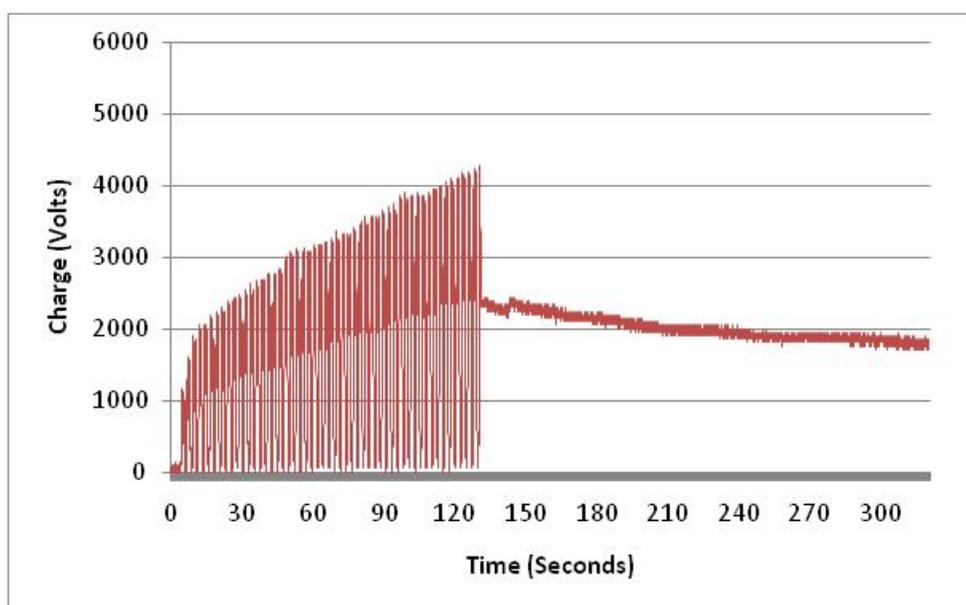
Appendix 19 Charge decay curve for nitric acid pretreated filament nylon 6,6 with electroless deposition sample 3



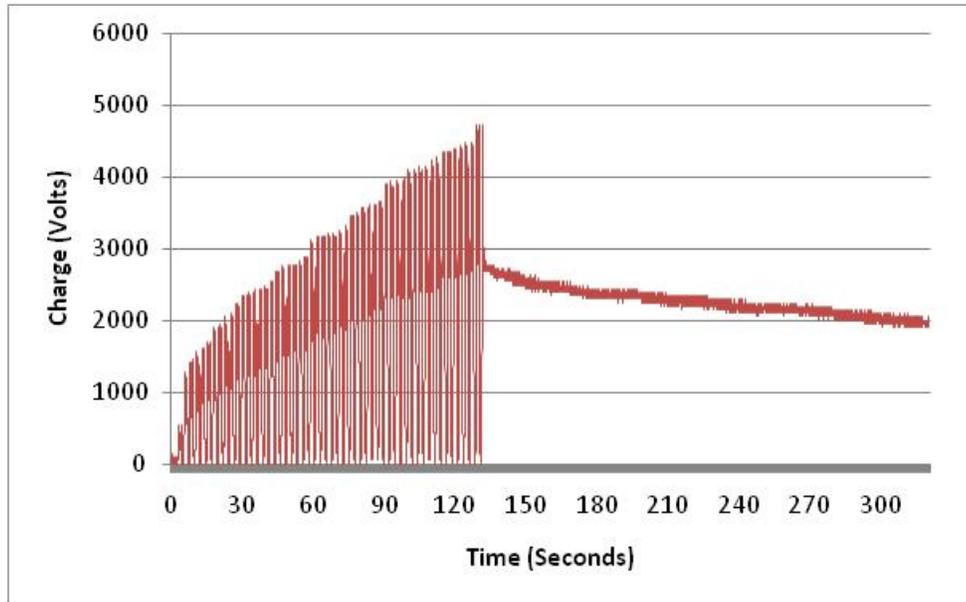
Appendix 20 Decay curves for nitric acid pretreated filament nylon 6,6 with electroless deposition samples 1-3



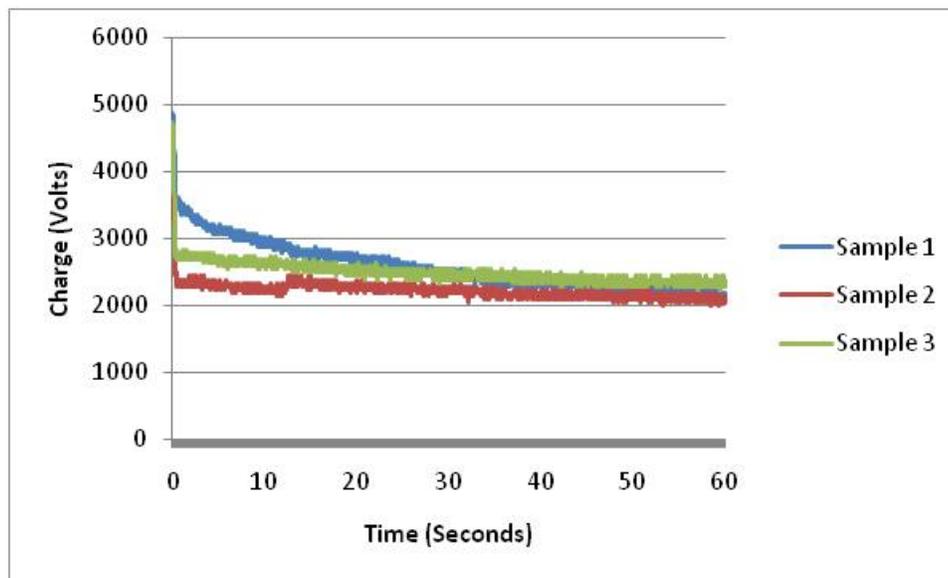
Appendix 21 Charge decay curve for NCAPS pretreated filament nylon 6,6 with electroless deposition sample 1



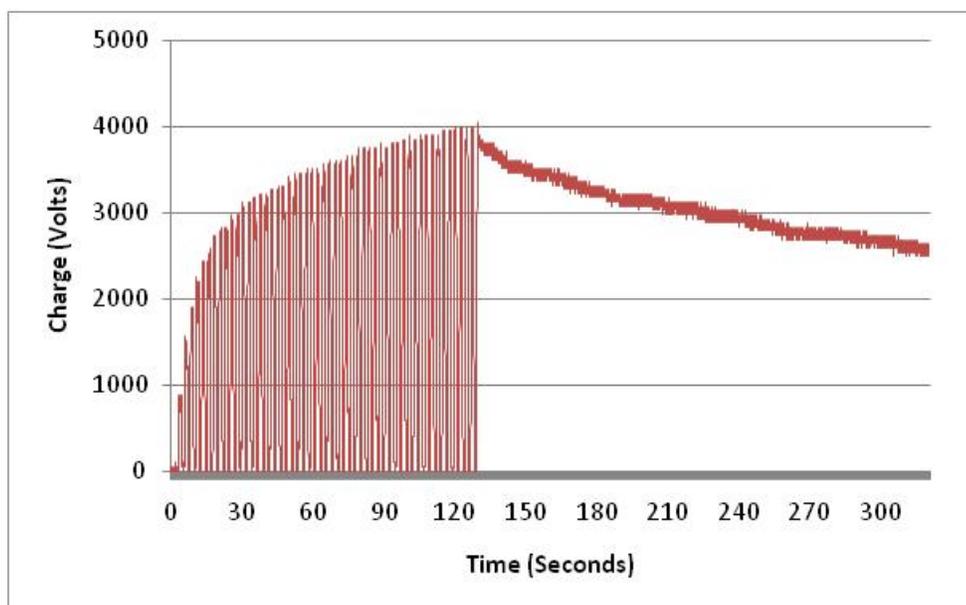
Appendix 22 Charge decay curve for NCAPS pretreated filament nylon 6,6 with electroless deposition sample 2



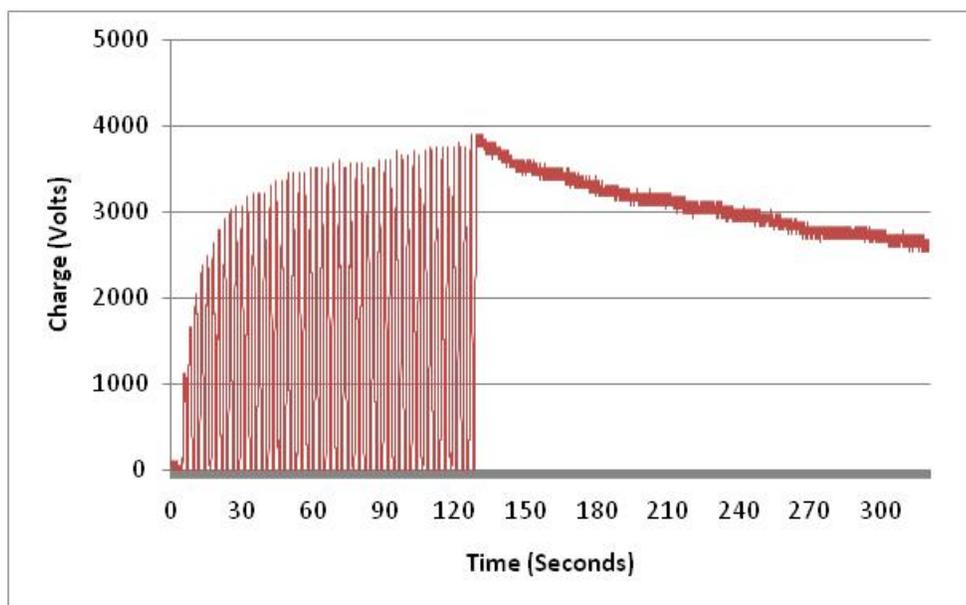
Appendix 23 Charge decay curve for NCAPS pretreated filament nylon 6,6 with electroless deposition sample 3



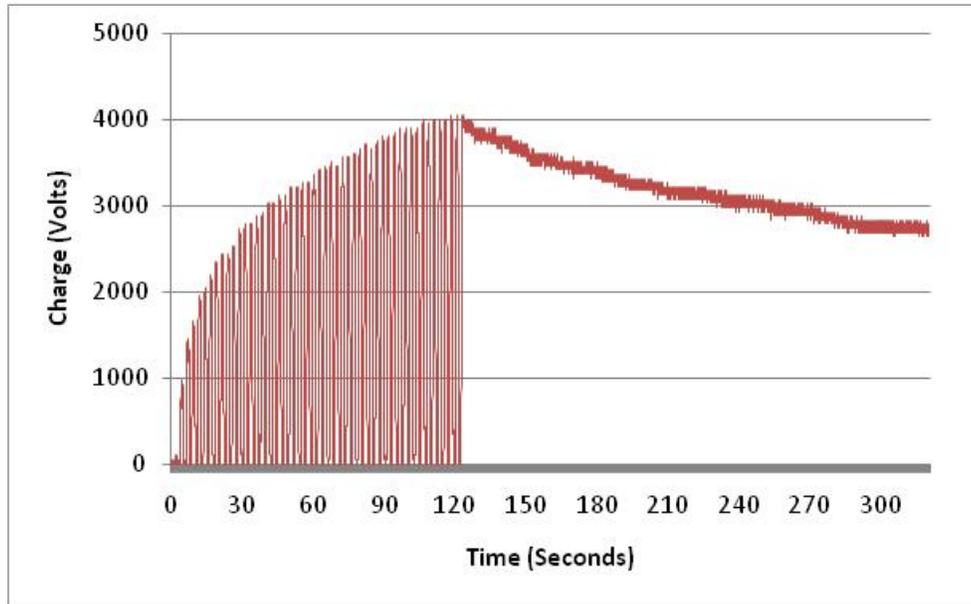
Appendix 24 Decay curves for NCAPS pretreated filament nylon 6,6 with electroless deposition samples 1-3



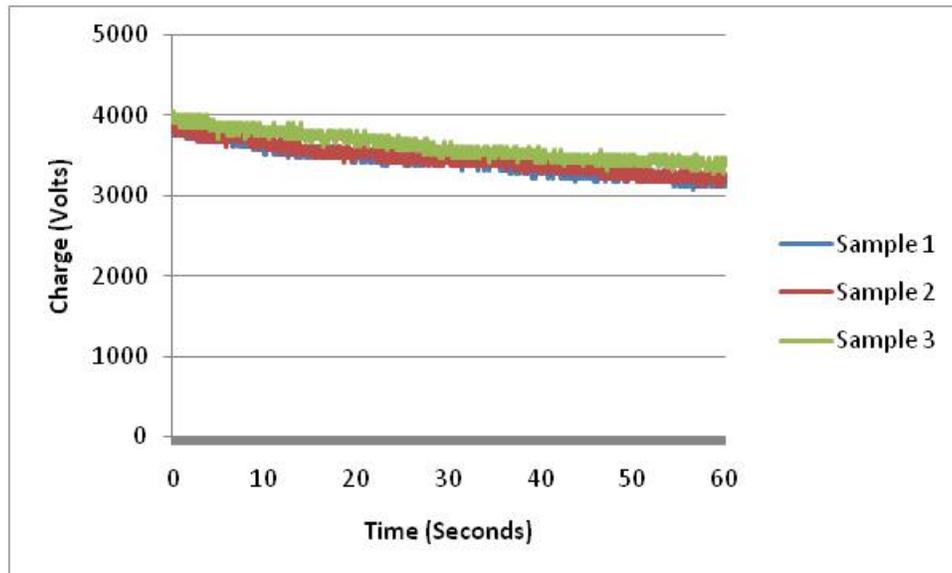
Appendix 25 Charge decay curve for untreated spun polyester sample 1



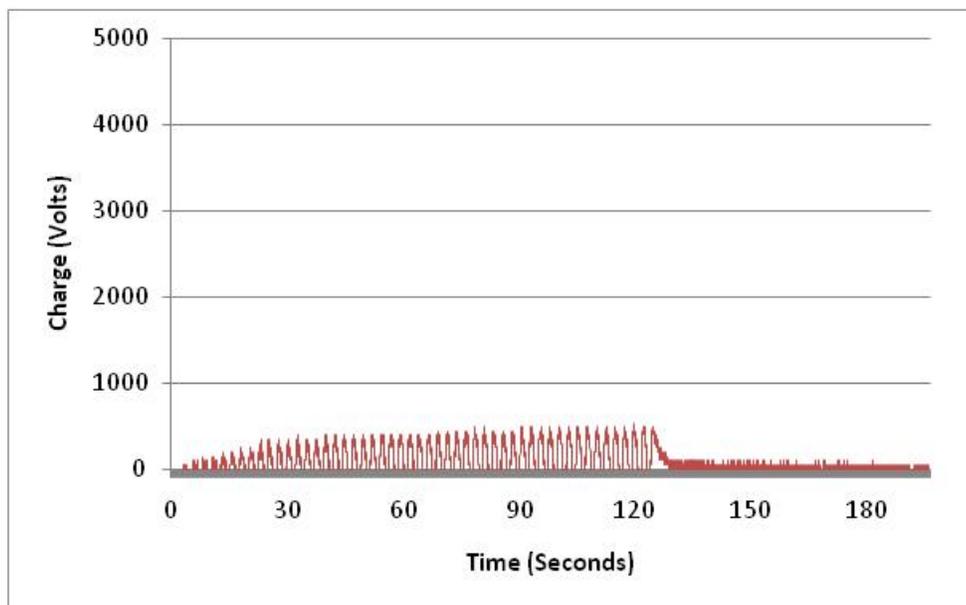
Appendix 26 Charge decay curve for untreated spun polyester sample 2



