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นางสาวสุพิชญา ชื่นชนม์

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ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

PROPERTIES AND MORPHOLOGY OF SURFACE MODIFIED MAN-MADE FIBERS
USING A THETA-PINCH DEVICE

Miss Supitchaya Chuenchon



สถาบันวิทยบริการ

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 พลาสมา โดยนำเส้นใยพอลิโพรพิลีน เส้นใยพอลิเอสเทอร์ และเส้นใยเรยอน มาดัดแปรด้วยเครื่อง
 เทตาพินช์ ซึ่งผลิตพลาสมาไนโตรเจนและพลาสมาออกซิเจน ที่จำนวนครั้งในการยิงต่างๆกัน
 แรงแลอเรนซ์เป็นสาเหตุที่ทำให้พลาสมาเกิดการเคลื่อนที่ไปสัมผัสบนผิวของเส้นใย ซึ่งการสัมผัสนี้
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 อิเล็กตรอนแบบส่องกราด ผลการทดลองแสดงให้เห็นว่า ชนิดของแก๊สและจำนวนครั้งของการยิง
 พลาสมามีผลต่อสมบัติด้านแรงดึง ความหนาแน่นเชิงเส้นและขนาดเส้นผ่านศูนย์กลางของเส้นใย
 ในขณะที่สมบัติด้านการติดไฟของเส้นใยที่ผ่านและไม่ผ่านการดัดแปรมีค่าใกล้เคียงกัน เส้นใยที่ผ่าน
 การดัดแปรทั้งหมดมีคุณลักษณะในการชอบน้ำเพิ่มขึ้นอย่างมาก เนื่องจากการเกิดหมู่ฟังก์ชันที่ชอบ
 น้ำขึ้นบนพื้นผิว ซึ่งสามารถยืนยันได้จากอินฟราเรดสเปกโทรสโกปี

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 จุฬาลงกรณ์มหาวิทยาลัย

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 สาขาวิชาวิทยาศาสตร์พอลิเมอร์ประยุกต์ฯ.ลายมือชื่ออาจารย์ที่ปรึกษา.....
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In this research, the properties and the morphology of plasma-treated man-made fibers were studied. Polypropylene, polyester and rayon fibers were treated by a theta-pinch device that produced oxygen and nitrogen plasma for different treatment times. The Lorentz force caused the induced plasma to move toward the fiber in an abrupt manner. The induced plasma caused an increase in the roughness on the fiber surface as revealed in SEM photographs. It was found that tensile properties, linear density and diameter of plasma-treated fibers were affected by the type of gases and the number of plasma shots. However, the flammability of both treated and untreated fibers were comparable. All plasma-treated fibers also exhibited a remarkable increase in hydrophilic characteristic due to the formation of hydrophilic groups on fiber surface confirmed by FT-IR spectroscopy.

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Department Materials Science.....Student's signature.....

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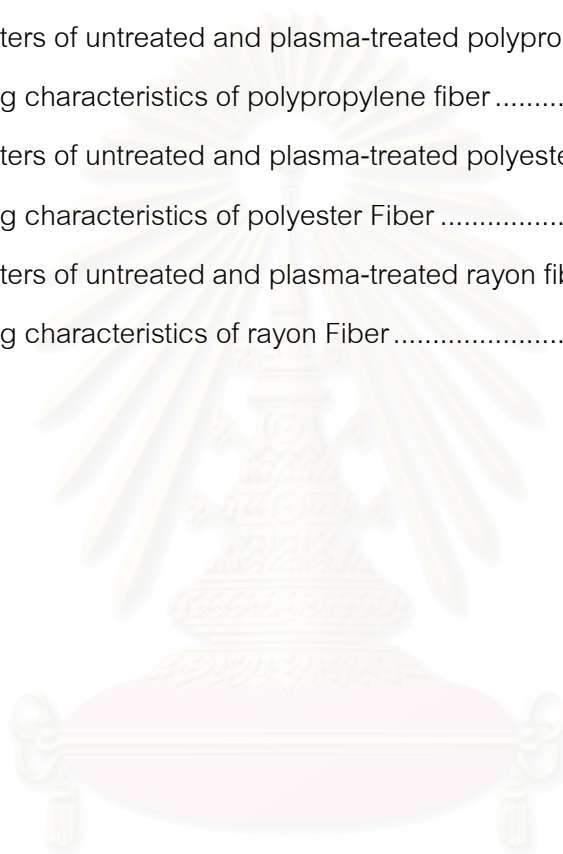
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CHAPTER I

INTRODUCTION

Polymer modification has become a major route to better polymer properties and wider polymer applications since the 1990's. The high cost of developing a completely new polymer having long-term performance and great characteristics has motivated many companies to innovate by polymer modification rather than synthesis of a new monomer and polymer [1].

Polymer modification can be divided into two types: chemical modification and physical modification. Chemical modification is the modification of a polymer, which the chemical structures of the polymers are changed by chemical reactions such as esterification, etherification, grafting and cross-linking, etc. On the other hand, physical modification is the modification of a polymer by physical methods such as blending with other polymers and mixing with suitable additives, etc. [2] The modification can be applied to polymers during or after their syntheses and during or after their manufacturing processes into products.

In the case of the polymers used as textile fibers, modification methods normally include blend or composite spinning of premixtures of resin and additives, coating the fiber surface with additive to give a thin and uniform additive layer in the finishing stage, and graft copolymerization or plasma treatment of the fiber surface. Of these processes, the spinning methods provide the best combination of durability and improved performance because the additives are uniformly distributed throughout the fiber. In blend spinning, conditions should be controlled to form a continuous filament of the modifier within the fiber. The other processes are useful in modification of the surface properties of fabricated products. Coating in the finishing stage requires fewer amounts of additives but often causes problem in fastness. Graft copolymerization gives a good fastness. However, it may affect bulk properties and required much complicated steps because it adopts a wet process. Plasma treatment can modify the surface properties of

fibers or fabrics. It has the following advantages over conventional modification processes: (1) only the surface is modified without changing any bulk properties; (2) almost all compounds including inert gases such as He and Ne and non-polymer forming gases such as NH_3 and CF_4 can be incorporated into a substrate polymer by chemical and physical reactions; and (3) its dry process requires much simpler steps than those conventional wet processes. [1-2]

From the advantages of plasma treatment mentioned above, many scientific literatures have revealed a growing interest in its applications on textiles. Previous works have emphasized on textile modification using cold plasmas generated from several devices. Because plasma generating processes of them are continuous, the control plasma exposure is difficult. Therefore, this research has been paid attention on using hot plasmas generated in shots from theta-pinch device instead. The dynamic of this device is an increase in magnetic field to induce an electric field opposite to the direction of the discharge current. Then it produces the plasma current sheath and ions that rapidly compress toward the tube axis. The ions produced are of high energy such that they can be used for modifying material surface [3-14]. Since plasma generating process of theta-pinch device is discontinuous, it is easier to control plasma exposure on a material.

Therefore, an alternative method for modifying the surface of man-made fibers using theta-pinch device is presented in this research. Three types of man-made fibers are selected based on their hydrophobic-hydrophilic characteristics. They are polypropylene fiber, polyester fiber and rayon fiber. They represent hydrophobic fiber, hydrophilic fiber, more hydrophilic fiber and, respectively. The effects of modification parameters including the type of gases and the number of plasma shots on morphology, chemical structure and properties of these fibers are investigated and compared to those of the unmodified ones.

CHAPTER II

THEORY AND LITERATURE REVIEW

2.1 Plasma

The fourth state of the matter is called as Plasma (The other states are solid liquid and gas.). Plasma is partially ionized gas, which consists of a gas of positively charged and negatively charges particles with approximately equal [15]. An electromagnetic field of sufficient power can produce and control ionizing of plasma. One useful form of gas plasma is made by introducing gas into a reaction chamber, maintaining pressure between 0.1 and 10 torr, and then applying energy [16].

2.1.1 Plasma States

Increasing the energy content of solid phase matter sufficiently leads to phase transformation processes. The processes depend on temperature, molecular weight, and the specific molecular structure. The system will be in the solid, liquid or gaseous phase or a mixture of these. However, not all materials can undergo all these transformation processes. Relatively low molecular weight, polar, and organic compounds [17].

Plasma states can be divided in two main categories: *Hot Plasmas* and *Cold Plasmas*. Hot plasmas have an extremely high energy content, which induce fragmentation of all organic molecules to atomic levels. Consequently, these plasmas can only be used for generating extremely high caloric energy or to modify thermally stable inorganic materials (metals, metal oxides, etc.). Hot plasma approaches considered have been initiated with the use of plasma arc heaters for materials processing, and later on, three distinct application areas emerged: synthesis, melting and deposition. An unsatisfactory understanding of the extremely complex reaction

kinetics, transport properties, heat transfer, and particle dynamics can be affected to progress in thermal plasma processing. As a result, it has only in a few instances progressed except laboratory and pilot scale stages. Furthermore, Accelerated electrons induce ionization, excitation and molecular fragmentation processes. They lead to a complex mixture of active species depending on the specific plasma mode (e.g. direct or remote plasma environments), recombination processes in the presence or absence of the plasma. However, the recombination reaction mechanisms are very different reaction mechanism from those for conventional chemical processes. Thus proper selection and control of plasma parameters are necessary for efficient approaches to specific applications. [18]

2.1.2 Applications of Plasma for Surface Modification

The plasma process modifies only several molecule layers, thus appearance and bulk properties are usually unaffected. In addition, plasma changes the molecular weight of the surface layer by scissoring, branching, and cross-linking organic materials. The chemistry of the plasma determines its effects on a polymer [19]. Polymeric materials are chosen and developed for their bulk properties; however, the inert nature of most polymer surfaces creates challenges for applications where coatings must be applied. Exposing them to an electrical discharge or plasma to modify their surface chemistry is a common approach to tailoring surface properties of polymer materials. This approach has been applied to a variety of polymers in order to improve adhesion (for layers coated on treated substrates or for bonding processes where adhesives are used to form mechanical joints between treated parts), improve inking and wetting in coating and printing processes, improved dyeing in textile products and in the development of biomaterials [20].

2.1.3 Plasma Reactor

The production process of plasma usually depends on the type of plasma generating devices. In this research, Theta-pinch device is used for fiber modification.

Theta-pinch device originated from the research in the field of controlled thermonuclear fusion. The theta-pinch discharge can produce the pulsed high-density plasma. The “high-density plasma” here means a fully ionized gas with density in excess of about 10^{16} particles/cm³ and with temperature in excess of some 10^6 K (1 eV=11,6000 °K).

Theta-pinch device is very useful in the applications. For example, this device has been utilized in deposition of thin-films, including amorphous carbon film, diamond-like carbon film, etc. It has also been used to process the super-conducting films for lithography, which reveals its potential advantages over other methods. Such plasma could be used as light sources giving copious emission of electromagnetic radiation at short wavelengths. In addition, the technique associated with the generation of pulsed high-density plasma has applications in a broad scope [21].

2.1.3.1 The Dynamic of Theta-pinch Device

The dynamic process of theta-pinch discharge is shown in Figure 2.1. After switch is closed, discharges through a single-turn coil and driving a current in the coil. The discharge current produces an axial magnetic field in the coil-encircled area (Figure 2.2). The rapidly increasing magnetic field induces an electric field opposite to the direction of the discharge current in the coil. The electric field in tube produces a plasma current sheath near the tube wall. The force exerts the plasma current sheath and rapidly compresses it toward the tube axis. Meanwhile, the plasma particles pass the surface of fibers and escape from the tube end (end loss). The plasma experiences shocks wave heating, adiabatic compression heating, and others. [21]

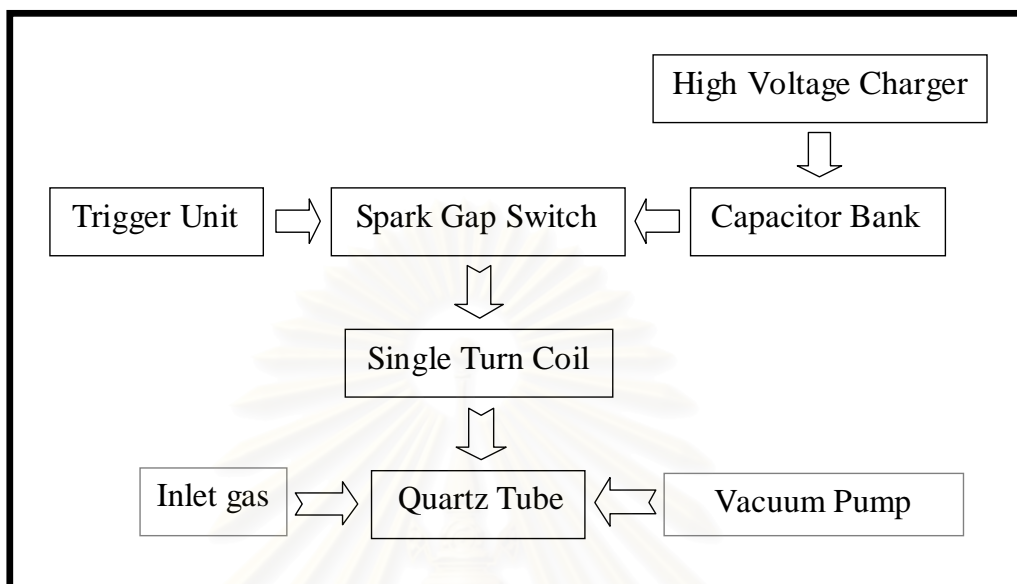


Figure 2.1 Dynamic process of Theta-pinch discharge [21]

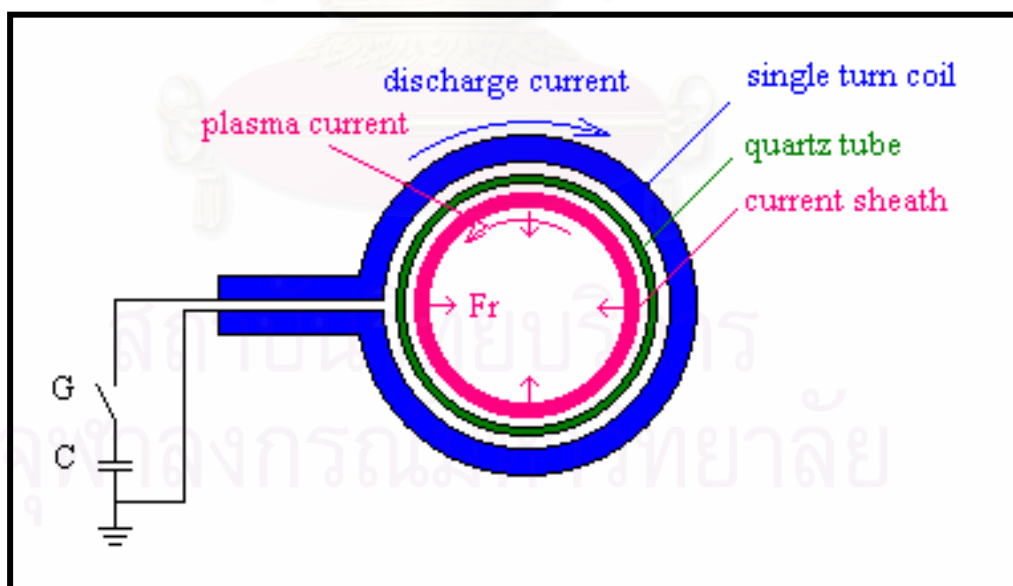


Figure 2.2 Theta-pinch discharges [21]

2.2 Man-Made Fibers

Man-made fibers are any fiber that derived from various sources. For example, the natural material of cellulose has been taken from cotton linters and wood pulp, processed chemically, and changed in form and several other characteristics into fibers of various lengths. These are classified as man-made cellulosic fibers

However, noncellulosic polymer fibers are other group of man-made fibers. These synthetics have been and are still being created by companies strives to imitate properties of other fibers, to develop other characteristics, or to combine certain properties. These fibers are synthesized by combining carbon, oxygen, hydrogen, and other simple chemical elements into large, complex molecular combinations or structures called polymers [22].

2.2.1 Polypropylene Fiber

Propylene is a by-product of the oil refineries; it comes from the cracking process. When propylene is polymerized under contain conditions it will give fiber-forming polymer; i.e., polypropylene [23]. Propylene with its methyl side-chains had every opportunity to form irregular or regular form. Polypropylene may also be extruded in sheet form for various uses. The sheets may be fibrillated, or slit, to form monofilaments or bundles of multifilament or melted, and spun by gear pump pressure through spinnerets [24]

2.2.1.1 Synthesis of Polypropylene

The polymerization reaction is a low-pressure process that utilizes Ziegler-Natta catalysts (aluminum alkyls and titanium halides). The catalyst may be slurried in a hydrocarbon mixture to facilitate heat transfer. The reaction is carried out in batch or

continuous reactors operating at temperatures between 50 and 80 °C and pressure in the range of 5 to 25 atm [25].

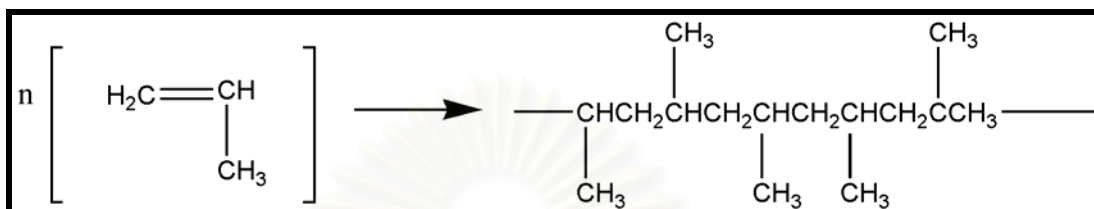


Figure 2.3 Synthesis of polypropylene [25]

The repeat unit is

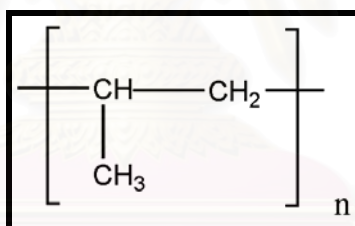


Figure 2.4 Repeating unit of polypropylene [25]

Polypropylene can be made in isotactic, syndiotactic and atactic forms. Ziegler-Natta type catalysts are used to produce stereoregular polypropylene. Usually 90% or more of the polymer is in the isotactic form. In this form, all the methyl groups are arranged on the same side of the plane of the main carbon chain. It can be form the fiber structure. On the other hand, figure 2.3 represents atactic polypropylene which is

not fiber forming because it is too irregular in the structure for the molecules to line up nicely [26].

2.2.1.2 Properties and Identification of Fiber

2.2.1.2.1 Microscopic Properties

Olefin fibers resemble glass rods in both longitudinal and cross-section views. They are even, clear, and round. Polypropylene can be extruded from specially shaped spinnerets, and it will then be irregular in cross section [27].

2.2.1.2.2 Physical and Mechanical Properties

Length and diameter of olefin fibers are controlled by the manufacturer. Olefin fibers are smooth and white.

