ABSTRACT

MALSHE, PRIYADARSHINI PRAKASH. Enhancing Electrostatic Properties and Hydroentangling Efficiency via Atmospheric Plasma Treatment. (Under the direction of Marian G. McCord and Mohamed A. Bourham.)

Hydroentangling is the fastest growing nonwoven bonding technology. Known for the production of most textile-like nonwoven fabric, hydroentangling is a mechanical bonding technique which involves impingement of high velocity water jets onto a nonwoven fiber web. The mechanical action of needle-like water jets entangles fibers and consolidates the web into a fabric. The final properties of a hydroentangled web are reported to depend on the textile material and its intrinsic properties such as strength, modulus, bending rigidity and the fiber surface properties such as friction, fiber shape etc. Hydroentangling efficiency is also shown to depend on fiber to water interaction by way of hydraulic drag force.

In previous works by other research groups, water pooling problem has been reported when hydroentangling hydrophobic fibers such as polypropylene. The focus of this work is to eliminate the problem via atmospheric plasma treatment prior to hydroentangling. The purpose of this study is to determine the effects of atmospheric plasma pre-treatment on nonwoven webs due to plasma induced hydrophilicity and other surface modifications such as roughness/smoothness. Different fiber substrates were treated with atmospheric plasma in a continuous run and hydroentangled at different times post-plasma treatment to determine the effect of aging on hydroentangling efficiency.

Enhancing Electrostatic Properties and Hydroentangling Efficiency via Atmospheric Plasma Treatment

by
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BIOGRAPHY

Priyadarshini Malshe was born on October 31, 1984 in India to Leena and Prakash Malshe. She earned her Bachelor's degree in fiber and textile processing technology from Institute of Chemical Technology, Mumbai, India in May, 2006.

She went on to work with BASF India Ltd. in their product development division for performance chemicals for textiles for a year.

Following her academic inclination, she joined NC State University for a Master of Science degree in Textile Chemistry in August 2007 under the guidance of Dr. Marian G. McCord and Dr. Mohamed Bourham. Her work focused on incorporation of continuous plasma treatment prior to hydroentangling to improve the ultimate process efficiency.

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Chapter 1

Introduction

Surface properties such as roughness or smoothness influence fibers' bending rigidity, fiber-to-fiber friction and their ability to entangle. In a previous study by Zheng et al, they studied the effects of different fiber properties on the mechanical properties of hydroentangled webs. In their work, they proposed that machine direction (MD) fabric tensile strength is determined by fiber flexural rigidity for fine mesh, friction with coarse mesh, or a combination of both flexural rigidity and friction for intermediate screens. They also indicated that cross direction (CD) tensile strength is generally governed by the friction mechanism, and that fabric tensile strength potential is a function of fiber modulus and friction [1]. In Zheng's work, the relationship between fiber properties and web properties was inferred by comparison of the web properties using different fiber types, and correlation of the mechanical properties of each fiber type with the measured web mechanical properties. However, little has been studied about the effects of surface chemistry on physical or mechanical properties of hydroentangled webs. Though it may be well imagined that surface modification of fibers may possibly influence the interaction of water jets with the nonwoven fibers during the hydroentangling process owing to change in substrate surface properties [2], to the author's knowledge no systematic study has been presented in the literature which quantifies this effect.

The present work is an attempt to quantify the effect of hydrophilicity on the ultimate properties of hydroentangled webs. The samples used in this study were obtained by pre-treating with atmospheric plasma and aging prior to hydroentangling process, resulting in a set of samples with different fiber surface hydrophilicities, roughnesses, and tensile properties among otherwise identical webs.

It is well established that plasma treatment affects surface properties of the substrate altering the wetting behavior, making the substrate much more hydrophilic by way of introduction of functional groups [3-7]; it also physically modifies the surface of the substrate, altering it by way of etching and ablation [8, 9] which may vary fiber to fiber friction or total surface area [10], as well as surface chain scission and cross-linking. In addition to this, plasma treatment can also change the substrate's electrostatic properties by making dipoles to polarize in the direction of the applied electric field. These alterations charge up the substrate and may also cause an increase in the charge holding capacity of the substrate [11], improving the electrostatic filtration efficiency. This study therefore, is an attempt to characterize the effects of plasma pretreatment on hydroentangling efficiency in terms of fabric mechanical and electrostatic properties. Characterization data for the untreated and plasma treated webs is analyzed to establish a trend between the plasma treatment and the substrate's response to it.

The specific objectives of this study are the enhancement of filtration efficiency, improvement of the overall hydroentangling efficiency, and cost reduction of processing by

reducing the amount of energy required to generate sufficient strength and electrostatic properties via atmospheric plasma treatment.

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Chapter 2

Literature Review

Nonwovens are highly engineered fabrics, made from webs of loose fibers by appropriate bonding technique [1]. Nonwovens are different from their woven and knitted counterparts and can be made to possess a variety of properties by varying any of the processes ranging from web formation to bonding. The nonwoven manufacturing technology encompasses incorporation of particles, films and making composites with wovens and knits [2, 3]. Nonwoven fabrics can be engineered to have highly specialized properties to serve in several fields such as medical, hygiene, geotextiles, automotives, horticulture, industrial applications etc. The main steps included in nonwoven manufacturing are web formation followed by web bonding and final finishing treatments required for specific end uses. Web formation may be carried out via air laying, wet laying, carding-crosslapping meltblowing, spunbonding etc. Web bonding involves various techniques which can primarily be divided into the three main techniques as follows:

2.1 Thermal Bonding [4, 5]

Thermal bonding is carried out by using heated rolls through which nonwoven fiber webs are passed. The temperature of the metal rolls is adjusted according to the polymer type of the fibers. The bonding carried out by the heated rollers may be area or point bonding. In

area bonding a heated roller distributes heat uniformly all over the surface, whereas in point bonding, the rollers are engraved and thus, heat is applied only in specific locations.

The shapes and sizes of bond points are adjusted according to the end use of the product.

2.2 Chemical bonding [6]

Chemical bonding technique uses an adhesive to provide structural integrity to nonwoven webs. The commonly used adhesives are chemical binders which are most generally water-borne latexes. Most latex binders are made from vinyl materials like polyvinyl acetate, polyvinylchloride, styrene/butadiene resin, butadiene or their combinations. Latexes are used as nonwoven binders because they are economical, easily applied and effective. Chemical composition of a binder is most important factor as it determines stiffness/softness, water affinity, elasticity, durability and ageing. It also determines solvent resistance, cross-linking nature and adhesive characteristics. Surfactants are used to stabilize the latex suspensions. The type and quantity of surfactant used influences the polymerization process, polymer stability and the application method.

2.3 Mechanical bonding

Mechanical bonding is carried out by application of pure mechanical energy and there is no addition of any external bonding agents. The two mechanical bonding techniques are:

2.3.1 Needle Punching [5, 6]: Needle punching involves a quick movement of barbed needles through a fiber web, pushing the fibers in z-dimension, which finally results in entanglement. Needle punching has a limitation on its speed because of friction between needles and fibers.

Increased friction can heat up the needles significantly, which may result in local melting of low melting fibers. It is also limited in processing very high basis weight webs due to high lofts and increased friction.

2.3.2 Hydroentangling [7]: Hydroentangling is the fastest growing web bonding technique because of the most textile-like hand it gives to the resulting nonwoven fabric. Hydroentangling involves impingement of very high velocity of water jets onto a nonwoven fiberweb, which push fibers down the web in z-dimension resulting in an inter-locking between the fibers. Depending on the hydroentangling energy, the jet pressures and several other parameters, the extent of fiber inter-locking varies and gives the web its final integrity and strength. Most often fabrics undergoing hydroentangling are passed through the process for two or more times. For every other pass, the fabric is reversed and the water jets are impinged on the other face of the fabric [9]. This is done to give both the faces of the nonwoven fabric an equal distribution of energy. Hydroentangling is very similar to needle punching in the sense that both techniques mechanically push fibers in z-dimension. In needle-punching, barbed needles also pull the fibers back whereas in hydroentangling, the same action is realized when the process involves multiple passes and reversing of the fabric.

Hydroentangling is much faster compared to needle-punching as it has no problems of heat build-up due to friction.

2.4 Hydroentangling

Hydroentangling or spunlacing is a relatively newer technique of web bonding and it first came into existence with Chicopee introducing the technology in 50's. However, the commercial entry of hydroentangling was marked with DuPont introducing a spunlacing line in early 70's [8]. It is the fastest growing mechanical bonding process for nonwovens, which involves impinging of high velocity water jets onto nonwoven fiber-webs. The high velocity water jets lead to entanglement of fibers around each other, thus providing a consolidated structure to the web. Hydroentangling has received growing interest in recent years and numerous studies have been carried out to understand the mechanism of the process. Known to impart most textile-like properties to the nonwoven, hydroentangling process gives the fabric several important characteristics such as high drapability, low linting, high absorbency, conformability and comfort, and high bulk [9]. Figure 2.1 illustrates a schematic of a hydroentangling device.

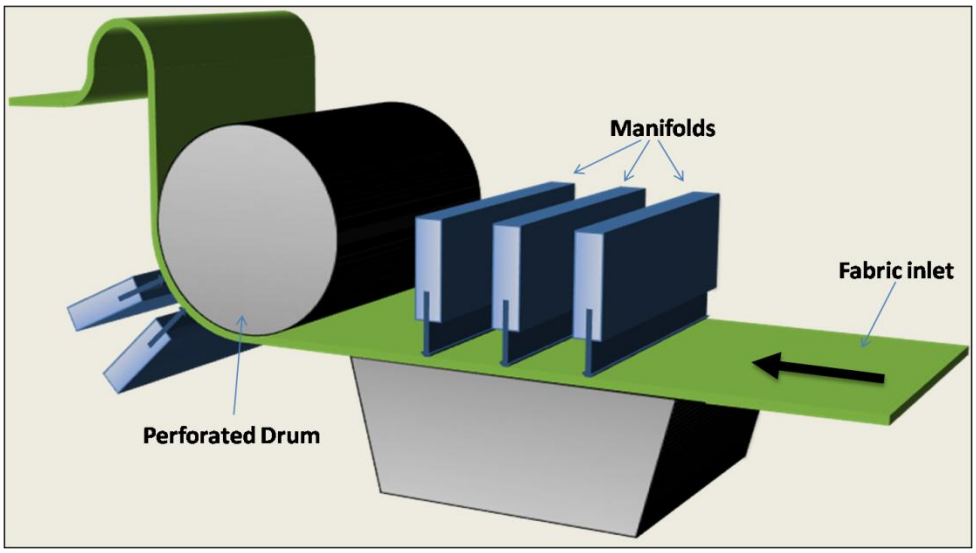

Figure 2.1: Hydroentangling device schematic

The efficiency of the Hydroentangling process depends upon the way in which fibers interact with high pressure water jets. The effectiveness of the process is influenced by fiber properties such as modulus, staple length, friction etc. The ultimate properties of hydroentangled fabrics depend on many hydroentangling parameters. Various studies have been performed to characterize the hydroentangling parameters which may affect the energy consumption and ultimate properties of the hydroentangled fabric [10, 11]. Some of these variables have been identified to be:

a) Water-jet pressure: The water jet velocity or jet pressure is an important parameter as it is directly responsible for entanglement of fibers [12]. In a typical hydroentangling line, there are a number of manifolds each having a number of orifices generating an equal number of

water jets. The water pressures at each manifold are regulated in a way that the loose fibers do not get washed away due to excessive pressure and yet entangle properly to achieve a consolidated structure.

b) Specific energy supplied to the web: Specific energy supplied to the fiber web determines the ultimate tensile properties of the hydroentangled web. As the specific energy increases, tensile strength of the web also increases. The relation between the two reaches a plateau after a threshold specific energy when tensile properties do not change anymore. It is recommended to supply specific energy to attain that maximum tensile strength of the web [13]. In most hydroentangling processes, threshold specific energies are often reached by subjecting the web to multiple passes through the line.

c) The carrier mesh: The geometry of forming wire is an important factor which influences the ultimate web properties. Forming wire creates an impression on the web because the water jets work their way out through the holes in the mesh. The fibers within the structure of a nonwoven web tend to get pushed towards the points of wires' intersection in the mesh as the water jets flow through the holes [14]. The finer this mesh the more consolidated is the structure of the web. However, too fine a mesh may restrict water flow through the web and result in standing water on the web surface.

d) Peeling forces: Peeling forces come into play when nonwoven web is peeled off the carrier mesh. Due to high pressure water jets, the nonwoven web is pressed hard against the

forming wires. The peeling forces result in partial alignment of fibers in MD thus resulting in restructuring of the nonwoven web [9, 16].

e) Jet orifices: Various studies have been performed to establish the best orifice geometry in achieving maximum jet energy [15]. Cone down geometry is concluded to be the most desirable orifice geometry which gives a longer jet at same pressure compared to cone-up geometry thus delivering maximum power [16].

Figure 2.2 shows a photograph of the Nonwoven Cooperative Research Center (NCRC) hydroentangling device which was used in the present work.

Figure 2.2: Photograph of the NCRC Hydroentangling device

Studies have been carried out and various potential losses of the energy supplied to fiber web during hydroentangling are reported in the literature. These potential losses may include:

1) compression of web and deformation of the fibers: A part of the energy applied to nonwoven web through water jets is lost to compress the web in z-dimension. Also, thermoplastic fibers may undergo deformation due to high velocity jets [11].

2) frictional forces due to fiber reorientation: The resisting frictional forces posed by the fibers entangling around each other need to be overcome to entangle the fibers effectively [17].

3) absorption of energy by water pools in the web: A substantial loss of energy to standing water or water pools formed on hydrophobic fiberwebs may be observed.

It is important that the stagnant water in the form of pools or film is removed from the fiberweb surface so that the water jet strikes the fibers and not water film/pool for the process to be efficient [18, 19].

4) fluid drag resistance of fibers and capillary resistance of pores in hydrophobic fibers [17].

It is observed that some of the potential losses of energy in the hydroentangling step may be associated with hydrophobicity of the associated fibers. However, to the author's best knowledge, there is no publication in the open literature which mentions hydrophilicity of the

nonwoven fibers as a parameter which could affect the process efficiency. One of the main objectives of this project is to minimize the water pooling effect and drag and capillary resistance due to hydrophobic fibers by rendering them hydrophilic by way of atmospheric plasma treatment prior to hydroentangling.

2.5 Atmospheric plasma

Plasma, also known as the fourth state of matter, can be defined as gaseous states of matter that consist of a dynamic mix of ions, electrons, free radicals, meta-stable excited species, molecular and polymeric fragments, and large amounts of visible, UV and IR radiations [20-22]. Industrial plasmas have found lots of applications in areas like manufacturing computer chips and semiconductors using plasma-aided techniques, machine tools, and medical implants.

There is a broad scope of plasma applications. Plasma, because of its ability to impart functionality to a surface can be used to modify the surface in any desired way to make it hydrophilic, hydrophobic, charged, etched, implanted or multi-functional [23, 24]. These properties, when induced, can make the surface behave differently in different setups of surrounding conditions.

The definition of plasma is a very refined one; there are three requirements which a system should fulfill to be classified as ‘plasma’, these are the quasi-neutrality, the collective behavior and the motion of the plasma charged particles controlled by electromagnetic forces.

Quasi-neutrality is when an equal number of electrons and ions coexist, which takes place when the gas dissociates and ionizes to give rise to ions and electrons and hence the number of these species is equal. The implications of this property can be understood by the concept of Debye shielding in which a charged particle can be surrounded by a cluster of opposite charges, and hence the particle is shielded. For example, if a positive charge is placed within plasma it is immediately surrounded by electrons and these electrons form a shield around the positive charge at a certain distance, known as the Debye Length and the bulk plasma can no longer feel the presence of the positive charge. For Debye shielding to occur, it is required that the length of plasma is greater than the length of the Debye shield. The Debye length is determined by the kinetic temperature and the number density $\lambda_D = \sqrt{\frac{\varepsilon_o k T_e}{n e^2}}$; where ε_o is the permittivity of free space, k is Boltzmann's constant, T_e is the plasma electron kinetic temperature, e is the unit charge and n is the plasma quasi-neutral number density [23].

The Debye length is very small in very high temperature plasmas such as fusion plasmas, but it can be quite large for laboratory plasmas.

By collective behavior, it is meant that the plasma particles are not only influenced by their immediate surroundings, but also by the regions which can be significantly distant [25]. In plasma, if a charge is displaced from its neutral position, it generates an electric field which influences the neighboring particles generating a wave that hits the whole body of

plasma. Thus, each particle feels attraction or repulsion resulting in a lot of motion within the plasma. These moving particles can generate currents and magnetic fields.

Lastly, plasma must have sufficient degree of ionization. The acceptable amount of ionization that allows for classification of an ionized gas mix as plasma is a combination of two factors, the collective behavior and the collision times between ionized particles and neutral gas. If ω is the frequency of plasma oscillations and τ is the time between ion/neutral collisions, then the value of $\omega\tau$ must be greater than or equal to 1 to classify an ionized gas as plasma [25-27]. The plasma frequency ω is a sole function of the plasma number density $\omega = \sqrt{\frac{ne^2}{m_e \varepsilon_o}}$, where m_e is the mass of the electron and all other parameters are same as previously defined.

Plasma can be created by applying energy to a gas sufficient enough to induce ionization. This energy organizes the electronic structure of the species (atoms and molecules) and produces excited species and ions. This energy may be thermal, or may be carried by electric current or via launched electromagnetic radiations into the gas. Depending on the type and amount of energy supplied to the gas, the plasma generated acquires different properties in terms of electron number density and kinetic temperatures of both electrons and ions. Plasmas are generated using electrical energy input to the working gas, where the electric field breaks down the gas and induces ionization followed by electrons' impact on the surrounding neutral species in various forms of reactions determined by the reaction rate

coefficient of each process. These collisions can either be elastic or inelastic or a combination of both. In elastic collisions the internal energy of the neutral species does not change and the colliding species maintain their individual identities.

Inelastic collisions are more complex and the electronic structure of the neutral species gets modified. This means that excited species such as ions and free radicals are generated from the interaction between colliding species and the individual identities of the species can be altered due to dissociation and recombination processes [27]. The excited species have a very short life and de-excite to the ground state by emitting photons, which appears as optical emission spectra in the visible, ultraviolet and infrared spectra. Some species in meta-stable states may have longer lifetime and thus are maintained active in the plasma [28].