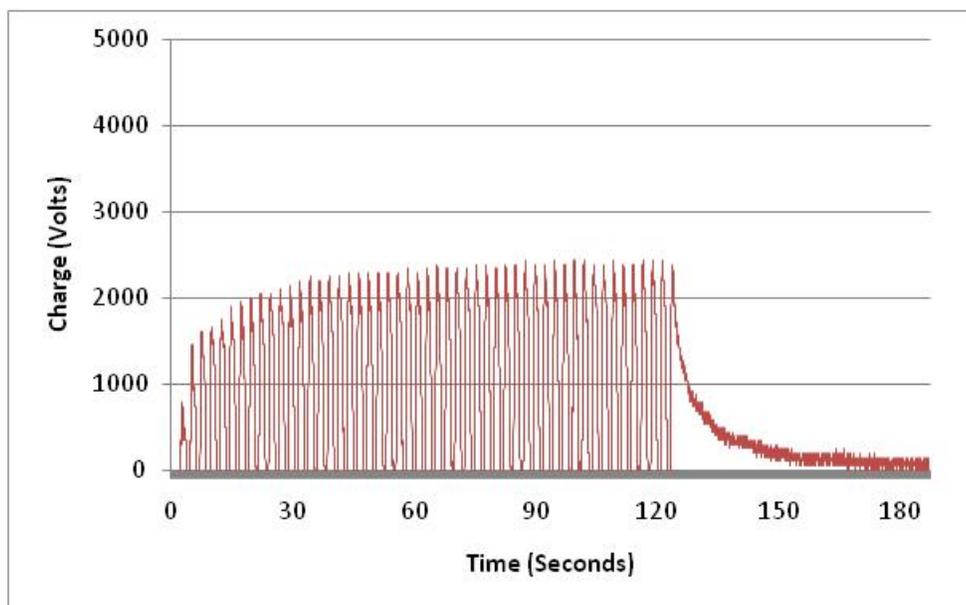
Appendix 27 Charge decay curve for untreated spun polyester sample 3



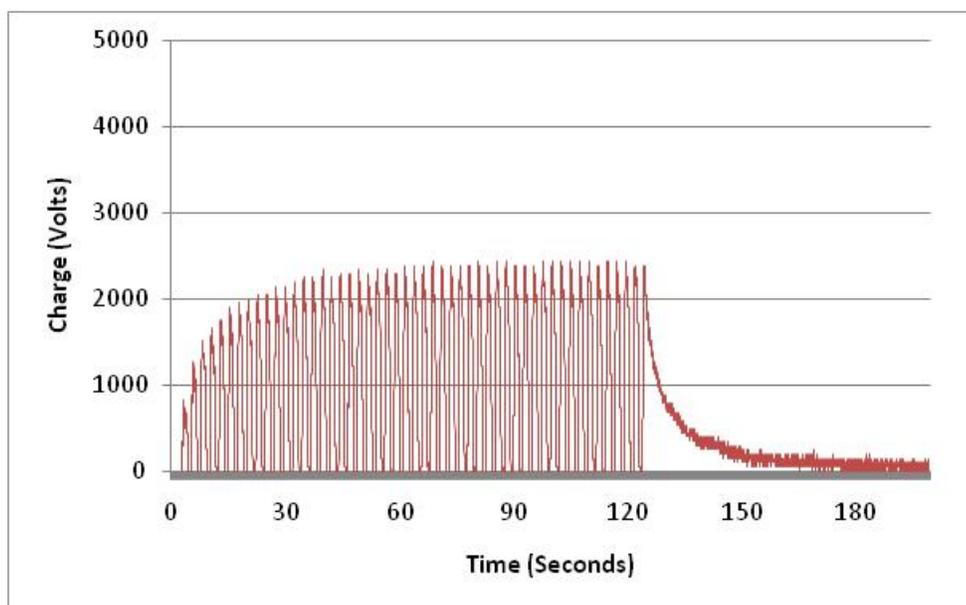
Appendix 28 Decay curves for untreated spun polyester samples 1-3



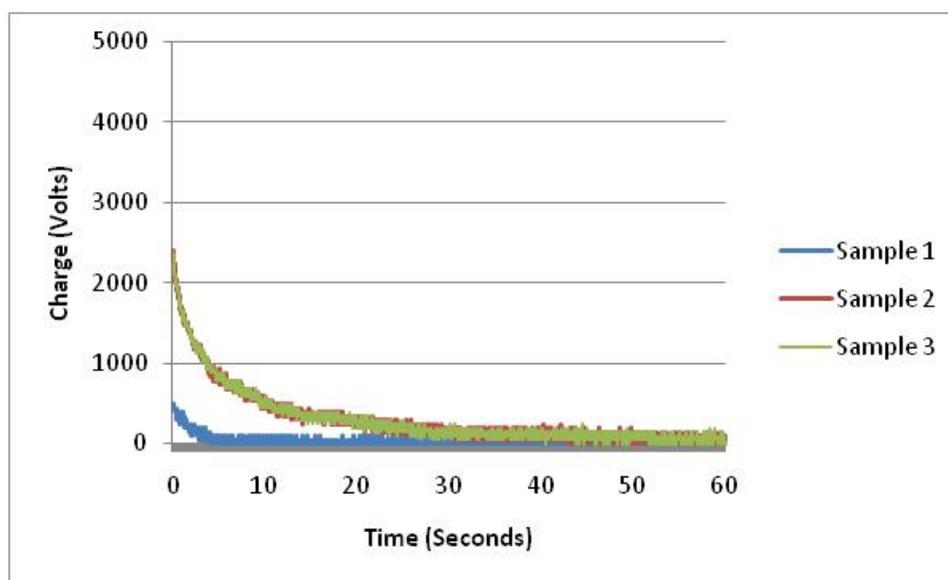
Appendix 29 Charge decay curve for nitric acid pretreated spun polyester with electroless deposition sample 1



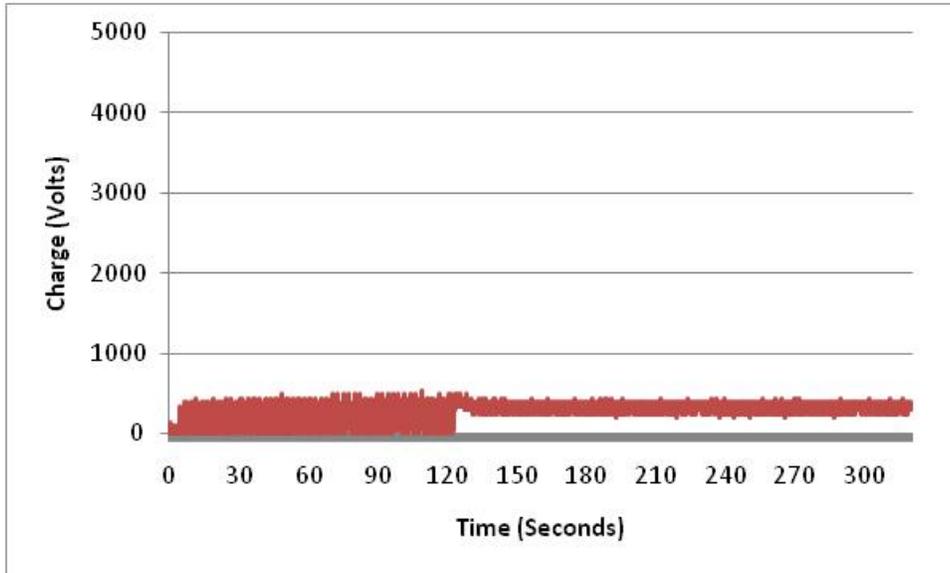
Appendix 30 Charge decay curve for nitric acid pretreated spun polyester with electroless deposition sample 2



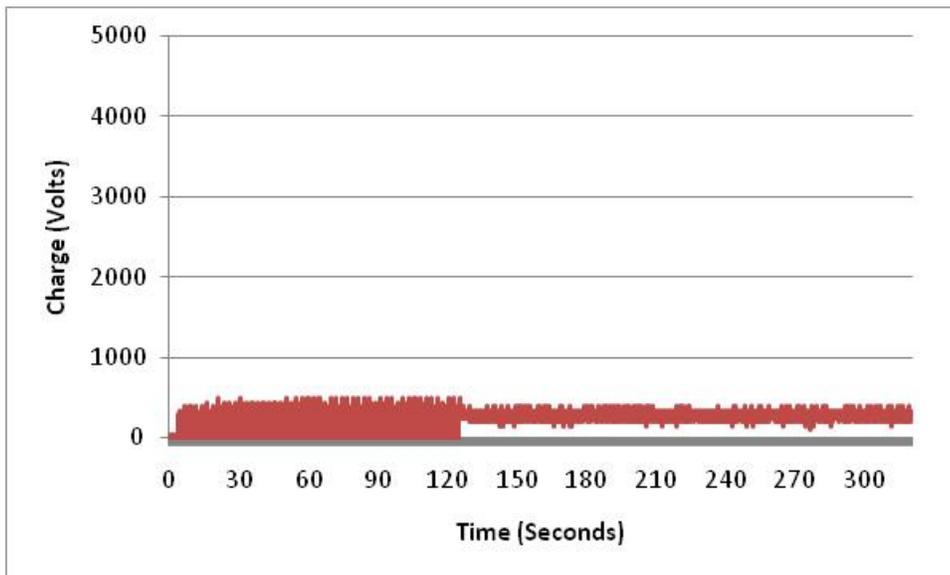
Appendix 31 Charge decay curve for nitric acid pretreated spun polyester with electroless deposition sample 3



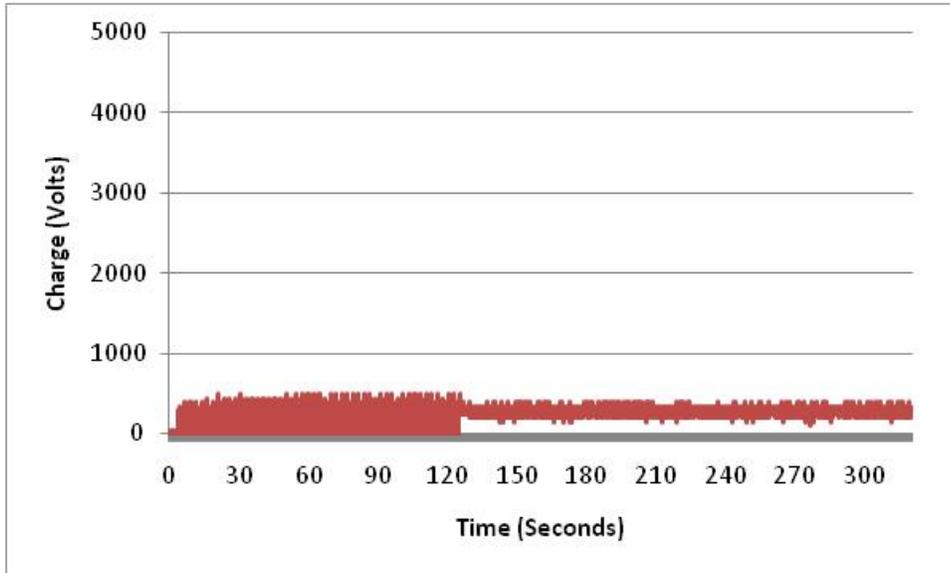
Appendix 32 Decay curves for nitric acid pretreated spun polyester samples 1-3



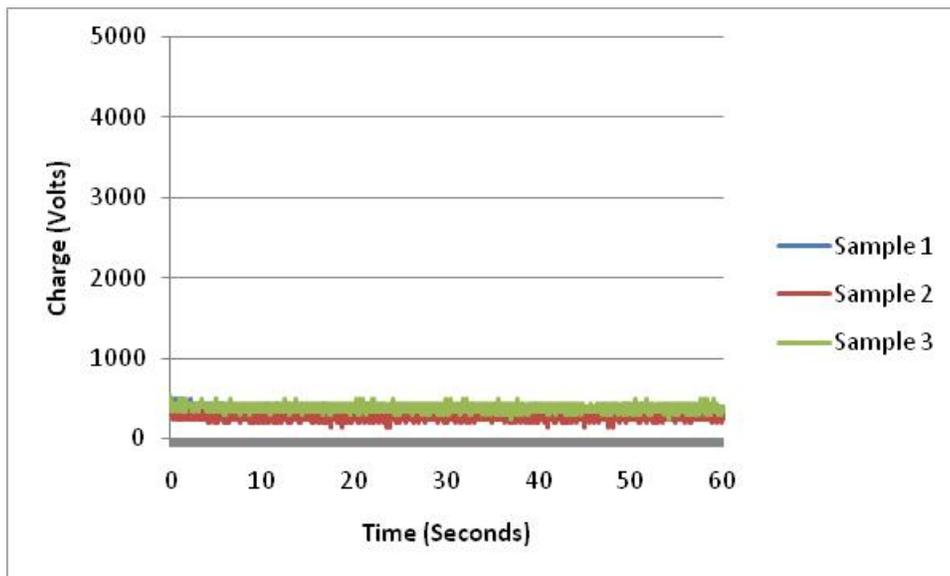
Appendix 33 Charge decay curve for NCAPS pretreated spun polyester with electroless deposition sample 1



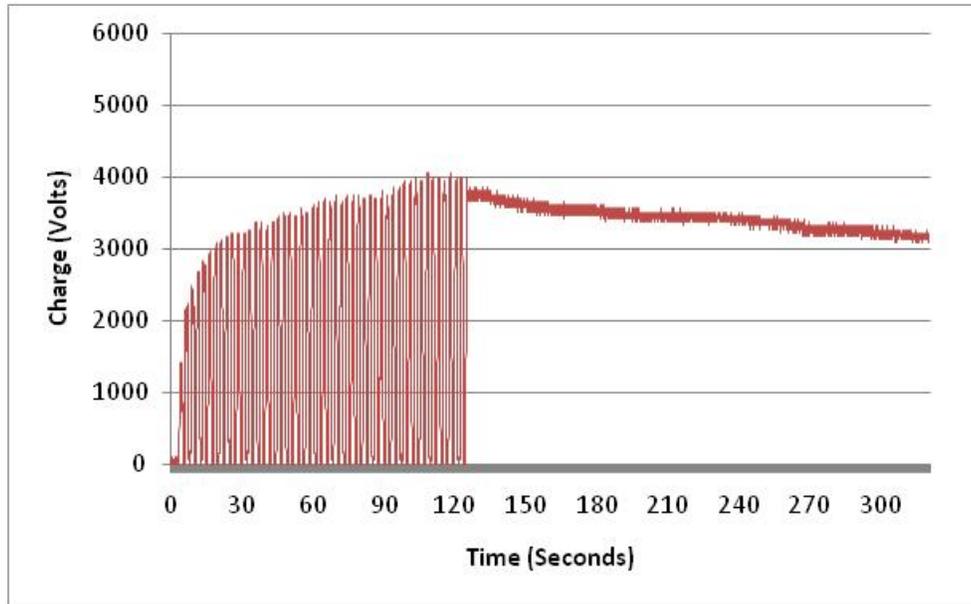
Appendix 34 Charge decay curve for NCAPS pretreated spun polyester with electroless deposition sample 2