The strength of olefin fibers is good, but it varies with degree of polymerization and molecular orientation from about 3.5 to 8.0 grams per denier. The elongation of polypropylene also varies widely, while the elastic recovery is outstanding. Properly heat-treated olefin fibers will retain their size and shape. They will stretch or shrink only if subjected to temperatures higher than the heat setting.

The fibers have little or no moisture absorption, which creates a serious problem in dyeing. Olefin fibers have good resistance to crushing. Because of this property polypropylene gives good service in carpeting [27].

2.2.1.2.3 Chemical Properties

Olefin fibers are highly resistant to alkaline substances. They also have good resistance to acids, except for oxidizing acids that weaken them. Cleaning solvents containing chlorinated hydrocarbons should never be used, for they cause olefin fibers to swell and eventually to degrade. Laundry soaps, synthetic detergents, and bleaches are safe [27].

Olefin will lose strength after prolonged exposure to sunlight. They are subject to staining by oil and grease, but normal laundering usually removes the stains. Olefins do have static electric buildup, but it is considered to be less than on nylon, polyester or wool [27].

2.2.1.2.4 Thermal Properties

Olefin fibers burn slowly and give off a sooty, waxy smoke. They shrink at low temperatures unless heat-set, and even then the fibers soften at temperatures above $65^{\circ}\text{--}71^{\circ}\text{C}$ ($150^{\circ}\text{--}160^{\circ}\text{F}$). Olefins should be ironed at the lowest setting possible with a press cloth [27].

2.2.2 Polyester Fiber

Polyester fiber is produced from poly(ethylene terephthalate) i.e. PET polymer. The molecular weight of polymer used for the preparation of polyester fiber is in the range of 20,000-40,000. The raw materials for the preparation of polyester are dimethyl ester of terephthalic acid (DMT) or terephthalic acid (TPA) and monoethylene glycol (MEG). Most of the earlier plants were based on DMT, as the raw material since high purity TPA required for polymerization was not available. However, methods for preparing purified TPA are now available and TPA is increasingly used as the raw material for the production of polyester [28].

2.2.2.1 Synthesis of PET

PET is a condensation polymer and is industrially produced by terephthalic acid with ethylene glycol. Linear polyester, PET is polymerized by a direct esterification as shown in Figure 2.5 .

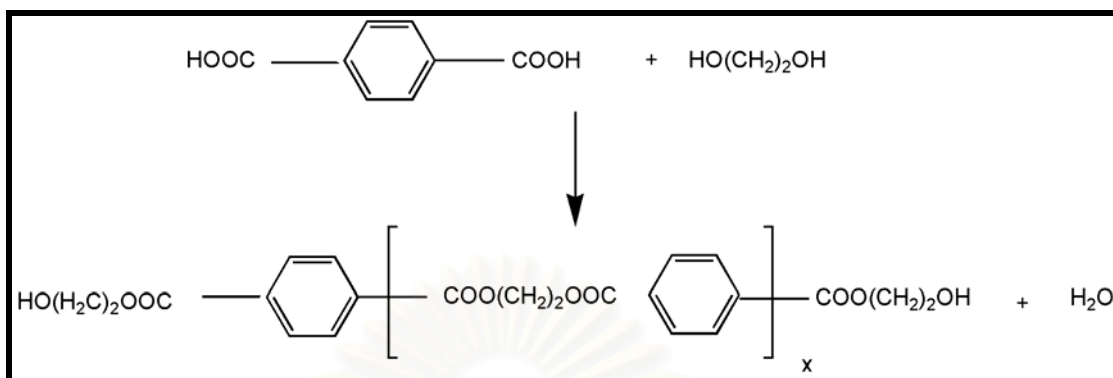


Figure 2.5 Esterification stage of PET production [28]

The major portion in the esterified product is diglycol- terephthalate, DGT in which case $X = 0$. It has the following structure:

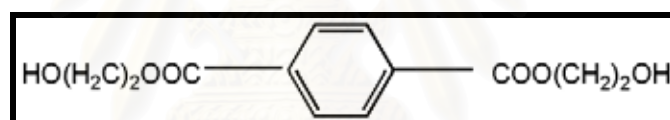


Figure 2.6 Chemical structure of diglycol terephthalate [28]

The second stage of preparation of PET polymer is called a polycondensation process. In the polycondensation process, DGT containing some quantity of dimer, trimer and tetramer is heated at high temperature and under vacuum to get with the liberation of H_2O [28].

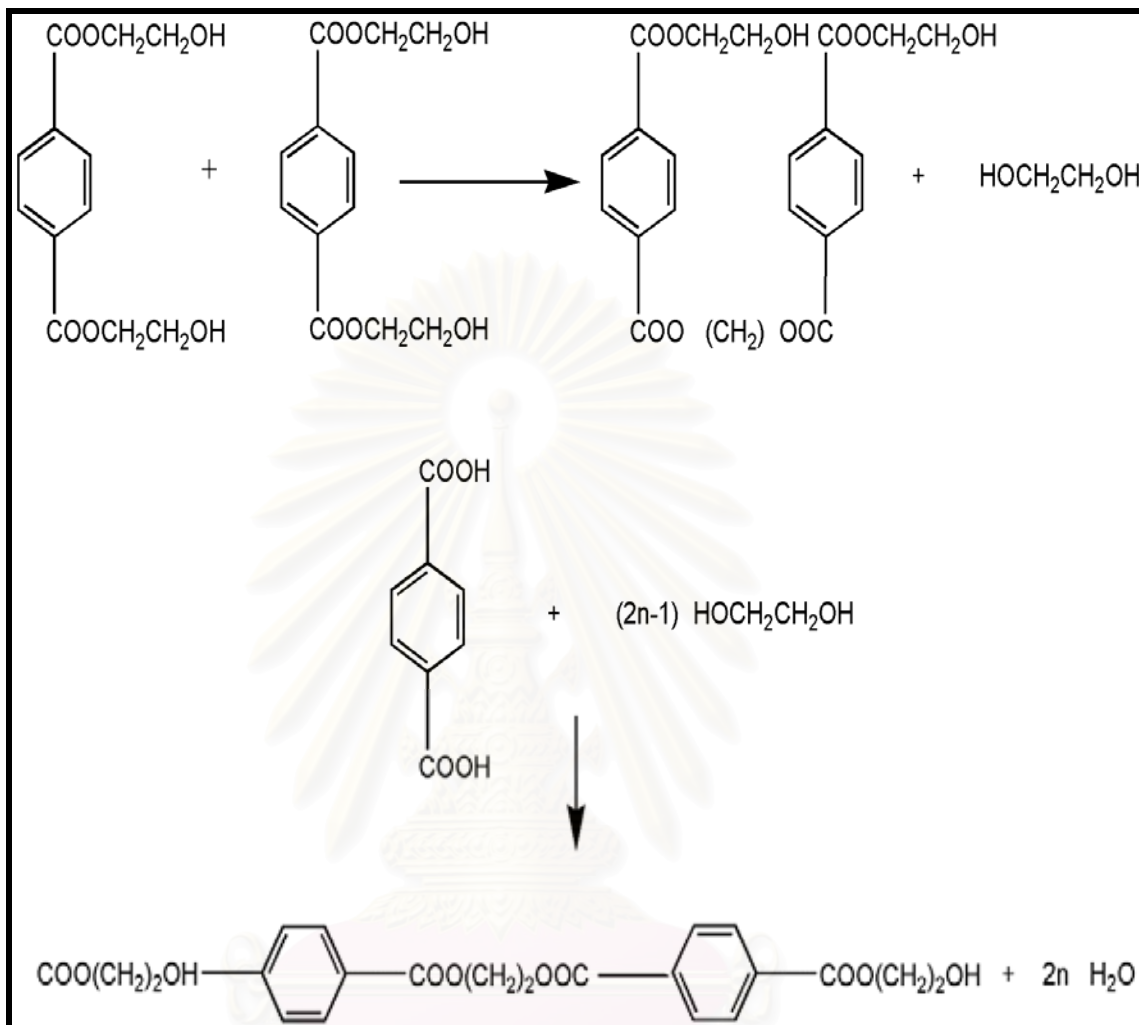


Figure 2.7 Polycondensation stage of PET production [28]

2.2.2.2 Properties and Identification of Fiber

2.2.2.2.1 Microscopic Properties

A longitudinal view of polyester fiber exhibits uniform diameter, smooth surface, and a rodlike appearance. The cross section is usually round, but modifications can include trilobal and pentalobal filaments [28].

2.2.2.2.2 Physical and Mechanical Properties

Polyester can be made in any length or diameter required for end-use. The fiber is partially transparent and white or slightly off-white in color. Pigment can be combined with the spinning solution, which permits control of the degree of luster. Optical brighteners are frequently added to produce clear, bright fibers.

The strength of polyesters varies widely. It, too, depends on end-use and is controlled by the manufacturer. Some fibers have the low tenacity to comparable rayon fiber, while others exceed the strongest nylon. There is no loss of strength when polyester fibers are wet.

Elongation is another controlled property in polyesters. But the fiber's elastic recovery overall is very good, and its resiliency is excellent. When properly heat-set, polyesters require little or no pressing to retain a smooth appearance.

The moisture regain of polyester fibers is very low -less than 0.5 percent. Because of the low regain, moisture has little effect on fiber strength, and static electric charges are accentuated. Furthermore, the low moisture absorption demands special techniques in dyeing and finishing. Like cotton and linen, polyesters have a high degree of wickability. This wicking property can produce end-use products that carry exterior moisture though to the inside, or body perspiration though to the outside [27].

2.2.2.2.3 Chemical Properties

Polyester has good resistance to weak alkaline but only moderate resistance to strong ones. Weak acids do not affect the fiber, nor do strong acids at room temperature. Strong acid at high temperatures, however, will destroy polyester. In general, the fiber resists organic solvents. Chemicals used in stain removal and cleaning do not damage it. Bleaches can be used safely.

Direct sunlight weakens polyester, but it has good resistance to sunlight when behind glass. It is, therefore, satisfactory for window curtains and drapes [27].

2.2.2.2.4 Thermal Properties

Polyester fibers melt at temperatures from 238 ° to 290 °C (460 ° –554 °F) depending upon type and modification. As the fiber melts, it forms a gray or tawny-colored bead that is hard and noncrushable. Polyesters will burn and produce a dark smoke and an aromatic odor. In light fabric constructions the fibers melt and drip away from the source of ignition, preventing the propagation of flame [27].

2.2.3 Rayon Fiber

Viscose rayon is regenerated cellulose. The cellulose comes from wood, of which it is the major constituent. It is purified, treated with caustic soda, which converts it into alkaline cellulose, then treated with carbon disulphide, which converts it into sodium cellulose xanthate and then dissolved in a dilute solution of caustic soda. This solution is then “ripened”, the solution becoming at first less viscous and then increasing nearly to its original viscosity; it is then spun into an acid coagulating bath consisting mainly of H_2SO_4 and $ZnSO_4$, which precipitates the cellulose in the form of a viscose filament [27].

2.2.3.1 Synthesis of Viscose Rayon

The sheets of cellulose are steeped in an alkali solution until the cellulose is converted to soda or alkali cellulose. The reaction is usually expressed:

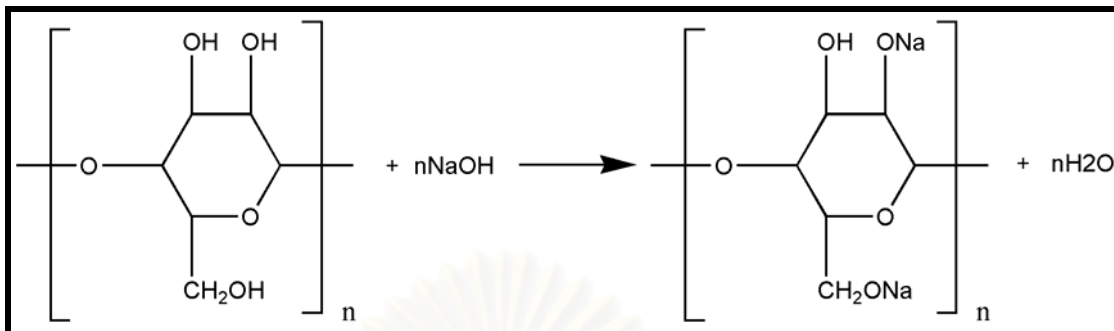


Figure 2.8 Chemical reaction of soda cellulose [29]

The reaction between the soda cellulose and the carbon disulphide may be represented:

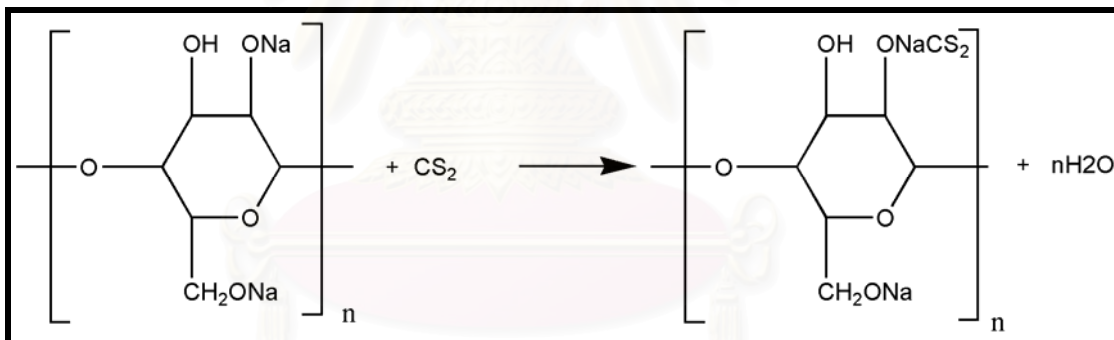


Figure 2.9 Chemical reaction of sodium cellulose xanthate [29]

When the sodium cellulose xanthate is dissolved in weak caustic soda to form the viscous solution known as “viscose” [29].

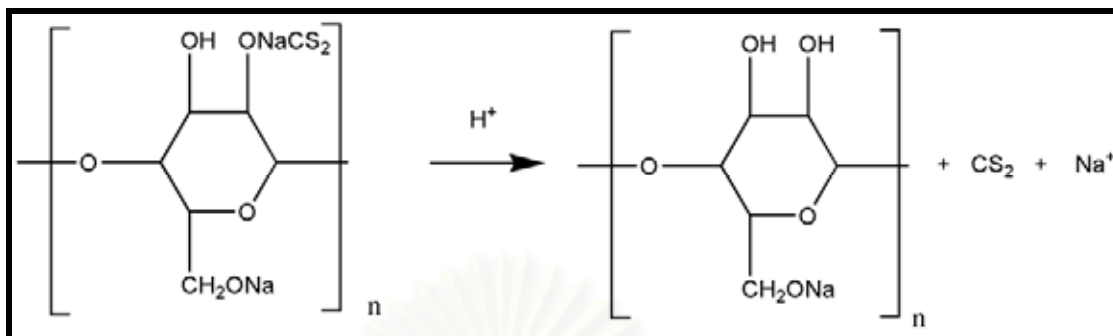


Figure 2.10 Chemical reaction of viscose rayon [29]

2.2.3.2 Properties and Identification of Fiber

2.2.3.2.1 Microscopic Properties

Since rayon is a manufactured fiber, the maker can control its size and shape to a great extent, and different types of rayon fibers, thus, can vary considerably in appearance.

The length or longitudinal appearance of regular viscose rayon exhibits uniform diameter and interior parallel lines called *striations*. These striations are the result of light reflection by the irregular surface contour. If the fiber has been delustered, it will have a grainy, pitted appearance; bright fiber is relatively transparent. The cross section of the fibers shows highly irregular or serrated edges. Here, too, the presence of delusterants is indicated by a spotted effect, while bright fiber appears crystal clear.

High-tenacity viscose is similar to regular viscose, except that it may have a less irregular contour and therefore show fewer striations in the longitudinal view. It may appear almost round in cross section. The same holds true for high-wet-modulus rayon [27].

2.2.3.2.2 Physical and Mechanical Properties

The length, width (diameter), and luster of rayon fibers can be controlled and may be determined by end-use application.

The strength of most rayon is relatively low and is further decreased when fibers are wet, which means that rayon fabrics require careful handling in laundering. High-tenacity and high-wet-modulus viscose fibers are considerably stronger, with dry and wet strength equal to or better than cotton. They can, therefore, be laundered like cotton. Elastic recovery and resiliency of regular viscose and cuprammonium rayon are low, while elongation for both fibers is high. These fabrics tend to wrinkle and stretch easily-weakness that can be corrected by finishes. High-wet-modulus rayon is less subject to stretching and wrinkling, but even these products are better when properly finished.

Rayon fibers have good moisture absorbency, which makes them accept dyes well. The same property also contributes to the high degree of comfort in rayon apparel fabrics.

While regular rayon are subjected to stretching in yarn and fabric manufacture, followed by relaxation shrinkage after laundering, the new high-wet-modulus viscose rayon fibers do not stretch easily, and thus are less likely to suffer relaxation shrinkage. Fabric construction as well as fiber characteristics contribute to the degree of relaxation shrinkage. Tightly woven fabrics will exhibit less size change because of the compact arrangement of yarns and fibers. Finishes are used in most rayon fabrics to control the dimensional stability [27].

2.2.3.2.3 Chemical Properties

The chemical properties of rayon are very similar to those of cotton. Rayon is resistant to most organic solvents, and is not readily decomposed by dilute acids. Resistance to alkaline ranges from poor to good, so chlorine bleaches should be

used according to directions. Rayon fiber is destroyed by concentrated mineral acids [27].

2.2.3.2.4 Thermal Properties

Rayon fibers are cellulose, so they burn rapidly with a yellow flame. A small amount of light gray or off-white fluffy residue is left. When the flame is extinguished. The fibers do not melt.

Hot water and iron temperatures from 300 ° - 350 °F can be used safely, but exposure to high temperatures for and extended period of time results in fiber degradation [27].

2.3 Literature Review

In these recent years, many scientific literatures have revealed a growing interest in plasma treatment on textiles as a method for allowing modification of the properties of textile fibers without changing the manufacturing process. The applications are quite varied.

The finishing of textile fabrics by plasma technologies can advantageously replace some wet chemical applications as an environmental friendly process. Plasma treatment modifies the fiber surface and leaves the bulk characteristics unaffected. By controlling the plasma variables, such as the nature of gas, discharge power, pressure and exposure time, a great variety of surface properties can be improved, mainly wettability, dyeability, printability, soil resistance, and other.

Kan C.W. et. al. [3] induced surface properties of wool fabrics by the sputtering of low-temperature plasma treatment, such as surface luster, wettability, surface electrostatic and dyeability. After low-temperature plasma treatment, the treated wool fabric specimens exhibited better hydrophilicity and surface electrostatic properties at room temperature, together with improved dyeing rate. Study of the surface of the

specimen showed that low-temperature plasma treatment did not effect any changes on the fiber specimens that might possibly provide a pathway for a faster dyeing rate.