Atmospheric plasmas are highly collisional and are characterized by low kinetic temperatures and relatively low number densities. Atmospheric plasma treatment of textiles, and other substrates has been shown to be an economical and effective method for improving the physical and mechanical properties of fibers and textiles, including tensile strength, modulus, bending rigidity, friction and wettability. These changes can be different in terms of the functionality introduced and the ultimate effects depending on the selection of plasma processing parameters [20-24, 26, 27].

2.6 Influence of Plasma on Substrate Properties

Plasma has the capability to introduce functional groups on the substrate surface. These functional groups can modify the way the substrate behaves or responds to certain chemical environments. Plasma treatment can render a hydrophobic surface hydrophilic. It can improve electrical conductivity of the substrate by way of introduction of polar functional groups and also by means of polarization of dipoles [26-27, 29-31].

2.6.1 Etching and Re-deposition

It is known that plasma treatment may result in a reduction in weight of the substrate due to physical etching effects. Surface etching by reactive species may break molecular chains in the substrate. The surface particles acquire kinetic energies enough to get physically knocked off and mix in the plasma system. After these particles lose their energies, they re-deposit back onto the surface. Both etching and re-deposition affect surface roughness. In some cases when the substrate is already rough, plasma can result in reduction of the roughness or in some cases, surface cleaning [32, 33].

2.6.2 Chain Scission and Functionalization

Ion bombardment onto a substrate surface during plasma exposure causes chain scission of molecules on the surface, resulting in the formation of ions and free radicals. The radicals are highly reactive and can interact with reactive species in the plasma to generate new functional groups. Depending upon the working gas being used for the plasma

generation, it is possible to introduce different functional groups onto the substrate surface ranging from –OH to >C=O to –COOH. For example, oxygen plasma yields oxygen containing functional groups such as hydroxyl, carbonyl, and carboxyl whereas fluorine plasma can generate fluoride ions, fluoro-methyl and oxy-fluoro groups [34, 35].

2.6.3 Cross-linking

The process of plasma induced cross-linking refers to a physical or chemical reaction that breaks chemical bonds on the surface of the material. This is done by the highly reactive and energetic species in the plasma. The resulting radicals in the polymer chain can potentially react with adjoining chains forming new links [36]. A fully grown cross-linked surface can improve the surface's barrier and biocompatibility characteristics. Some surfaces can be effectively sealed with this process.

Sealing reduces the potential for gases or liquids to penetrate into the surface and/or it also prevents internal mobile species from migrating to the surface or blooming [37]. The various processes of plasma-substrate interaction are illustrated in Figure 2.3 in which plasma effects on the substrate may induce etching, cross-linking, chain scission, generation of functional groups and surface roughness and degradation.

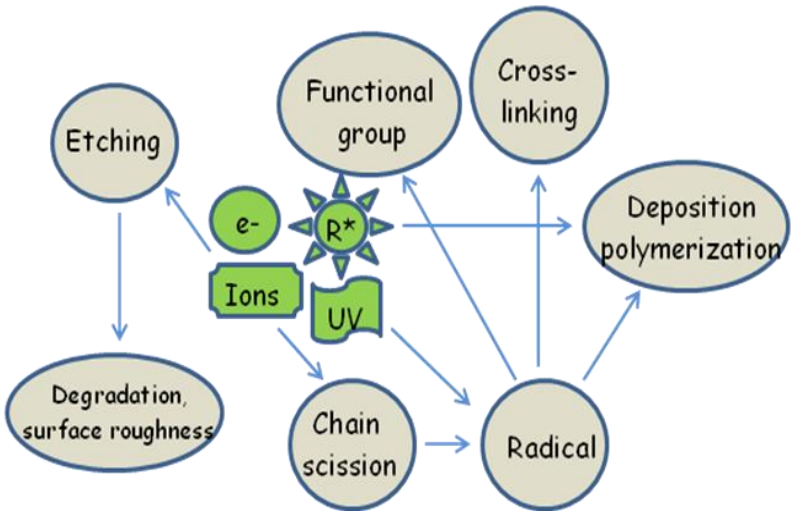

Figure 2.3: Plasma effects on substrate and various processes in plasma-substrate interaction

In essence, plasma treatment affects surface properties of the substrate altering the wetting behavior, making the substrate much more hydrophilic by way of introduction of functional groups; it also physically modifies the surface of the substrate, and alters it by way of etching and ablation which may vary fiber-to-fiber friction or total surface area or surface chain scission and cross-linking. Additionally, plasma can also change the substrate's electrostatic properties by causing dipoles to polarize in the direction of the applied electric field. These alterations charge up the substrate and may also cause an increase in the charge holding capacity of the substrate, improving the electrostatic filtration efficiency. This study, therefore, is an attempt to characterize the effects of plasma pretreatment on hydroentangling efficiency in terms of fabric mechanical and electrostatic properties.

Hydroentangling creates mechanical bonds within the nonwoven web by way of high pressure water jets. When the fiber substrate is hydrophobic, it does not effectively wet the surface and sometimes, small water pools form on the web surface. The water jets coming out of the subsequent manifolds hit these small pools and lose a portion of their energy. This may cause some fabric imperfection at such sites because of non-uniform distribution of energy. Plasma treatment of such hydrophobic webs prior to hydroentangling can make them hydrophilic. According to the proposed hypothesis in this study, the improved hydrophilicity is likely to cause the webs to wet properly thus, completely eliminate the water pooling problem. The plasma treatment also etches the surface which is likely to make it rough, potentially changing the fiber-to-fiber friction. The increased fiber surface roughness is likely to pose a greater resistance to hydroentangling because of higher fiber-to-fiber friction. Introduction of functional groups may also work towards increasing web strength by way of formation of different bonds such as hydrogen bonds, dipole-dipole interactions, Van der Waal's forces etc. The ultimate response of a web to hydroentangling then becomes a resultant of many forces such as fiber-to-fiber friction, fiber-to-forming wire adhesion, fiber-to-fiber cohesion, dipole-dipole interactions, H-bonding etc.

2.7 Influence of Plasma on Electrostatic Properties

The phenomenon of plasma introducing polar functional groups onto a substrate surface and thus altering its electrostatic properties is well understood. An important

mechanism in plasma systems is ‘Electret’ formation [38, 39]. An electret is a material that retains its electric polarization after being subjected to a strong electric field. The positive charge within the material becomes permanently displaced in the direction of the field, and the negative charge becomes permanently displaced in the direction opposite to the field. One end of the electret is somewhat positive, and the other is somewhat negative, though the net charge remains zero. Electrets are prepared from certain waxes, plastics, and ceramics, the individual molecules of which are permanently polarized but are randomly arranged before being subjected to an electric field so that there is no overall polarization in the material [39-41]. The strong electric field rotates the polar molecules into an alignment that persists when the external field is removed. Sometimes electrets are made by allowing a molten material to solidify in a strong electric field. The behavior of electrets in an electric field is analogous to that of permanent magnets in a magnetic field. An electret, for example, lines up in an electric field with its positive end pointing in the direction of the field. Electrets, discovered in 1925, have found applications in many applications ranging from electrostatic microphones to filters. Electrets are typically formed by using AC and DC corona discharges [42].

In case of fibrous material that is subjected to plasma treatment, it is expected that the dipoles will be aligned due to the strong electric field of the plasma and remain in that polarization unless other processes, such as melting, dominate. The electric polarization of electrets is very important with respect to some special applications such as filtration. The

ability of such materials to hold charges is much higher than a non-electret. Such materials can prove to be better electrostatic filters in terms of performance [43-45].

2.8 Fiber Friction

Friction is a surface phenomenon. Hence, it depends on a material's morphology, its physical and chemical structure and properties [46]. Friction is defined as the force resisting lateral relative motion of solid surfaces, fluid layers or two different phases. Frictional force is divided into two categories [47]:

(a) Static friction: It is the threshold force required to get a surface into motion relative to another surface in contact. This element of frictional force arises from the inter-locking of the surface irregularities of two surfaces.

The equation for static friction is:

$$F_s = F_n \mu_s$$

Where,

F_s = the force due to static friction, F_n = the normal force, μ_s = coefficient of static friction

(b) Kinetic friction: It is the force required to keep the surface in motion once static friction has been overcome. For the same set of surfaces, static friction is generally up to 20 % greater than the kinetic friction.

The equation for kinetic friction is:

$$F_k = F_n \mu_k$$

Where,

F_k = the force due to kinetic friction, F_n = the normal force, μ_k = coefficient of kinetic friction

In hydroentangling, both the elements of friction play important roles. Literature suggests that fiber-to-fiber friction is an important parameter which affects hydroentangling efficiency [17-18]. Higher friction between the fibers results in higher energy requirements for entangling. In a nonwoven web, fibers are stationary with respect to each other. The surfaces of fibers interact and the surface irregularities get inter-locked. When this web is subjected to hydroentangling, the water jets apply force to the fibers. This force is resisted by the static friction. Once this force is overcome, kinetic friction comes into play and resists the water jet force until the hydroentangling pass is complete. Thus, both the elements of friction play a role in influencing the hydroentangling efficiency.

Smooth surfaces at the molecular level have high contact area between them. For such a case, coefficient of static friction is very high.

However, the coefficient of kinetic friction is much smaller because there is lesser resistance to motion once the surface starts to slide. Conversely, if the surfaces are relatively more rough, the points of contact are fewer and the coefficient of static friction drops down.

However, the coefficient of kinetic friction increases because of the asperities [46]. Friction is a complex force and it heavily depends on the scale at which a surface is rough or smooth [48-49].

In addition to fiber-to-fiber friction, there may also be fiber-to-fluid friction or drag [17]. Drag force is proportional to reference area, or the area of the fibers. Increasing surface roughness of fiber may result in an increased area and hence, an increased drag force. Hence, for the fibers of same dimension, rough fibers are likely to show increased drag and kinetic friction whereas for smooth fibers, static friction should be most dominant.

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Chapter 3

Experimental Approach

3.1 Atmospheric Plasma Unit

The atmospheric plasma device NCAPS (**N**orth **C**arolina Atmospheric **P**lasma **S**ystem) was used in this project [1, 2]. It is a dielectric barrier discharge (DBD) plasma device with two 60 x 60 cm copper electrodes imbedded in 2 cm thick polycarbonate (Lexan) dielectric plates. The electrodes are located inside a chamber that allows for venting of the plasma gas and the whole assembly is further enclosed within another chamber which provides shielding from the high voltage and houses winding mechanisms for continuous treatment of fabrics. Voltage is supplied to the device by 8 Pyramid 15V direct current power supplies coupled together in parallel and alternated by a Model 4011 BK Precision Function Generator. The voltage passes through two step-up transformers 180˚ out of phase to both the upper and lower electrodes. The function generator is tunable, but most operations are performed at 1.3 kHz where power is maximized. Voltage, current, and ambient gas temperature are all monitored by an oscilloscope and a computer interfaced with a LabView® software program with in-house developed plasma models and mathematical solvers for power, electron number density, electron temperature, and electron-neutral collision frequency [3]. The program also monitors the flow of gases out of the Mass Flow

controllers and the ambient gas temperature via a Teflon-coated thermocouple. Data can be viewed in real time on both the oscilloscope and via the program interface.

To generate a plasma discharge, the working gas is allowed to flow between the electrodes and the electric field is applied via applying the voltage from the power supplies across the electrodes. Helium is always used as the primary discharge gas due to its low ionization potential. Other gases including oxygen, CF_4, methane and nitrogen can be introduced into the helium stream where they dissociate and ionize through collisional processes. These secondary gases are added in very small percentages, usually on 1-2% by mass. All gases are regulated by MKS Mass-Flow Controllers which are controlled by a MKS 4-channel controller and readout unit. The gas is then introduced into a gap between the electrodes and the plasma is generated. Figure 3.1 shows a schematic of the NCAPS atmospheric pressure plasma device for continuous treatment of fabrics.

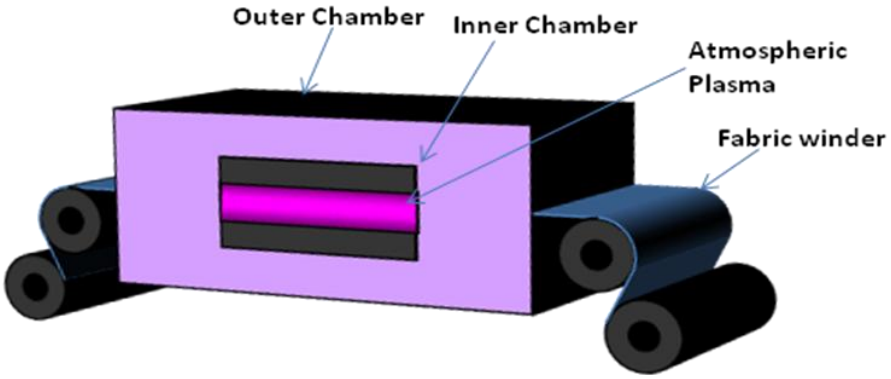


Figure 3.1: Schematic of the NCAPS atmospheric pressure plasma unit

Samples were treated using the continuous run treatment cell and external let-out and pick-up rollers at a gap space of 0.5 inch. The continuous run treatment cell is composed of a set of detachable panels and silicone flaps that can be added to the upper and lower electrodes and allow for the helium gas to be concentrated between the electrodes without restricting fabric flow. Figure 3.2 shows a schematic drawing of the continuous run cell, while Figure 3.3 illustrates the details of the cell and the fabric treatment.

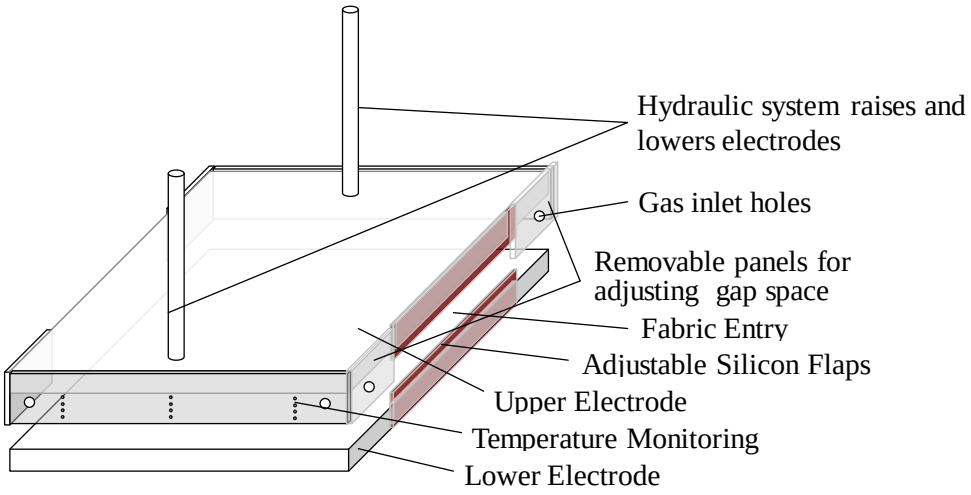
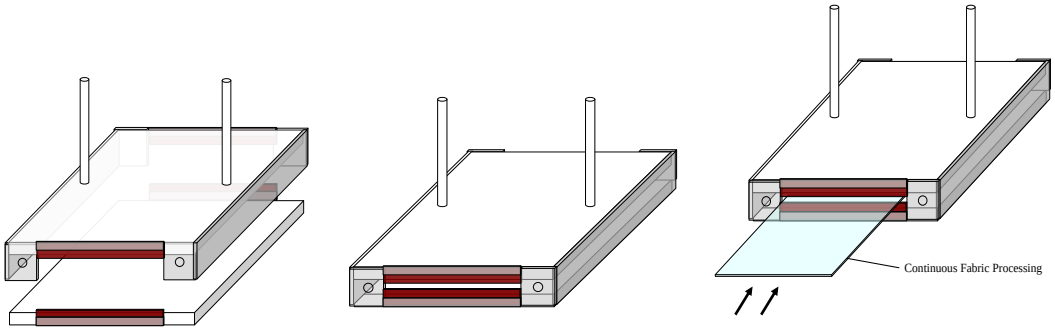


Figure 3.2: Continuous Run "Cell" Design



a b c

Figure 3.3: Continuous run cell with upper electrode raised (a), upper electrode lowered (b) and with fabric treatment (c).

3.2 Materials

One of the objectives of this project is to investigate the effect of plasma treatment on hydroentangling efficiency. To mark this effect, a set of fibers of varying degree of hydrophobicity were used as shown in Table 3.1 below:

Table 3.1: Fibers composition and their characteristics for plasma treatment

Fiber Type	**Composition**	**Fiber Denier**	**Basis Weight(gsm)**
Polypropylene	100%	1.7	111
Polyester	100%	1.5	110
Nylon 6	100%	3	113
Polypropylene-Lyocell	50-50%	1.7, 1.7	106

3.3 Plasma Parameters

3.3.1 Working Gas

As previously mentioned, helium is used as the seed gas for atmospheric plasma treatments. The first set of plasma trials were conducted with pure helium plasma while second and third trials were conducted with He-1% O_2 plasma and He-1% CF_4 plasma respectively.

3.3.2 Exposure Time

Exposure time for the trials was fixed as 1 min in a continuous run.

3.3.3 Plasma Aging

The webs were treated with He, He-1% O_2 plasma and He-1% CF_4 plasma for 1 min exposure time prior to hydroentangling. The webs were cut to a width of 12 inches and passed through the plasma chamber in a continuous run by incorporating an external fabric winder to the facility. The plasma treated webs were divided into three sets A, B and C. Set A was hydroentangled with an approximate lag of 10 minutes between the plasma treatment and hydroentangling; set B was hydroentangled 4 hours after the treatment while set C was allowed to age for 24 hours before being hydroentangled.

3.4 Hydroentangling Parameters

The hydroentangling parameters were kept constant throughout the course of the experiments at the following values:

(a) Line speed: 10 m/min

(b) Manifold Pressures:

Manifold	**Pressure (bars)**
Manifold 1	30
Manifold 2	90
Manifold 3	120
Manifold 4	150
Manifold 5	150

(c) No. of passes: 1

(d) Drying temperature: 120 deg C

References:

[1] Matthews, SR 2005, ‘Plasma aided finishing of textile materials’, PhD thesis, North Carolina State University, USA.

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Chapter 4

Results and Discussion

Characterization of hydroentangled webs and plasma treated fibers

The above mentioned three sets of plasma-treated and hydroentangled nonwoven webs were characterized for the following properties:

4.1 Vertical Wicking/Absorbency

The control nonwoven webs were divided into 3 groups: He plasma, He-O_2 plasma and He-CF_4 plasma and tested for wicking after 60 seconds of plasma treatment with respective plasmas against the control. The test strips (6" X 1") were immersed 3 mm in standing water and wicking height was measured after 15 minutes.