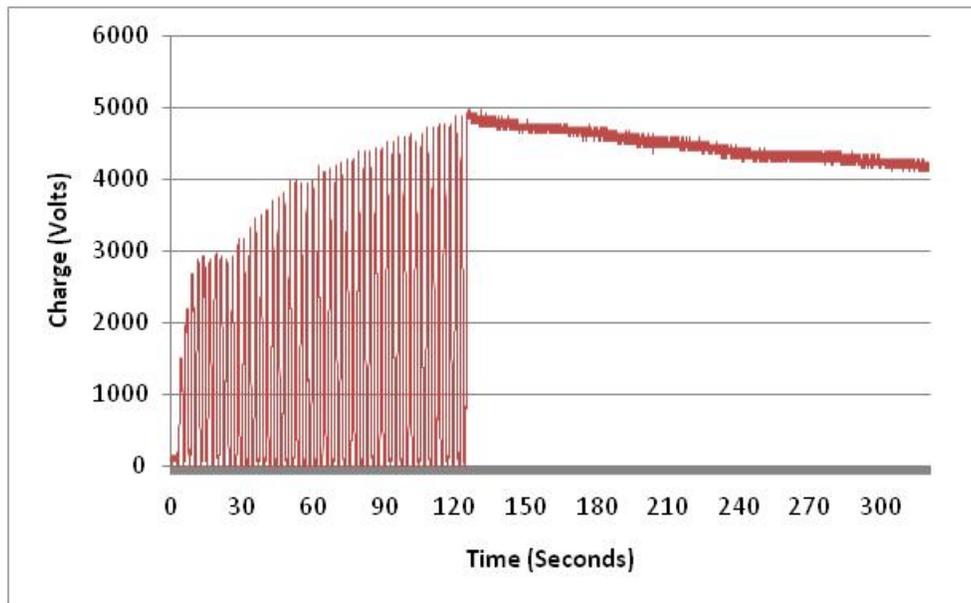
Appendix 35 Charge decay curve for NCAPS pretreated spun polyester with electroless deposition sample 3



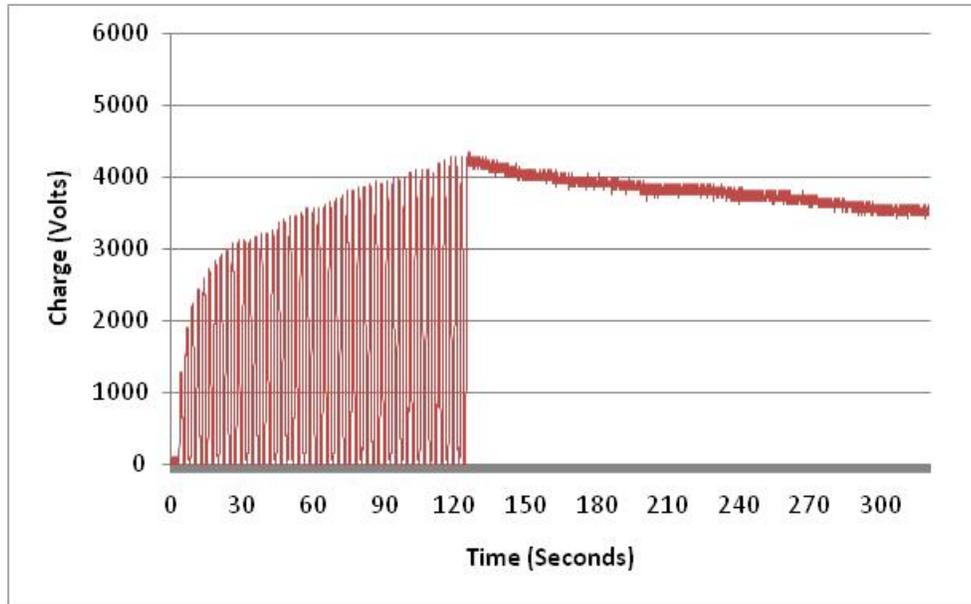
Appendix 36 Decay curves for NCAPS pretreated spun polyester samples 1-3



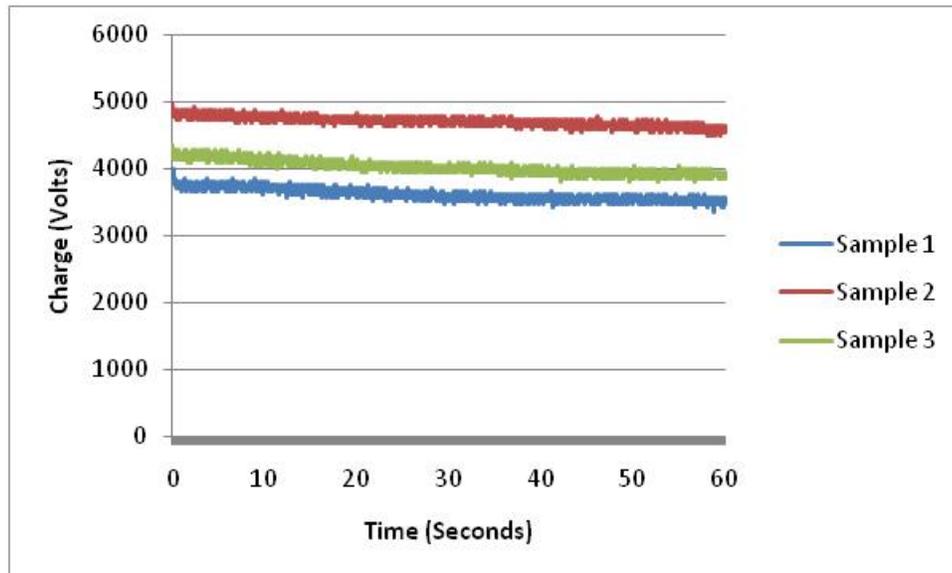
Appendix 37 Charge decay curve for untreated filament polyester sample 1



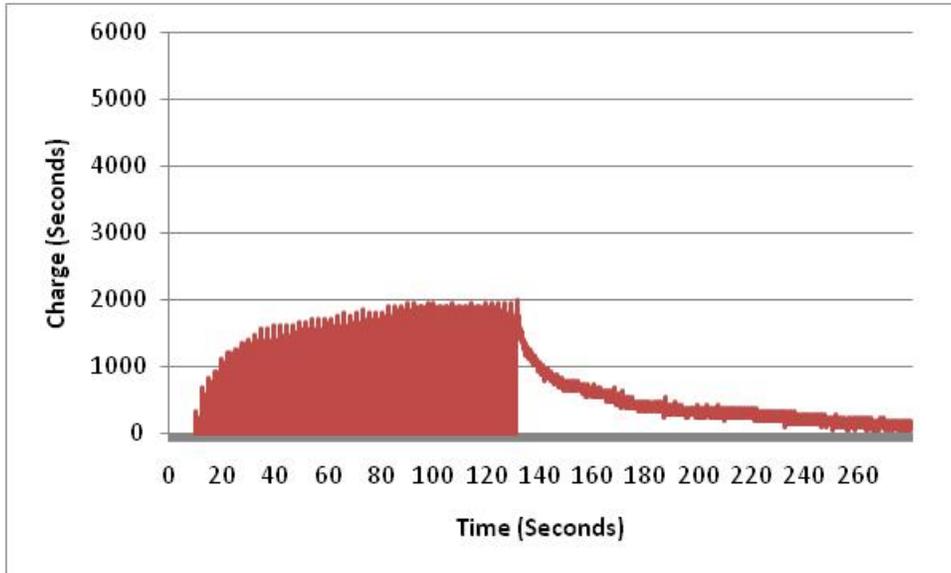
Appendix 38 Charge decay curve for untreated filament polyester sample 2



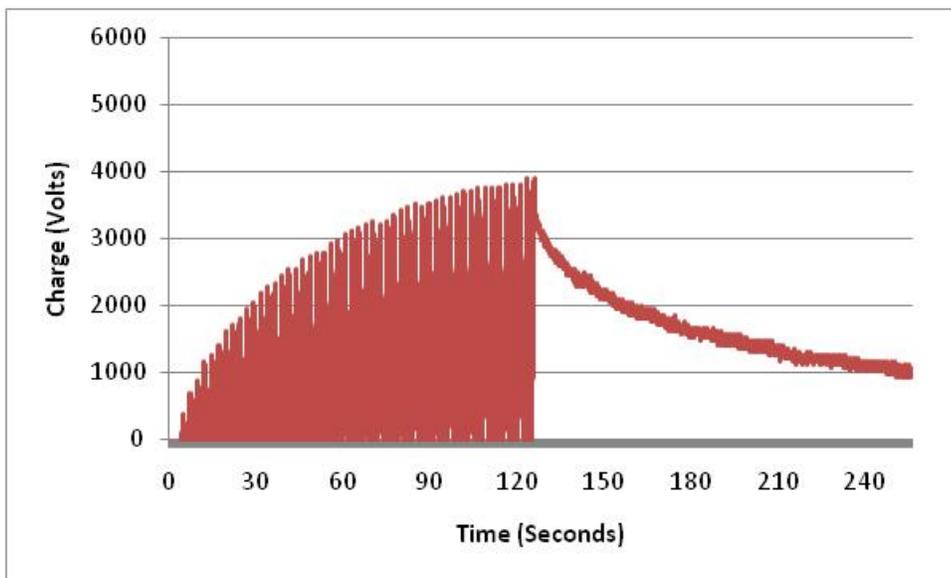
Appendix 39 Charge decay curve for untreated filament polyester sample 3



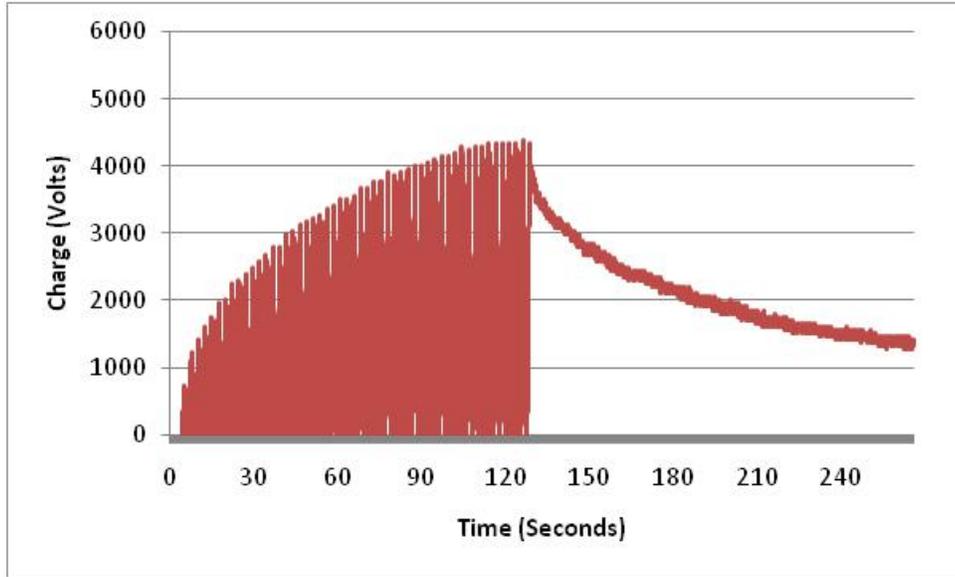
Appendix 40 Decay curves for untreated filament polyester samples 1-3



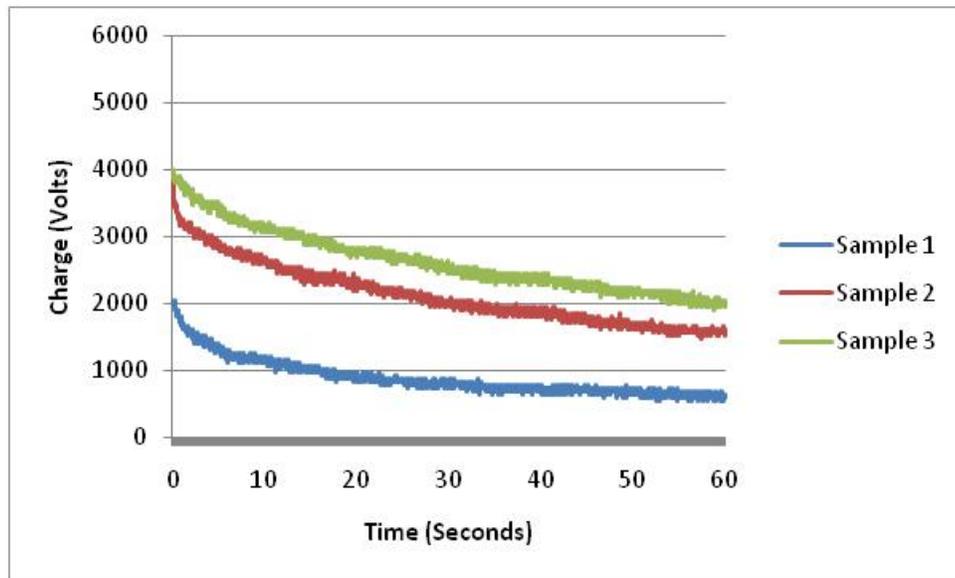
Appendix 41 Charge decay curve for nitric acid pretreated filament polyester with electroless deposition sample 1



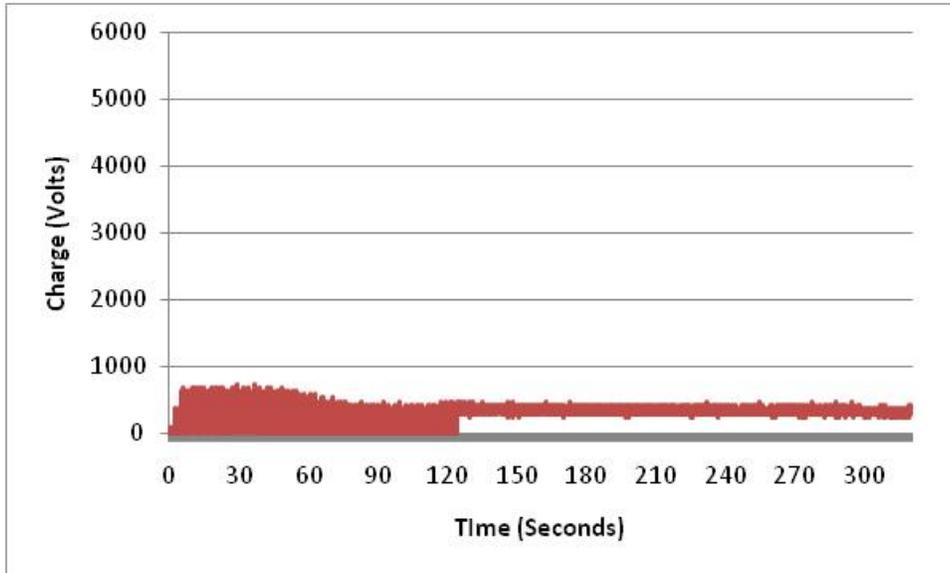
Appendix 42 Charge decay curve for nitric acid pretreated filament polyester sample with electroless deposition sample 2