Ueno, et. al. [4] provides a method for increasing the color density of a dyed fabric material of, especially, a synthetic fiber as well as the fastness of the color to rubbing and washing. The inventive method comprising (a) pretreating the dyed fabric material with an organopolysiloxane, which is an amino-modified organopolysiloxane or a dimethylpolysiloxane, and then (b) exposing the thus pretreated dyed fabric material to low temperature plasma of an inorganic gas under a reduced pressure. The inorganic gas is preferably oxygen or a gaseous mixture containing at least 10% by volume of oxygen. The color deepening effect is particularly remarkable when the color of the dyed fabric material is black to impart increased graveness and vividness of the color

Ferrero F., et. al. [5] applied the capillary rise method to evaluate the improvement in water uptake of polyester and acrylic fabrics obtained by plasma treatment. Power, exposure time and gas type (nitrogen, air and oxygen) were considered as plasma variables. After plasma treatment, fabric samples were tested through capillary rise of water. The processing a set of height absorption values vs. time, until equilibrium is reached, allows characterization of the wettability of samples by three parameters. The influence of plasma variables on these parameters was studied.

Riccardi C., et. al. [6] induced the surface chemical and physical modifications of poly(ethylene terephthalate) (PET) fibers by radio frequency air plasma treatments were correlated with the characteristics of the discharge parameters and the chemical composition of the plasma itself, to identify the plasma-induced surface processes prevailing under different operating conditions. Treated polymer surfaces were characterized by water droplet absorption time measurements and XPS analysis, as a function of the aging time in different media, and by AFM analysis. They exhibited a remarkable increase in hydrophilicity, accompanied by extensive etching and by the implantation of both oxygen- and nitrogen-containing polar groups. Etching was mainly a consequence of ion bombardment, yielding low molecular weight, water-soluble

oxidation products, while surface chemical modifications were mainly due to the action of neutral species on the plasma-activated polymer surface.

Poletti G., et. al. [7] investigated the morphology changes in the surface of poly(ethylene terephthalate) (PET) fabrics due to cold plasma treatments. This has resulted in the possibility to measure quantitatively the root-mean-square (rms) surface roughness and the surface area of the samples developed after the treatment. The morphology changes, mainly rms surface roughness and surface area, on the PET fabric's surface due to air cold plasma have been measured also for He, Ar, SF₆ and CF₄ gases. The changes in morphology in the cases of air, He and Ar gases seems to be due mainly to etching effects. The situation is different for SF₆ and CF₄ gases where reorganization of the surface, possibly due to fluorine atoms grafting, seems to be effective.

Cioffi M.O.H., et. al. [8] studied mechanical strength of polyethylene terephthalate (PET) fibers and polymethyl methacrylate (PMMA) matrix composites with particular interest on the effects of oxygen and argon plasma treated fibers. PET fibers were treated in a radio frequency plasma reactor using argon or oxygen for different treatment times to increase the interface adhesion. Fiber volume fraction was measured through digital image analysis. Elastic modulus resulted between 3 GPa for untreated to 6 GPa for treated composites. Tensile tests on PET fibers showed that plasma treatment caused a decrease in average tensile strength compared to untreated fibers. Fracture analysis confirmed the increase in interfacial adhesion due to plasma treatment

Jeremy M., et. al. [20] investigations of the effects of plasma treatment, the nature of the plasma environment, and the mechanisms that drive the plasma-surface interaction. We then discuss low-radio-frequency capacitively coupled nitrogen plasmas and their characteristics, suggesting that they provide significant plasma densities and populations of reactive species for effective plasma treatments on a variety of materials, particularly when placing the sample surface in the cathode sheath region. We further discuss surface chemical characterization of treated polymers, including some results

on polyesters treated in capacitively coupled nitrogen plasmas driven at 40 kHz. Finally, we connect plasma characterization with surface chemical analysis by applying a surface sites model to nitrogen uptake of poly(ethylene terephthalate) (PET) and poly(ethylene naphthalate) (PEN) treated in a 40 kHz nitrogen plasma. This example serves to suggest an interesting practical approach to comparisons of plasma treatments. In addition, it suggests an approach to defining the investigations required to conclusively identify the underlying treatment mechanisms.

The above application are also employed o plastics as follows:

Carrino L., et. al. [9] deals with air cold plasma treatment of polypropylene surface, the influence of AC electrical discharge cold plasma parameters on wettability and adhesion of polymeric surface was studied. Also, the nature of the relationship between wettability and adhesive was investigated. Owing to the complexity of plasma-work piece interaction, an experimental approach was followed. A set of process variables (voltage, time and air flow rate) was identified and used to conduct some experimental tests on the basis of design of experimental techniques. The experimental results show that the proposed plasma process may considerably increase polypropylene wettability and adhesive properties.

Cueff R., et. al. [10] studied the chemical effects of a reactive plasma treatment on the PET surface by X-ray photoelectron spectroscopy and investigated the chemical interactions to involve at the alumina/PET interface. The treatment of the PET by carbon dioxide plasma introduces new oxygen reactive species on the polymer surface. Evidence of this chemical modification is given by the appearance of additional carbonyl bonds. The interfacial study reveals a reaction of alumina with the oxygen atoms of the carbonyl bonds of PET (plasma-induced carbonyl bonds and carbonyl bonds of the ester groups). This chemical interaction results in the formation of Al-O-C interfacial bonds in the early stages of deposition.

Inagaki., et. al. [17] studied the polyethylene film that was treated with oxygen and nitrogen plasma. The activated species interact with the polymer's surface and then

special function, such as hydroxyl, carbonyl, carboxyl, amino and amide groups are formed at the surface of the polymers. As result, the implantation reactions lead to large changes in the surface properties of the polymer, for example, the polymers change from hydrophobic to hydrophilic.



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CHAPTER III

EXPERIMENT

3.1 Scope of Experiment

In this chapter, the properties and morphology of plasma-treated man-made fibers were studied. Man-made fibers were treated by oxygen or nitrogen plasmas generated from theta-pinch device at various numbers of plasma shots. A summary of the scope of the experiment is shown in Figure 3.1.

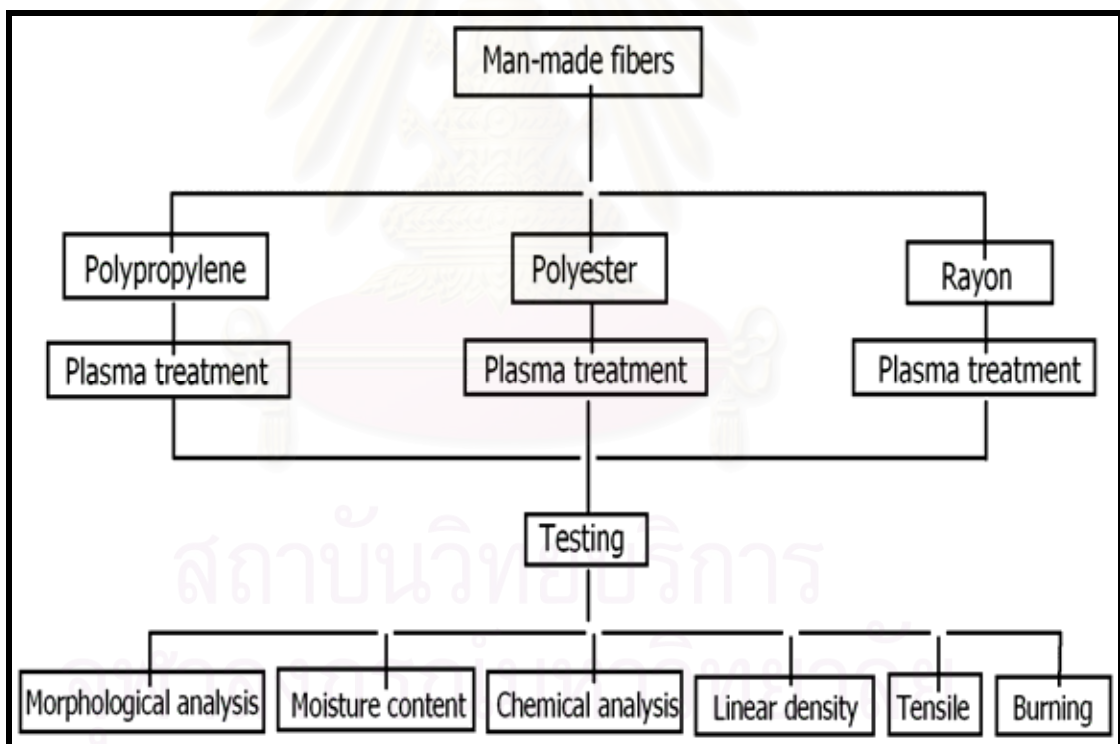


Figure 3.1 Scope of experiment

3.2 Materials and Equipments

3.2.1 Materials

3.1.1 Polyester fiber was provided by Num Rung Rayon Co., Ltd. The fiber was received in the form of yarn. It consists of 36 filaments with the size of 75 Denier and having melting temperature of 460 – 554 °F

3.1.2 Polypropylene fiber was provided by Num Rung Rayon Co., Ltd. The fiber was received in the form of yarn. It consists of 24 filaments with the size of 70 Denier and having melting temperature of 225 – 255 °F

3.1.3 Rayon fiber was provided by Num Rung Rayon Co., Ltd. The fiber was received in the form of yarn. It consists of 24 filaments with the size of 75 Denier and having melting temperature of 350 °F

3.2.2 Equipments

3.2.1 Theta-pinch Device

3.2.2 Universal Testing Machine (LLOYD 500)

3.2.3 Fourier Transform Infrared Spectrophotometer (Thermo Nicolet Nexus 670)

3.2.4 Infrared Moisture Determination Balance (AND AD-4715)

3.2.5 Weighing Balance (AB 204)

3.2.6 Optical Microscope (OLYMPUS BH-2)

3.2.7 Scanning Electron Microscope (JSM-6400)

3.3 Procedure

3.3.1 Fiber Modification by Theta-pinch Device

A single fiber was removed from each type of the yarns. Each fiber was wound around of metal bar and it is placed in the middle of the theta-pinch device chamber. Theta-pinch was then operated. After the treatment completed, the fiber was removed from the chamber. The processed fiber was characterized and was subjected to mechanical tests.

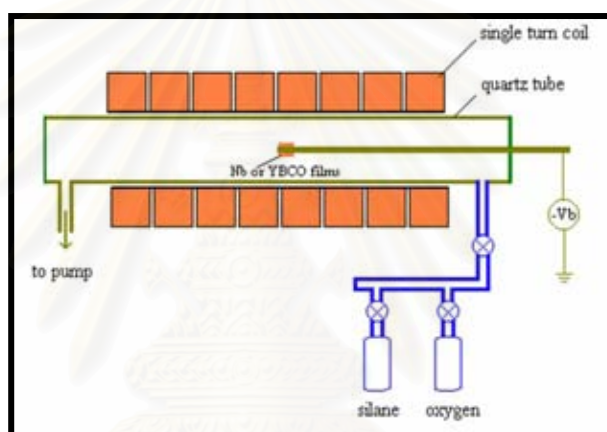


Figure 3.2 Configuration of theta-pinch device



(a)



(b)

Figure 3.3 a) Front view of Theta-pinch device

b) Side view of Theta-pinch device

Table 3.1 shows the conditions used for modification. The type of gases and the number of plasma shots were varied in order to study the effects of these parameters on the properties of each man-made fiber. From this table, it can be seen that each man-made fiber was modified at different number of plasma shots depending on the melting temperature of each man-made fiber. The number of plasma shots increased corresponding to an increase in fiber melting temperatures in order of polypropylene fiber, Rayon fiber and polyester fiber, respectively.

Table 3.1 Modification conditions used in surface modification of man-made fibers by theta-pinch device

Type \ Gas	Number of plasma Shots							
	Nitrogen				Oxygen			
Polyester fibers	20	30	40	50	20	30	40	50
Polypropylene fibers	3	5	7	10	3	5	7	10
Rayon fibers	10	20	30	40	10	20	30	40

3.3.2 Characterization and Mechanical Testing

3.3.2.1 Morphological Analysis

Optical microscope (OLYMPUS BH-2) as shown in Figure 3.4a was used to observe the surface of the fibers. Prior to test, a sample was placed on the glass slide in horizontal axial. Then it were observed by magnification of 20X.

JSM-6400 Scanning electron microscope (SEM) was also used to characterize the sample morphology. Prior to test, a sample was placed on the stub with double sticky tape. Then it was coated with thin evaporated layer of gold in order to improve conductivity and prevent electron charging on the surface. SEM was operated at 15 keV

acceleration voltages. The SEM photographs were taken at different angles of view with magnification of 2300X.

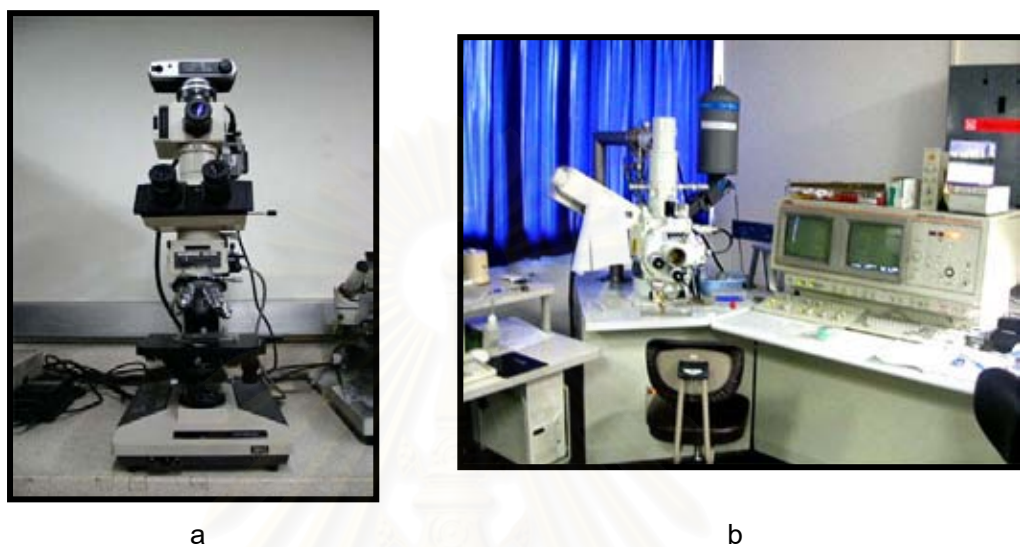


Figure 3.4 a) Optical microscope

b) SEM

3.3.2.2 Determination of Moisture Content

Moisture content of the fibers was measured according to ASTM D2654. Each sample was exposed in normal atmosphere at room temperature for 24 hours. After that, the sample was weighed in the condition state and dried at $105 \pm 2^{\circ}\text{C}$ and weighed again to determine the weight change. The moisture content was calculated as the weight difference and reported as percentage of weight decrease from the initial weight using equation 3.1.

$$\text{Moisture Content, \%} = 100 (A-D)/A \quad (3.1)$$

Where:

A = the mass of fiber, mg. and

D = the mass of dried fiber, mg.



Figure 3.5 Infrared moisture determination balance

3.3.2.3 Analysis of Chemical Structure

Fourier transform infrared spectroscopy (FT-IR) was performed using Thermo Nicolet Nexus 670 to characterize the chemical structures of the fibers. A sample was mixed with potassium bromide (KBr) and pressed into the disc form by the hydraulic compression. The samples were scanned at the frequency range of $4000\text{-}400\text{ cm}^{-1}$ with 32 consecutive scans and 4 cm^{-1} resolutions.



Figure 3.6 Fourier transform infrared spectrophotometer

3.3.2.4 Determination of Linear Density

Linear Density of the fibers was measured according to ASTM D 1577. The untreated and treated fibers were tested in the standard atmosphere for testing of textiles, which was $21 \pm 1^{\circ}\text{C}$ ($70 \pm 2^{\circ}\text{F}$) and $65 \pm 2\%$ relative humidity. Each fiber was cut and its length was measured to the nearest estimated 0.1 mm. Then each fiber was weighed to the nearest 0.0001 mg and its mass was recorded. Linear density of a fiber was calculated using equation 3.2 with $N=1$.

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$$D = 9000 W / (L \times N) \quad (3.2)$$

Where:

D = average fiber linear density, denier,

W = mass of bundle specimen, mg,

L = length of bundle specimen, mm, and

N = number of fibers in the bundle specimen.



Figure 3.7 Weighing balance

3.3.2.5 Determination of Tensile Properties

Tensile Properties of the fibers was determined according to ASTM D2256 using Universal testing machine (LLOYD 500). A sample was tested at a testing speed of 300 ± 10 mm./min using a gauge length of 250 ± 3 mm. and load cell of 1 N.



Figure 3.8 Universal testing machine

3.3.2.6 Burning Test (AATCC 20)

Burning test of the fibers based on AATCC 20 standard was performed. At first, a sample was placed close to the side of a small flame and it was noted if the sample melted or shrink from the flame. After that, the sample was moved into the flame and it was noted whether the sample burnt when held in the flame. Then it was slowly and carefully removed from the flame and it was noted whether the sample continued to burn after removing from the flame. In addition, the ash residues were observed.

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Research Objectives

The main objective of this research is to investigate an alternative method for surface modification of man-made fibers using hot plasmas generated as discontinuous pulse from theta-pinch device. This research also aims to determine the optimum conditions for modification of each type of selected fibers. The selection of man-made fibers was based on hydrophobic-hydrophilic characteristics of the fibers which are the main characteristics that significantly change after cold plasma-treatment as previously reported by many literatures. [3-14, 17, 20] Therefore, polypropylene fiber, polyester fiber and rayon fiber which respectively represent hydrophobic, hydrophilic and more hydrophilic fibers were selected. The following sections will cover the comparison of morphology and properties of untreated and treated fibers for each type of selected fibers.

4.2 Morphology and Properties of Polypropylene Fiber

4.2.1 Cross-section

The cross-sections of polypropylene fibers observed after they were treated with nitrogen and oxygen plasmas produced by theta-pinch device are shown in Figures 4.1 and 4.2, respectively. It can be seen that plasma treatment changed the shape of cross-sections of polypropylene fibers. This change increased with increasing the number of plasma shots and it also depended on the type of gas used. However, average diameters of these plasma-treated fibers were slightly decreased, it is due to the etching effects on surface of polypropylene fiber as shown in Table 4.1.