Though tested with a model to predict the behavior of unbonded webs, vertical wicking gave a direct measure of fabric wettability. From the results, it was concluded that plasma enhances wicking or wettability significantly. The effects of plasma treatment on surface chemistry are known to decrease with aging. However in most cases, the effect is not completely reversible even after 24 hours of aging. The effect of hydrophilicity was visually observed during the hydroentangling wherein plasma pre-treated webs showed no sign of water-pooling on the surface compared to the controls where water pools persisted on the control samples, specifically for polypropylene and polyester.

Hence, it is clear that due to plasma pre-treatment, webs were more hydrophilic compared to their original state at all times before they were hydroentangled.

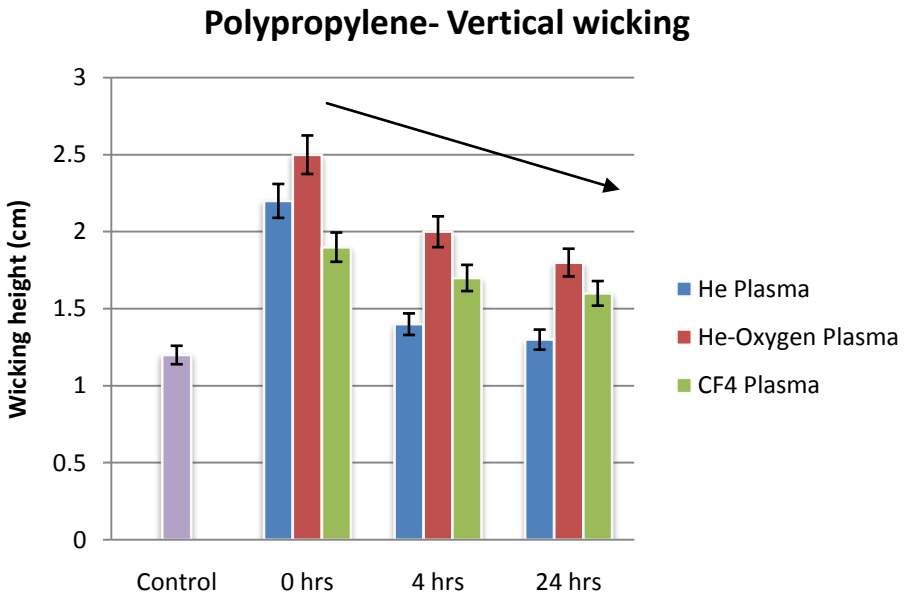

Figure 4.1: Vertical wicking for PP web for the three plasma systems

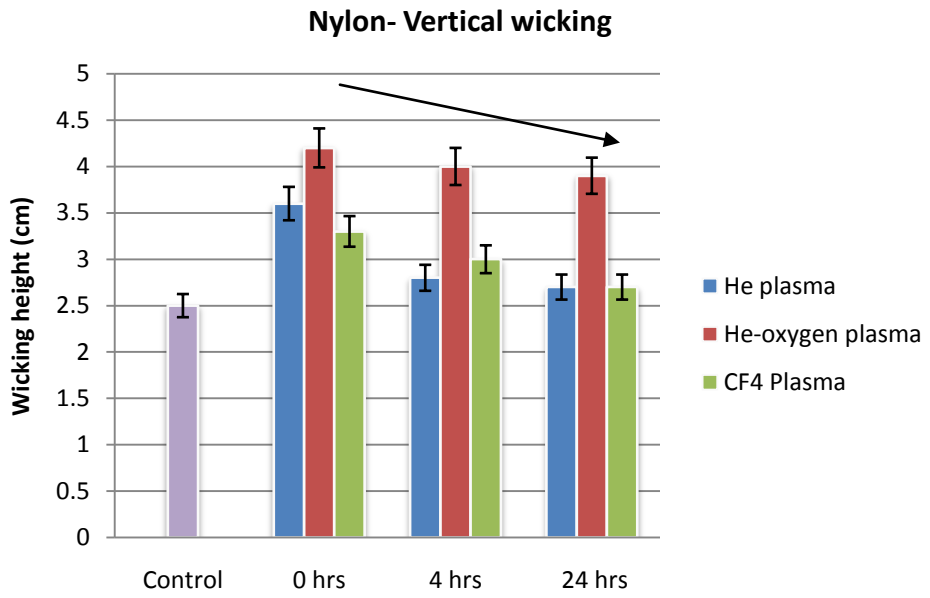

Figure 4.2: Vertical wicking for Nylon web for the three plasma systems

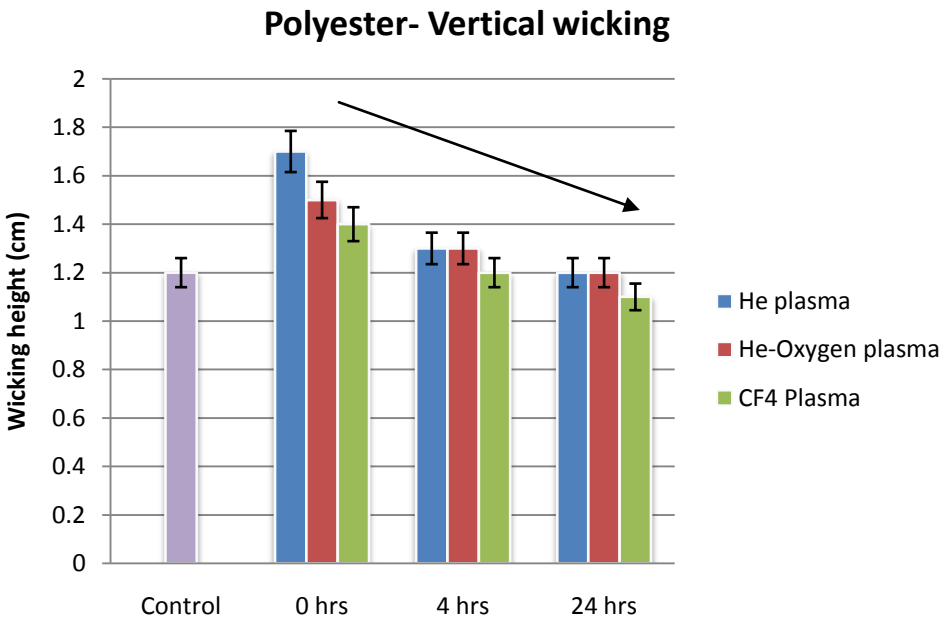

Figure 4.3: Vertical wicking for PET web for the three plasma systems

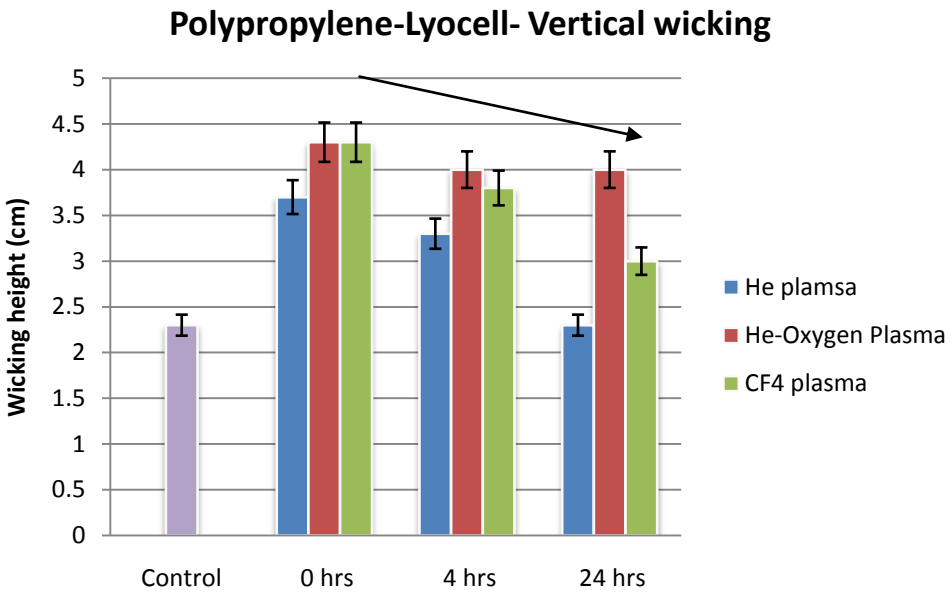

Figure 4.4: Vertical wicking for PP-Lyocell web for the three plasma systems

4.2 Tensile Strength (webs)

The tensile strength of the webs was characterized by test method ASTM D5034: Standard Test Method for Breaking Strength and Elongation of Textile Fabrics in machine and cross directions for all the webs corresponding to A, B and C sets with aging times of 0, 4 and 24 hrs and compared against the control web.

The tensile strengths of webs corresponding to sets A, B and C were then plotted against the control for both MD and CD.

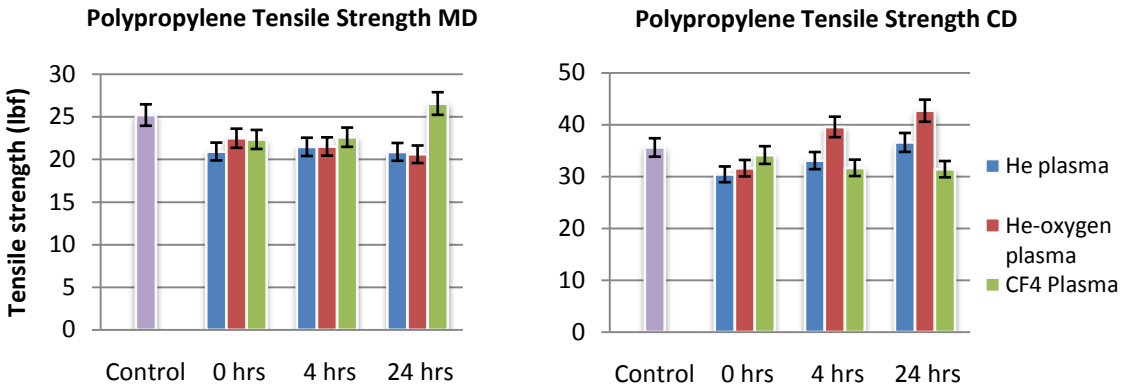

Figure 4.5: MD and CD tensile strength of PP webs pre-treated with plasmas for 60 sec

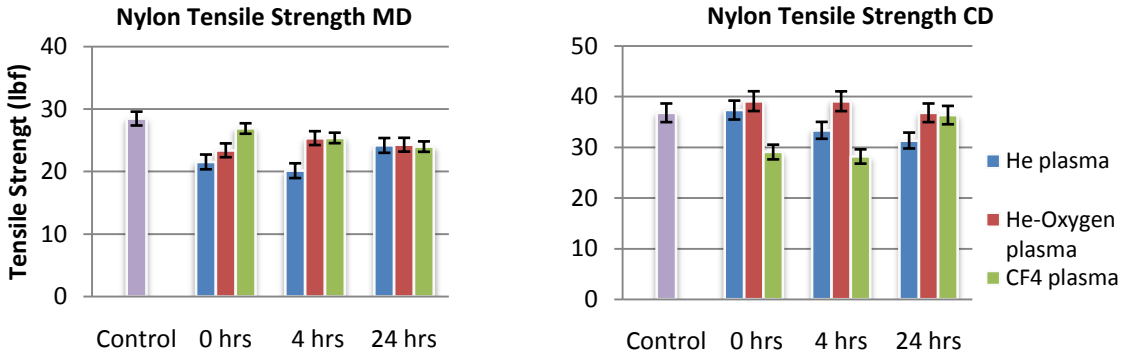

Figure 4.6: MD and CD tensile strength of Nylon webs pre-treated with plasmas for 60 sec

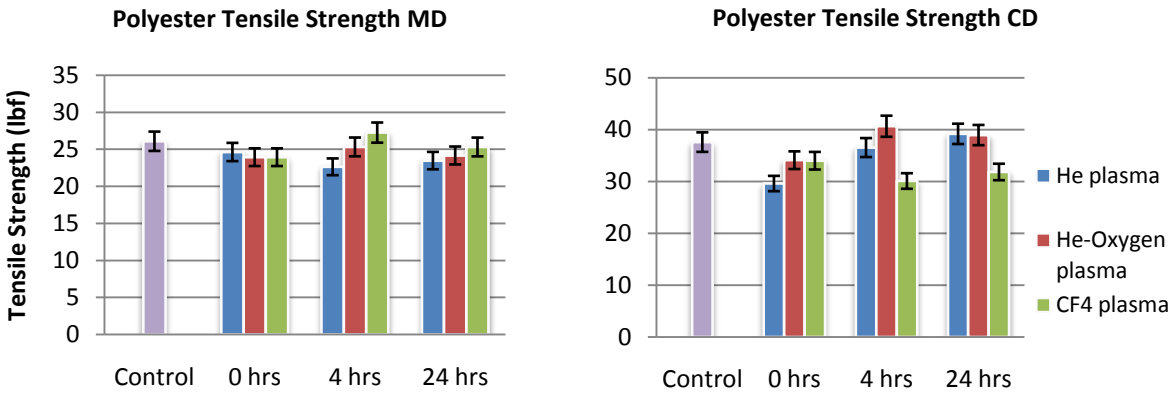

Figure 4.7: MD and CD tensile strength of Polyester webs pre-treated with plasmas for 60 sec

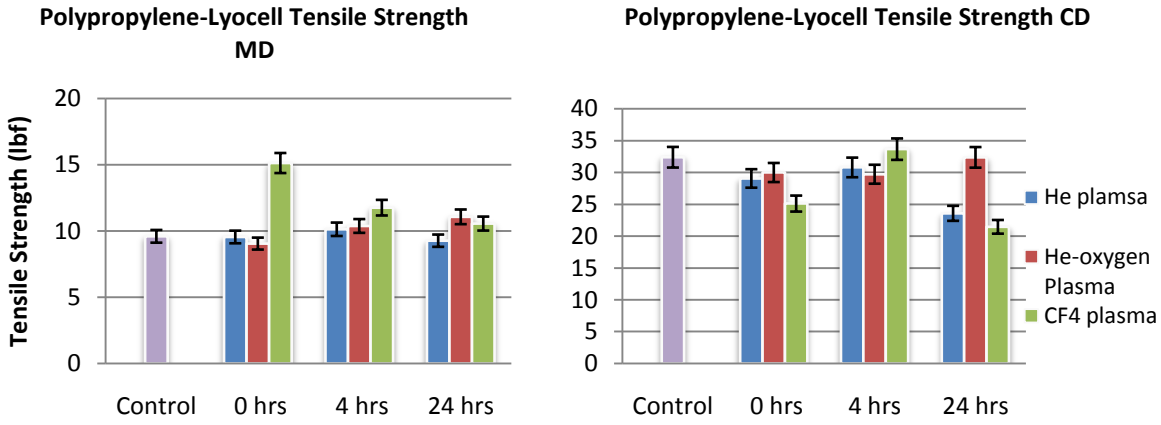

Figure 4.8: MD and CD tensile strength of Polypropylene-Lyocell webs pre-treated with plasmas for 60 sec

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Tensile strength of a nonwoven fabric depends on the degree of entanglement of the fibers. Other factors influencing the same are individual strength of fibers, fiber-to-fiber interaction and orientation of fibers in the web structure. From the above results, it is noted

that the tensile strength is showing a general decline in both MD and CD. There are few specific cases in which slight enhancements may be noted. However, these points may be outliers. It was also observed for some cases where a decrease in the tensile strength in MD or CD was compensated by a corresponding increase in the same in the opposite directions. For example, in specific cases of polypropylene-lyocell blend pre-treated with CF_4 plasma and hydroentangled after 0 hrs and polypropylene pre-treated with CF_4 plasma and hydroentangled after 24 hrs, it can be noticed that the strength in CD has declined for an increase in the same in MD.

4.3 Trapezoid Tear Strength (webs)

The trapezoid tear strength of the webs was characterized by test method ASTM D1117: Standard Test Method for Evaluating Nonwoven Fabrics, in both machine and cross directions was plotted against control.