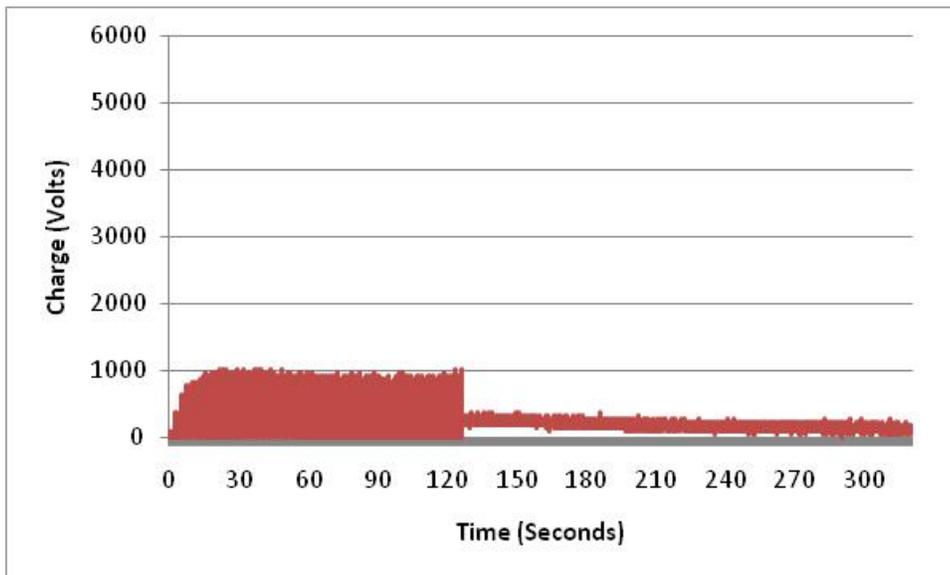
Appendix 43 Charge decay curve for nitric acid pretreated filament polyester sample with electroless deposition sample 3



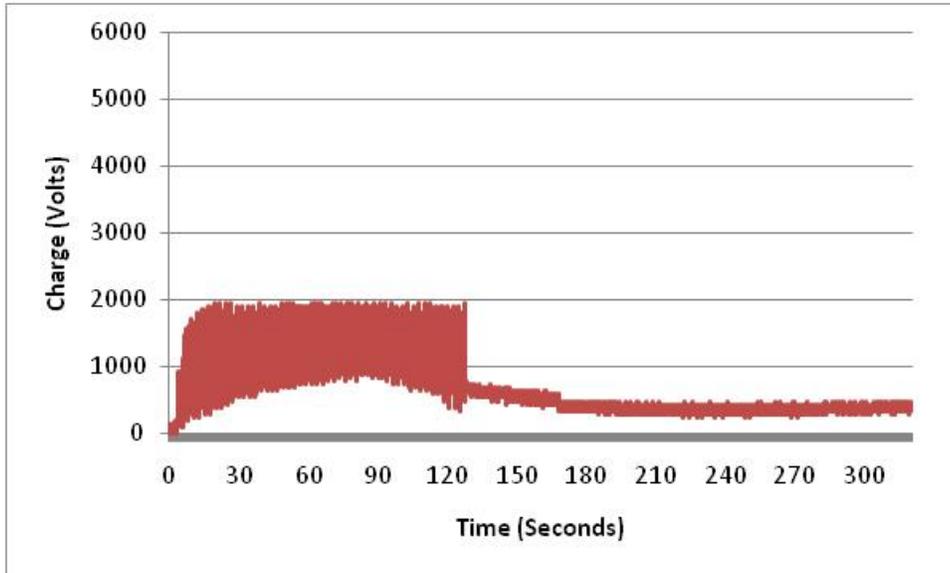
Appendix 44 Decay curves for nitric acid pretreated filament polyester samples 1-3



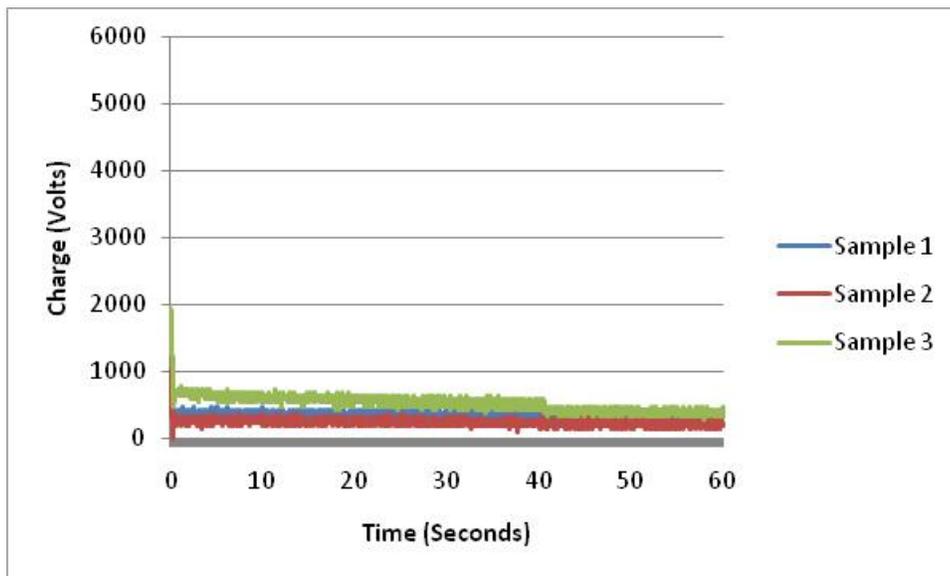
Appendix 45 Charge decay curve for NCAPS pretreated filament polyester sample with electroless deposition sample 1



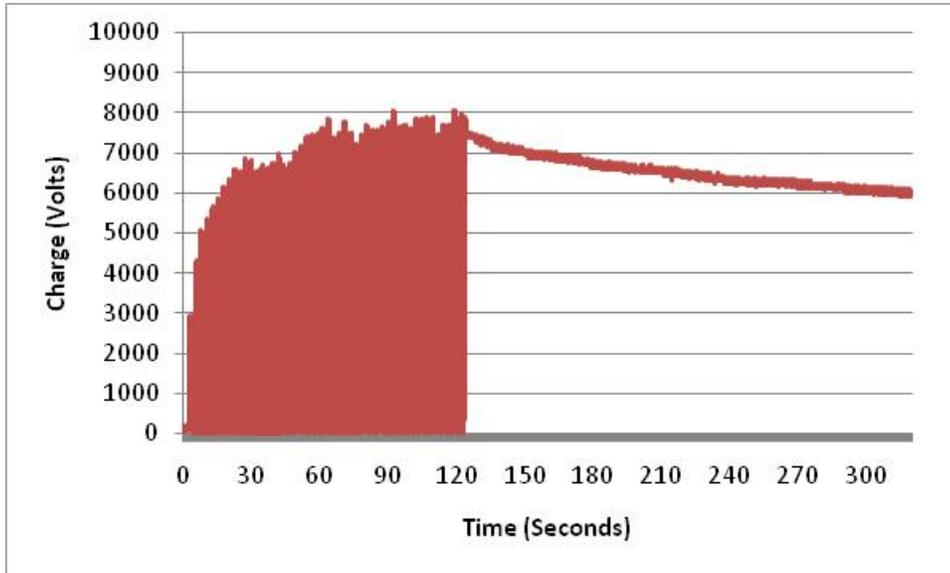
Appendix 46 Charge decay curve for NCAPS pretreated filament polyester sample with electroless deposition sample 2



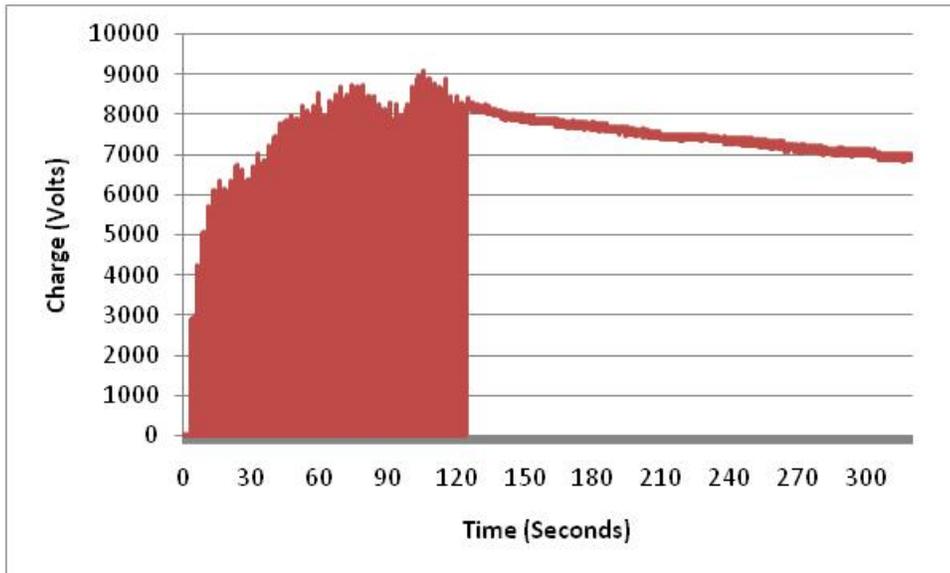
Appendix 47 Charge decay curve for NCAPS pretreated filament polyester sample with electroless deposition sample 3



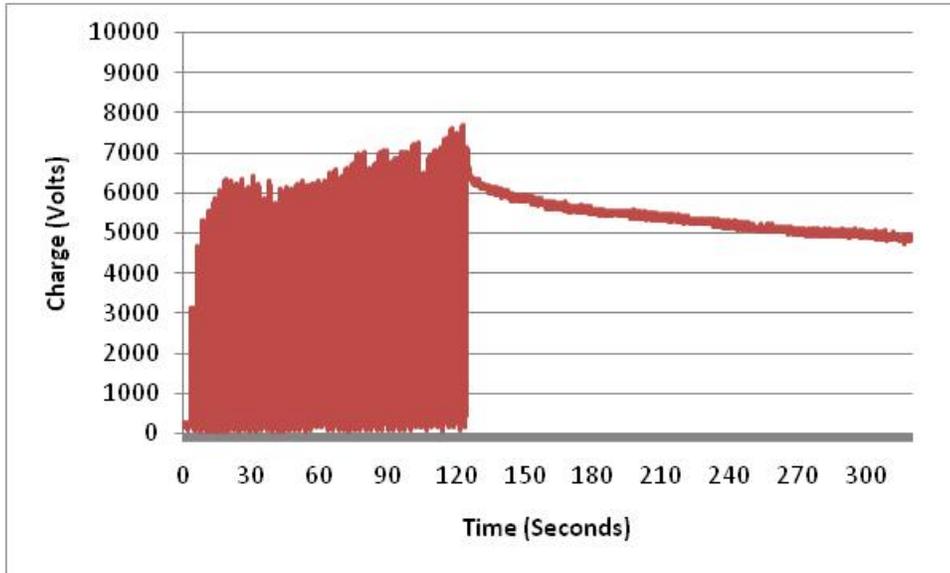
Appendix 48 Decay curves for NCAPS pretreated filament polyester samples 1-3



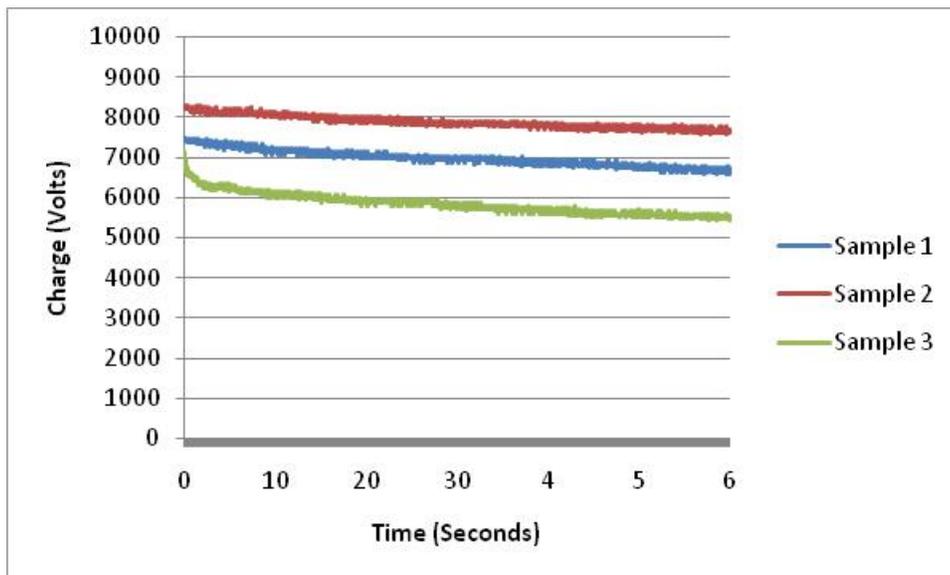
Appendix 49 Charge decay curve for untreated spun acrylic sample 1



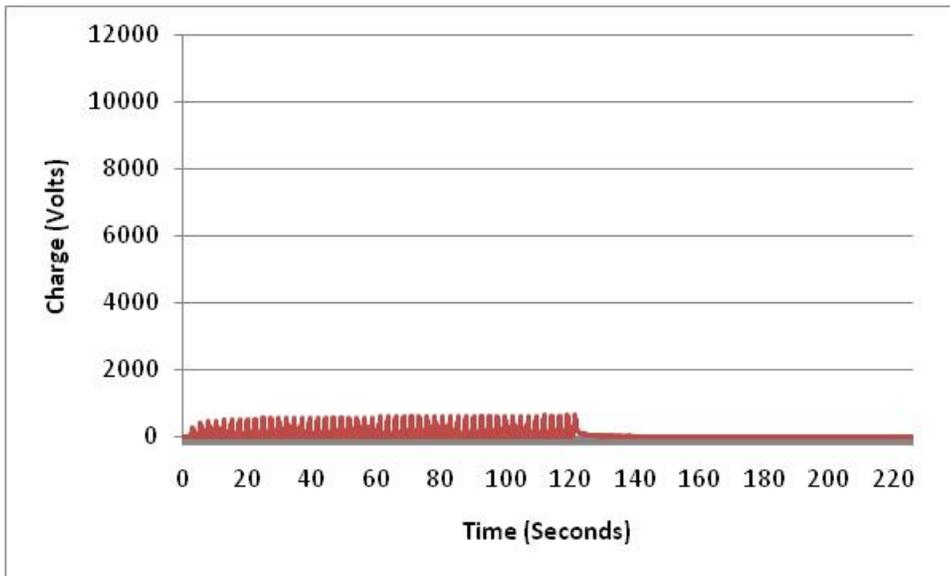
Appendix 50 Charge decay curve for untreated spun acrylic sample 2