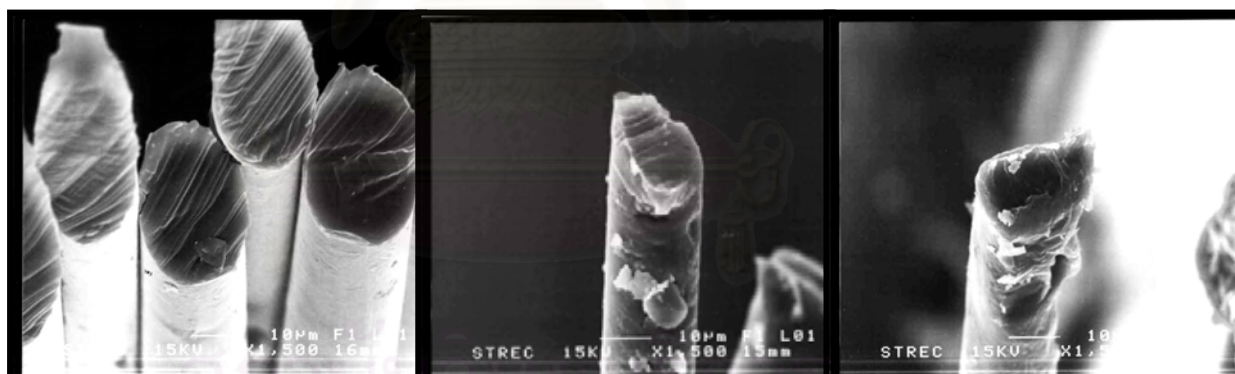


a

b

c

Figure 4.1 SEM photographs of cross-sections of untreated polypropylene fiber (a) and polypropylene fibers treated with 3 (b) and 10 (c) shots of nitrogen plasma



a

b

c

Figure 4.2 SEM photographs of cross-sections of untreated polypropylene fiber (a) and polypropylene fibers treated with 3 (b) and 10 (c) shots of oxygen plasma

Table 4.1 Diameters of untreated and plasma-treated polypropylene (PP) fibers

Type of PP Fiber	Diameter (μm)	
	Nitrogen	Oxygen
Untreated	25.83 ± 3.26	25.83 ± 3.80
Treated with 3 plasma shots	22.03 ± 3.26	22.54 ± 3.80
Treated with 10 plasma shots	19.35 ± 3.26	18.26 ± 3.80

4.2.2 Morphology

The drastic change on polypropylene fiber surface after plasma treatment was observed by optical microscopy and scanning electron microscopy at 20X and 2300X magnifications as shown in Figures 4.3 and 4.4. The induced plasma caused an increase in the roughness of the fiber surface. This change is possibly caused by the reactions between plasma and polypropylene molecules. Therefore, as the number of plasma shots increases, higher reaction can occur. Consequently, it can be seen from both figures that the roughness of the surface increases as the number of plasma shots increases. Furthermore, it was also observed that the change in roughness depended on the type of gases. Nitrogen plasma treatment resulted in higher etching of the surface than oxygen plasma. This may be due to the difference in the reactions occurred in both gases.

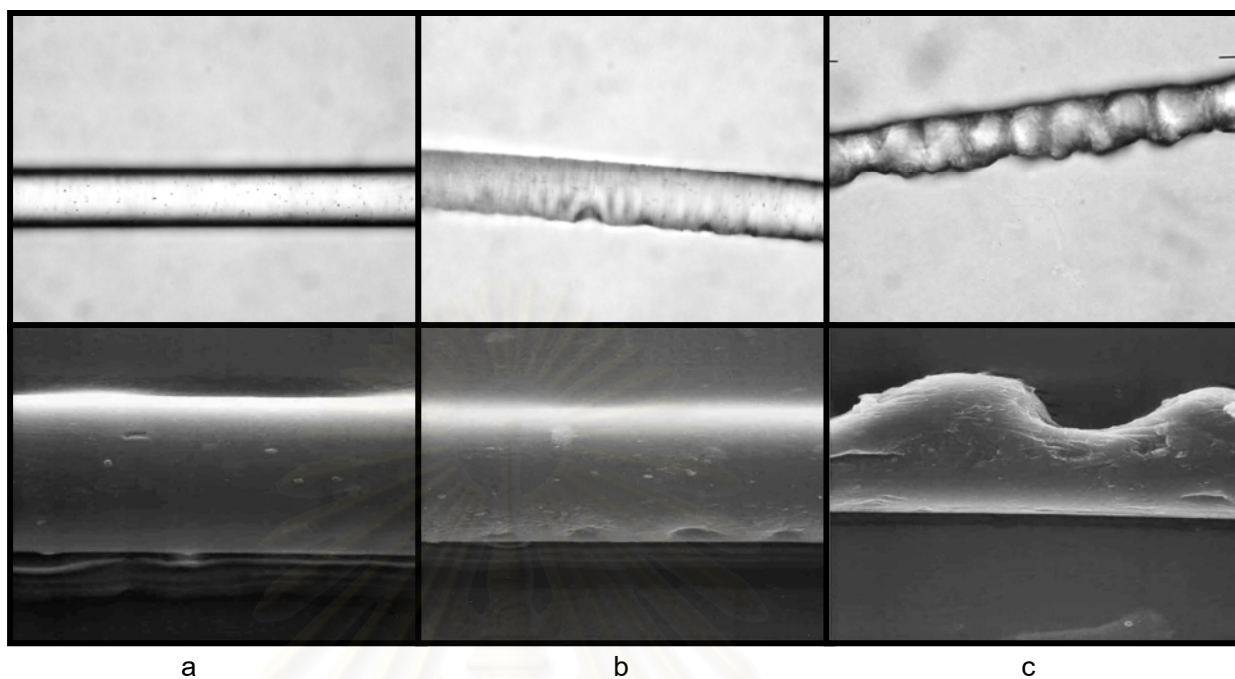


Figure 4.3 Optical (top) and SEM photographs (bottom) of untreated polypropylene fiber (a) and polypropylene fibers treated with 3 (b) and 10 (c) shots of nitrogen plasma

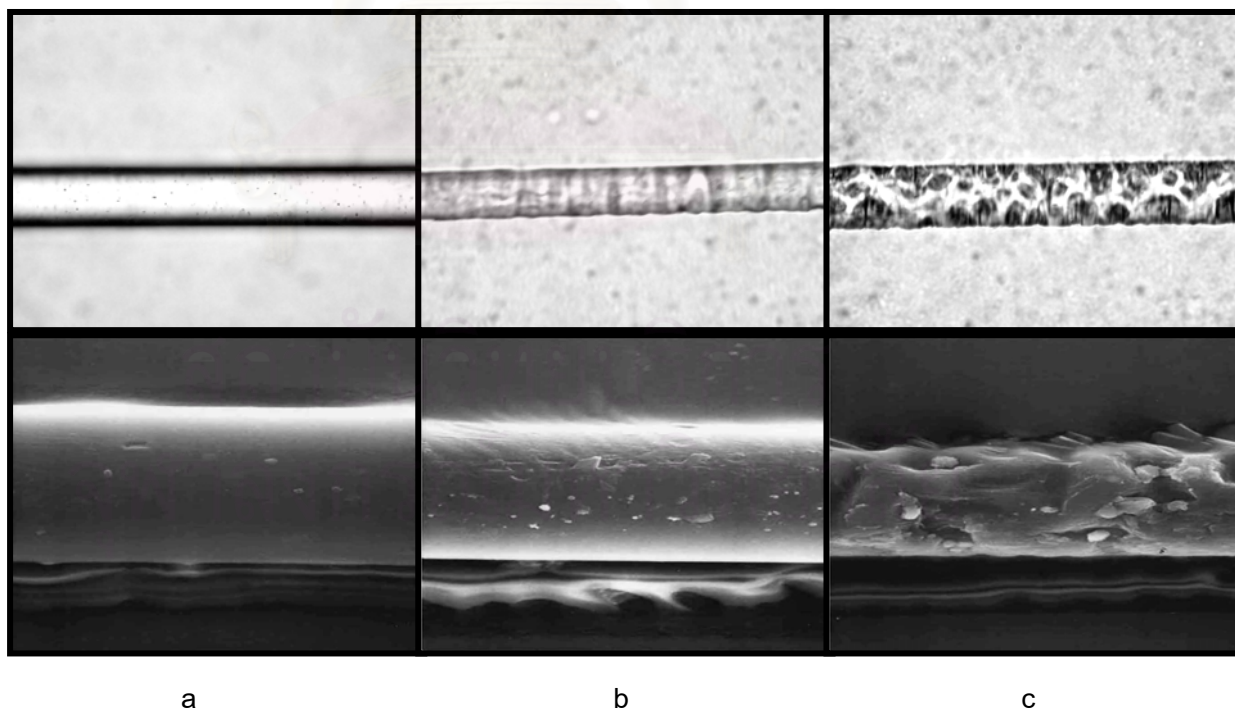


Figure 4.4 Optical (top) and SEM photographs (bottom) of untreated polypropylene fiber (a) and polypropylene fibers treated with 3 (b) and 10 (c) shots of oxygen plasma

4.2.3 Linear Density

Linear density was also used to determine the weight of fibers that may change during surface modification. It is shown in form of the weight per length at 9000 meters (Denier).

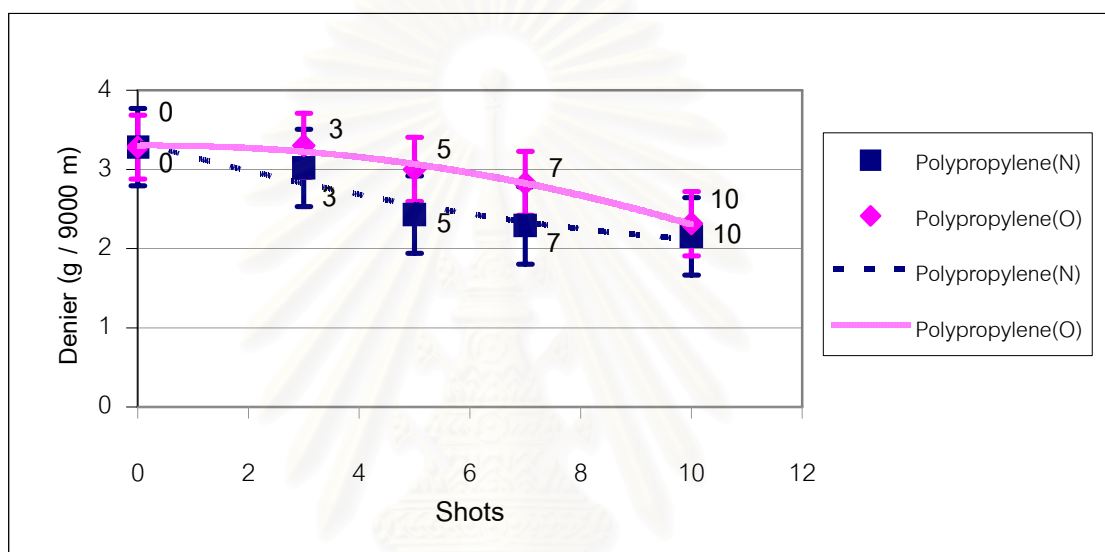


Figure 4.5 Linear densities of untreated and plasma-treated polypropylene fibers

It was found that deniers of treated fibers using both nitrogen and oxygen plasmas were lower than that of untreated fiber as shown Figure 4.5. This is a result of the etching of fiber surface by plasma as can be seen from Figures 4.3 and 4.4. Therefore, the weight of the fiber decreases with increasing the number of plasma shots and denier of the fiber decreases. Furthermore, it can be seen that nitrogen plasma-treated polypropylene fiber exhibit lower denier than that treated with oxygen-plasma. These results also correspond to those observed by SEM as shown in Figures 4.3 and 4.4.

4.2.4 Chemical Structure

As was reported by Inagaki [17], oxygen and nitrogen plasma treatment can cause the formation of many functional groups such as carboxylic, aldehyde, ketone, amino, etc. depending on the type of the gas and fiber. They proposed the mechanism of oxygen plasma treatment on polyethylene indicating possible formation of hydroxyl, ketone, aldehyde or carboxylic groups. As for this research, the following mechanisms in Figures 4.6 and 4.7 are proposed for both nitrogen plasma and oxygen plasma treatments on polypropylene fiber. For nitrogen plasma treatment, two functional groups can be formed: amino ($-\text{NH}_2$) and azo ($-\text{N}=\text{N}-$). In the case of oxygen plasma treatment, four functional groups can be formed: hydroxyl ($-\text{OH}$), ketone ($-\text{C}=\text{O}$), aldehyde ($-\text{CH}=\text{O}$) or carboxylic ($-\text{COOH}$).



FT-IR spectra of polypropylene fibers treated with nitrogen plasma compared to the untreated fiber are shown in Figures 4.8. All plasma-treated fibers exhibit the appearance of the peak corresponding to NH stretching at wavenumber range of 3400-3500 cm^{-1} and peak corresponding to N=N at wavenumber of 1744 cm^{-1} while the intensities of the peak corresponding to CH_3 bending at 1461 cm^{-1} decreases. These results suggest the formation of amine and azo groups at methylene (CH_2) position of polypropylene molecule.

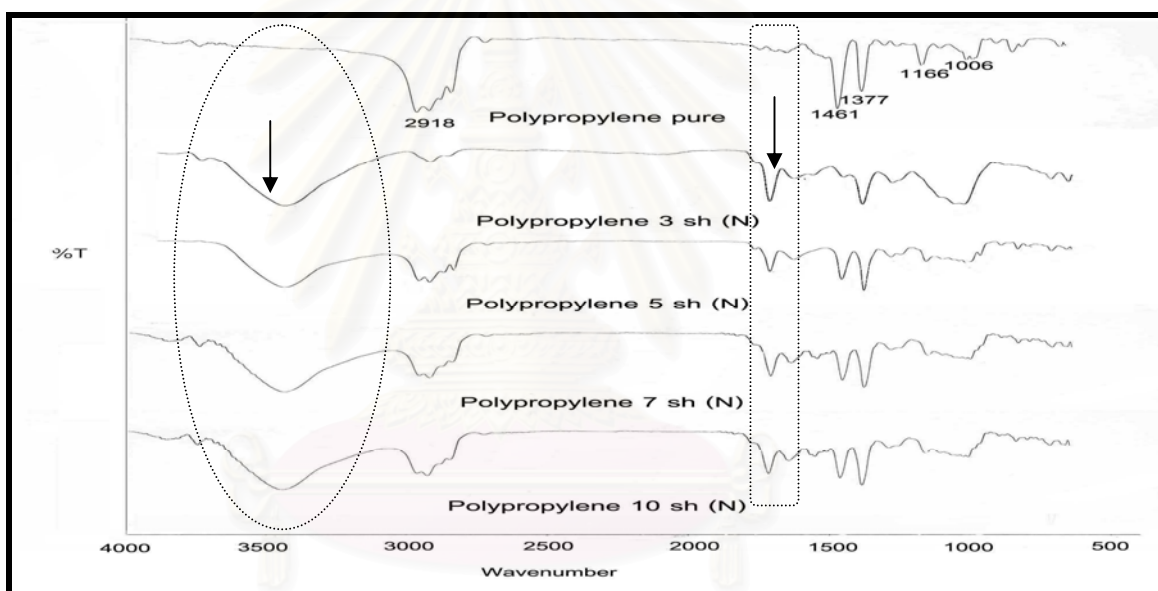


Figure 4.8 FT-IR spectra of untreated and nitrogen plasma-treated polypropylene fibers

In the case of oxygen treated polypropylene fiber, there are no peak corresponding to OH stretching at wavenumber 3300-3500 cm^{-1} . However, the appearance of the peak corresponding to C=O around 1800 cm^{-1} indicates the formation of ketone groups.

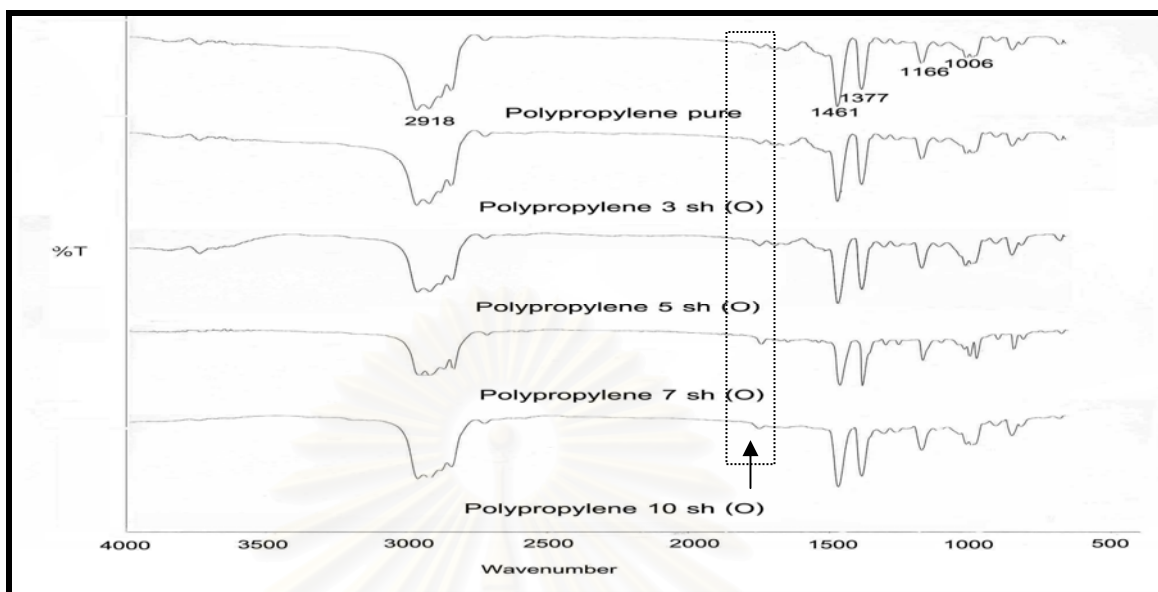


Figure 4.9 FT-IR spectra of untreated and oxygen plasma-treated polypropylene fibers

4.2.5 Moisture Content

Moisture content of polypropylene fibers treated with nitrogen and oxygen plasmas compared to the untreated fiber is exhibited in Figure 4.10.

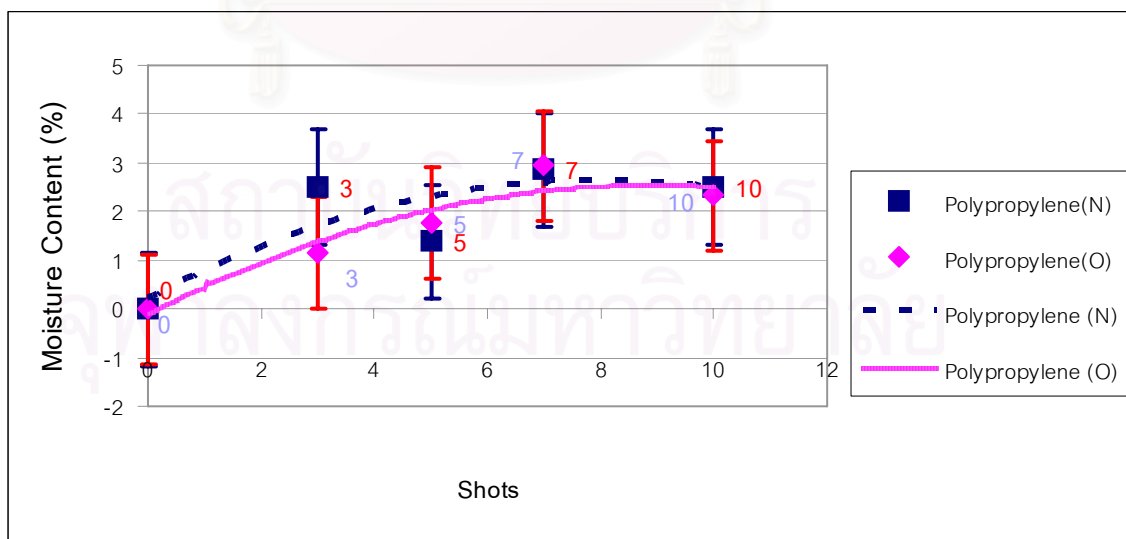


Figure 4.10 Moisture contents of untreated and plasma-treated polypropylene fibers

It is clearly seen that moisture content of all treated polypropylene fibers are higher than that of the untreated fiber. This may be due to the formation of hydrophilic functional groups on the fiber as previously discussed. Thus, it is easier for water molecules to form hydrogen bonds with these hydrophilic groups on the surface of treated fibers than with hydrophobic groups of the untreated fiber. In addition, it was also found that as the number of plasma shots increases indicating more hydrophilic groups and higher surface area, moisture content of the fiber increases. Since surface area of nitrogen plasma treated fiber is higher than that of oxygen plasma treated fiber as shown in Figures 4.3 and 4.4, moisture content of the former is higher than that of the latter.