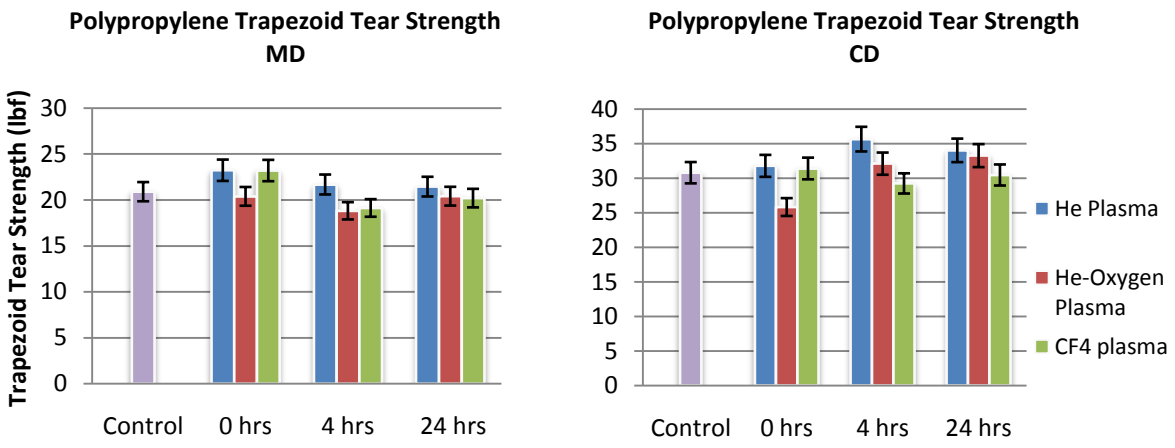


Figure 4.9: MD and CD trapezoid tear strength of Polypropylene webs pre-treated with plasmas for 60 sec

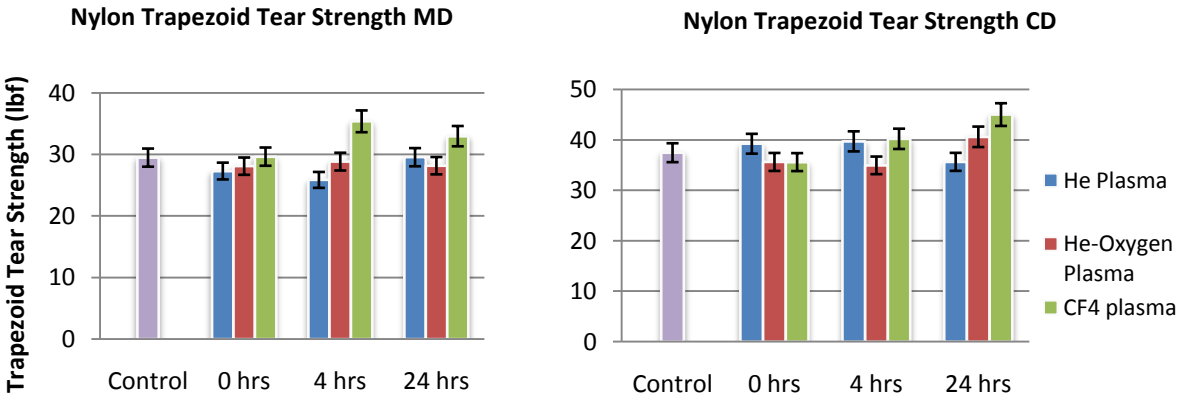


Figure 4.10: MD and CD trapezoid tear strength of Nylon webs pre-treated with plasmas for 60 sec

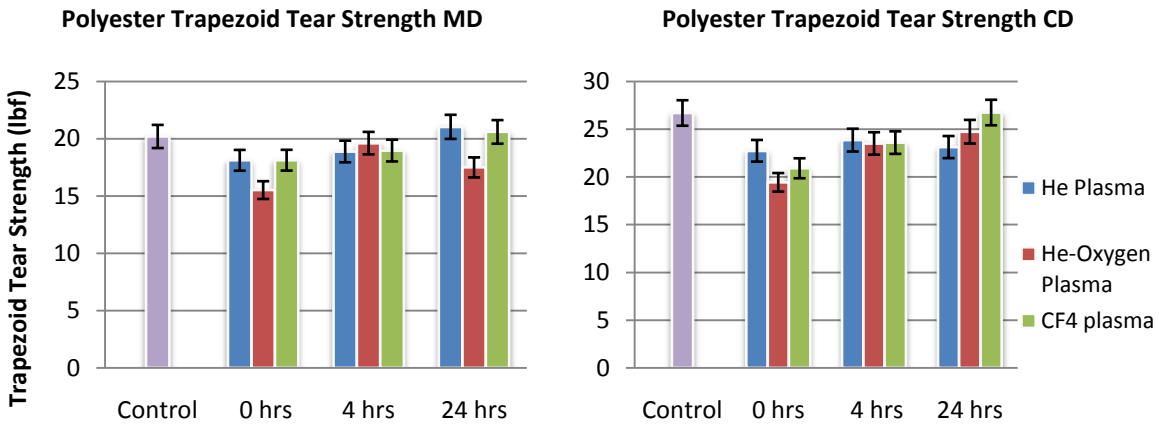


Figure 4.11: MD and CD trapezoid tear strength Polyester webs pre-treated with plasmas for 60 sec

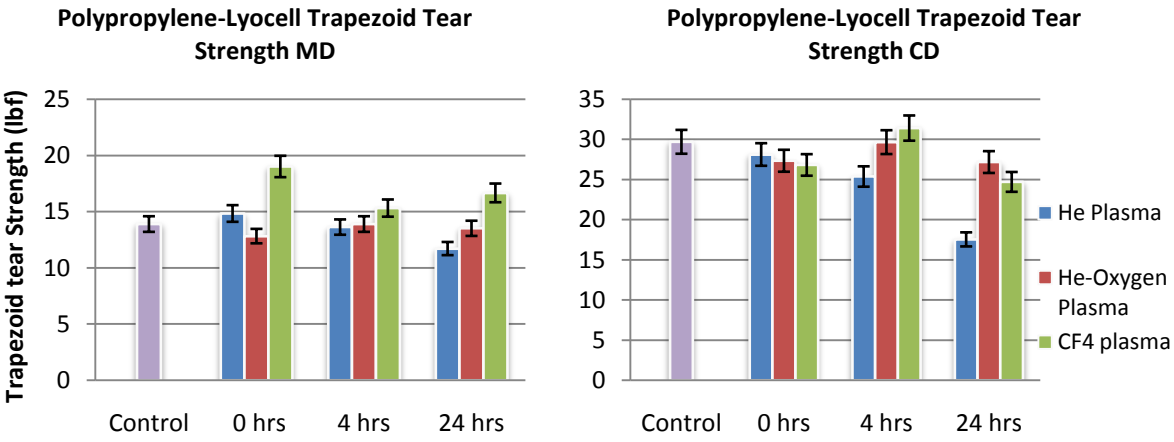


Figure 4.12: MD and CD trapezoid tear strength Polypropylene-Lyocell webs pre-treated with plasmas for 60 sec

4.4 Fiber Modulus and Tenacity Testing

The individual fibers used in the nonwovens were tested for tenacity and modulus values using an MTS Q-Tester (Test method: ASTM D3822 Standard Test Method for Tensile Properties of Single Textile Fibers) at a constant crosshead speed of 15 mm/min. While tenacity is the amount of force needed to break the fiber, modulus is a measure of fiber stiffness. These two properties are important fiber properties which affect the ultimate web properties. Flexural rigidity of a fiber, defined as the couple required to bend the fiber to unit curvature is one of the factors considered to have significant effects on hydroentangling efficiency. Fibers with low flexural rigidity can bend around small radii easily and require less energy to entangle compared to those with high flexural rigidity [1]. Flexural rigidity shows a direct relationship with fiber modulus values. It is reported in the literature that plasma does have an effect on the fiber mechanical properties [2] which, in turn can affect the hydroentangling efficiency.

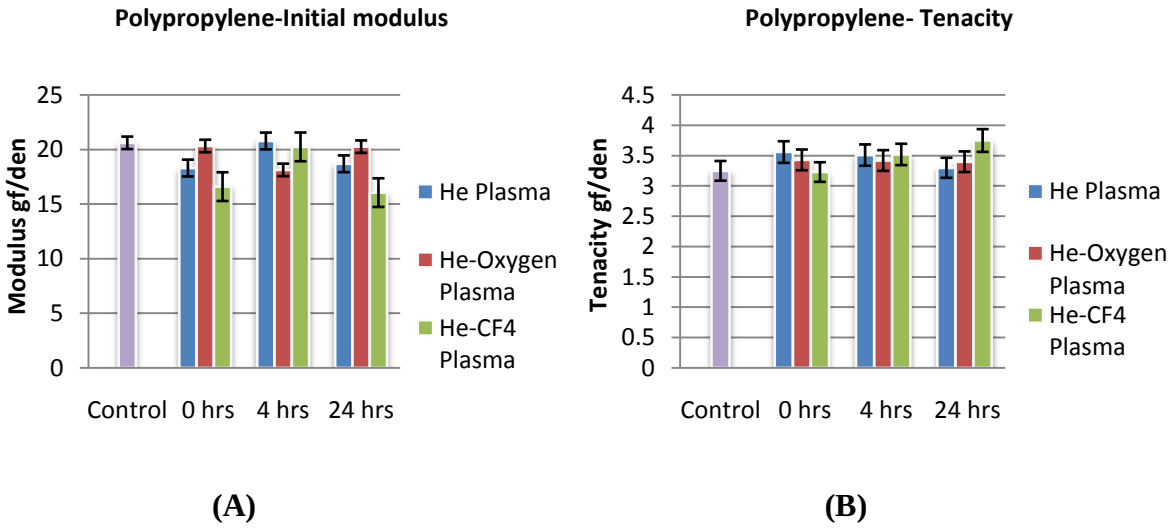


Figure 4.13: (A) Change in Modulus, (B) Change in Tenacity for Polypropylene fiber for the three plasma systems, exposed for 60 sec

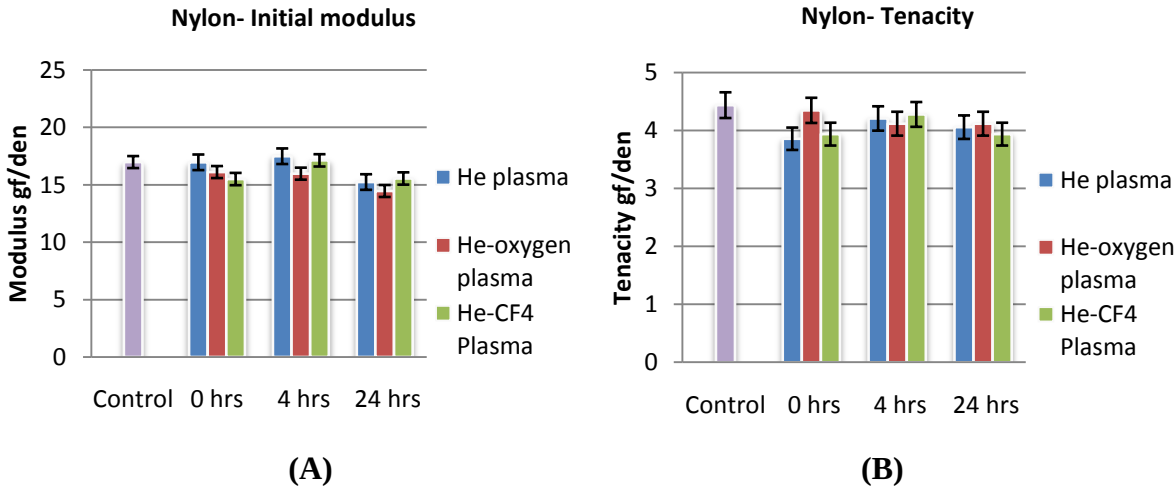
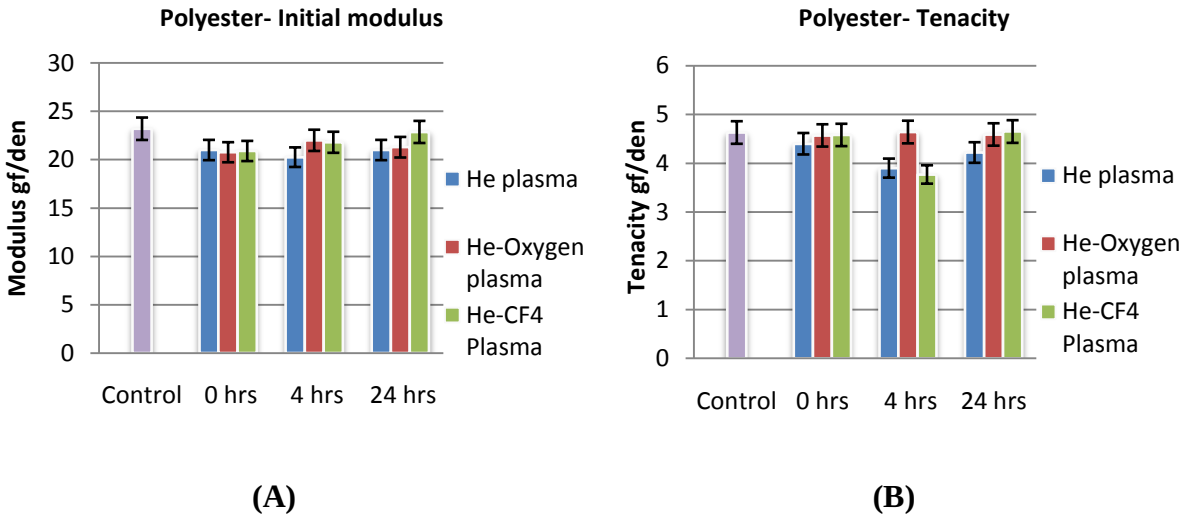
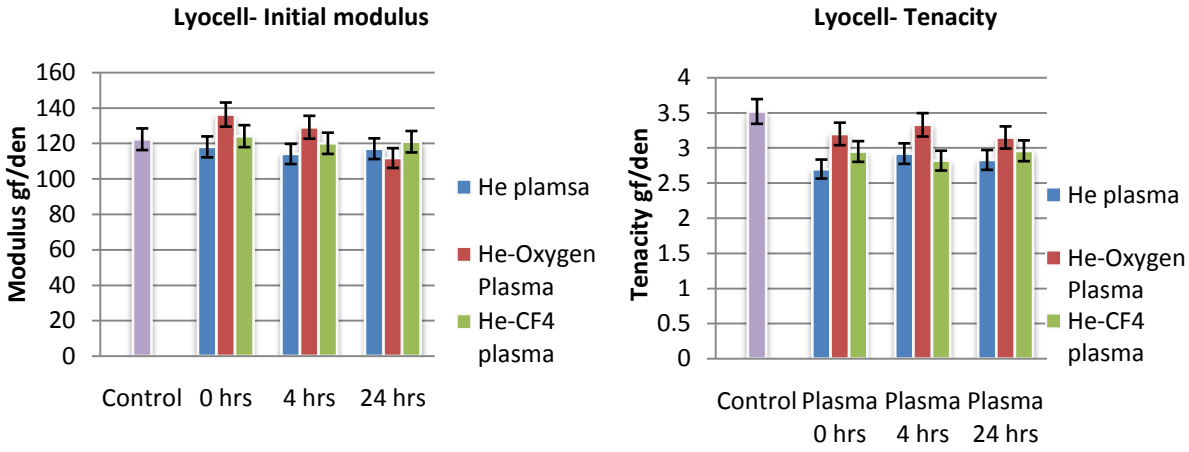


Figure 4.14: (A) Change in Modulus, (B) Change in Tenacity for Nylon fiber for the three plasma systems, exposed for 60 sec



(A) **(B)**

Figure 4.15: (A) Change in Modulus, (B) Change in Tenacity for Polyester fiber for the three plasma systems, exposed for 60 sec



(A) **(B)**

Figure 4.16: (A) Change in Modulus, (B) Change in Tenacity for Lyocell fiber for the three plasma systems, exposed for 60

From the above results, it was seen that in most plasma-substrate combinations, the initial modulus values were either not significantly different or lower than controls. The tenacity showed a slight enhancement for polypropylene but declined in general for nylon, polyester and lyocell. A positive change in initial modulus may render the fibers difficult to entangle because of increased bending rigidity. Though not too clear from the data, the change in modulus values may be contributing towards the change in fabric mechanical properties. The change in tenacity values is upto 10% in some cases which may play a significant role in altering the final properties. Hence, the changes in individual fiber properties may influence the overall strength of the fabric.

4.5 Tenacity and Elongation of the webs

The tenacity of the webs was calculated by using the following equation [3]:

Fabric tenacity = 0.00981F / 0.0254EW

Where,

F=force at break,

E= elongation at break, and

W= basis weight

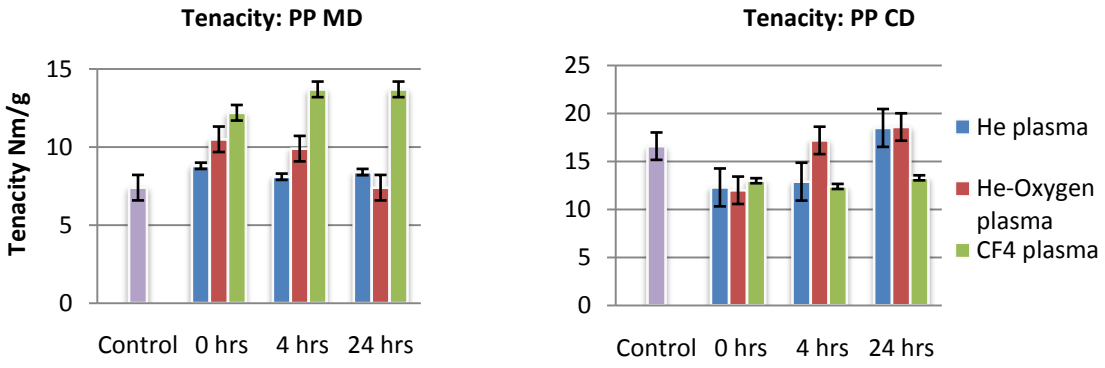


Figure 4.17: MD and CD tenacity of Polypropylene webs pre-treated with plasmas for 60 sec

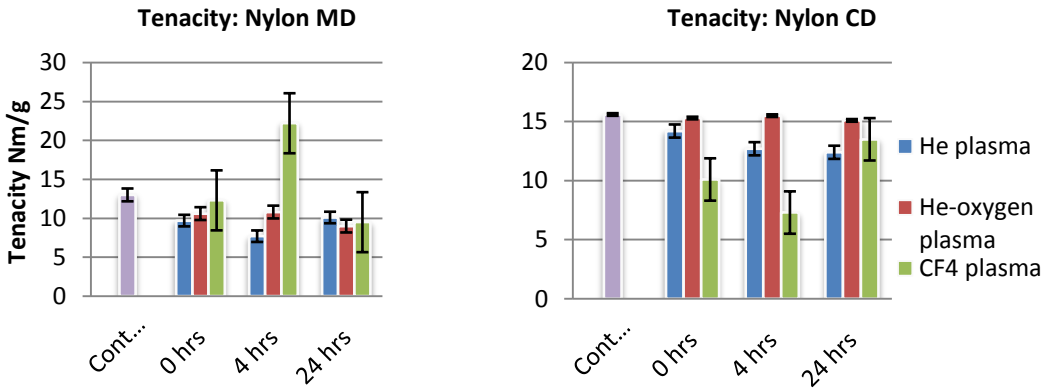


Figure 4.18: MD and CD tenacity of Nylon webs pre-treated with plasmas for 60 sec

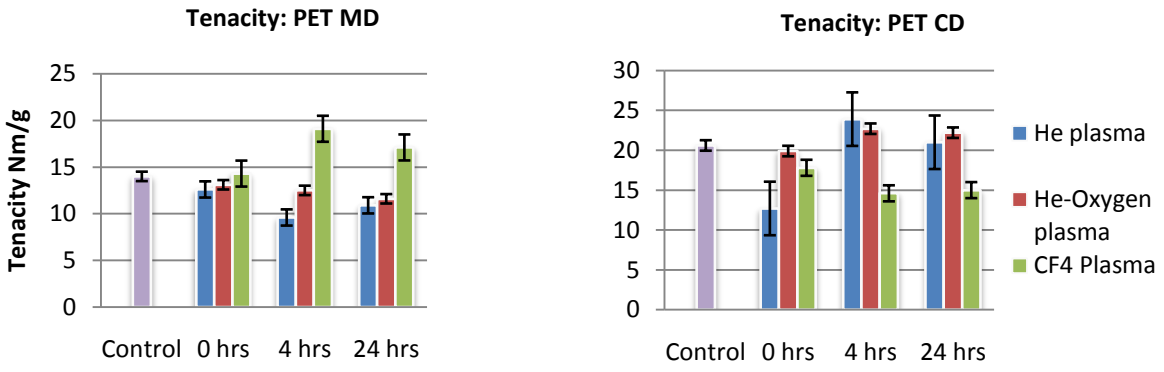


Figure 4.19: MD and CD tenacity of Polyester webs pre-treated with plasmas for 60 sec

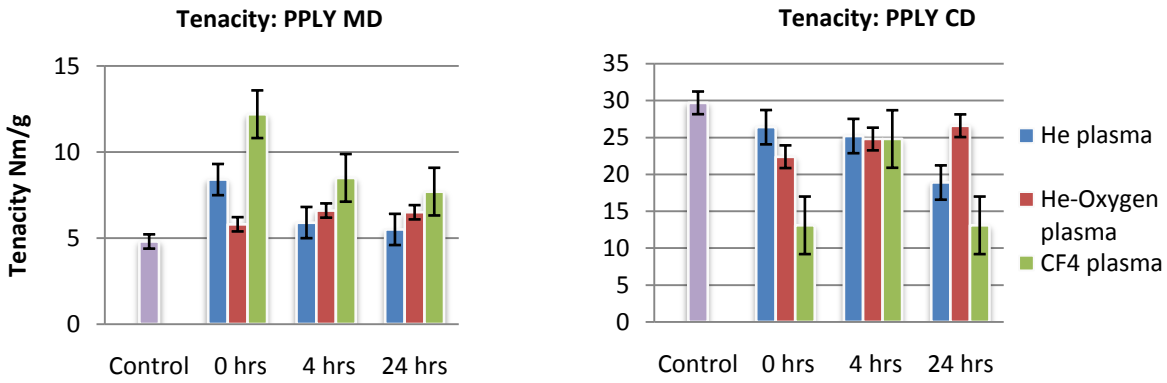


Figure 4.20: MD and CD tenacity of Polypropylene-Lyocell webs pre-treated with plasmas for 60 sec

Elongation of the fabric was measured by the Instron and plotted for the webs.