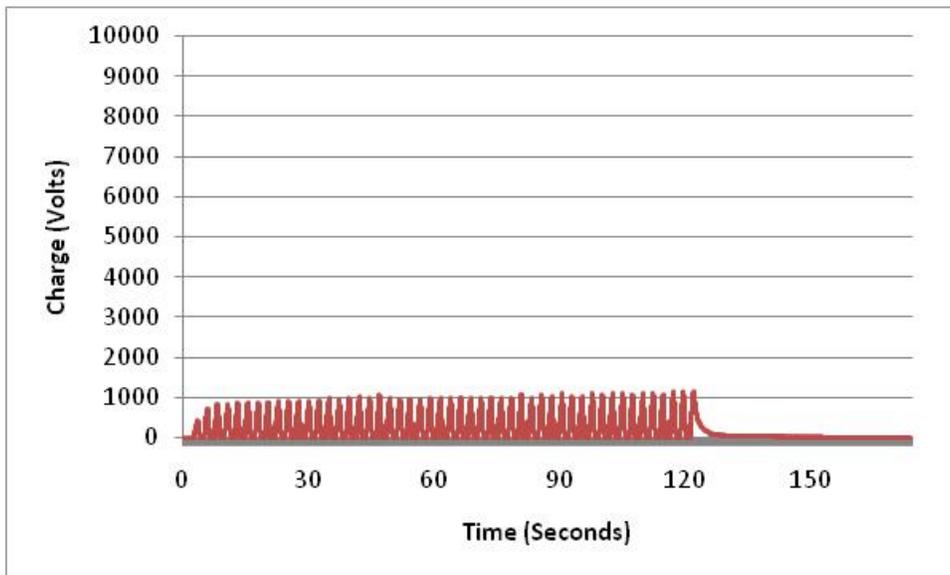
Appendix 51 Charge decay curve for untreated spun acrylic sample 3



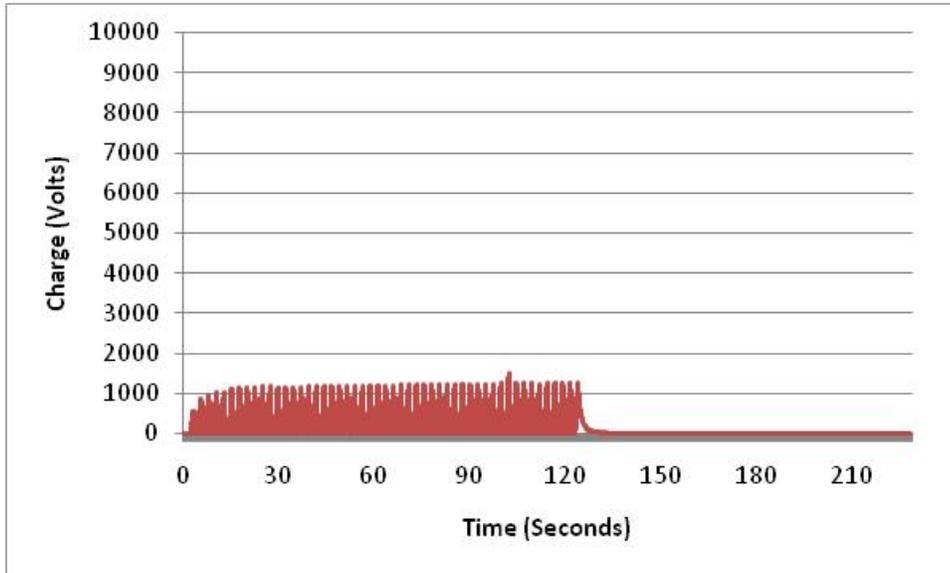
Appendix 52 Decay curves for untreated spun acrylic samples 1-3



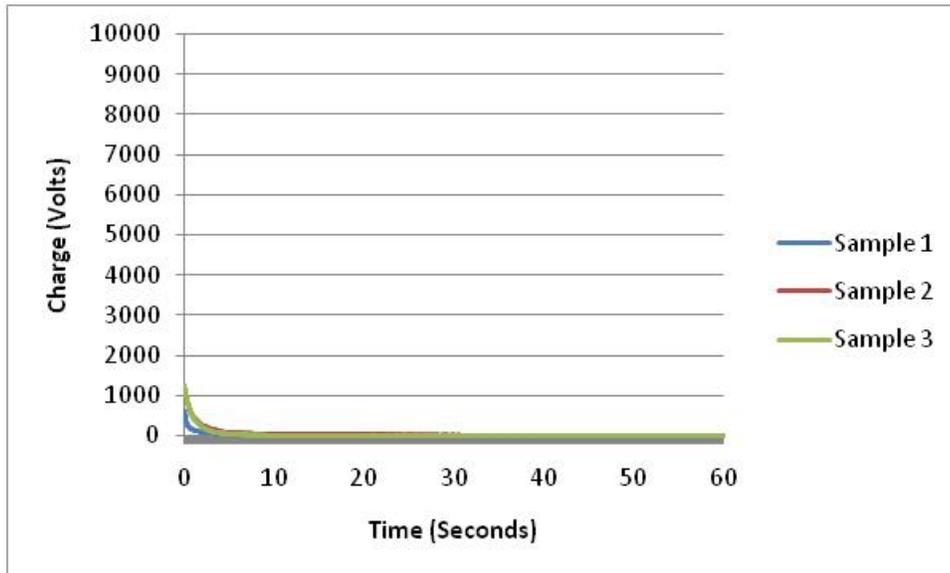
Appendix 53 Charge decay curve for nitric acid pretreated spun acrylic with electroless deposition sample 1



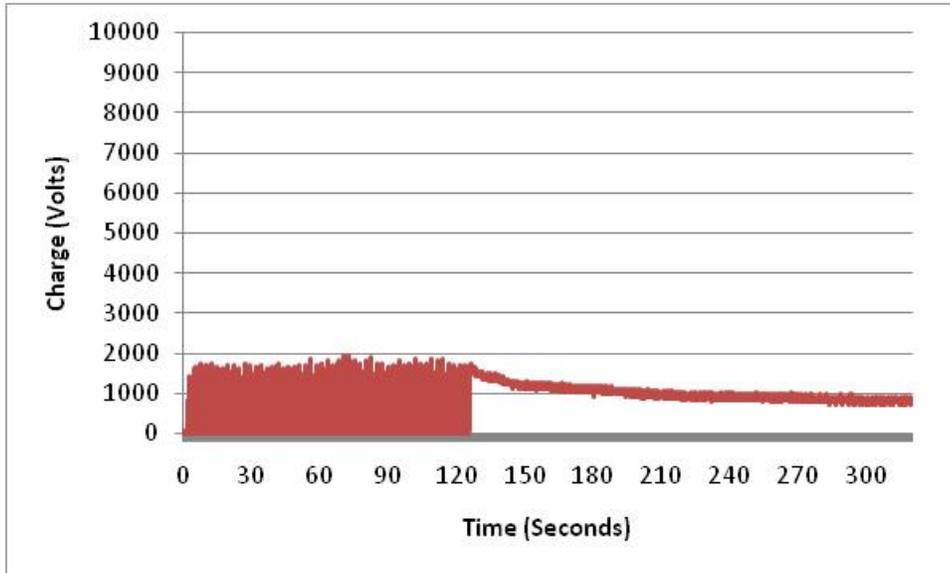
Appendix 54 Charge decay curve for nitric acid pretreated spun acrylic with electroless deposition sample 2



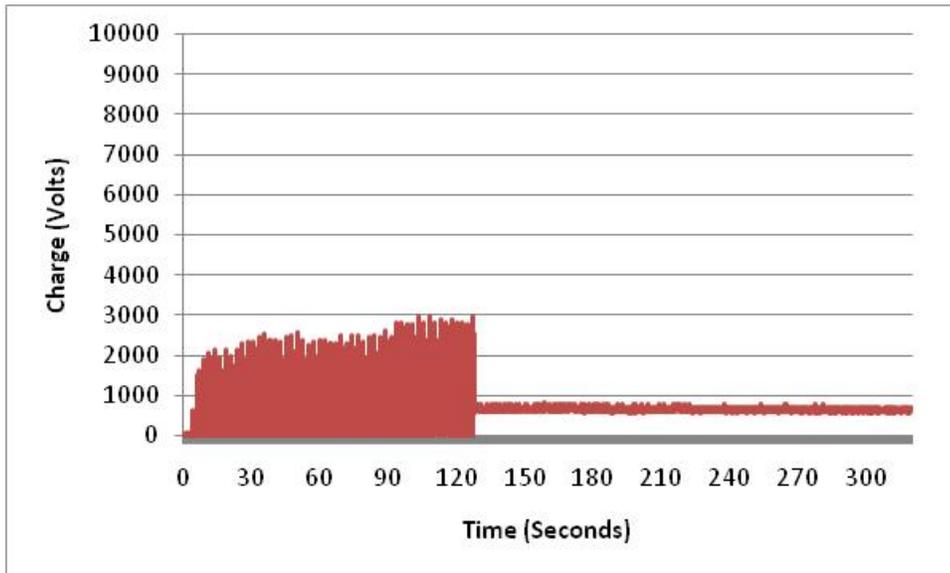
Appendix 55 Charge decay curve for nitric acid pretreated spun acrylic with electroless deposition sample 3



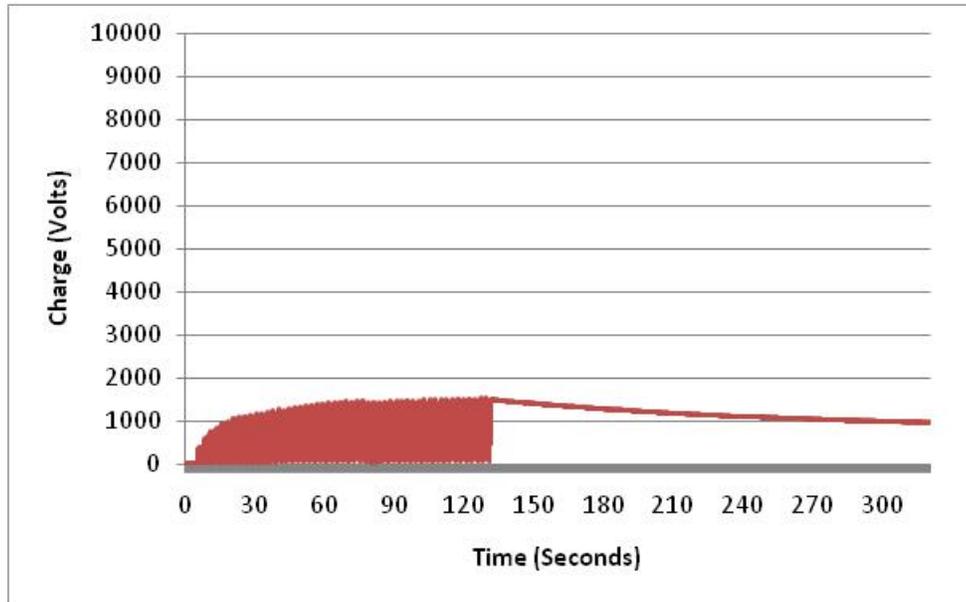
Appendix 56 Decay curves for nitric acid pretreated spun acrylic samples 1-3



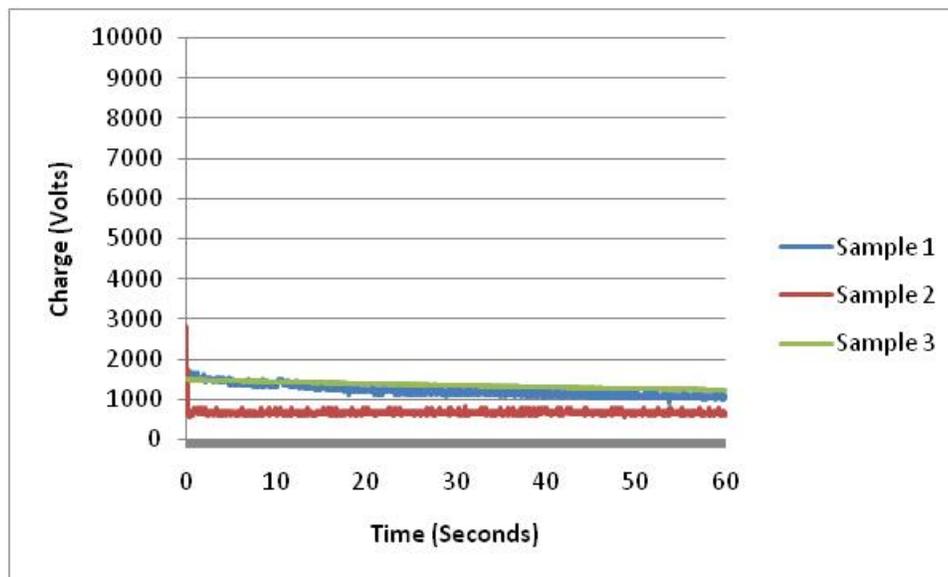
Appendix 57 Charge decay curve for NCAPS pretreated spun acrylic with electroless deposition sample 1



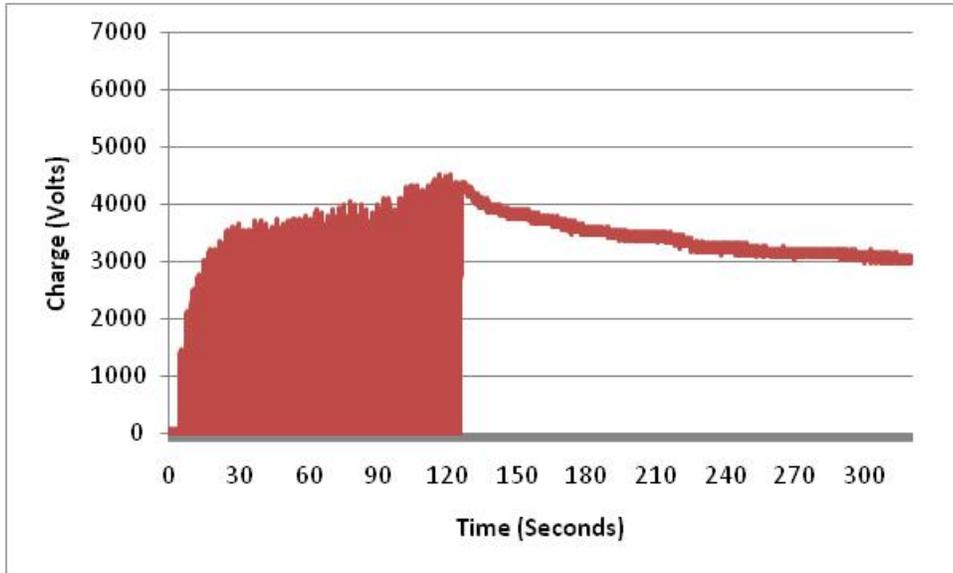
Appendix 58 Charge decay curve for NCAPS pretreated spun acrylic with electroless deposition sample 2