4.2.6 Tensile Properties

The effects of plasma treatment on tensile properties of treated polypropylene fibers are shown in form of tenacity, elongation and modulus as exhibited in Figures 4.11 to 4.13.

Figure 4.11 shows that tenacity of all treated polypropylene fibers is slightly lower than that of the untreated fiber. This may be due to plasma etching of the fiber as previously discussed. This etching can result in lower molecular weight polypropylene chains which have lower tensile strength when compared to those higher molecular weight molecules in untreated fiber. However, the formation of hydrophilic groups after plasma treatment yields stronger intermolecular bonds such as hydrogen bonds. These two effects are in the contrary but the former effect is slightly higher; therefore, the results occurred as follows.

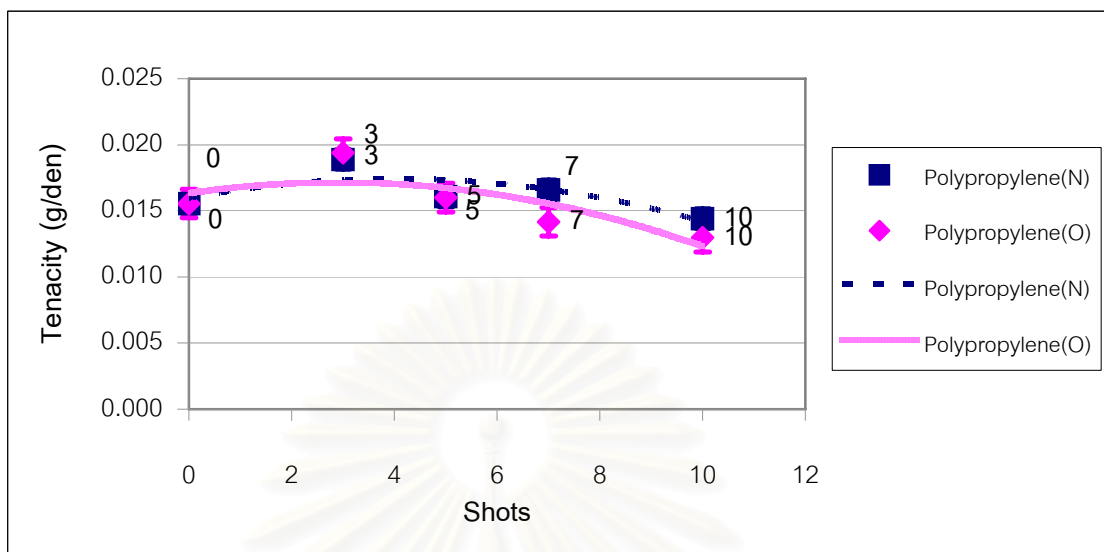


Figure 4.11 Tenacity of untreated and plasma-treated polypropylene fibers

%Elongation of all plasma-treated polypropylene fibers is lower than that of the untreated fiber as shown in Figure 4.12. This is due to stronger intermolecular forces between treated polypropylene molecules caused by the formation of hydrophilic groups. Thus, it is more difficult for treated polypropylene molecules to move apart from each other due to these stronger forces. These effects on tenacity and %elongation also results in an increase in modulus as shown in Figure 4.13.

The effect of the type of the gas is not clearly seen. Nitrogen and oxygen plasma-treated polypropylene fibers exhibit comparable tensile properties.

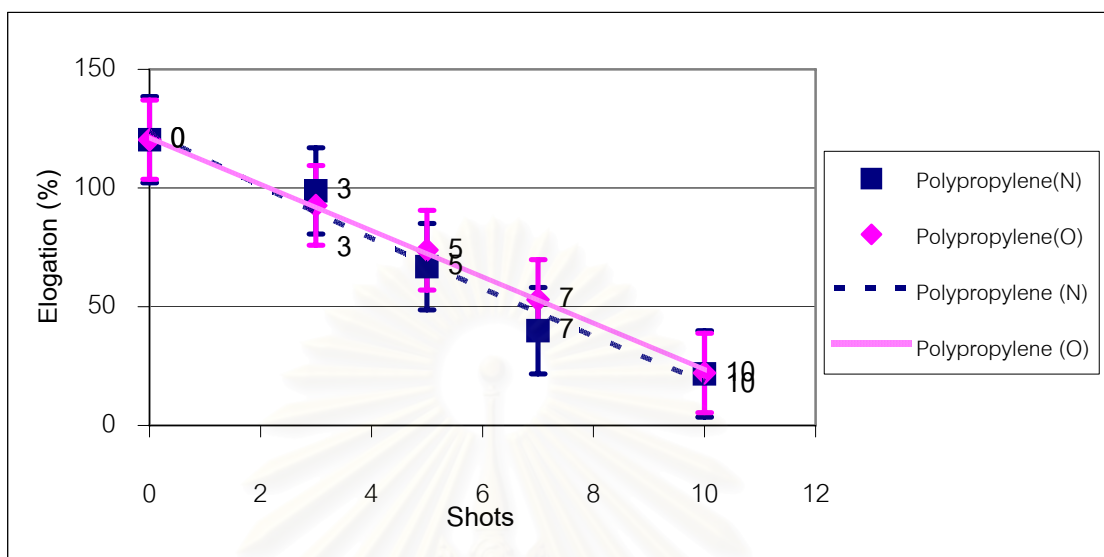


Figure 4.12 Elongation of untreated and plasma-treated polypropylene fibers

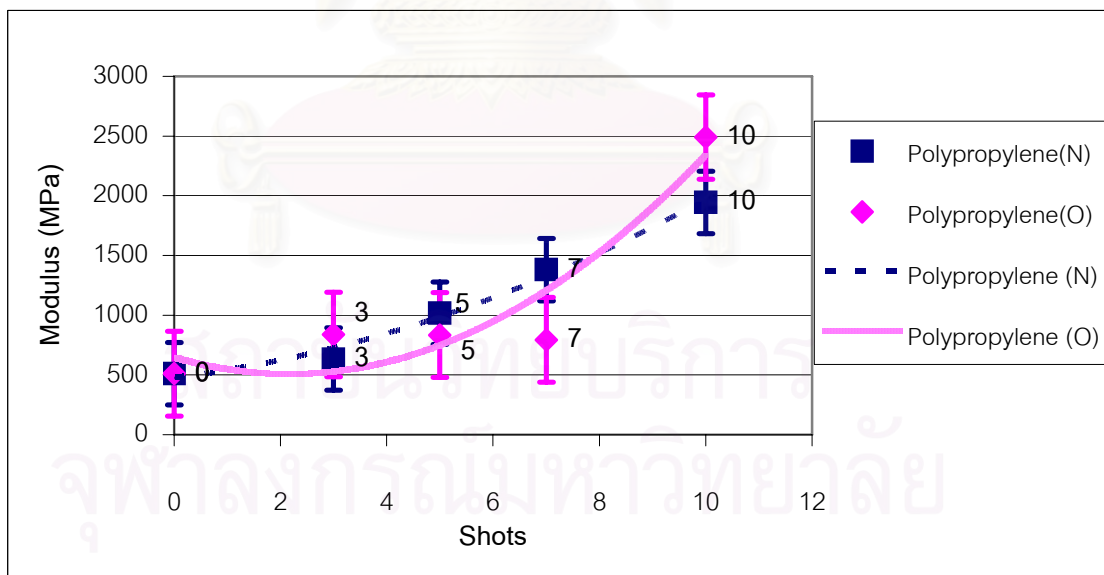


Figure 4.13 Modulus of untreated and plasma-treated polypropylene fibers

4.2.7 Burning Characteristics

The results of burning characteristics of untreated and plasma-treated polypropylene fibers are given in Table 4.2. It shows that their characteristics are similar indicating that surface modification by plasma does not affect burning characteristics of polypropylene fiber.

Table 4.2 Burning Characteristics of Polypropylene Fiber

Fiber	Approaching Flame	In Flame	Removed from Flame	Residue
Untreated	Shrinks and curls away from flame	Melts and burns	Continues to burn and melt	Hard, tough, gray bead
Nitrogen plasma treated	Shrinks and curls away from flame	Melts and burns	Continues to burn and melt	Hard, tough, gray bead
Oxygen plasma treated	Shrinks and curls away from flame	Melts and burns	Continues to burn and melt	Hard, tough, gray bead

4.3 Morphology and Properties of Polyester Fiber

4.3.1 Cross-section

The cross-sections of polypropylene fibers observed after they were treated with nitrogen and oxygen plasmas produced by theta-pinch device are shown in Figures 4.1 and 4.2, respectively. It can be seen that plasma treatment changed the shape of cross-sections of polypropylene fibers. This change increased with increasing the number of plasma shots and it also depended on the type of gas used. However, average

diameters of these plasma-treated fibers were comparable to that of the untreated one as shown in Table 4.3.

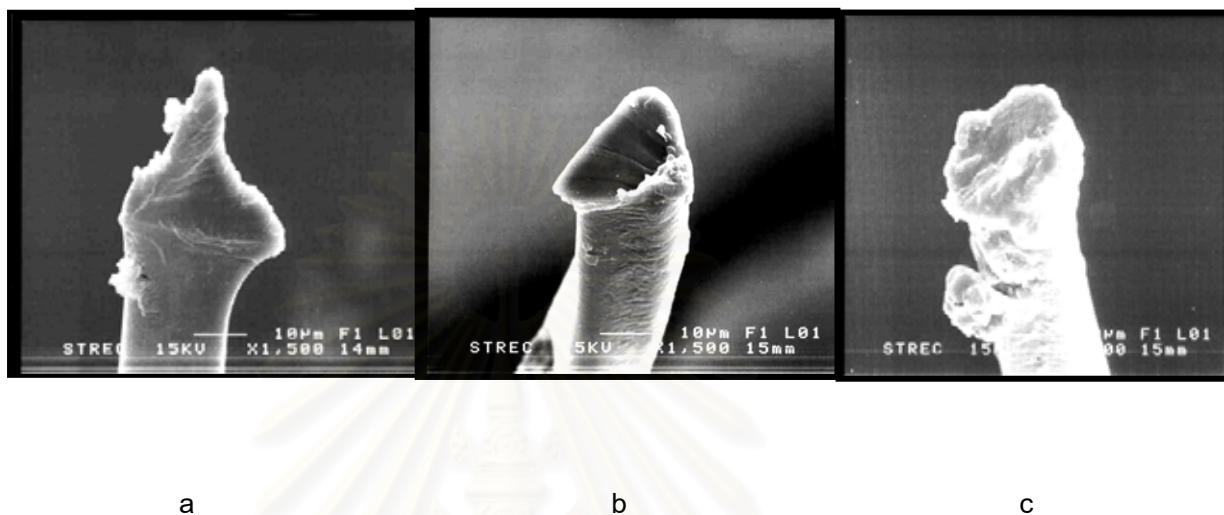


Figure 4.14 SEM photographs of cross-sections of untreated polyester fiber (a) and polyester fibers treated with 20 (b) and 50 (c) shots of nitrogen plasma

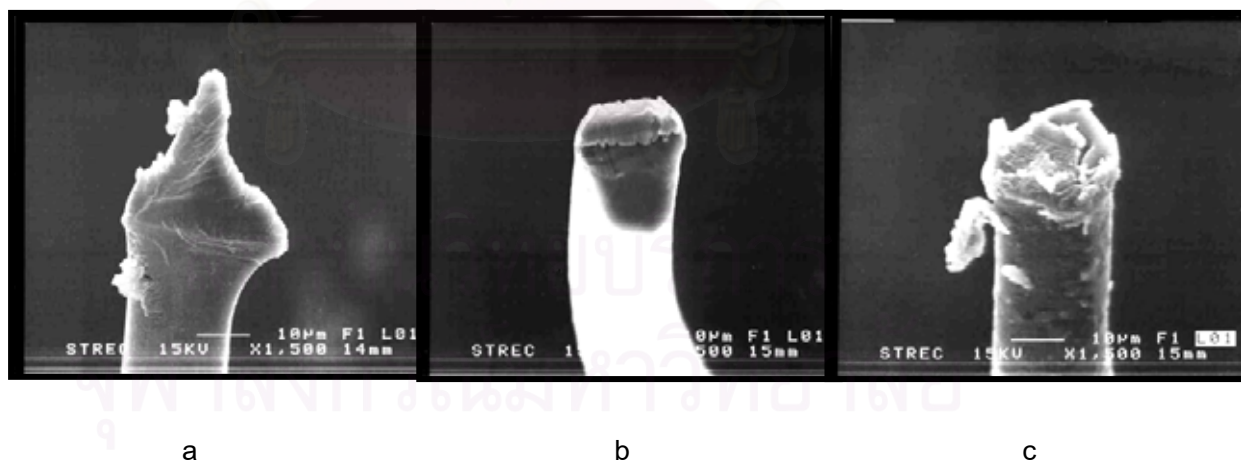


Figure 4.15 SEM photographs of cross-sections of untreated polyester fiber (a) and polyester fibers treated with 20 (b) and 50 (c) shots of oxygen plasma

Table 4.3 Diameters of untreated and plasma-treated polyester (PET) fibers

Type of PET Fiber	Diameter (μm)	
	Nitrogen	Oxygen
Untreated	21.50 ± 0.21	21.50 ± 0.38
Treated with 20 plasma shots	21.08 ± 0.21	20.78 ± 0.38
Treated with 50 plasma shots	21.25 ± 0.21	21.33 ± 0.38

4.3.2 Morphology

Morphology of untreated and treated polyester fibers was characterized using optical microscope and scanning electron microscope. Both optical and SEM photographs in Figures 4.16 and 4.17 show the drastic change on the fiber surface after plasma treatment. The induced plasma caused an increase in the roughness of the fiber surface. The same phenomenon was also reported by Poletti and et al. who studied the surface morphology characteristic of cold plasma treatment of PET fabrics by AFM [8]. This change in roughness is possibly caused by the reaction between plasma and polyester molecules. Therefore, as the number of plasma shots increases, higher reaction can occur. Consequently, it can be seen from both figures that the roughness of the surface increases as the number of plasma shots increases. Furthermore, it was also observed that the change in roughness depended on the type of gases. This may be due to the difference in the reactions occurred in both gases.

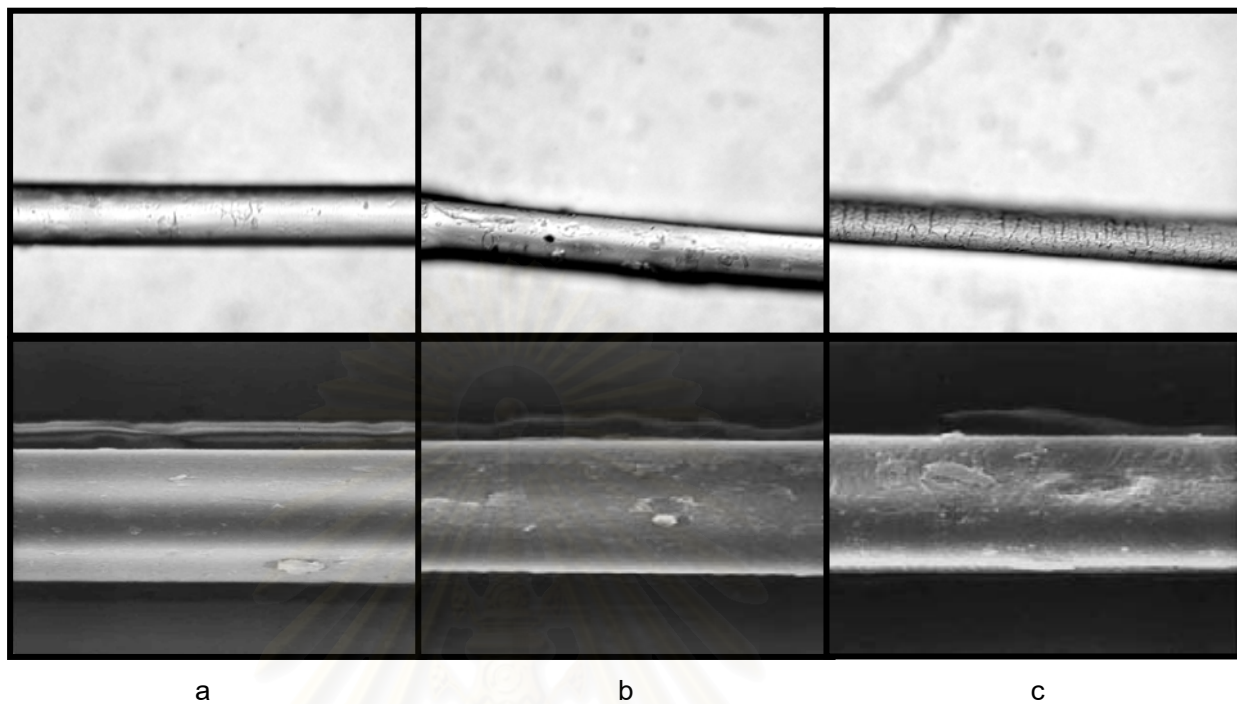


Figure 4.16 Optical (top) and SEM photographs (bottom) of untreated polyester fiber (a) and polyester fibers treated with 20 (b) and 50 (c) shots of nitrogen plasma