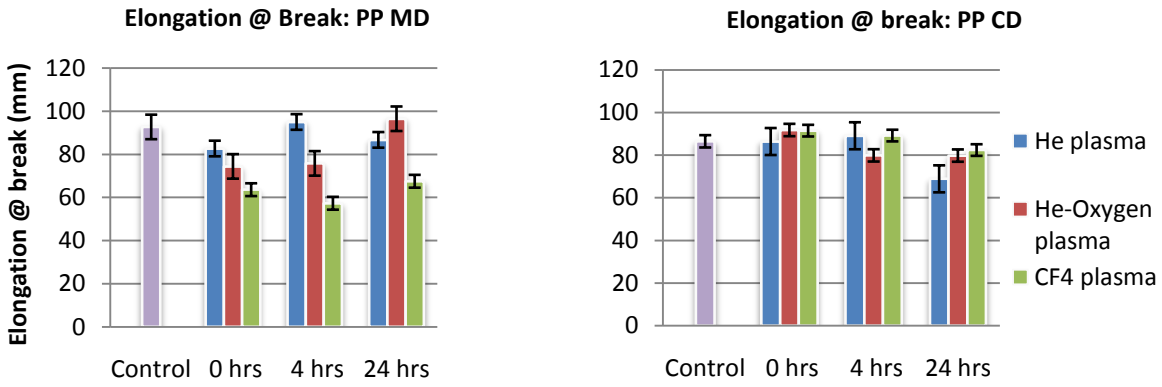


Figure 4.21: MD and CD elongation of Polypropylene webs pre-treated with plasmas for 60 sec

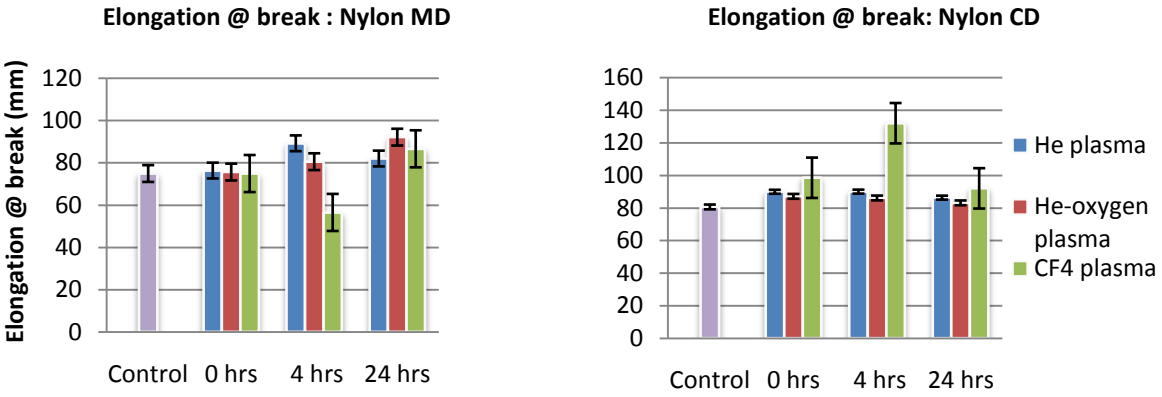


Figure 4.22: MD and CD elongation of Nylon webs pre-treated with plasmas for 60 sec

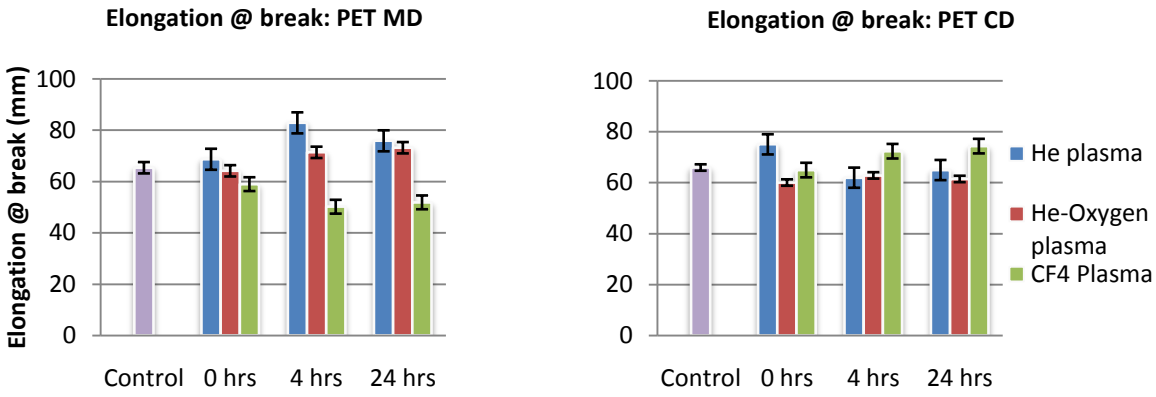


Figure 4.23: MD and CD elongation of Polyester webs pre-treated with plasmas for 60 sec

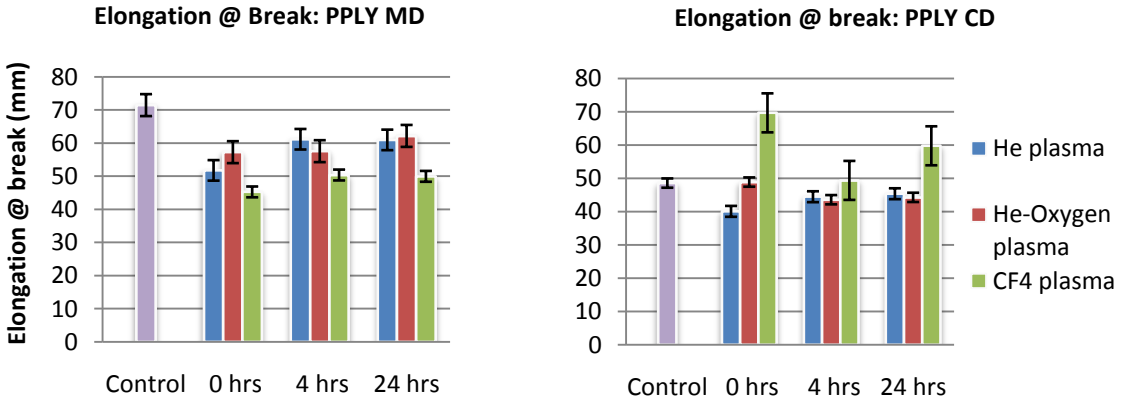


Figure 4.24: MD and CD elongation of Polypropylene-Lyocell webs pre-treated with plasmas for 60 sec

From the above results, it is observed that the tenacity value which is also representing the secant modulus of the fabric is showing a significant enhancement in MD specifically for Polypropylene for all plasma systems. It is interesting to notice a corresponding decrease in the tenacities in CD. For nylon and polyester, there is an enhancement in secant modulus in MD only for the webs treated with helium-CF_4 plasma and

there is a decline for the same in CD. The polypropylene-lyocell blend is showing a behavior similar to that of polypropylene wherein there is an enhancement in secant modulus values in MD and there is corresponding decline in the same in CD.

The elongation at break has declined in MD for polypropylene and the polypropylene-lyocell blend while it showed slight enhancement in CD for nylon especially for helium-CF_4 plasma. Elongation at break did not change much for polyester in either direction.

An enhancement in modulus and decline in elongation at break in MD suggest a re-orientation or compaction of fibers in MD. It is suggested that the secondary bonding due to plasma treatment may give rise to two additional forces:

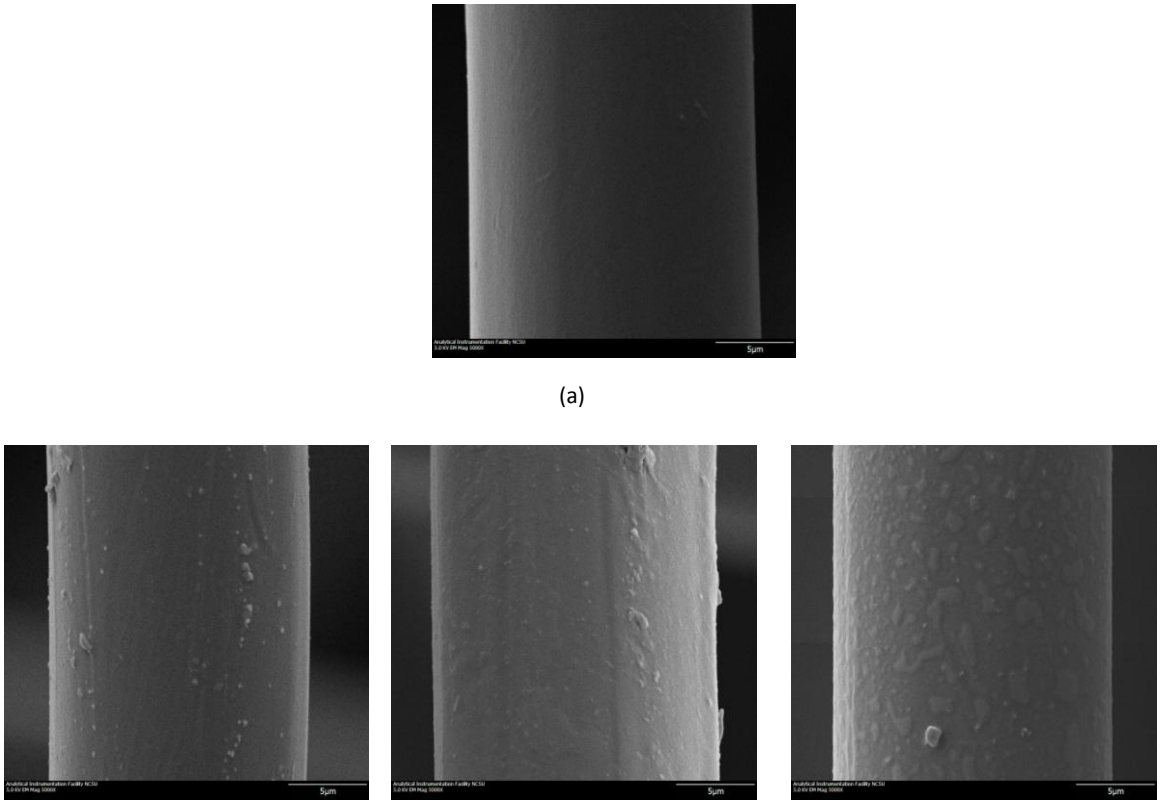
- Fiber-to-fiber cohesive forces due to excessive H-bonding and van der Waal's forces
- Fiber-web to carrier wire-mesh adhesion. This adhesive force may resist the peeling of web from the wire-mesh inducing the fibers to align more in MD.

Due to the above mentioned secondary bonding, there could be a possible rearrangement of fibers in the nonwoven structure.

4.6 Scanning Electron Microscopy

Plasma is known to have a significant effect on substrate's surface in terms of its roughness or smoothness. Plasma etching is strongly evident in reactive plasmas such as oxygen or CF_4 plasma. However, to a great extent it depends on the combination of substrate

and plasma and exposure time. The individual fibers were plasma treated prior to taking SEM images on Hitachi S-3200 Scanning Electron Microscope. SEM images were taken at 5000 X resolution.



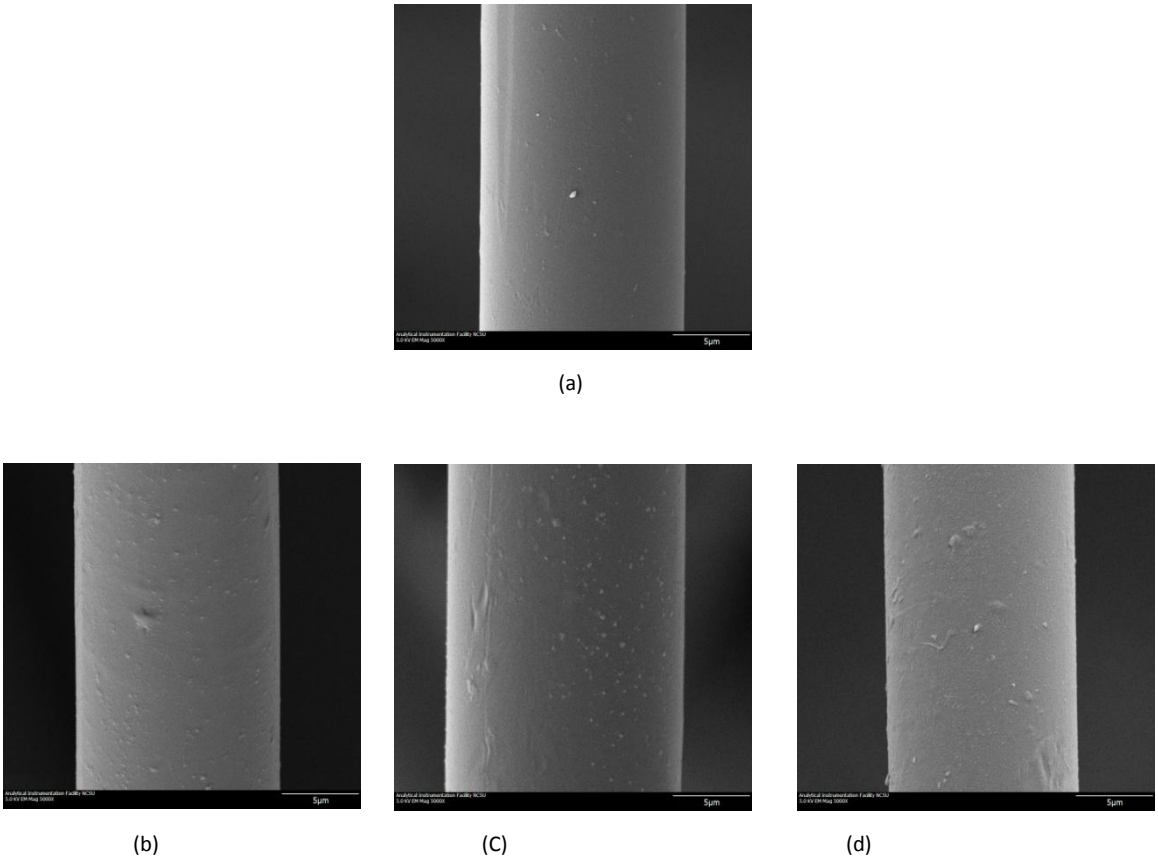
(a)

(b) (c) (d)

Figure 4.25: SEM images for polypropylene (a) PP Control sample, (b) PP, He plasma treated, 60 sec, (c) PP, He-O2 plasma treated, 60 sec, (d) PP, He-CF4 plasma treated, 60 sec

The SEM images of Polypropylene indicate a significant surface roughness added to the plasma-treated surfaces. The fiber treated with helium plasma is significantly rougher than the control at the micro scale. Helium-oxygen plasma has also added to the final surface roughness of the fiber. Helium-CF_4 plasma has displayed the most profound effect on fiber

surface and the surface is much rougher compared to the control as well as the fibers treated with pure helium and helium-oxygen plasmas.



(a)

(b) (C) (d)

Figure 4.26: SEM images for polyester (a) PET Control sample, (b) PET, Helium plasma treated, 60 sec, (c) PET, He-O2 plasma treated, 60 sec (d) PET, He-CF4 plasma treated, 60 sec

The polyester control fiber appears rough before the treatment. From the above SEM images, pure helium plasma and helium-CF_4 plasmas do not appear to have added to the

existing surface roughness significantly. The fiber treated with helium-oxygen plasma has displayed maximum surface roughness compared to the control and the fibers.