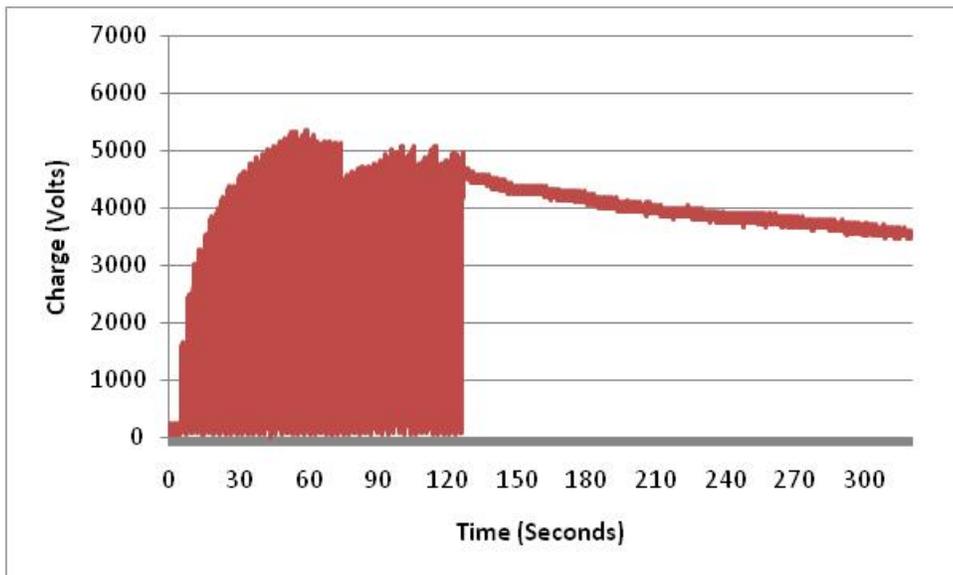
Appendix 59 Charge decay curve for NCAPS pretreated spun acrylic with electroless deposition sample 3



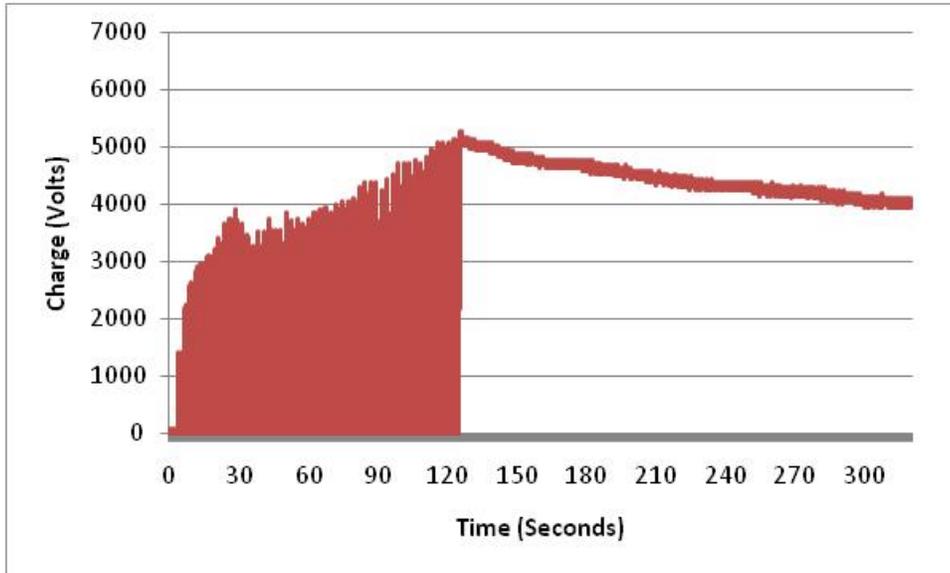
Appendix 60 Decay curves for NCAPS pretreated spun acrylic samples 1-3



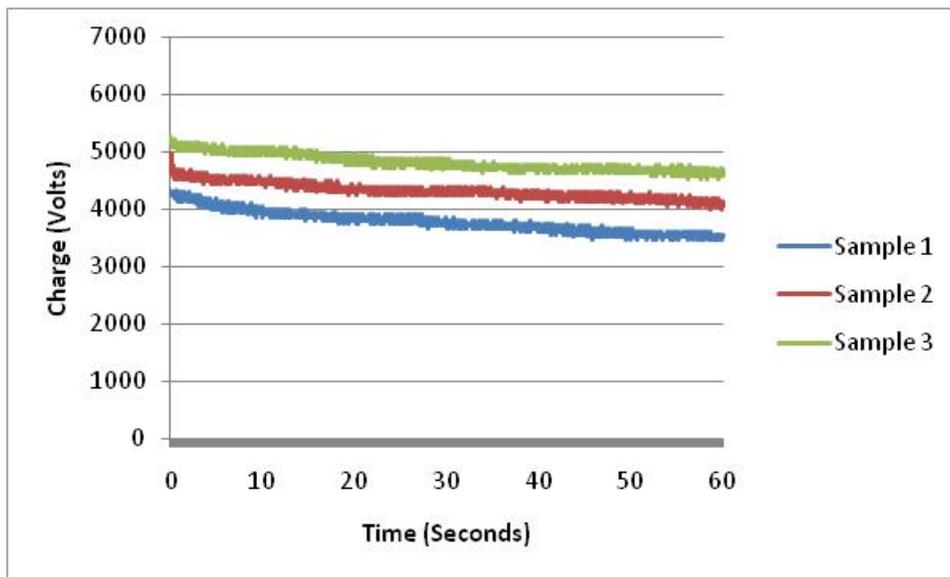
Appendix 61 Charge decay curve for untreated spun polypropylene sample 1



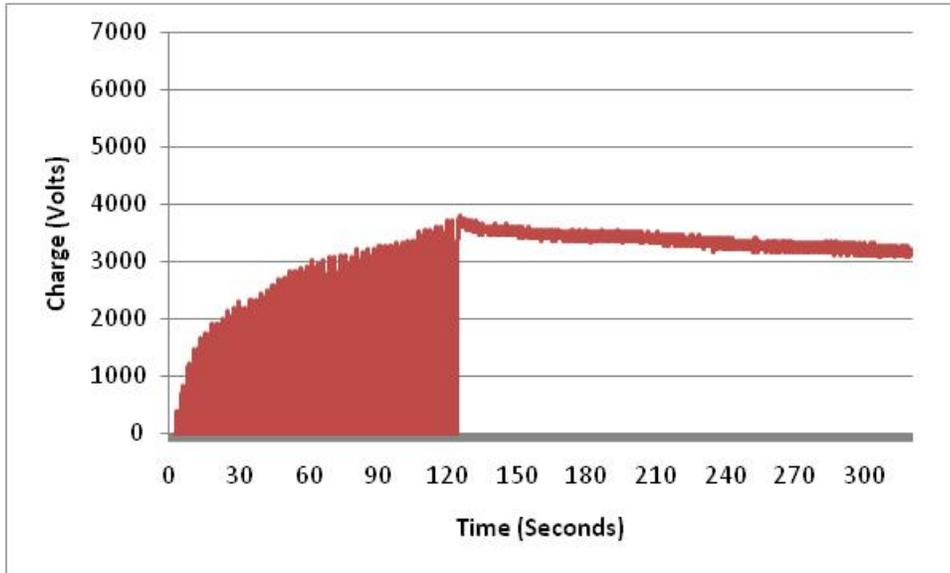
Appendix 62 Charge decay curve for untreated spun polypropylene sample 2



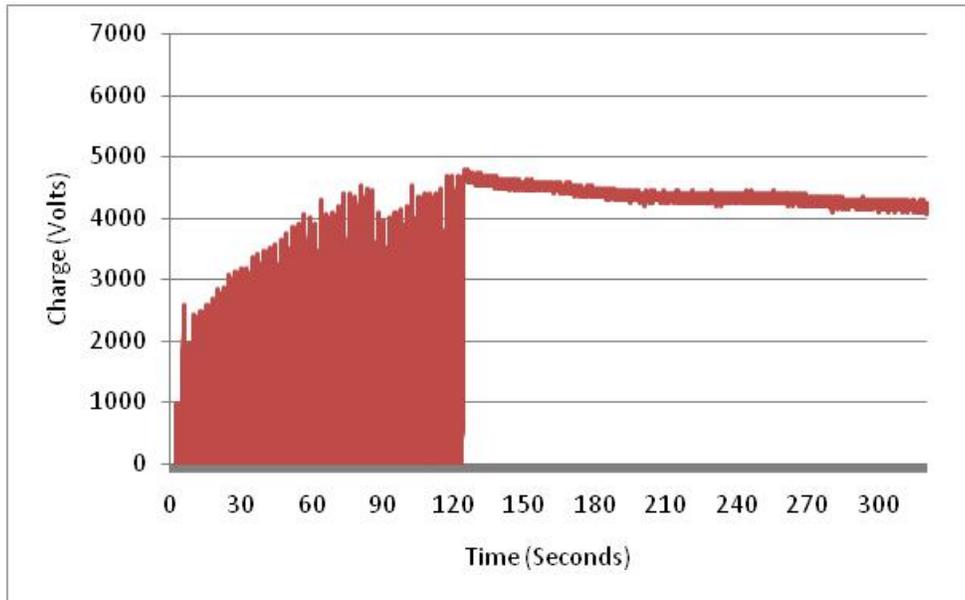
Appendix 63 Charge decay curve for untreated spun polypropylene sample 3



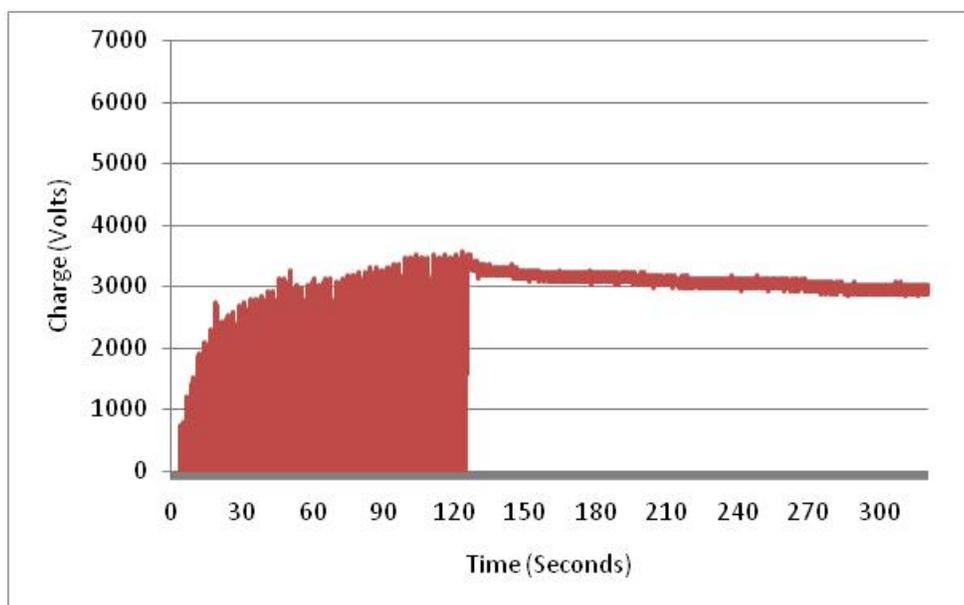
Appendix 64 Decay curves for untreated spun polypropylene samples 1-3



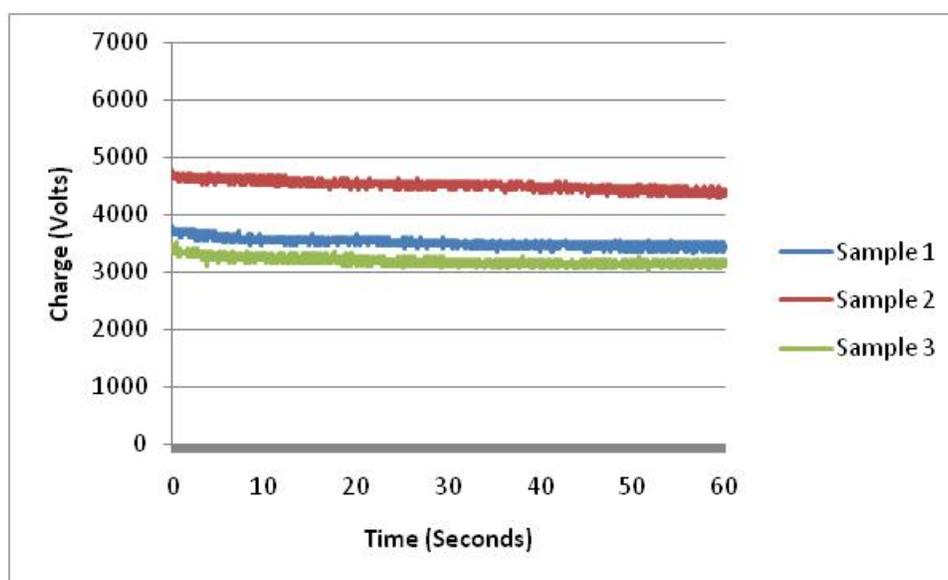
Appendix 65 Charge decay curve for nitric acid pretreated spun polypropylene with electroless deposition sample 1



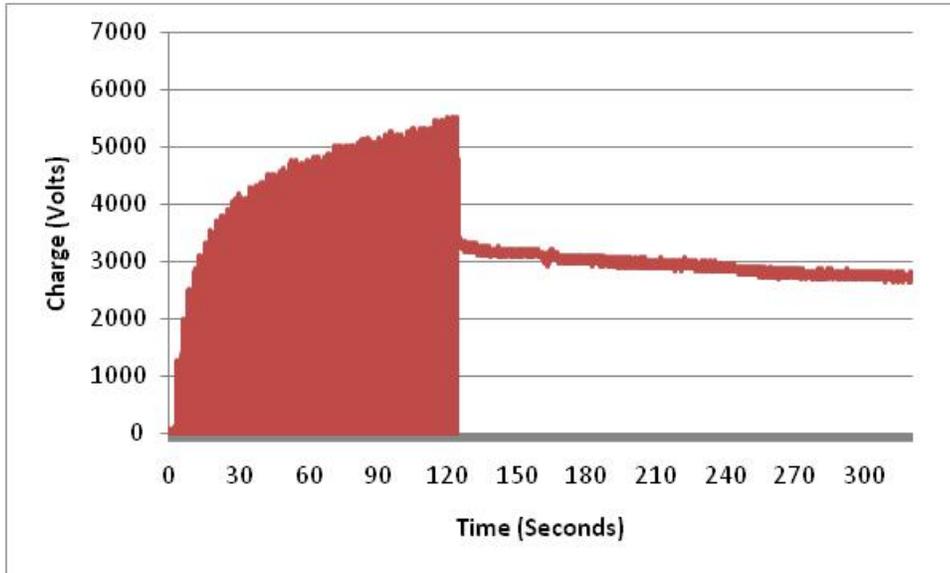
Appendix 66 Charge decay curve for nitric acid pretreated spun polypropylene with electroless deposition sample 2