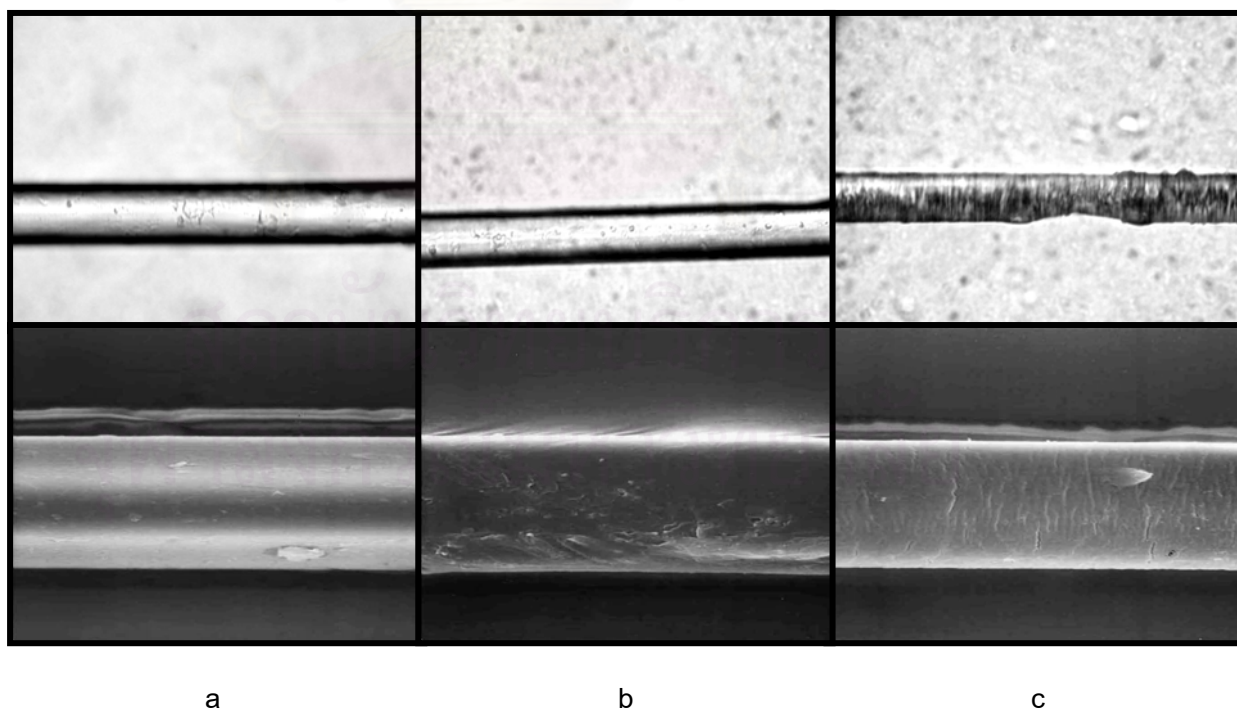


Figure 4.17 Optical (top) and SEM photographs (bottom) of untreated polyester fiber (a) and polyester fibers treated with 20 (b) and 50 (c) shots of oxygen plasma

4.3.3 Linear Density

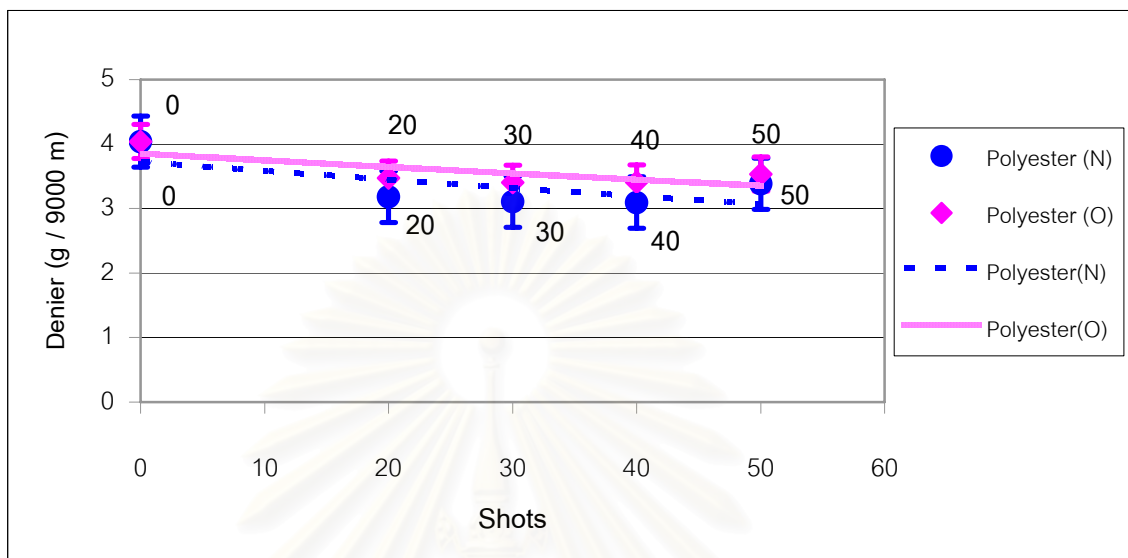


Figure 4.18 Linear densities of untreated and plasma-treated polyester fibers

It was found that deniers of treated fibers using both nitrogen and oxygen plasmas were lower than that of untreated fiber as shown Figure 4.18. The results are in conclusive with those of treated polypropylene fibers in which the weight of the fiber decreases with increasing the number of plasma shots and denier of the fiber decreases and nitrogen plasma-treated polypropylene fiber exhibit lower denier than that treated with oxygen-plasma. These results are caused by the same effects as described for polypropylene fibers.

4.3.4 Chemical Structure

FT-IR spectra of untreated and nitrogen plasma treated polyester fibers are shown in Figure 4.19. An increase in relative intensity of the peak at wavenumber 3400-3500 cm^{-1} indicates the formation of amine groups.

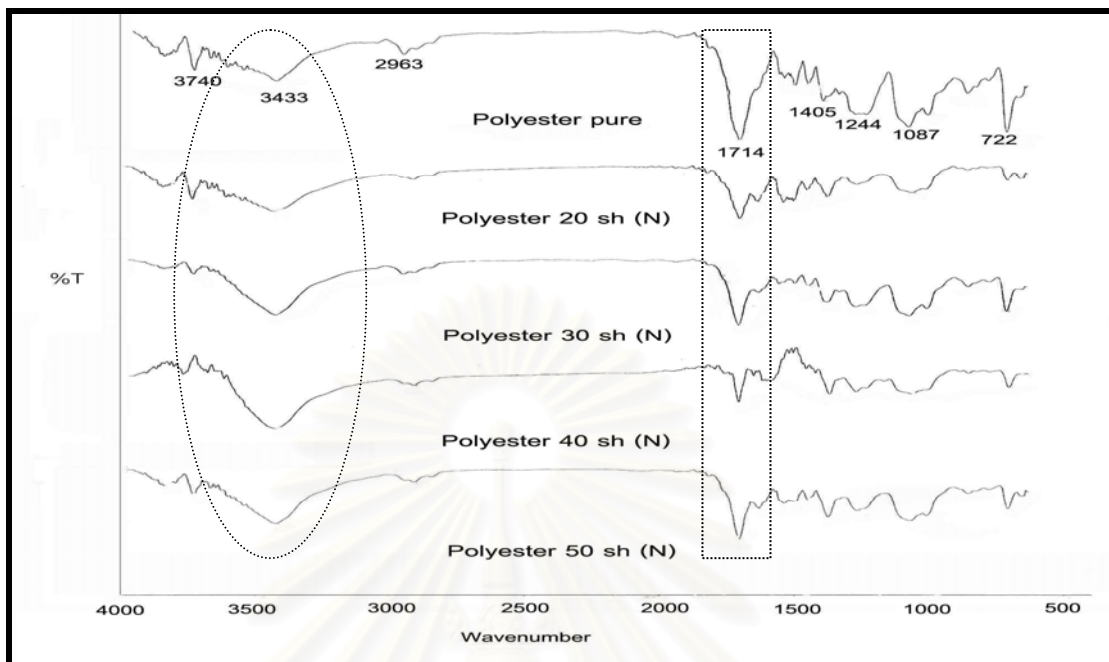


Figure 4.19 FT-IR spectra of untreated and nitrogen plasma-treated polyester fibers

On the other hand, FT-IR spectra of untreated and oxygen plasma treated polyester fibers shown in Figure 4.20 suggests the possibilities of the formation of several groups on polyester fiber. The disappearance or increasing intensities or decreasing intensities of OH stretching at wavenumber range of $3300\text{-}3500\text{ cm}^{-1}$ and splitting of the peak corresponding to C=O group at 1714 cm^{-1} indicate the formation of hydroxyl, carboxylic, ketone and aldehyde groups.

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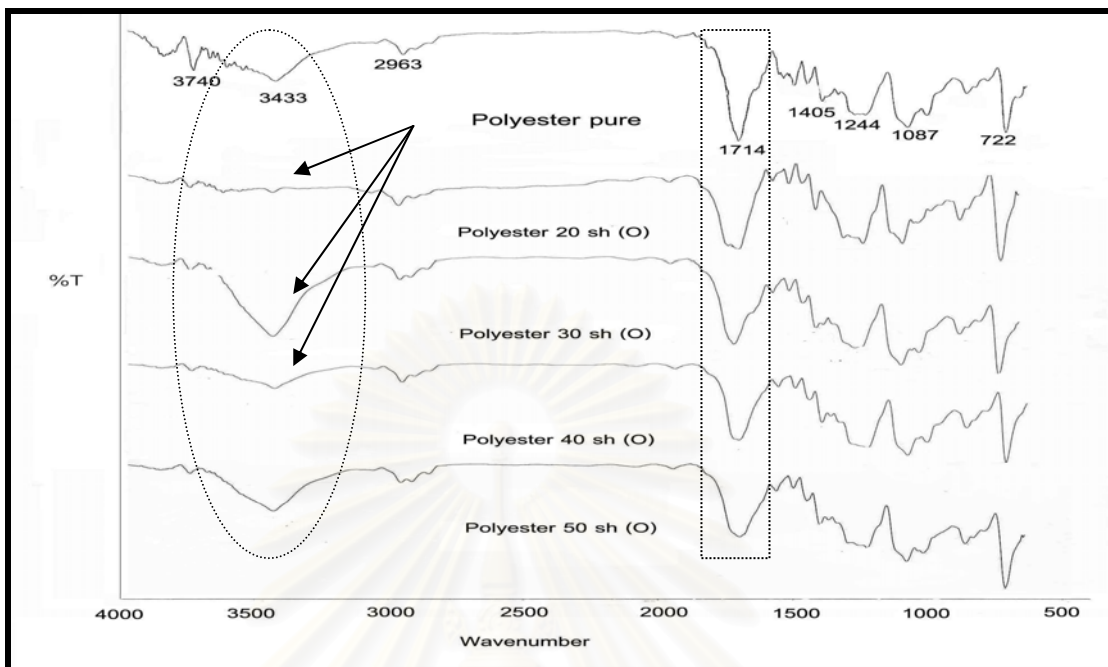


Figure 4.20 FT-IR spectra of untreated and oxygen plasma-treated polyester fibers

4.3.5 Moisture Content

Moisture content of polyester fibers treated with nitrogen and oxygen plasmas compared to the untreated fiber is exhibited in Figure 4.21.

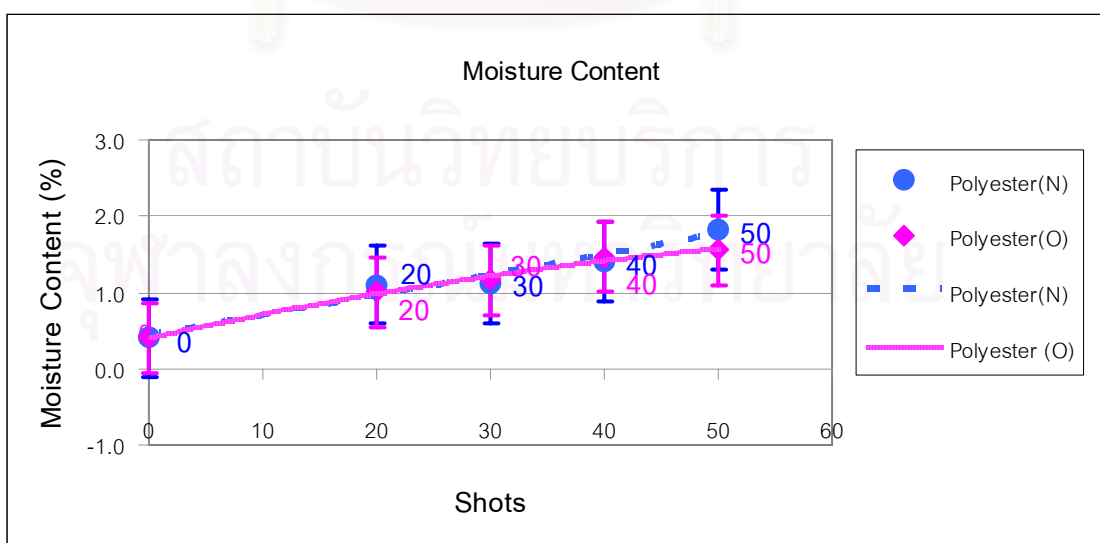


Figure 4.21 Moisture contents of untreated and plasma-treated polyester fibers

It is clearly seen that moisture content of all treated polyester fibers are higher than that of the untreated fiber. This may be due to the formation of hydrophilic functional groups on the fiber as previously discussed. This result is in good agreement with the result obtained by Cioffi and et. al. who studied the properties of oxygen plasma treated PET [10].

4.3.6 Tensile Properties

The effects of plasma treatment on tensile properties of treated polyester fibers are shown in form of tenacity, elongation and modulus as exhibited in Figures 4.22 to 4.24.

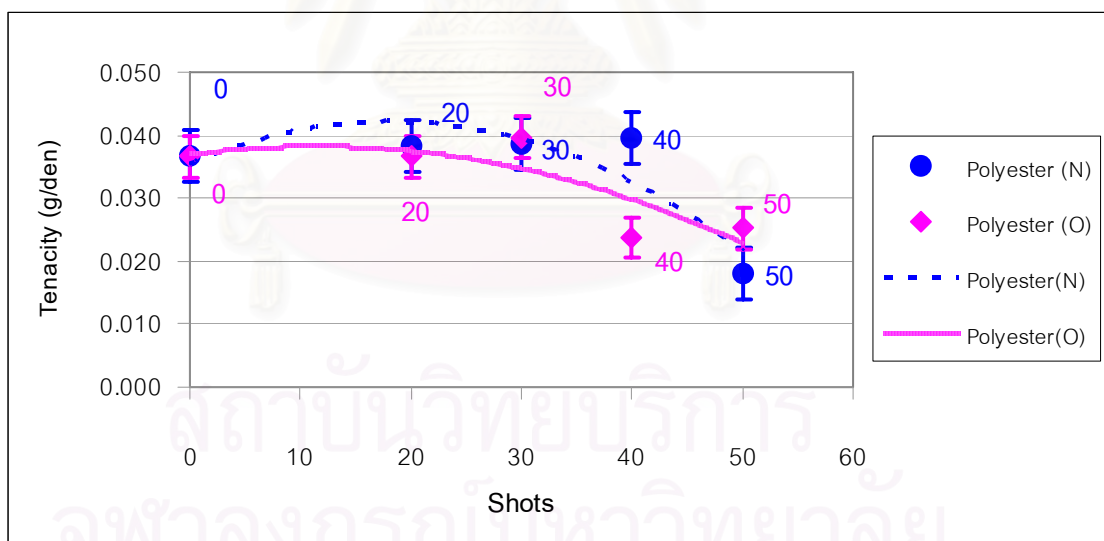


Figure 4.22 Tenacity of untreated and plasma-treated polyester fibers

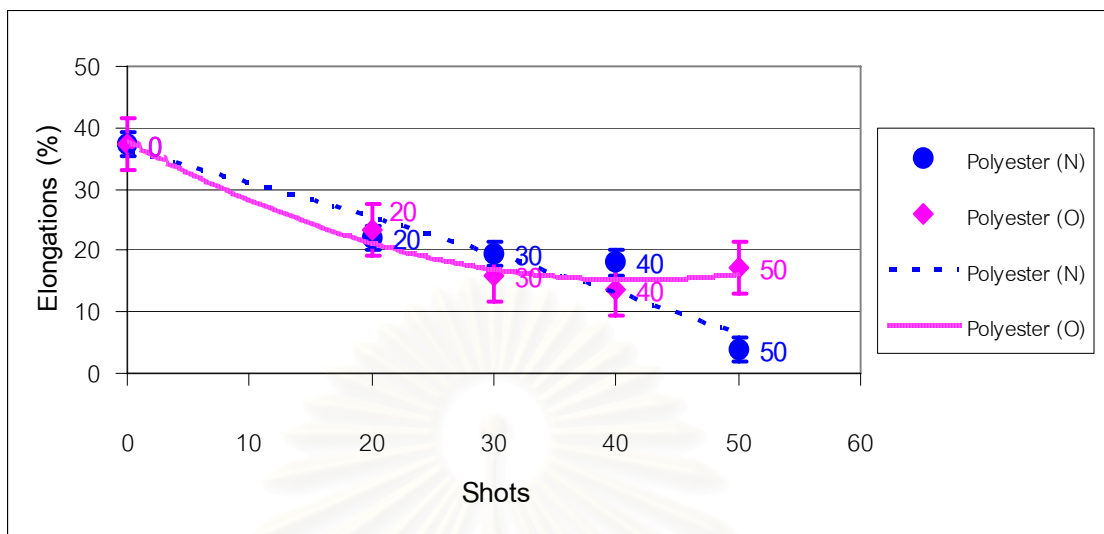


Figure 4.23 Elongation of untreated and plasma-treated polyester fibers

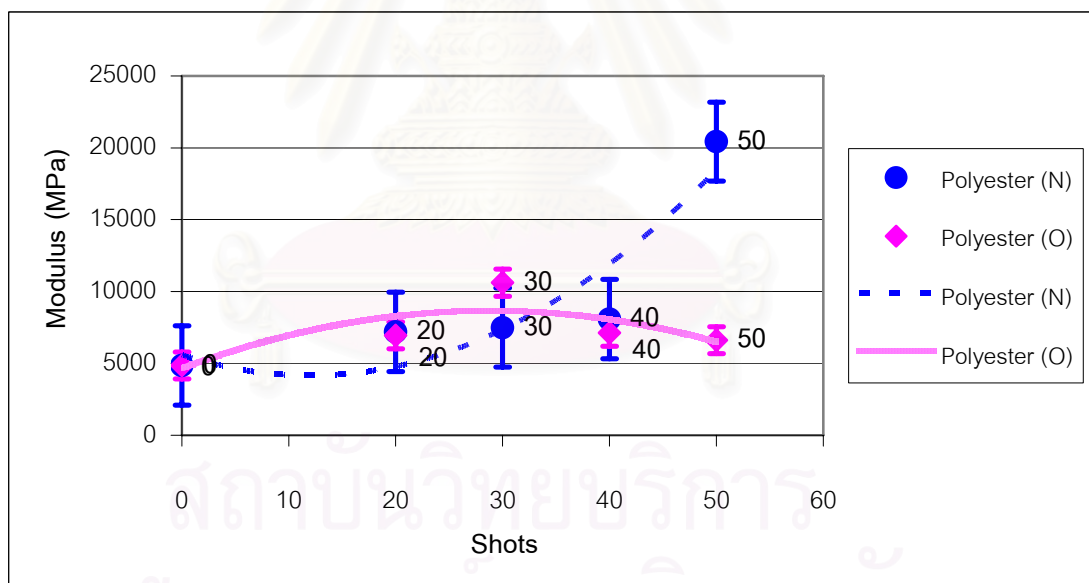


Figure 4.24 Modulus of untreated and plasma-treated polyester fibers

These properties are in the same trend as was observed for polypropylene fibers.