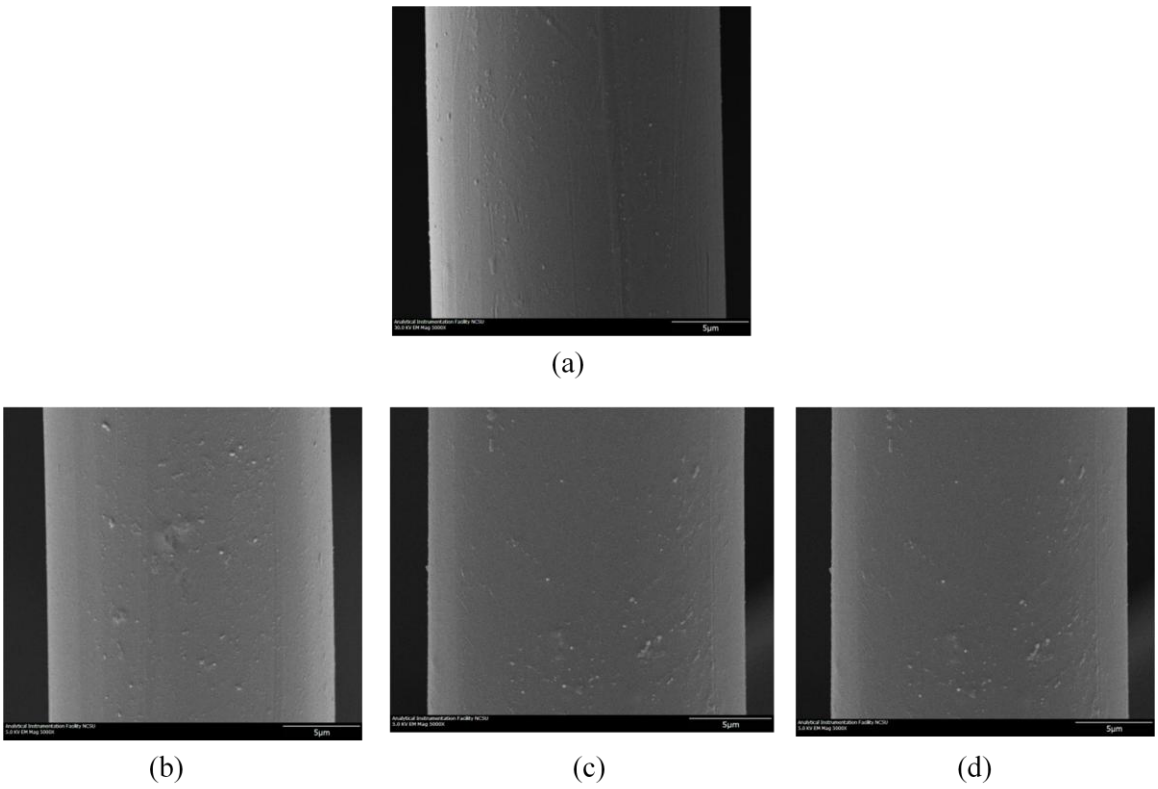


Figure 4.27: SEM images for nylon (a) Nylon control sample, (b) Nylon, Helium plasma treated, 60 sec, (c) Nylon, He-O2 plasma treated, 60 sec (d) Nylon, He-CF4 plasma treated, 60 sec

Nylon control fiber has significant existing surface roughness. From the above SEM images, none of the plasma systems appear to have added to the existing surface roughness significantly.

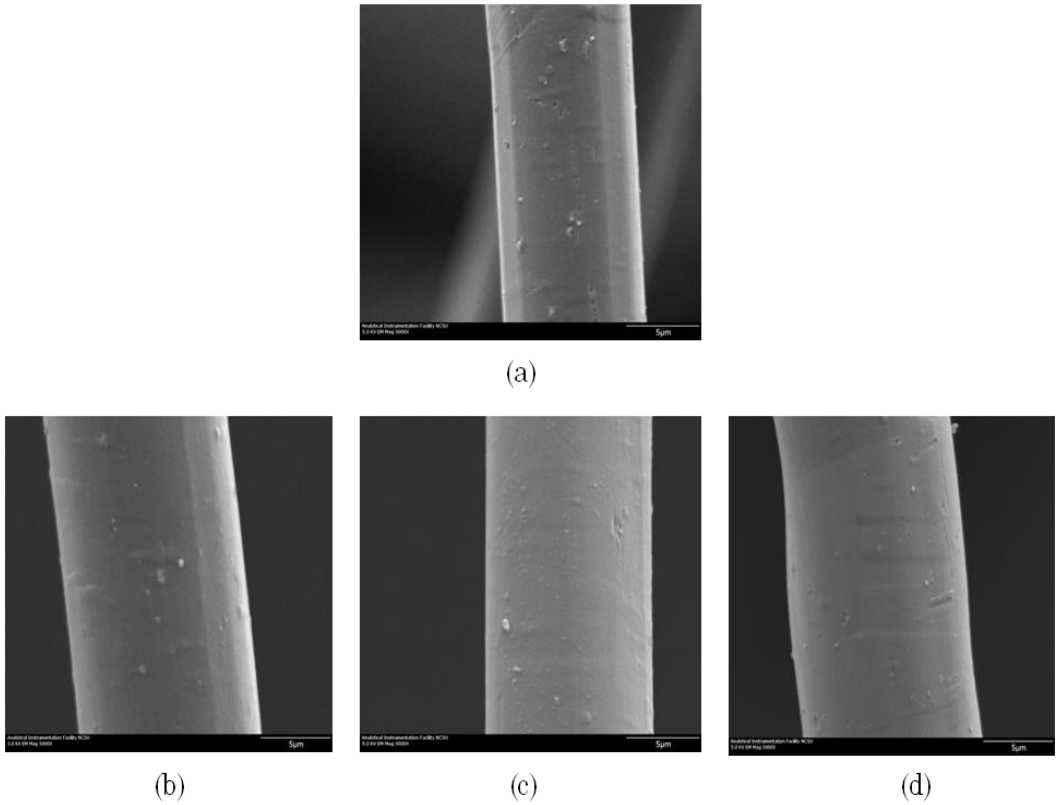


Figure 4.28: SEM images for Lyocell (a) Lyocell control sample, (b) Lyocell, Helium plasma treated, 60 sec, (c) Lyocell, He-O2 plasma treated, 60 sec (d) Lyocell, He-CF4 plasma treated, 60 sec

Like polyester and nylon control fibers, Lyocell control fiber is also already rough before the treatment. From the above SEM images, it appears that the all the plasma systems have a smoothing effect on the existing surface roughness and the surfaces have become smoother compared to the control surface.

4.7 Electrostatic Filtration Efficiency

Electrostatic filtration efficiency tests were conducted on TSI 3160 Fractional Filtration Efficiency Tester. The test was performed with NaCl aerosol with a particle size of 0.3 microns. The results were plotted against the controls.

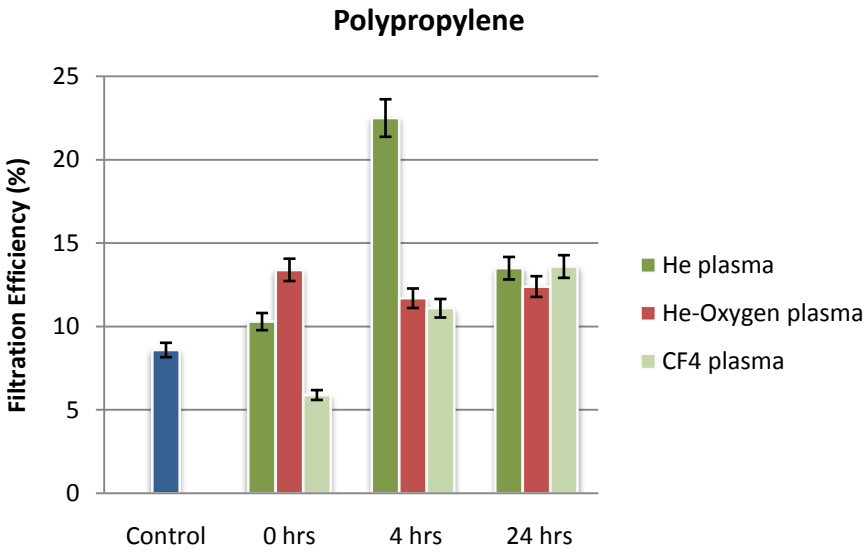


Figure 4.29: Air filtration efficiency for Polypropylene webs

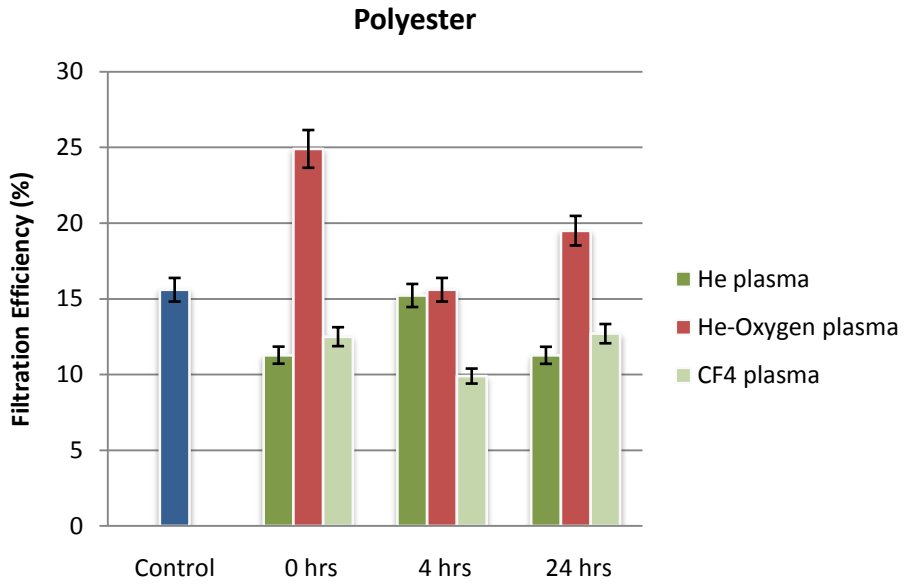


Figure 4.30: Air filtration efficiency for Polyester webs

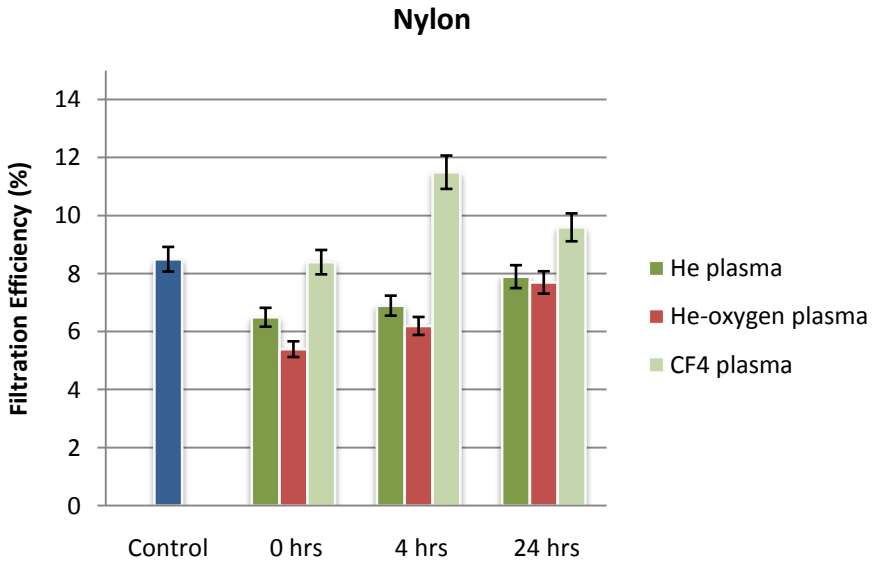


Figure 4.31: Air filtration efficiency for Nylon webs

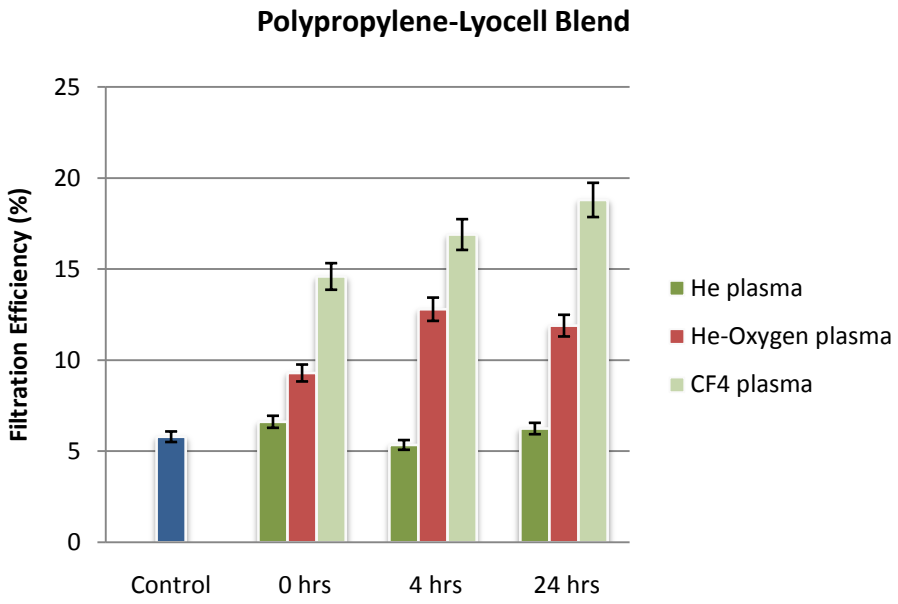


Figure 4.32: Air filtration efficiency for Polypropylene-Lyocell blend

From the above filtration data, it can be seen that there is a significant enhancement in the filtration efficiency of polypropylene fabrics for all three plasma treatments. In case of Polyester, the filtration efficiency has dropped for pure helium and helium-CF_4 plasma systems. However, the filtration efficiency has enhanced significantly for the webs treated with helium-oxygen plasma. Nylon fabrics have shown a general decline in the filtration efficiency for all plasma systems except for the specific case of web treated with helium-CF_4 plasma and hydroentangled after 4 hours of aging. Polypropylene-Lyocell blend fabrics have shown significant enhancement in filtration efficiency for the reactive plasma systems. The enhancement in filtration efficiency after plasma treatment is reported to be due to addition in surface area due to plasma-substrate interaction. Tracing the filtration results back to SEM images in the previous section, it can be observed that the fibers responding to plasma in

terms of addition of surface roughness must have enhanced surface area and hence enhanced filtration efficiency.

4.8 Air Permeability

The control and treated webs were characterized for air permeability using ASTM D737: Standard Test Method for Air Permeability of Textile fabrics. Air is blown through the nonwoven web and permeability is calculated by measuring the pressure difference across it.