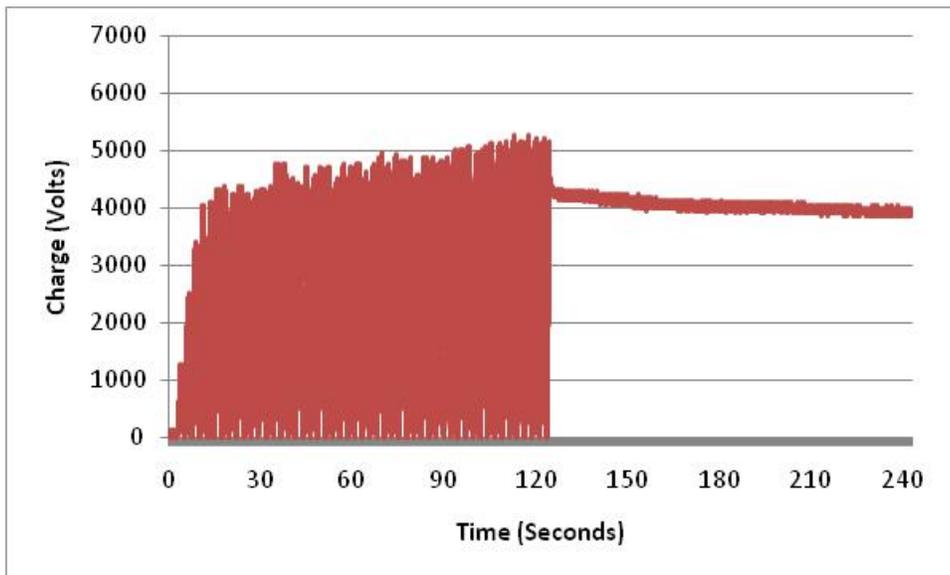
Appendix 67 Charge decay curve for nitric acid pretreated spun polypropylene with electroless deposition sample 3



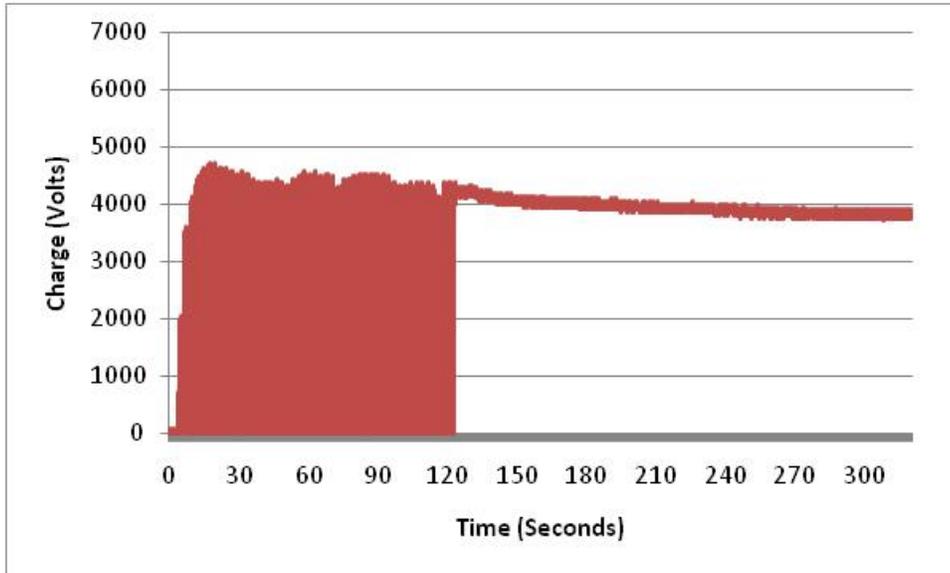
Appendix 68 Charge decay curves for nitric acid pretreated spun polypropylene samples 1-3



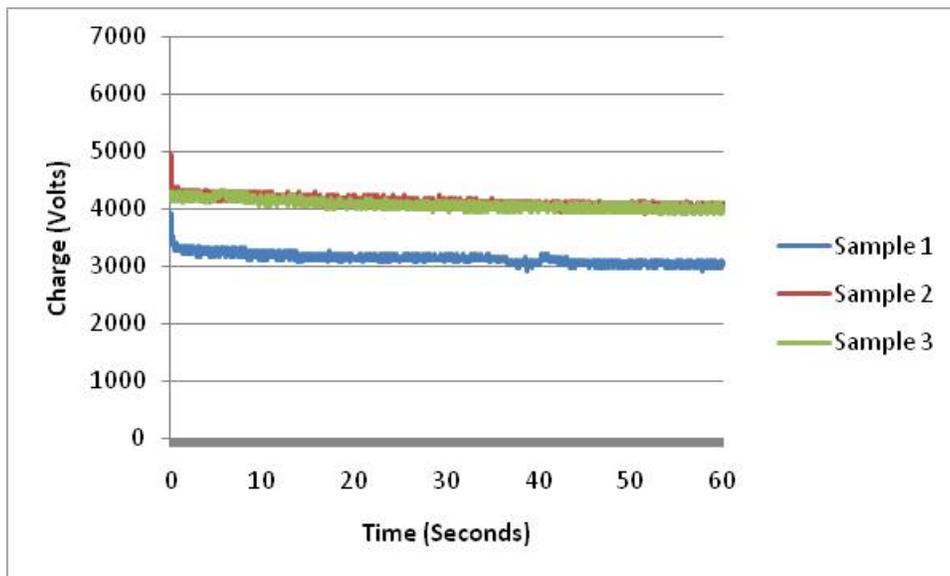
Appendix 69 Charge decay curve for NCAPS pretreated spun polypropylene with electroless deposition sample 1



Appendix 70 Charge decay curve for NCAPS pretreated spun polypropylene with electroless deposition sample 2



Appendix 71 Charge decay curve for NCAPS pretreated spun polypropylene with electroless deposition sample 3



Appendix 72 Charge decay curves for NCAPS pretreated spun polypropylene samples 1-3



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205 East Meadow Road - Suite A
Eden, NC 27288
(336)623-8921

Pace Analytical Services, Inc.
2225 Riverside Dr.
Asheville, NC 28804
(828)254-7176

Pace Analytical Services, Inc.
9800 Kinsey Ave. Suite 100
Huntersville, NC 28078
(704)875-9092

March 10, 2011

Matt Crutchfield
NCSU Institute of Textile Tech
1000 Main Campus Drive
Raleigh, NC 27695

RE: Project: MATT CRUTCHFIELD THESIS
Pace Project No.: 9288905

Dear Matt Crutchfield:

Enclosed are the analytical results for sample(s) received by the laboratory on March 01, 2011. The results relate only to the samples included in this report. Results reported herein conform to the most current NELAC standards, where applicable, unless otherwise narrated in the body of the report.

Analyses were performed at the Pace Analytical Services location indicated on the sample analyte page for analysis unless otherwise footnoted.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Kevin Herring

kevin.herring@pacelabs.com
Project Manager

Enclosures

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Pace Analytical Services, Inc.
2225 Riverside Dr.
Asheville, NC 28804
(828)254-7176

Pace Analytical Services, Inc.
9800 Kinsey Ave. Suite 100
Huntersville, NC 28078
(704)875-9092

CERTIFICATIONS

Project: MATT CRUTCHFIELD THESIS
Pace Project No.: 9288905

Asheville Certification IDs

2225 Riverside Dr., Asheville, NC 28804
Connecticut Certification #: PH-0106
Florida/NELAP Certification #: E87648
Massachusetts Certification #: M-NC030
New Jersey Certification #: NC011
North Carolina Bioassay Certification #: 9
North Carolina Drinking Water Certification #: 37712

North Carolina Wastewater Certification #: 40
Pennsylvania Certification #: 68-03578
South Carolina Bioassay Certification #: 99030002
South Carolina Certification #: 99030001
Virginia Certification #: 00072
West Virginia Certification #: 356

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 9800 Kinsey Ave. Suite 100
 Huntersville, NC 28078
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SAMPLE ANALYTE COUNT

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
9288905001	UNTREATED SPUN NYLON 6.6	EPA 6010	JMW	1	PASI-A
9288905002	UNTREATED FILAMENT NYLON 6.6	EPA 6010	JMW	1	PASI-A
9288905003	UNTREATED SPUN POLYESTER	EPA 6010	JMW	1	PASI-A
9288905004	UNTREATED FILAMENT POLYESTER	EPA 6010	JMW	1	PASI-A
9288905005	UNTREATED SPUN ACRYLIC	EPA 6010	JMW	1	PASI-A
9288905006	UNTREATED SPUN POLYPROPYLENE	EPA 6010	JMW	1	PASI-A
9288905007	NA+ED SPUN NYLON 6.6	EPA 6010	JMW	1	PASI-A
9288905008	NA+ED FILAMENT NYLON 6.6	EPA 6010	JMW	1	PASI-A
9288905009	NA+ED SPUN POLYESTER	EPA 6010	JMW	1	PASI-A
9288905010	NA+ED FILAMENT POLYESTER	EPA 6010	SHB	1	PASI-A
9288905011	NA+ED SPUN ACRYLIC	EPA 6010	SHB	1	PASI-A
9288905012	NA+ED SPUN POLYPROPYLENE	EPA 6010	SHB	1	PASI-A
9288905013	PLASMA+ED SPUN NYLON 6.6	EPA 6010	SHB	1	PASI-A
9288905014	PLASMA+ED FILAMENT NYLON 6.6	EPA 6010	SHB	1	PASI-A
9288905015	PLASMA+ED SPUN POLYESTER	EPA 6010	SHB	1	PASI-A
9288905016	PLASMA+ED FILAMENT POLYESTER	EPA 6010	SHB	1	PASI-A
9288905017	PLASMA+ED SPUN POLYPROPYLENE	EPA 6010	SHB	1	PASI-A
9288905018	PLASMA+ED SPUN ACRYLIC	EPA 6010	SHB	1	PASI-A

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 2225 Riverside Dr.
 Asheville, NC 28804
 (828)254-7176

Pace Analytical Services, Inc.
 9800 Kinney Ave. Suite 100
 Huntersville, NC 28078
 (704)875-9092

HITS ONLY

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Lab Sample ID Method	Client Sample ID Parameters	Result	Units	Report Limit	Analyzed	Qualifiers
9288905001	UNTREATED SPUN NYLON 6.6					
EPA 6010	Copper	13.4	mg/kg	0.33	03/07/11 15:18	
9288905002	UNTREATED FILAMENT NYLON 6.6					
EPA 6010	Copper	9.5	mg/kg	0.28	03/07/11 15:31	
9288905003	UNTREATED SPUN POLYESTER					
EPA 6010	Copper	19.4	mg/kg	0.41	03/07/11 15:34	
9288905004	UNTREATED FILAMENT POLYESTER					
EPA 6010	Copper	4.9	mg/kg	0.44	03/07/11 15:37	
9288905005	UNTREATED SPUN ACRYLIC					
EPA 6010	Copper	8.3	mg/kg	0.42	03/07/11 15:40	
9288905006	UNTREATED SPUN POLYPROPYLENE					
EPA 6010	Copper	16.2	mg/kg	0.43	03/07/11 15:44	
9288905007	NA+ED SPUN NYLON 6.6					
EPA 6010	Copper	1680	mg/kg	8.3	03/07/11 17:30	
9288905008	NA+ED FILAMENT NYLON 6.6					
EPA 6010	Copper	439	mg/kg	0.45	03/07/11 15:50	
9288905009	NA+ED SPUN POLYESTER					
EPA 6010	Copper	930	mg/kg	8.9	03/07/11 17:33	
9288905010	NA+ED FILAMENT POLYESTER					
EPA 6010	Copper	1300	mg/kg	22.3	03/09/11 16:03	
9288905011	NA+ED SPUN ACRYLIC					
EPA 6010	Copper	1090	mg/kg	23.1	03/09/11 16:10	
9288905012	NA+ED SPUN POLYPROPYLENE					
EPA 6010	Copper	2270	mg/kg	25.0	03/09/11 17:14	
9288905013	PLASMA+ED SPUN NYLON 6.6					
EPA 6010	Copper	8270	mg/kg	20.5	03/09/11 17:19	
9288905014	PLASMA+ED FILAMENT NYLON 6.6					
EPA 6010	Copper	17100	mg/kg	25.0	03/09/11 17:23	
9288905015	PLASMA+ED SPUN POLYESTER					
EPA 6010	Copper	57400	mg/kg	44.6	03/10/11 11:48	
9288905016	PLASMA+ED FILAMENT POLYESTER					
EPA 6010	Copper	5960	mg/kg	22.3	03/09/11 17:29	
9288905017	PLASMA+ED SPUN POLYPROPYLENE					
EPA 6010	Copper	21800	mg/kg	24.5	03/09/11 17:32	

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 2225 Riverside Dr.
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 (828)254-7176

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 9800 Kincey Ave. Suite 100
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 (704)875-9092

HITS ONLY

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Lab Sample ID Method	Client Sample ID Parameters	Result	Units	Report Limit	Analyzed	Qualifiers
9288905018	PLASMA+ED SPUN ACRYLIC					
EPA 6010	Copper	22200	mg/kg	20.8	03/09/11 15:08	

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2225 Riverside Dr.
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Pace Analytical Services, Inc.
9800 Kinsey Ave. Suite 100
Huntersville, NC 28078
(704)875-9092

PROJECT NARRATIVE

Project: MATT CRUTCHFIELD THESIS
Pace Project No.: 9288905

Method: EPA 6010
Description: 6010 MET ICP
Client: NCSU Institute of Textile Technology
Date: March 10, 2011

General Information:

18 samples were analyzed for EPA 6010. All samples were received in acceptable condition with any exceptions noted below.

Hold Time:

The samples were analyzed within the method required hold times with any exceptions noted below.

Sample Preparation:

The samples were prepared in accordance with EPA 3050 with any exceptions noted below.

Initial Calibrations (including MS Tune as applicable):

All criteria were within method requirements with any exceptions noted below.

Continuing Calibration:

All criteria were within method requirements with any exceptions noted below.

Method Blank:

All analytes were below the report limit in the method blank with any exceptions noted below.

Laboratory Control Spike:

All laboratory control spike compounds were within QC limits with any exceptions noted below.

Matrix Spikes:

All percent recoveries and relative percent differences (RPDs) were within acceptance criteria with any exceptions noted below.

QC Batch: MPRP/7959

A matrix spike and matrix spike duplicate (MS/MSD) were performed on the following sample(s): 9288597002

M0: Matrix spike recovery and/or matrix spike duplicate recovery was outside laboratory control limits.

- MS (Lab ID: 573295)
- Copper

Duplicate Sample:

All duplicate sample results were within method acceptance criteria with any exceptions noted below.

QC Batch: MPRP/7959

R1: RPD value was outside control limits.

- DUP (Lab ID: 573296)
- Copper

Additional Comments:

This data package has been reviewed for quality and completeness and is approved for release.