4.3.7 Burning Characteristics

The results of burning characteristics of untreated and plasma-treated polyester fibers are given in Table 4.4. It shows that their characteristics are similar to indicating that surface modification by plasma does not affect burning characteristics of polyester fiber.

Table 4.4 Burning Characteristics of Polyester Fiber

Fiber	Approaching Flame	In Flame	Removed from Flame	Residue
Untreated	Melts and shrinks away from flame	Burns slowly and continues to melt	Self-extinguishing	Hard, tough, black bead
Nitrogen plasma treated	Melts and shrinks away from flame	Burns slowly and continues to melt	Self-extinguishing	Hard, tough, black bead
Oxygen plasma treated	Melts and shrinks away from flame	Burns slowly and continues to melt	Self-extinguishing	Hard, tough, black bead

4.4 Morphology and Properties of Rayon Fiber

4.4.1 Cross-section

The effects of oxygen plasma treatment on the cross-sections of rayon fibers are in good agreement with those of polyester fibers. However, an increase in cross-sections of nitrogen plasma-treated fibers was observed. The results are shown in Figures 4.25 and 4.26 and Table 4.5.

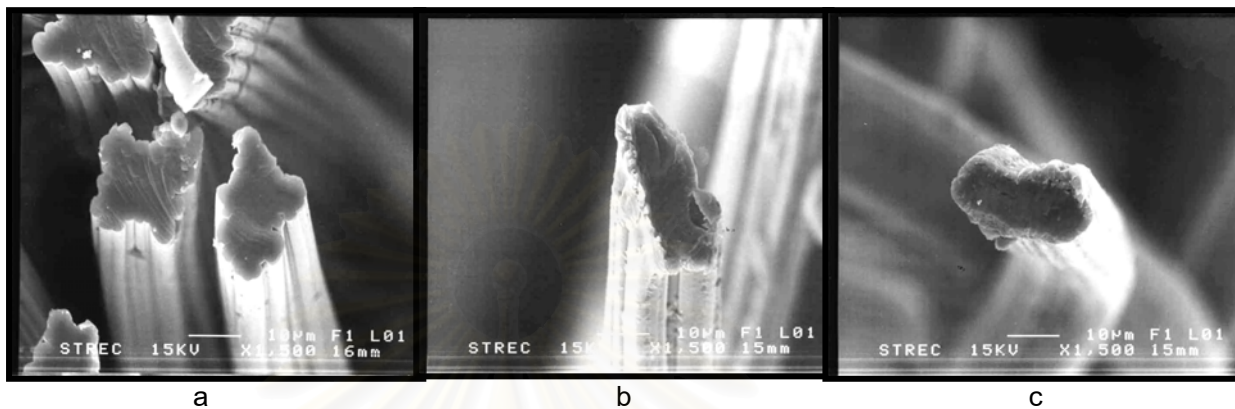


Figure 4.25 SEM photographs of cross-sections of untreated rayon fiber (a) and rayon fibers treated with 10 (b) and 40 (c) shots of nitrogen plasma

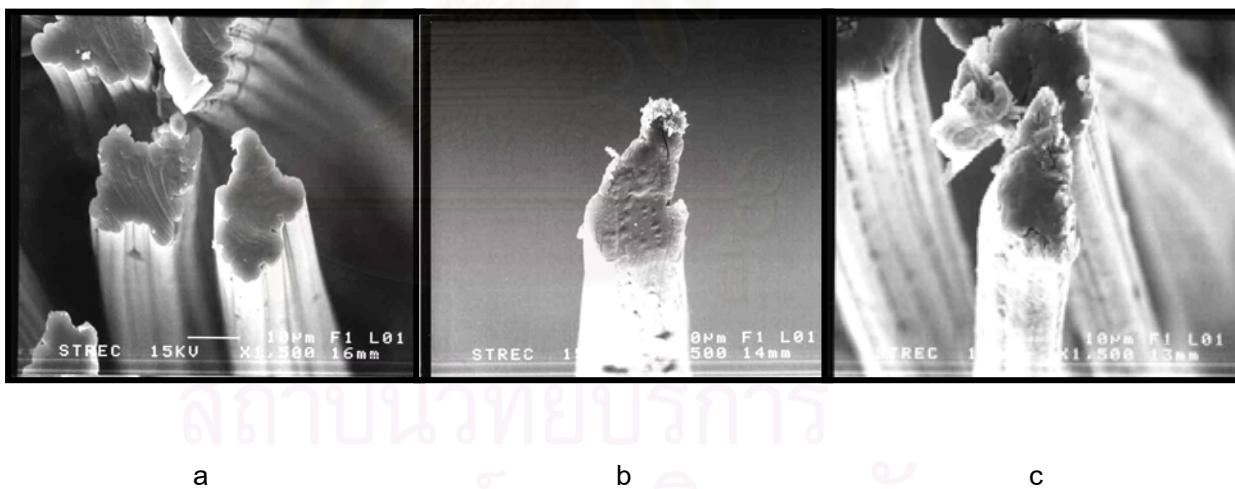


Figure 4.26 SEM photographs of cross-sections of untreated rayon fiber (a) and rayon fibers treated with 10 (b) and 40 (c) shots of oxygen plasma

Table 4.5 Diameters of untreated and plasma-treated rayon fibers

Type of rayon Fiber	Diameter (μm)	
	Nitrogen	Oxygen
Untreated	20.94 ± 2.48	20.94 ± 0.15
Treated with 10 plasma shots	25.58 ± 2.48	20.87 ± 0.15
Treated with 40 plasma shots	24.78 ± 2.48	20.65 ± 0.15

4.4.2 Morphology

The effects of plasma treatment on the morphology of rayon fibers are similar to those of polypropylene and polyester fibers. The results are shown in Figures 4.27 and 4.28.

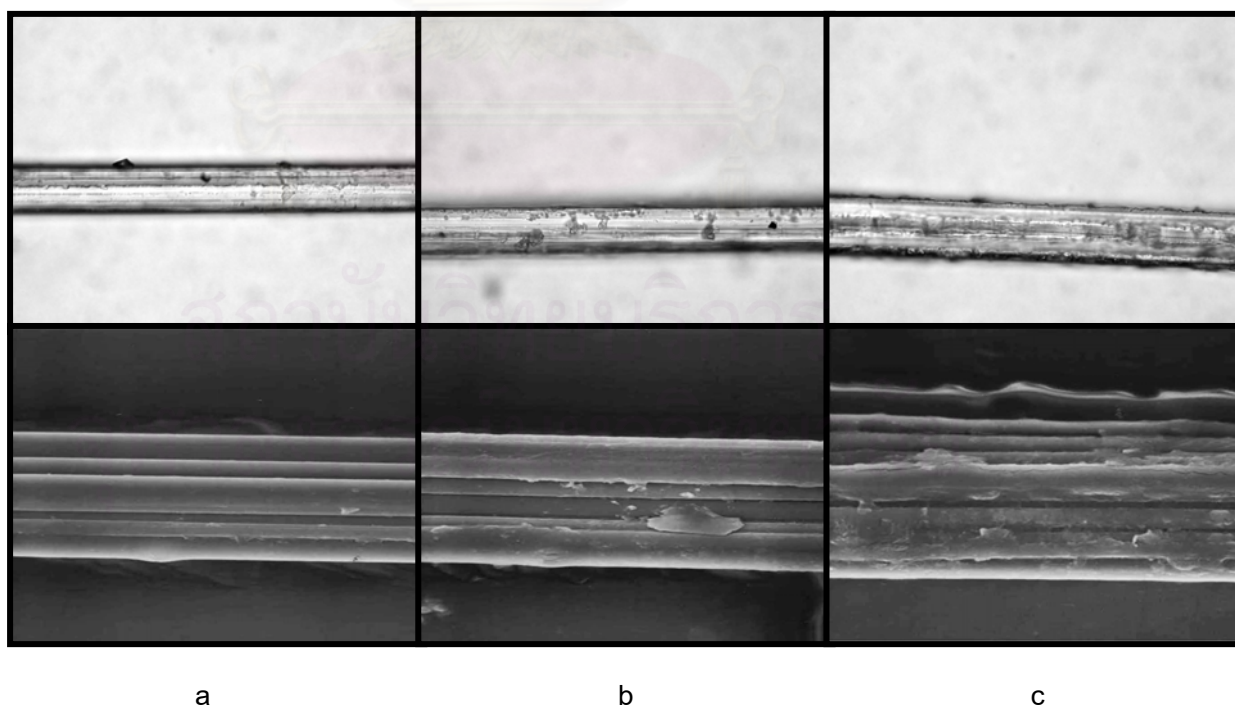


Figure 4.27 Optical (top) and SEM photographs (bottom) of untreated rayon fiber (a) and rayon fibers treated with 10 (b) and 40 (c) shots of nitrogen plasma

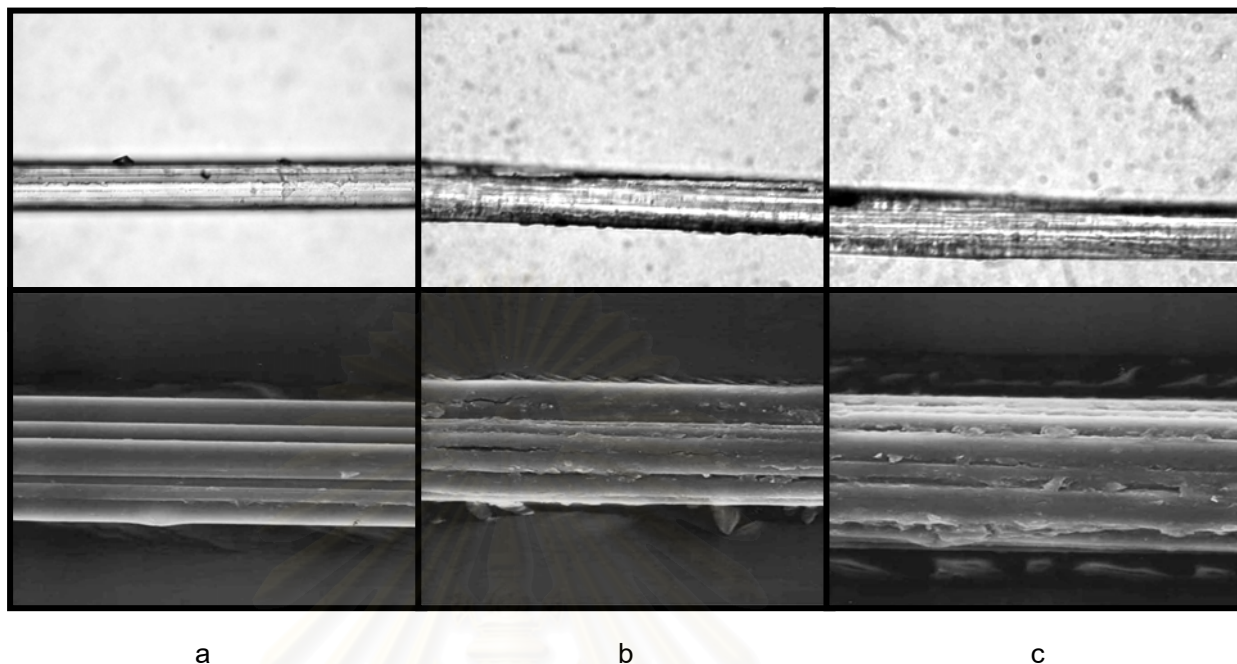


Figure 4.28 Optical (top) and SEM photographs (bottom) of untreated rayon fiber (a) and rayon fibers treated with 10 (b) and 40 (c) shots of oxygen plasma

4.4.3 Linear Density

The effects of plasma treatment on linear density of rayon fibers are in contrast to those of polypropylene and polyester fibers. It can be seen that in this case, linear density of plasma-treated rayon fibers especially nitrogen-plasma treated ones increases as the number of plasma shots increases as shown in Figure 4.29. It is expected that plasma etching as was found in plasma treatment of polypropylene and polyester fibers should decrease the weight of treated fibers. However, for rayon, even SEM photographs as previously shown exhibited the change of fiber surface, linear density of treated fiber is higher than that of the untreated one. This may be because treated rayon fibers can absorb higher amount of moisture than untreated fiber possibly due to some stronger hydrophilic functional groups, which presented on the surface of the fiber. These functional groups are different from those observed on polypropylene

and polyester fibers. This moisture absorption results in increasing weight of the fiber during the measurement.

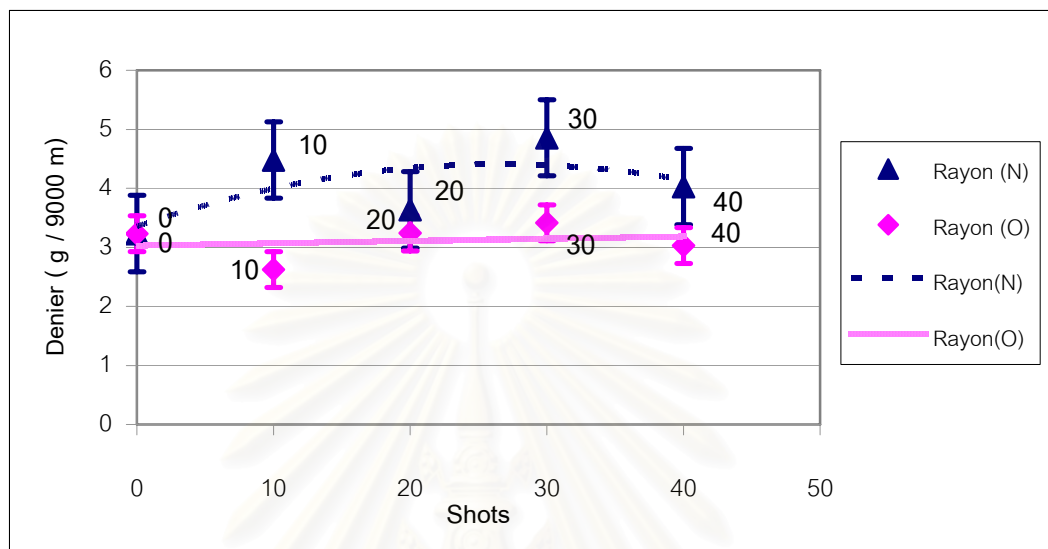


Figure 4.29 Linear densities of untreated and plasma-treated rayon fibers

4.4.4 Chemical Structure

From FT-IR spectra of nitrogen and oxygen plasma treated fibers as shown in Figures 4.30 and 4.31, respectively, it can be seen that one functional group formed on nitrogen plasma treated fiber is different from other functional groups formed on oxygen plasma treated fiber. This group is responsible for higher moisture absorption. From FT-IR spectra, it can be concluded that this group is amide group (HN-C=O).

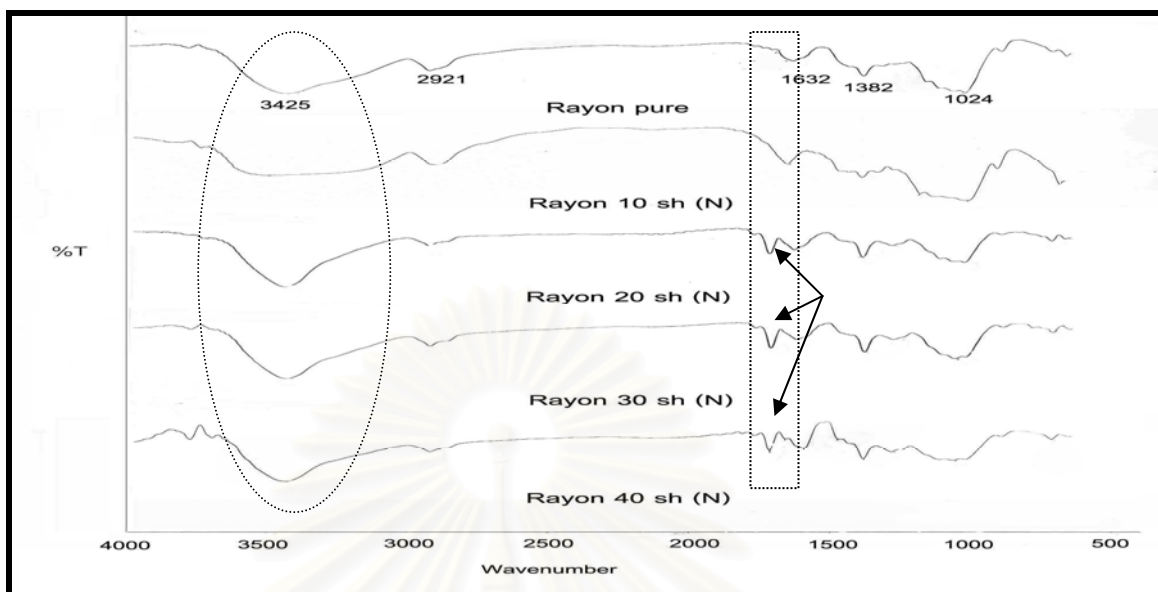


Figure 4.30 FT-IR spectra of untreated and nitrogen plasma-treated rayon fibers

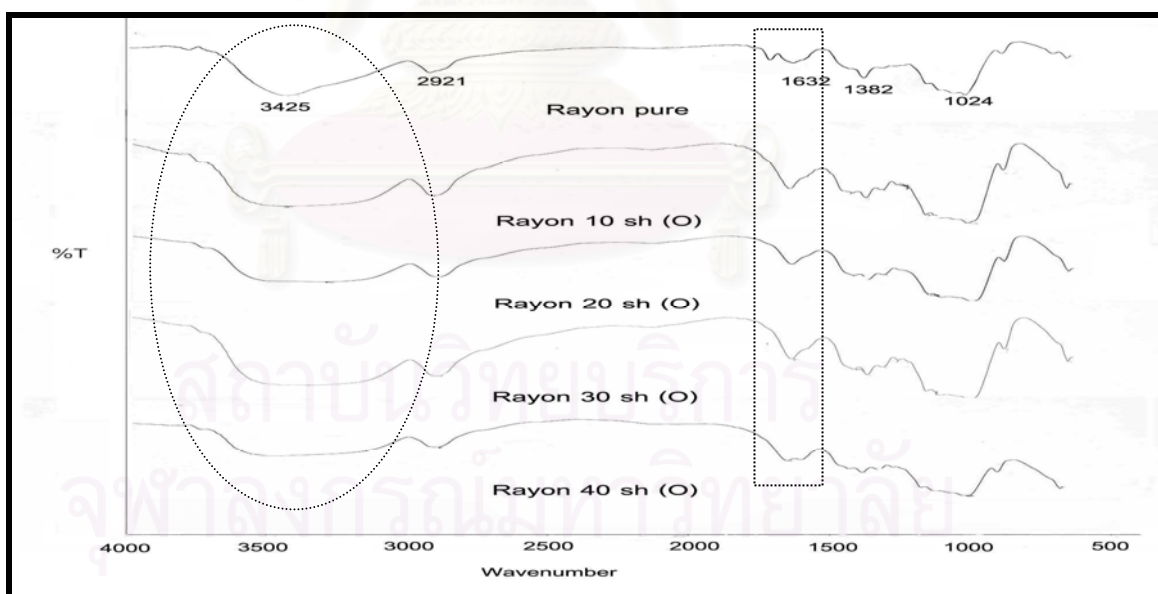


Figure 4.31 FT-IR spectra of untreated and oxygen plasma-treated rayon fibers

4.4.5 Moisture Content

The effects of plasma treatment on moisture content of rayon fibers are in good agreement with those of polypropylene and polyester fibers. The results are shown in Figure 4.32.