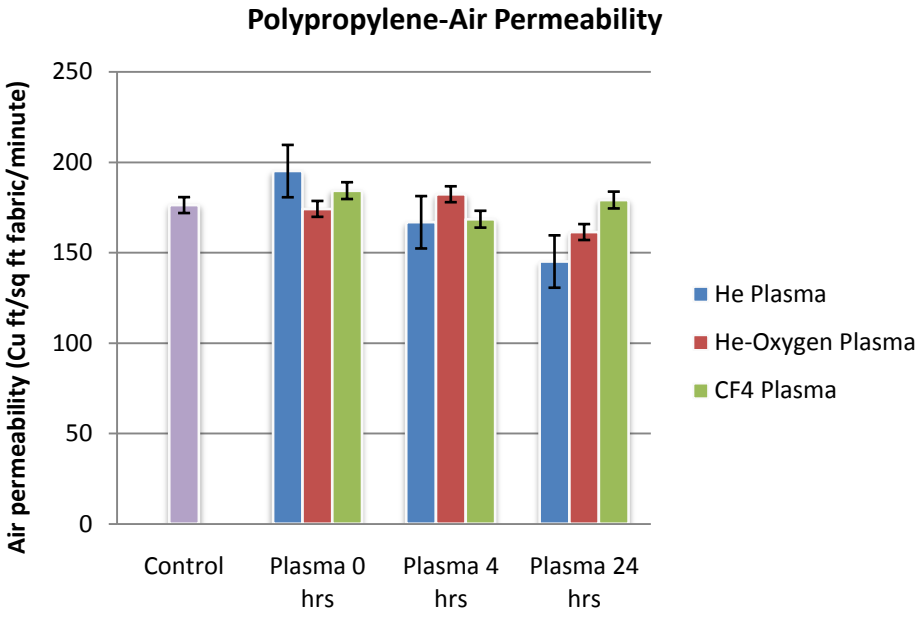


Figure 4.33: Air permeability for Polypropylene webs

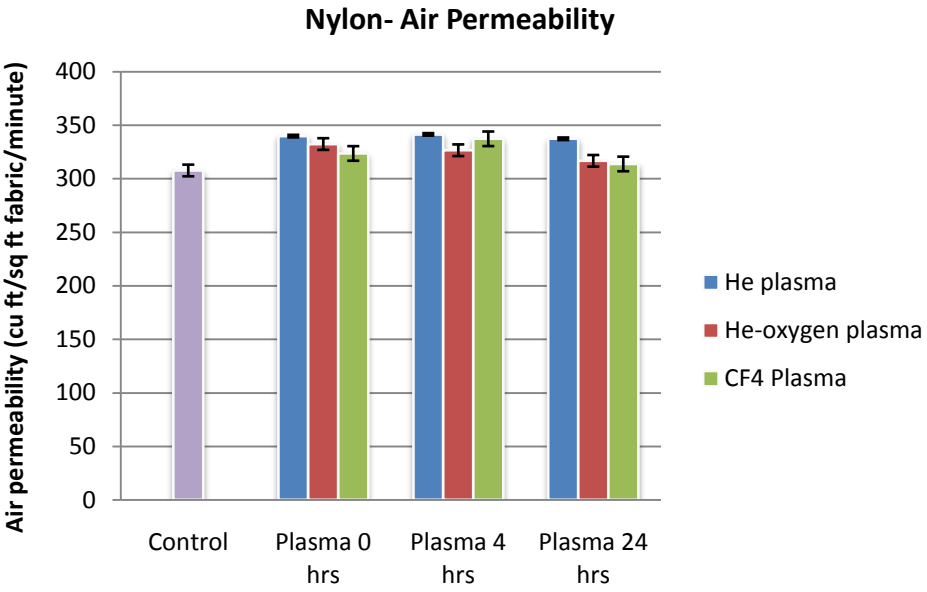


Figure 4.34: Air permeability for Nylon webs

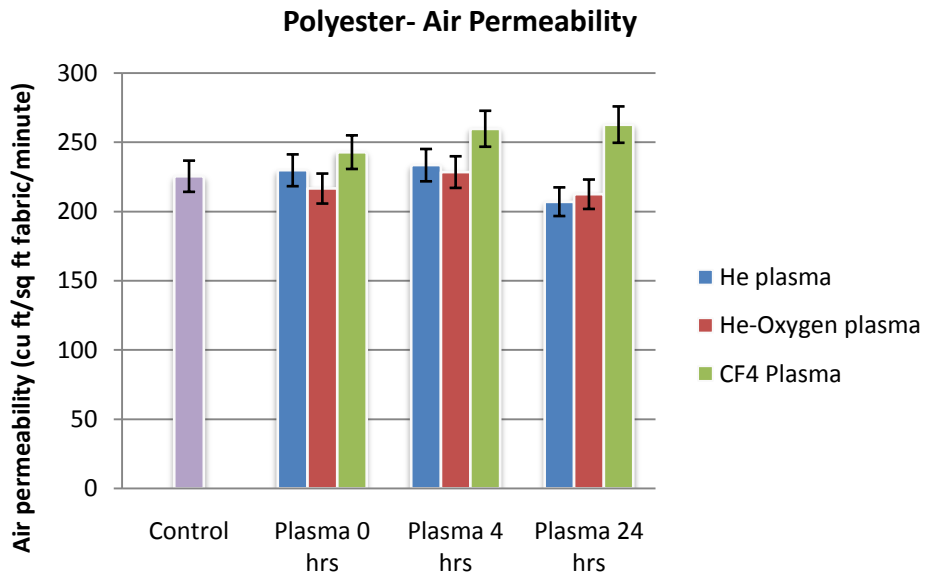


Figure 4.35: Air permeability for Polyester webs

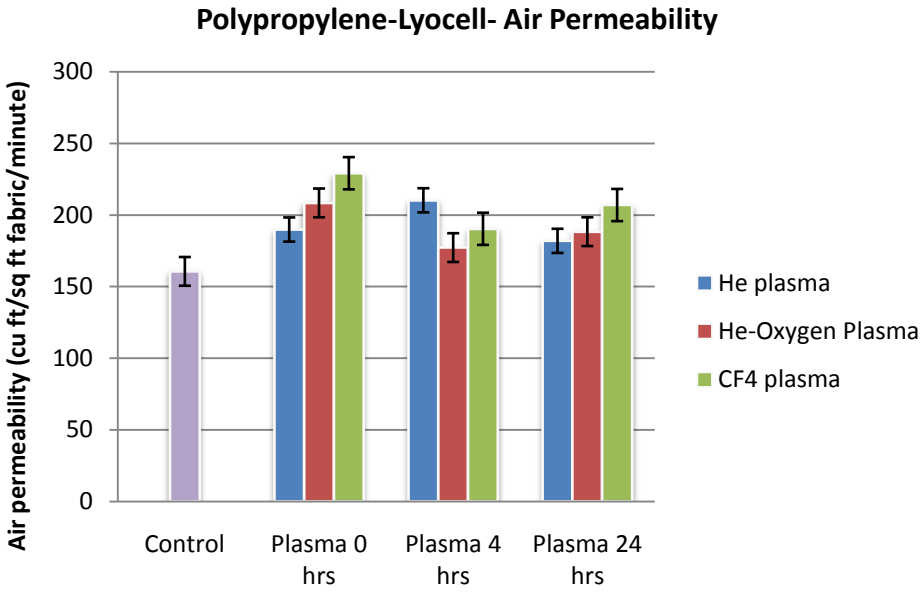


Figure 4.36: Air permeability for Polypropylene-Lyocell blend

4.9 Web Density

Web densities were calculated for control webs as well as the plasma-pretreated and hydroentangled webs. The results were compared to get an insight into changes in the web properties caused by adding the plasma pretreatment step to the entire cycle. Ten pieces of known dimensions were cut from all the samples, stacked together and measured for thickness with an electronic thickness tester. The stacks were then weighed and volume was calculated. The mass was divided by the calculated volume and the density was thus obtained.

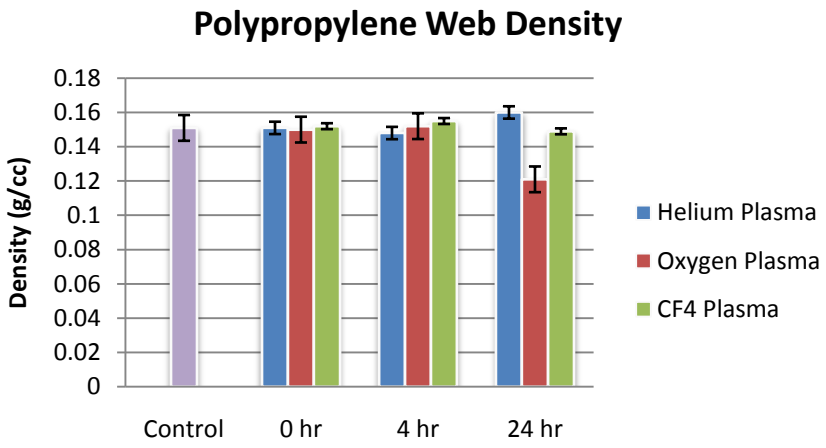


Figure 4.37: Web densities for Polypropylene webs

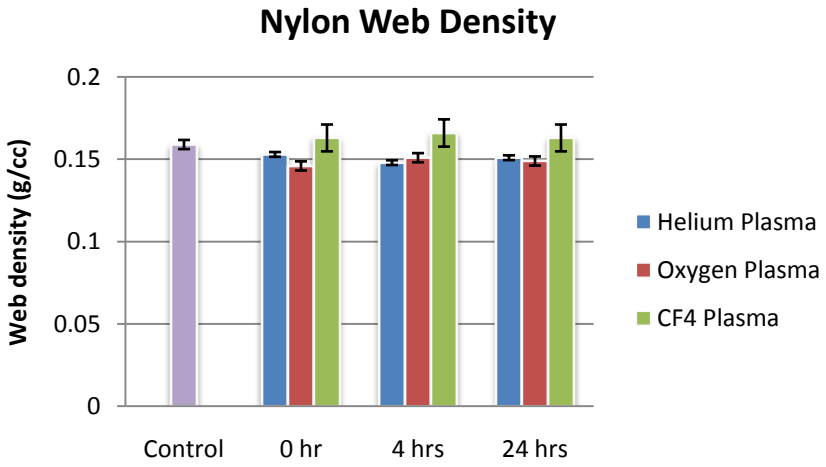


Figure 4.38: Web densities for Nylon webs

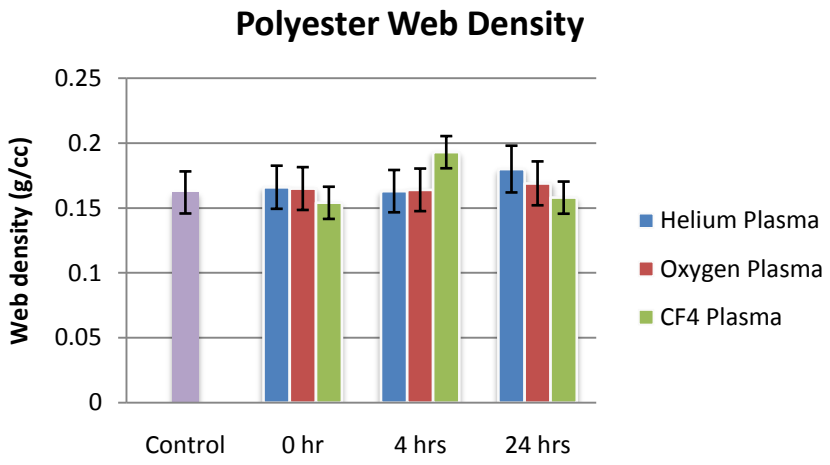


Figure 4.39: Web densities for Polyester webs

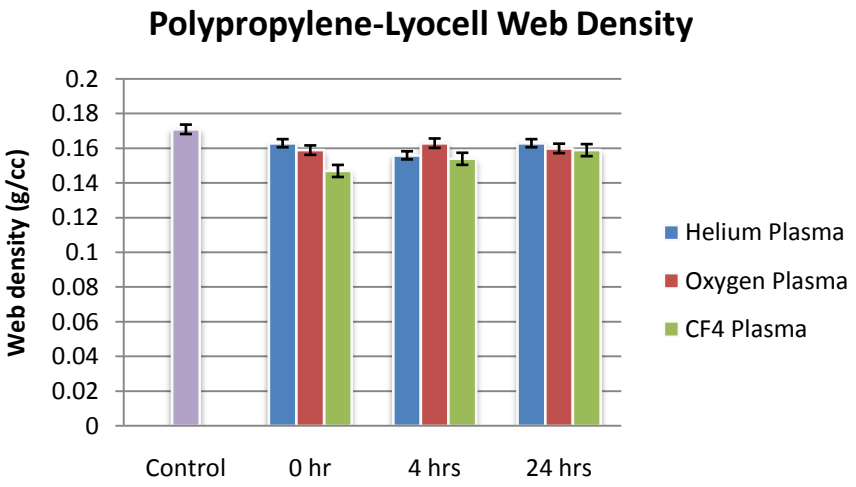


Figure 4.40: Web densities for Polypropylene-Lyocell webs

The tests for web densities suggested that in most cases, there was very limited weight loss due to plasma. This suggests that the reorientation is small not to change the density but big enough to alter secant modulus and elongation at break values.

4.10 Orientation Distribution Function

Orientation distribution function (ODF) is a parameter which determines the distribution of fibers within a range of angles in a nonwoven fabric structure. ODF determines the dominant angle in which the fibers are oriented. The dominant angle of fiber orientation influences many mechanical properties such as tensile strength, tear strength, bending rigidity etc. ODF is determined by capturing digital images which provides a discrete input function. This function is then converted into a frequency function by a complex algorithm by Fast Fourier Transform (FFT) analysis [4]. The results of ODF analysis are presented in the form of a table/plot which depicts the percent frequency of fibers in a range of angles also known as bin range.

ODF test was performed on controls and webs hydroentangled immediately after plasma treatment.

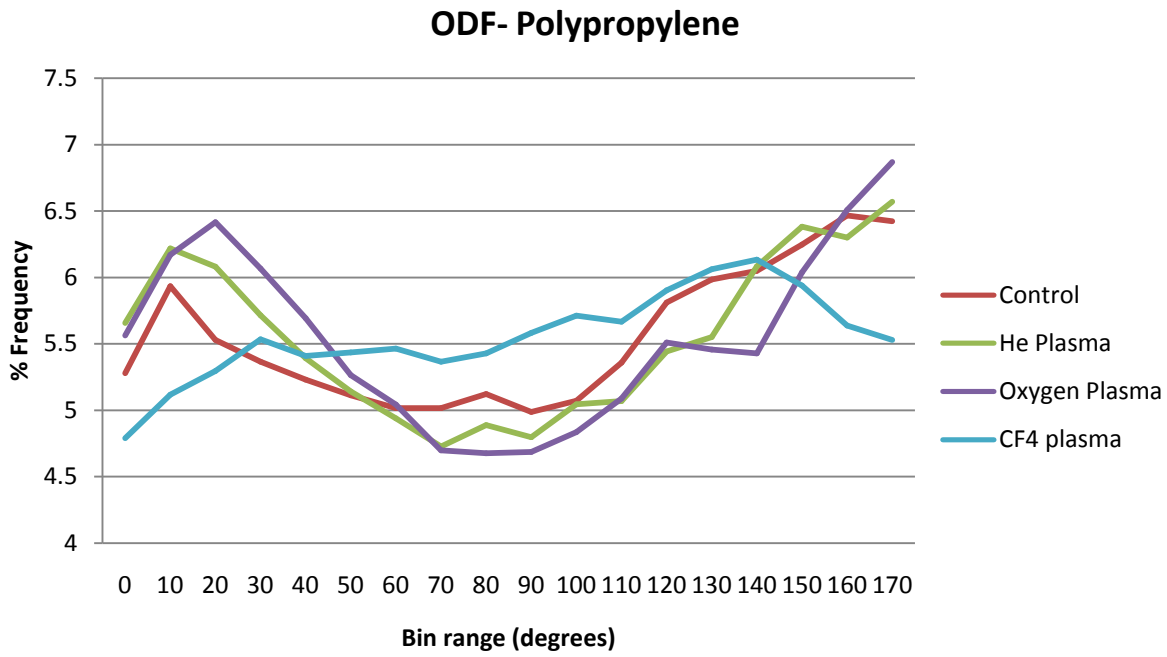


Figure 4.41: A comparison of ODF for Polypropylene webs with and without plasma pretreatment

From the above graph it can be seen that there is a re-orientation of fibers treated with helium-CF_4 plasma specifically in bin ranges from 50 to 120 suggesting that there is significant rearrangement of fibers in machine direction. For helium-oxygen plasma system, the rearrangement has taken place in bin ranges 10 to 40 which means that fibers are more oriented in cross direction. The rearrangement of fibers may because of plasma-substrate interaction. Plasma is known to introduce functional groups onto a surface. Because of added functionality and surface energy, the interaction of fibers with forming wire and amongst themselves is likely to change in terms of adhesive and cohesive forces respectively. If the adhesive forces are enhanced, the force resisting the peeling of web from the forming wire

would be higher and this may cause a reorientation in MD. However, if cohesive forces are enhanced, the fibers are likely to stick together in the directions they are cross-lapped in.

Depending on the type of substrate and the plasma system, one of the forces may be dominant and the final web may have different orientation distribution as seen in the ODF graphs.

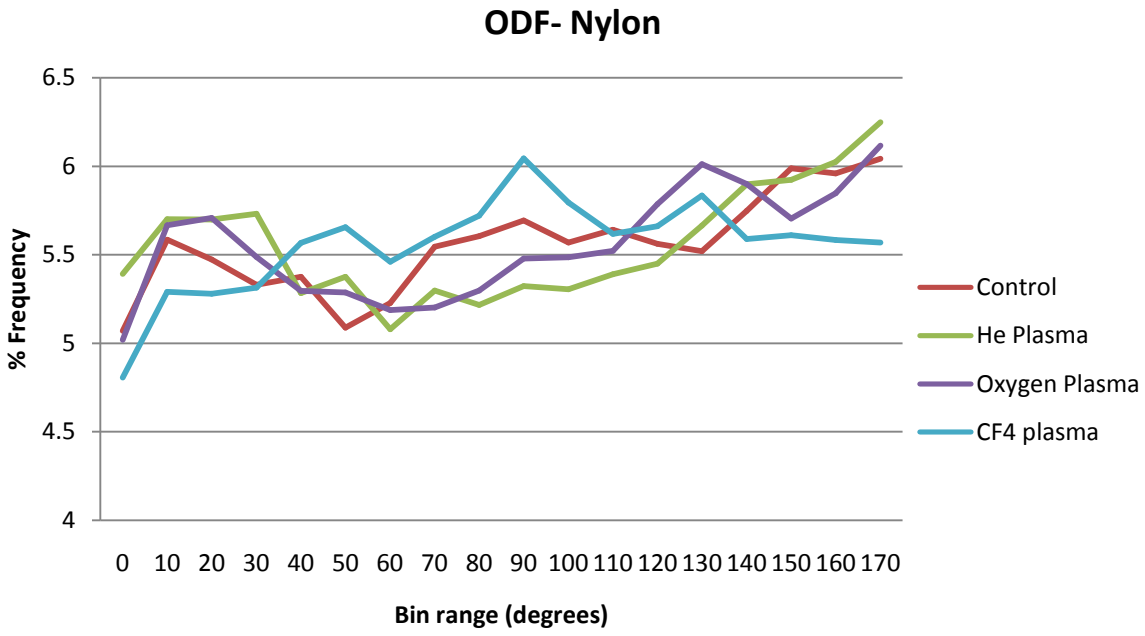


Figure 4.42: A comparison of ODF for Nylon webs with and without plasma pretreatment

As seen in the above graph, helium and helium-oxygen plasmas have not caused nylon fibers to re-orient in any other dominant direction compared to the control. Helium-CF_4 plasma appears to have a significant effect in bin ranges 40 to 90 suggesting that there is more orientation in MD compared to the control.

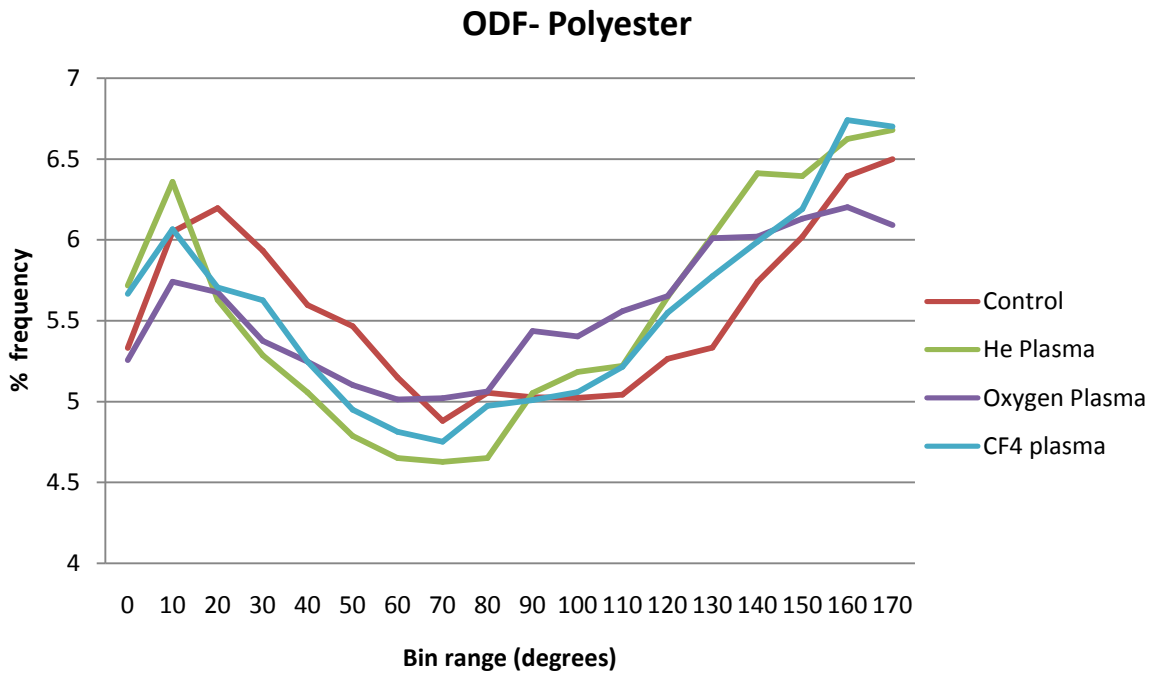


Figure 4.43: A comparison of ODF for Polyester webs with and without plasma pretreatment

The ODF data for polyester shows a reorientation of fibers in MD especially for helium-oxygen plasma system (bin ranges: 90-130). For pure helium plasma, the reorientation appears to be in CD (bin ranges: 0 -10 and 120-170). The curve for helium-CF_4 plasma system has flattened at the left end but an increase in the bin ranges from 120-170 suggests an orientation in CD.