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 Asheville, NC 28804
 (828)254-7176

Pace Analytical Services, Inc.
 9800 Kinsey Ave. Suite 100
 Huntersville, NC 28078
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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: UNTREATED SPUN NYLON Lab ID: 9288905001 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid
 6.6

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	13.4	mg/kg	0.33	1	03/04/11 16:00	03/07/11 15:18	7440-50-8	

Date: 03/10/2011 04:48 PM

REPORT OF LABORATORY ANALYSIS

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 Eden, NC 27288
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Pace Analytical Services, Inc.
 2225 Riverside Dr.
 Asheville, NC 28804
 (828)254-7176

Pace Analytical Services, Inc.
 9800 Kinsey Ave. Suite 100
 Huntersville, NC 28078
 (704)875-9092

ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: UNTREATED FILAMENT Lab ID: 9288905002 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid
NYLON 6.6

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	9.5	mg/kg	0.28	1	03/04/11 16:00	03/07/11 15:31	7440-50-8	

Date: 03/10/2011 04:48 PM

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: UNTREATED SPUN **Lab ID:** 9288905003 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid
 POLYESTER

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	19.4	mg/kg	0.41	1	03/04/11 16:00	03/07/11 15:34	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: UNTREATED SPUN **Lab ID:** 9288905005 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid
ACRYLIC

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	8.3	mg/kg	0.42	1	03/04/11 16:00	03/07/11 15:40	7440-50-8	





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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: UNTREATED SPUN POLYPROPYLENE **Lab ID: 9288905006** Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	16.2	mg/kg	0.43	1	03/04/11 16:00	03/07/11 15:44	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: NA+ED SPUN NYLON 6.6 **Lab ID: 9288905007** Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	1680	mg/kg	8.3	20	03/04/11 16:00	03/07/11 17:30	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: NA+ED FILAMENT NYLON 6.6 **Lab ID:** 9288905008 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	439	mg/kg	0.45	1	03/04/11 16:00	03/07/11 15:50	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: NA+ED SPUN POLYESTER Lab ID: 9288905009 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	930	mg/kg	8.9	20	03/04/11 16:00	03/07/11 17:33	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: NA+ED FILAMENT **Lab ID:** 9288905010 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid
 POLYESTER

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	1300	mg/kg	22.3	50	03/09/11 11:25	03/09/11 16:03	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: NA+ED SPUN **Lab ID:** 9288905012 **Collected:** 03/01/11 00:00 **Received:** 03/01/11 17:00 **Matrix:** Solid
 POLYPROPYLENE

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	2270	mg/kg	25.0	50	03/09/11 11:25	03/09/11 17:14	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: PLASMA+ED SPUN Lab ID: 9288905013 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid
 NYLON 6.6

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	8270	mg/kg	20.5	50	03/09/11 11:25	03/09/11 17:19	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: PLASMA+ED FILAMENT **Lab ID:** 9288905014 **Collected:** 03/01/11 00:00 **Received:** 03/01/11 17:00 **Matrix:** Solid
 NYLON 6.6

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	17100	mg/kg	25.0	50	03/09/11 11:25	03/09/11 17:23	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: PLASMA+ED SPUN POLYESTER **Lab ID:** 9288905015 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	57400	mg/kg	44.6	100	03/09/11 11:25	03/10/11 11:48	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: PLASMA+ED FILAMENT **Lab ID:** 9288905016 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid
POLYESTER

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP								
Analytical Method: EPA 6010 Preparation Method: EPA 3050								
Copper	5960	mg/kg	22.3	50	03/09/11 11:25	03/09/11 17:29	7440-50-8	

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: PLASMA+ED SPUN POLYPROPYLENE **Lab ID:** 9288905017 Collected: 03/01/11 00:00 Received: 03/01/11 17:00 Matrix: Solid

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP	Analytical Method: EPA 6010 Preparation Method: EPA 3050							
Copper	21800	mg/kg	24.5	50	03/09/11 11:25	03/09/11 17:32	7440-50-8	

Date: 03/10/2011 04:48 PM

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ANALYTICAL RESULTS

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Sample: PLASMA+ED SPUN **Lab ID:** 9288905018 **Collected:** 03/01/11 00:00 **Received:** 03/01/11 17:00 **Matrix:** Solid
ACRYLIC

Results reported on a "wet-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
6010 MET ICP	Analytical Method: EPA 6010 Preparation Method: EPA 3050							
Copper	22200	mg/kg	20.8	50	03/09/11 11:25	03/09/11 15:08	7440-50-8	

Date: 03/10/2011 04:48 PM

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QUALITY CONTROL DATA

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

QC Batch: MPRP/7959 Analysis Method: EPA 6010
 QC Batch Method: EPA 3050 Analysis Description: 6010 MET
 Associated Lab Samples: 9288905001, 9288905002, 9288905003, 9288905004, 9288905005, 9288905006, 9288905007, 9288905008, 9288905009

METHOD BLANK: 573293 Matrix: Solid
 Associated Lab Samples: 9288905001, 9288905002, 9288905003, 9288905004, 9288905005, 9288905006, 9288905007, 9288905008, 9288905009

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Copper	mg/kg	ND	0.50	03/07/11 14:08	

LABORATORY CONTROL SAMPLE: 573294

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Copper	mg/kg	50	48.0	96	80-120	

MATRIX SPIKE SAMPLE: 573295

Parameter	Units	9288597002 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Copper	mg/kg	307	44.8	20000	44034	75-125 M0	

SAMPLE DUPLICATE: 573296

Parameter	Units	9288597003 Result	Dup Result	RPD	Qualifiers
Copper	mg/kg	312	190	49 R1	





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QUALITY CONTROL DATA

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

QC Batch: MPRP/7971 Analysis Method: EPA 6010
 QC Batch Method: EPA 3050 Analysis Description: 6010 MET
 Associated Lab Samples: 9288905010, 9288905011, 9288905012, 9288905013, 9288905014, 9288905015, 9288905016, 9288905017, 9288905018

METHOD BLANK: 574055 Matrix: Solid
 Associated Lab Samples: 9288905010, 9288905011, 9288905012, 9288905013, 9288905014, 9288905015, 9288905016, 9288905017, 9288905018

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Copper	mg/kg	ND	0.50	03/09/11 13:30	

LABORATORY CONTROL SAMPLE: 574056

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Copper	mg/kg	50	50.4	101	80-120	

MATRIX SPIKE SAMPLE: 574057

Parameter	Units	9288905010 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Copper	mg/kg	1300	35.2	1020	-812	75-125	

SAMPLE DUPLICATE: 574058

Parameter	Units	9288905011 Result	Dup Result	RPD	Qualifiers
Copper	mg/kg	1090	1580	37	





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QUALIFIERS

Project: MATT CRUTCHFIELD THESIS
Pace Project No.: 9288905

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.
ND - Not Detected at or above adjusted reporting limit.
J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.
MDL - Adjusted Method Detection Limit.
S - Surrogate
1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.
Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.
LCS(D) - Laboratory Control Sample (Duplicate)
MS(D) - Matrix Spike (Duplicate)
DUP - Sample Duplicate
RPD - Relative Percent Difference
NC - Not Calculable.
SG - Silica Gel - Clean-Up
U - Indicates the compound was analyzed for, but not detected.
N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.
Pace Analytical is NELAP accredited. Contact your Pace PM for the current list of accredited analytes.

LABORATORIES

PASI-A Pace Analytical Services - Asheville

ANALYTE QUALIFIERS

M0 Matrix spike recovery and/or matrix spike duplicate recovery was outside laboratory control limits.
R1 RPD value was outside control limits.





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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: MATT CRUTCHFIELD THESIS
 Pace Project No.: 9288905

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
9288905001	UNTREATED SPUN NYLON 6.6	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905002	UNTREATED FILAMENT NYLON 6.6	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905003	UNTREATED SPUN POLYESTER	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905004	UNTREATED FILAMENT POLYESTER	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905005	UNTREATED SPUN ACRYLIC	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905006	UNTREATED SPUN POLYPROPYLENE	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905007	NA+ED SPUN NYLON 6.6	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905008	NA+ED FILAMENT NYLON 6.6	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905009	NA+ED SPUN POLYESTER	EPA 3050	MPRP/7959	EPA 6010	ICP/7357
9288905010	NA+ED FILAMENT POLYESTER	EPA 3050	MPRP/7971	EPA 6010	ICP/7374
9288905011	NA+ED SPUN ACRYLIC	EPA 3050	MPRP/7971	EPA 6010	ICP/7374
9288905012	NA+ED SPUN POLYPROPYLENE	EPA 3050	MPRP/7971	EPA 6010	ICP/7374
9288905013	PLASMA+ED SPUN NYLON 6.6	EPA 3050	MPRP/7971	EPA 6010	ICP/7374
9288905014	PLASMA+ED FILAMENT NYLON 6.6	EPA 3050	MPRP/7971	EPA 6010	ICP/7374
9288905015	PLASMA+ED SPUN POLYESTER	EPA 3050	MPRP/7971	EPA 6010	ICP/7374
9288905016	PLASMA+ED FILAMENT POLYESTER	EPA 3050	MPRP/7971	EPA 6010	ICP/7374
9288905017	PLASMA+ED SPUN POLYPROPYLENE	EPA 3050	MPRP/7971	EPA 6010	ICP/7374
9288905018	PLASMA+ED SPUN ACRYLIC	EPA 3050	MPRP/7971	EPA 6010	ICP/7374

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CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section A Required Client Information:		Section B Required Project Information:		Section C Invoice Information:	
Company: NCSU Institute of Textile Technology		Report for: Dr. Henry Boyer		Company Name: ITT-NCSU	
Address: 1000 Main Campus Drive Raleigh, NC 27695		Copy To: Matt Crutchfield		Attention: Patricia Hill	
Email To: matcrutchfield21@gmail.com		Purchase Order No.:		Address: 2401 Research Drive Raleigh 27695	
Phone: 919-749-0734		Project Name: Matt Crutchfield Thesis		Price Code: \$25 per sample	
Requested Due Date/TAT:		Project Number: 5-22741		Reference: Kevin Herring	
				Price Project Manager:	
				Price Profile #:	
				Requested Analysis Filtered (Y/N): <u>4096-1</u>	
				Regulatory Agency: <u>NC</u>	
				Site Location: <u>UST</u>	
				State: <u>NC</u>	
				NPDES: <u>GROUND WATER</u>	
				ROPA: <u>DRINKING WATER</u>	
				OTHER: <u> </u>	

ITEM #	Valid Matrix Codes Requested Client Information	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED			SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives	Analysis Test	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)	Sample No./Lab I.D.
				COMPOSITE START	COMPOSITE END/STOP	DATE							
1	Untreated Spun Nylon 6,6												
2	Untreated Filament Nylon 6,6												
3	Untreated Spun Polyester												
4	Untreated Filament Polyester												
5	Untreated Spun Acrylic												
6	Untreated Spun Polypropylene												
7	NA+ED Filament Nylon 6,6												
8	NA+ED Spun Nylon 6,6												
9	NA+ED Spun Polyester												
10	NA+ED Filament Polyester												
11	NA+ED Spun Acrylic												
12	NA+ED Spun Polypropylene												
ADDITIONAL COMMENTS				REQUISITIONED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS			
				<i>Patricia Hill</i>	3-1-11	8:40	<i>Patricia Hill</i>	3-1-11	8:10				
				<i>Patricia Hill</i>	3-1-11	1:00	<i>Patricia Hill</i>	3-1-11	1:30				

SAMPLER NAME AND SIGNATURE	
PRINT Name of SAMPLER: <u>MATT CRUTCHFIELD</u>	DATE Signed (MM/DD/YY): <u>3-1-10</u>
SIGNATURE of SAMPLER: <i>[Signature]</i>	

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

CHAIN-OF-CUSTODY / Analytical Request Document
The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section A Required Client Information:		Section B Required Project Information:		Section C Invoice Information:	
Company:	NCSU Institute of Textile Technology	Report To:	Dr. Henry Boyer	Attention:	Patrice Hill
Address:	1000 Main Campus Drive Raleigh, NC 27695	Copy To:	Matt Crutchfield	Company Name:	ITT-NCSU
Email To:	mattcrutchfield21@gmail.com	Purchase Order No.:		Address:	2401 Research Drive Raleigh 27695
Phone:	919-749-0734	Project Name:	Matt Crutchfield Thesis	Price Code:	\$25 per sample
Requested Due Date/AT:		Project Number:	5-22741	Reference:	
				Price Project Manager:	Kevin Herring
				Price Profile #:	
				REGULATORY AGENCY:	NPDES <input type="checkbox"/> GROUND WATER <input type="checkbox"/> DRINKING WATER <input type="checkbox"/> USF <input type="checkbox"/> RCRA <input type="checkbox"/> OTHER <input type="checkbox"/>
				Site Location:	STATE: NC
				Requested Analysis Filtered (Y/N)	

ITEM #	Valid Matrix Codes EPHRAIM/WATER WATER WASTE WATER SOIL/SOLID WINE OIL MILK OTHER TISSE	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED		SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives	Analysis Test	Residual Chlorine (Y/N)	Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
				DATE	TIME									
1	Plasma+ED Spun Nylon 6,6													
2	Plasma+ED Filament Nylon 6,6													
3	Plasma+ED Spun Polyester													
4	Plasma+ED Filament Polyester													
5	Plasma+ED Spun Acrylic													
6	Plasma+ED Spun Polypropylene													
7														
8														
9														
10														
11														
12														

REQUISITIONED BY / AFFILIATION:	DATE:	TIME:	RECEIVED BY / AFFILIATION:	DATE:	TIME:
<i>Patrice Hill</i>	3/11/11	8:40	<i>Patrice Hill</i>	3/11/11	8:40
	3/11/11	12:00	<i>Kevin Herring</i>	3/11/11	3:00

SAMPLER NAME AND SIGNATURE:		DATE SIGNED (MM/DD/YY):	
PRINT NAME OF SAMPLER:	<i>NOT CRUTCHFIELD</i>	DATE SIGNED:	3-1-11
SIGNATURE OF SAMPLER:	<i>[Signature]</i>		

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.



Sample Condition Upon Receipt

Client Name: NCSU Project # 0288905

Where Received: Huntersville Asheville Eden

Courier: Fed Ex UPS USPS Client Commercial Pace Other _____

Optional
Proj. Due Date:
Proj. Name:

Custody Seal on Cooler/Box Present: yes no Seals intact: yes no

Packing Material: Bubble Wrap Bubble Bags None Other _____

Thermometer Used: IR Gun : T904 Type of Ice: Wet Blue None Samples on ice, cooling process has begun

Temp Correction Factor: Add / Subtract 0 C

Corrected Cooler Temp.: N/A C Biological Tissue is Frozen: Yes No
Temp should be above freezing to 6°C

Date and Initials of person examining contents: mm-4/1/11

Chain of Custody Present:	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.
Chain of Custody Filled Out:	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	2. 1. Air filter 2. no date/time
Chain of Custody Relinquished:	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3. on coc or riploc. 3. Analy air
Sampler Name & Signature on COC:	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	4. Section not filled out for
Samples Arrived within Hold Time:	? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5. Will bother have no analy air
Short Hold Time Analysis (<72hr):	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	6. on them
Fast Turn Around Time Requested:	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.
Sufficient Volume:	? <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8. filter
Correct Containers Used:	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	9. riploc
-Pace Containers Used:	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	
Containers Intact:	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.
Filtered volume received for Dissolved tests	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11.
Sample Labels match COC:	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12. filter
-Includes date/time/ID/Analysis Matrix:		
If containers needing preservation have been checked.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	13.
If containers needing preservation are found to be in compliance with EPA recommendation.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	
Exceptions: VOA, coliform, TOC, O&G, WI-DRO (water)	<input type="checkbox"/> Yes <input type="checkbox"/> No	Initial when completed
Samples checked for dechlorination:	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	14.
Headspace in VOA Vials (>6mm):	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	15.
Trip Blank Present:	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	16.
Trip Blank Custody Seals Present	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	
Purchase Trip Blank Lot # (if purchased):		

Client Notification/ Resolution: _____ Field Data Required? Y / N

Person Contacted: _____ Date/Time: _____

Comments/ Resolution: _____

CURF Review: KCH Date: 3/2/11 SRF Review: KCH Date: 3/3/11

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)