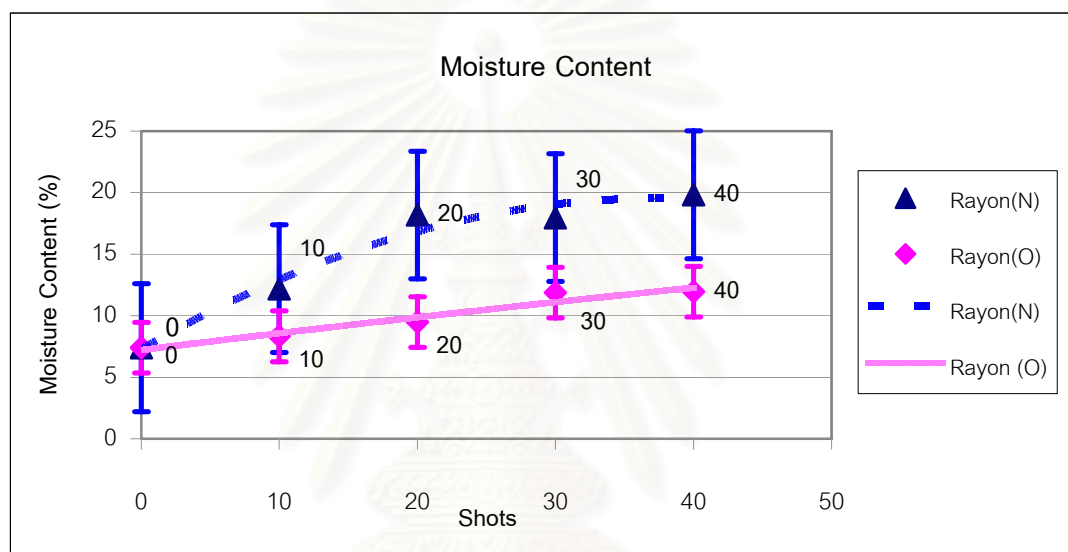


Figure 4.32 Moisture contents of untreated and plasma-treated rayon fibers

4.4.6 Tensile Properties

The effects of plasma treatment on moisture content of rayon fibers are in good agreement with those of polypropylene and polyester fibers. The results are shown in Figures 4.33-4.35.

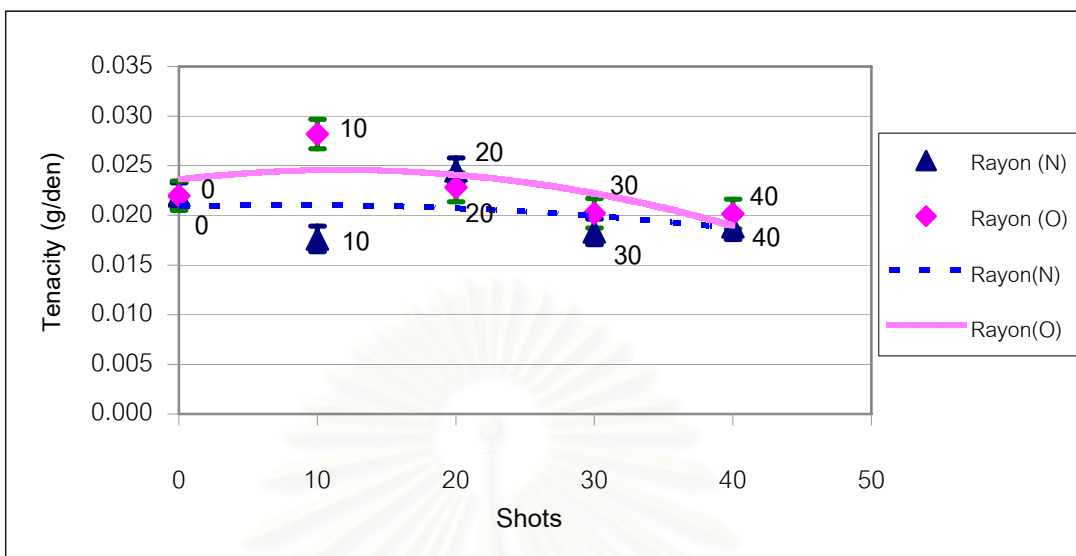


Figure 4.33 Tenacity of untreated and plasma-treated rayon fibers

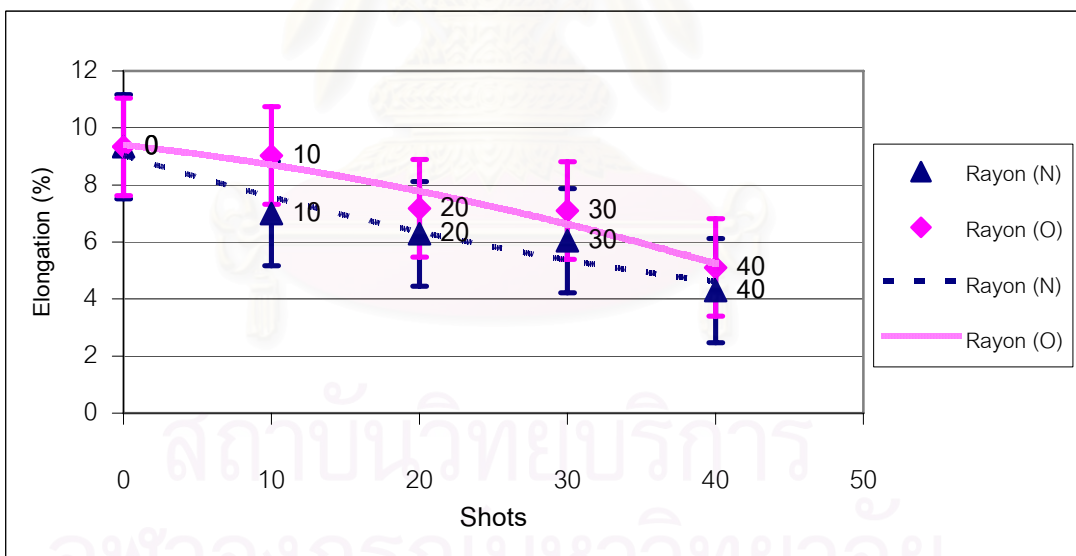


Figure 4.34 Elongation of untreated and plasma-treated rayon fibers

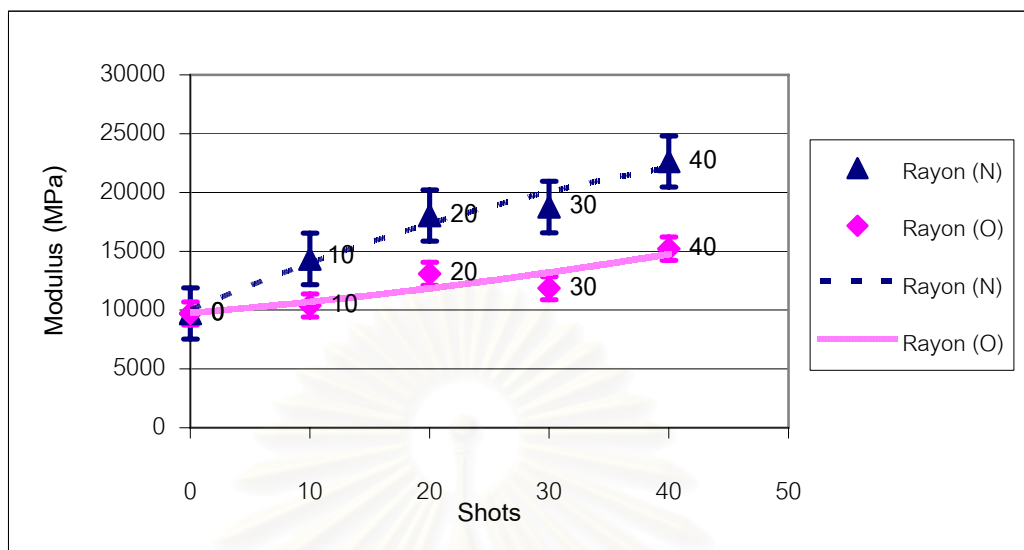


Figure 4.35 Modulus of untreated and plasma-treated rayon fibers

4.4.7 Burning Characteristics

The results of burning characteristics of untreated and plasma-treated rayon fibers are given in Table 4.6. It shows that their characteristics are similar indicating that surface modification by plasma does not affect burning characteristics of rayon fiber.

Table 4.6 Burning Characteristics of Rayon Fiber

Fiber	Approaching Flame	In Flame	Removed from Flame	Residue
Untreated	Does not shrink away	Burns quickly	Continues burning	None residue
Nitrogen plasma treated	Does not shrink away	Burns quickly	Continues burning	None residue
Oxygen plasma treated	Does not shrink away	Burns quickly	Continues burning	None residue

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

From this research, the surface of man-made fibers is modified by using a theta-pinch device. The morphology and properties of treated man-made fibers were affected by several parameters including the type of gases and the number of plasma shots. The results can be concluded as follows:

(1) Morphology and cross section of all plasma treated fibers were affected by the number of plasma shots and the type of gases. As the number of plasma shots increased, the roughness of fiber surface increased. Due to the difference of the reactions of nitrogen plasma and the fiber and those of oxygen plasma and the fiber, the change of fiber surface were different. However, the diameters of plasma treated fibers were affected by these parameters.

(2) The formation of hydrophilic functional groups on the fiber surface was confirmed by FT-IR spectroscopy. The type of functional groups formed on each fiber depended upon the type of gases.

(3) Linear densities of plasma treated polypropylene and polyester fibers were significantly affected by the number of plasma shots but not by the type of gases. Their linear densities decreased with increasing the number of plasma shots. On the other hand, the formation of amide groups on nitrogen plasma treated rayon fibers confirmed by FT-IR spectra caused the linear density of this fiber to increase as the number of plasma shots increased.

(4) Due to the formation of hydrophilic groups on plasma treated surface, moisture content of the fiber increased as the number of plasma shots increased.

(5) Tensile properties of plasma treated fibers were significantly affected by the number of plasma shots but not by the types of gases.

(6) It was found that the burning characteristics of plasma treated fibers were not different from untreated ones.

5.2 Recommendations

As the above results indicated that surface modification of polypropylene, polyester, rayon fibers using a theta-pinch device that morphology characteristic and properties of fibers was studied, therefore, further experiments can be also be studied by using these results as reference. These experiments can possibly be changing the type of the gas such as argon and mixing gases or changing form of textile products or changing charging voltage, etc.



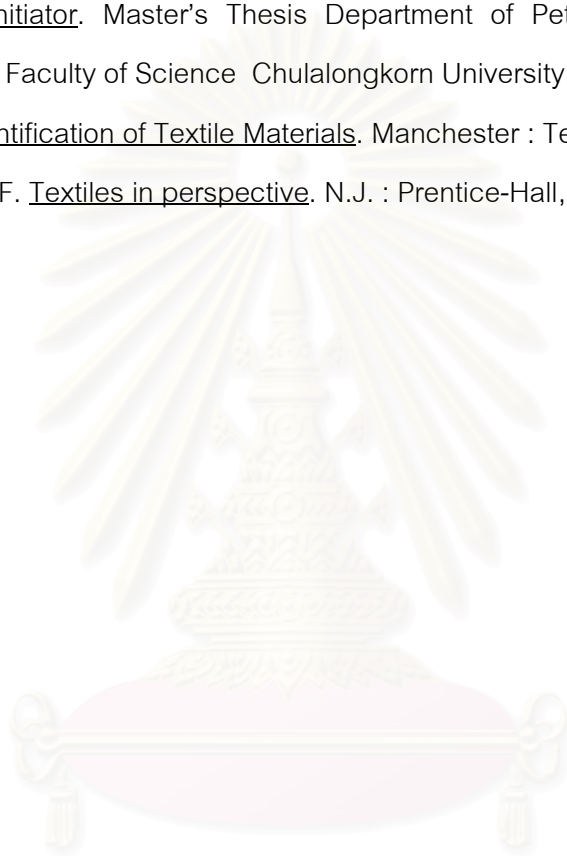
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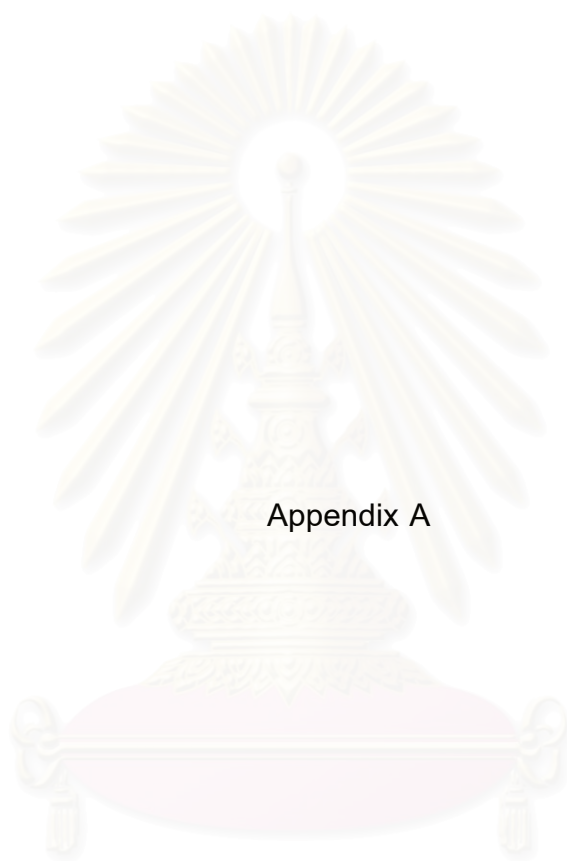


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Appendices

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Appendix A

สถาบันวิทยบริการ
จุฬาลงกรณ์มหาวิทยาลัย

Moisture Content of Untreated and Treated Man-made Fibers

Table A1. Data of Moisture Content of Untreated and Plasma-treated Polyester Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	0.4877	0.4877
20	1.1038	1.0002
30	1.1220	1.1669
40	1.4083	1.4586
50	1.8232	1.5558
S.D.	0.5203	0.4581

Table A2. Data of Moisture Content of Untreated and Plasma-treated Polypropylene Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	0.1241	0.1241
3	2.5000	1.1525
5	1.3913	1.7674
7	2.8571	2.9412
10	2.5000	2.3333
S.D.	1.1325	1.0952

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5	1.3913	1.7674
7	2.8571	2.9412
10	2.5000	2.3333
S.D.	1.1325	1.0952

Table A3. Data of Moisture Content of Untreated and Plasma-treated Rayon Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	7.4074	7.4074
10	12.1951	8.3333
20	18.1818	9.4828
30	17.9687	11.8750
40	19.8275	11.9565
S.D.	5.1887	2.0574



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Appendix B

Linear Density of Untreated and Treated Man-made Fibers

Table B1. Data of Denier of Untreated and Plasma-treated Polyester Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	4.0390	4.0390
20	3.1818	3.4709
30	3.1034	3.4041
40	3.0925	3.4099
50	3.3835	3.5342
S.D.	0.3971	0.2666

Table B2. Data of Denier of Untreated and Plasma-treated Polypropylene Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	3.2804	3.2804
3	3.0180	3.3028
5	2.4286	3.0000
7	2.2909	2.8235
10	2.1522	2.3137
S.D.	0.4892	0.4052

Table B3. Data of Denier of Untreated and Plasma-treated Rayon Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	3.2308	3.2308
10	4.4776	2.6231
20	3.6355	3.2412
30	4.8561	3.4146
40	4.0286	3.0288
S.D.	0.6473	0.3034

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Appendix C

สถาบันวิทยบริการ
จุฬาลงกรณ์มหาวิทยาลัย

Appendix C

Tensile Properties of Untreated and Treated Man-made Fibers

Table C1. Data of Tenacity of Untreated and Plasma-treated Polyester Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	0.0366	0.0366
20	0.0383	0.0366
30	0.0387	0.0397
40	0.0395	0.0238
50	0.0180	0.0252
S.D.	0.0091	0.0073

Table C2. Data of Tenacity of Untreated and Plasma-treated Polypropylene Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	0.0155	0.0155
3	0.0189	0.0194
5	0.0161	0.0160
7	0.0166	0.0142
10	0.0144	0.0130
S.D.	0.0017	0.0024

Table C3. Data of Tenacity of Untreated and Plasma-treated Rayon Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	0.0220	0.0220
10	0.0176	0.0282
20	0.0245	0.0228
30	0.0183	0.0202
40	0.0189	0.0201
S.D.	0.0029	0.0033

Table C4. Data of Elongation of Untreated and Plasma-treated Polyester Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	37.3530	37.3530
20	22.0970	23.3130
30	19.4500	15.8700
40	18.0270	13.6300
50	3.8050	17.1700
S.D.	11.9571	9.5784

Table C5. Data of Elongation of Untreated and Plasma-treated Polypropylene Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	120.3670	120.3670
3	98.7930	92.6530
5	66.8930	73.8100
7	39.8570	53.0200
10	21.6470	22.0970
S.D.	40.6846	37.4799

Table C6. Data of Elongation of Untreated and Plasma-treated Rayon Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	9.3390	9.3390
10	6.9990	9.0330
20	6.2870	7.1820
30	6.0430	7.1000
40	4.2930	5.1070
S.D.	1.8297	1.7107

Table C7. Data of Modulus of Untreated and Plasma-treated Polyester Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	4855.4350	4855.4350
20	7180.5700	6944.5240
30	7491.0030	10609.1160
40	8084.3200	7129.1270
50	20425.7900	6602.6010
S.D.	6170.6391	2094.1746

Table C8. Data of Modulus of Untreated and Plasma-treated Polypropylene Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	511.1050	511.1050
3	632.6430	838.7180
5	1016.5440	833.4910
7	1381.0320	793.9770
10	1943.9650	2491.0240
S.D.	583.8230	792.8571

Table C9. Data of Modulus of Untreated and Plasma-treated Rayon Fibers

No of plasma shots	Nitrogen gas	Oxygen gas
0	9710.1660	9710.1660
10	14344.6370	10395.5870
20	18029.6920	13077.2800
30	18761.0330	11857.1900
40	22631.4150	15217.3630
S.D.	4888.8401	2198.6679



สถาบันวิทยบริการ
จุฬาลงกรณ์มหาวิทยาลัย

VITA

Miss Supitchaya Chuenchon was born in Bangkok, Thailand, on August 6, 1982. She received a Bachelor of Science degree with a major in Polymer Science and Textile Technology from Chulalongkorn University in 2003. She started as a graduate student in Department of Materials Science with a major in Applied Polymer Science and Textile Technology, Chulalongkorn University in June 2003, and completed the programmed in March 2005



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