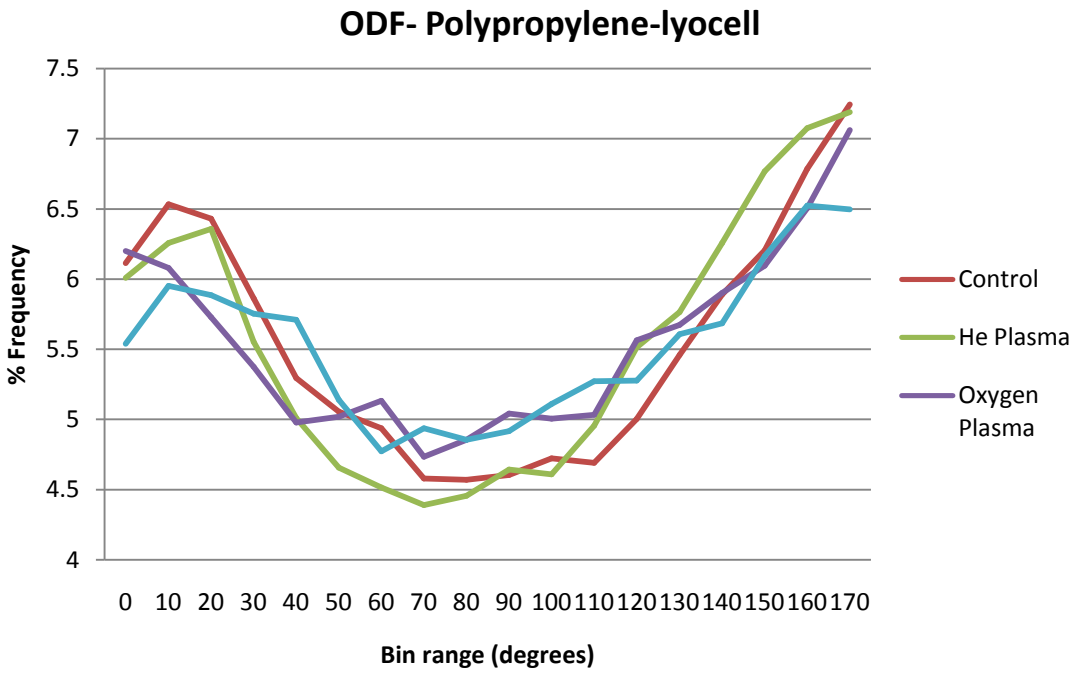


Figure 4.44: A comparison of ODF for Polypropylene-Lyocell webs with and without plasma pretreatment

From the above plot it may be seen that there is a significant reorientation in MD for webs treated with reactive plasmas. The curves for helium-oxygen and helium-CF_4 plasmas have in general flattened at both the ends and risen in the middle of the bin ranges especially between bin ranges from 70 to 120. Also, from the software statistics, the overall cosine squared anisotropy values are as tabulated in table 4.1:

Table 4.1: Cosine squared anisotropy values

	Control	He Plasma	He-O_2 Plasma	He-CF_4 Plasma
Polypropylene	-0.059	-0.074	-0.076	-0.048
Polyester	-0.058	-0.085	-0.051	-0.072
Nylon	-0.029	-0.044	-0.044	-0.028
PP-Lyocell	-0.105	-0.117	-0.061	-0.061

The values are quite close to 0 indicating that the webs are quite random. (Cos squared anisotropy close to 1 or -1 indicates a perfectly oriented web while it being close to 0 indicates randomness).

From the above ODF data, no significant changes in the frequency can be noticed. The plasma induced roughness as well as secondary bonding between the fibers may be responsible to make fibers even more difficult to move with respect to one another and therefore, the orientation is almost fixed in the state that the web had acquired when the web was first carded and cross-lapped.

4.11 Fiber Pullout Force

A test was devised in which the force required to pull a single fiber out of the nonwoven structure was measured. In this test, nonwoven fabric was firmly held by the bottom grip of the Instron and a single fiber was carefully attached to the top grip with a pair of tweezers. This fiber was pulled out of the fabric by applying a constant crosshead speed on 15mm/min. The force required to pull this fiber out is a function of fiber-to-fiber friction, elastic properties of the fiber and the total length pulled out of the structure.

Pullout force of individual fiber compares with the application of fabric tensile properties. In both the tests, fibers are subjected to similar forces. The typical force versus crosshead curve for a pull-out test is shown in Figure 11. The maximum force required to pull the fiber out is measured and divided by the staple length to disregard the effect of staple length on the pull-out force for better comparison. Recalling that previous results have shown that plasma treatment is not modifying fiber tenacity and modulus significantly, the change in pullout force can be related to change in fiber surface roughness. The pull-out force thus, is a measure of static friction which is the threshold force required to pull the fiber out of the structure.

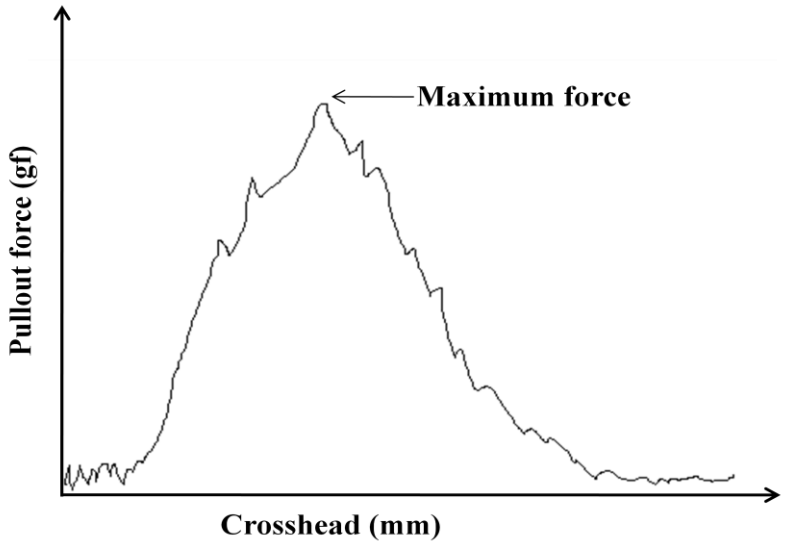


Figure 4.45: Typical curve for fiber pull-out force

To confirm the role of surface roughness, control fabrics were treated with plasma and fiber pullout test was performed on the same. It was found that fiber pullout strength for these fabrics was significantly more than the controls as well as the plasma pre-treated fabrics.

Plasma is known to incorporate surface roughness due to processes such as etching, ablation, surface cross-linking, re-deposition etc. The roughness is imparted on different scales ranging between nano- to micro-scales. The important conclusion derived from the pull-out tests is that the scale at which roughness has been introduced has an effect on the final mechanical properties of the nonwoven fabrics. This scale of roughness has an influence on the entangling efficiency of the fibers which is ultimately impacting the fabric properties.

There are also secondary forces existing between the fibers due to functionality induced on the fiber surface due to plasma treatment. The functional groups can give rise to forces such as hydrogen bonding and van der Waal forces which may also contribute towards increased pull-out strength.

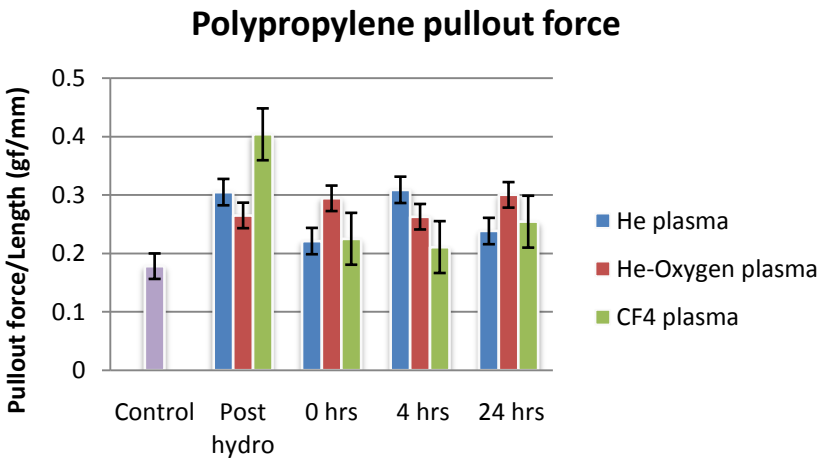


Figure 4.46: Change in fiber pullout force for Polypropylene webs

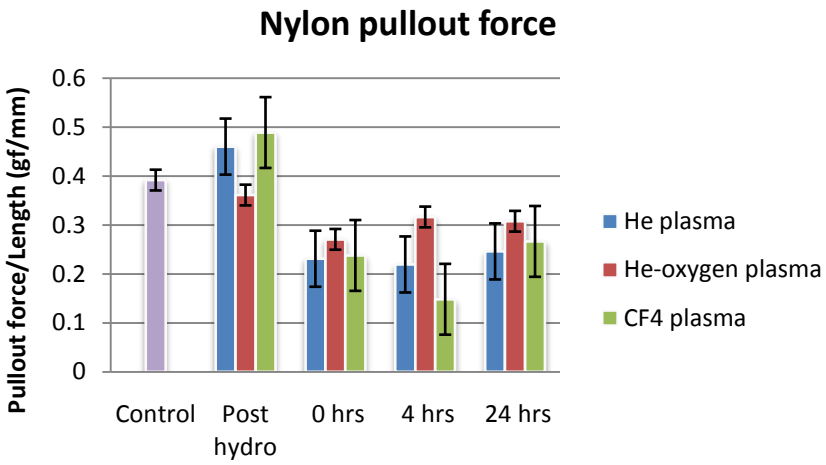


Figure 4.47: Change in fiber pullout force for Nylon webs

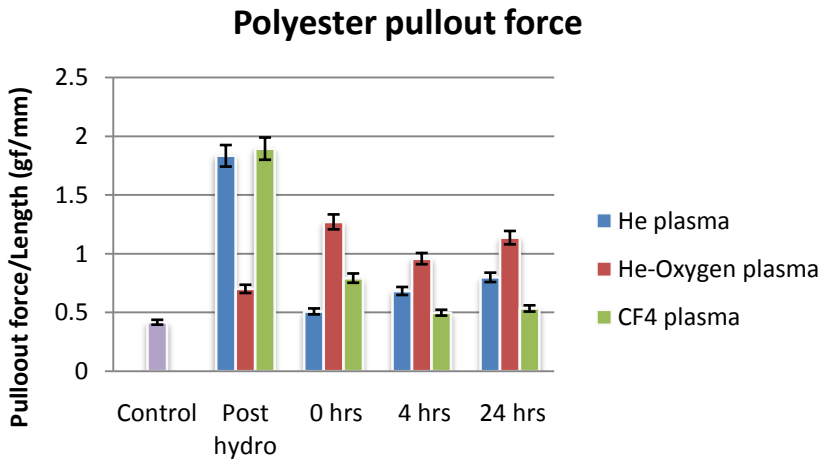


Figure 4.48: Change in fiber pullout for Polyester webs

Atmospheric plasma is a known technology for surface treatment of various substrates for a multitude of end uses [5].

Atmospheric plasma treatment has also been shown to cause significant changes in the wettability/hydrophilicity of fibers because of the modified chemistry at the fiber surface [6, 7]. Plasma also causes drastic changes in surface roughness properties due to a variety of effects including etching, re-deposition, surface cross-linking, diffusion of oligomers to the surface etc. [8]. The effect of these changes on the hydroentangling properties of the fiber were meticulously investigated in our work. The ultimate effect was found to be a non-trivial combination of the above mentioned properties. It is usually imagined that increasing the wettability of the hydrophobic fibers would lead to improved hydroentangling efficiency. The argument that is often presented in support of this hypothesis is that the improved wettability of the hydrophobic fibers leads to elimination of the pooling effect during the hydroentangling process, resulting in improved properties of the nonwoven webs. However, our results suggest a different relationship between plasma treatment and improvement in tensile properties of the nonwoven webs. The data indicates that the surface roughness introduced during plasma treatment may play a key role in determining the measured mechanical properties of the hydroentangled webs; however, the effects of plasma treatment on the hydroentangling process itself are complex. For hydrophobic fibers, the effect of plasma treatment on hydroentangling may be a result of two counter-acting effects:

1) improved wettability produced because of introduction of ions and free radicals at the surface, and

2) higher contact angle caused by increased surface roughness.

The net effect of plasma treatment was found to depend on the type of fiber and its degree of hydrophobicity and it is discussed in further detail in the following sections.

Polypropylene

Amongst all the fibers studied in this work polypropylene (PP) is most hydrophobic. Multiple studies in our laboratory [2, 6, 7, 8] have demonstrated that plasma treatment of PP significantly increases the hydrophilicity and surface functionality. The hydrophilicity of the webs was characterized by vertical wicking test and absorbency tests of post-hydroentangled plasma treated webs which serve as a model for the pre hydroentangled plasma treated webs, which cannot be tested due to their inherently loose structures. It was concluded that the plasma treated webs were significantly more hydrophilic compared to the controls even after 24 hours of aging. Visual observation of the hydroentangling suggested that water-pooling was completely eliminated for plasma-pretreated webs compared to the controls which showed significant water pooling. In addition to the hydrophilicity, plasma also introduced a high degree of micro-scale surface roughness. The roughness is evident in the SEM pictures. Fiber pullout tests were conducted to understand the role of surface roughness in affecting the ultimate web properties. Increased pull-out strengths of fibers indicated an increased fiber-to-fiber friction. Plasma pre-treated webs demonstrated lower tensile and trapezoid tear strength where individual fibers had higher pull-out strength while tenacity and initial modulus remained unchanged. This established that the scale of the introduced roughness has an influence on the entanglement of web and subsequently, the final web properties.

The tenacity of the fabrics enhanced significantly in MD and displayed a corresponding decrease in CD for all three plasma systems. Elongation at break decreased in MD and either did not change significantly or slightly decreased in CD. The results for fabric tenacity and elongation at break suggested re-orientation of fibers in MD. An enhanced fibers orientation in MD is likely to enhance fabric tenacity or secant modulus whereas it will cause a decrease in elongation at break.

The results for ODF showed an increased orientation in MD for CF_4 plasma treated webs specifically in bin ranges from 60 to 110 degrees. The webs pre-treated with helium and helium-oxygen plasmas did not appear to have changed dominant orientations in any particular bin range. The air filtration properties were enhanced significantly for plasma pre-treated webs. The air permeability and web density results did not change significantly due to plasma treatment. Previous studies [9] suggest that increased surface area of fibers due to plasma induced surface roughness increases air filtration. Our experimental work supports previous studies relating fiber surface area and filtration, but the roughness has been confirmed by fiber pullout tests.

This led to a hypothesis that the fibers are more resistant to entanglement due to increased fiber-to-fiber friction which was further supported by fiber pull-out strength testing of webs treated with plasma after hydroentangling. These webs showed higher fiber pull-out strengths compared to control and plasma pre-treated webs. This established that increasing fiber roughness or fiber-to-fiber friction before hydroentangling leads to higher resisting

force to entanglement which may be responsible for inferior mechanical properties. It may also be added that change in fiber-to-fiber friction is unlikely to contribute towards change in air permeability and web density but extremely likely to increase fiber surface area and hence the air filtration efficiency. Two more forces which are likely to appear due to plasma induced functionality are:

(1) fiber-to-fiber cohesion, and

(2) fiberweb to carrier mesh adhesion.

The fiber-to-fiber cohesive force may arise due to introduction of functional groups such as hydroxyl and carboxyl groups. These functional groups are capable of forming H-bonds and also enhance chemical cross-linking and van der Waal's forces. The fiberweb to carrier mesh adhesive force may appear due to change in surface energy and increased functionality of the fibers due to plasma. Due to the increased adhesive force between fiberweb and carrier mesh, the peeling force is resisted causing a slight re-orientation of fibers in the direction of peeling, or MD. Depending on the interaction of a particular substrate and plasma, these forces are likely to vary in magnitude. It may be hypothesized that due to adhesive forces, there may be re-orientation of fibers in the MD which is observed in ODF results; increased fabric tenacities and decreased elongation at break in MD. All these points were well supported by our experimental results discussed above.

Polyester

Like polypropylene, polyester demonstrated a decline in tensile and trapezoid tear strengths. The individual fibers showed no change in tenacity and modulus while the fiber pull-out strength was significantly higher.

As suggested by the SEM pictures, oxygen plasma has a significant effect on fiber surface. This result falls in congruence with the filtration efficiency, which showed an improvement for He-O_2 plasma alone. The web density and air permeability were not significantly altered. The ODF of polyester nonwoven fabrics showed an increased orientation of fibers in MD. Specifically, for all plasma pre-treated webs, a significant reorientation was observed in bin ranges 90 to 140 degrees.

The orientation may be a result of secondary bonding between fibers and carrier mesh. Therefore the hypothesis proposed for polypropylene holds good for PET.

Nylon

Absorbency and vertical wicking results established that plasma makes nylon much more hydrophilic compared to the control as well as other fibers used in this study. Nylon displayed a significant decline in tensile and trapezoid tear strength of the web while the individual fibers displayed no change in tenacity and initial modulus. The SEM images suggested very less change in surface roughness and this was further confirmed by a decrease

in fiber pull-out strength. The air filtration efficiency of the nylon webs dropped while web density and ODF recorded no change but web air permeability increased considerably. This may probably be because of the surface chemistry introduced by the plasma, which results in secondary bonding such as hydrogen bonds [10, 11] and van der Waal forces. The H-bonds, if formed in excess can be very strong as they have strength up to 10% of a covalent bond. This may increase adhesion between adjacent fibers resulting in a poor entanglement and an increased permeability because the fibers are more likely to be closer to one another. This adhesion is unlikely to change web densities but likely to influence the average pore size of the web because of fiber bundles sticking together. A thorough visual observation indicated that the plasma pre-treated nylon webs were non-uniform compared to the controls and appeared to have more inhomogeneous structure. However, more testing and analysis needs to be carried out to confirm this hypothesis. The ODF results showed that helium-CF_4 plasma treated webs exhibited enhanced orientation in MD specifically in bin ranges 40 to 110 degrees. This may be due to increased adhesive forces between fibers and carrier mesh.

Polypropylene-Lyocell Blend

The blended webs displayed a much better hydrophilicity compared to the controls. The tensile and trapezoidal tear strengths declined but the individual fiber tenacity and initial modulus remained unaffected. The web densities and air permeability exhibited no significant effects while the air filtration increased considerably. The ODF results showed a considerable enhancement in orientation in MD especially for webs treated with helium-

oxygen and helium-CF_4 plasma in bin ranges 60 to 120 degrees. These webs showed a behavior similar to 100% PP webs and had most of the results almost congruent to those of PP webs. Also, because of incorporation of Lyocell fiber which is closer to Nylon in behavior, there may be significant secondary bonding between the fibers.

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Chapter 5

Summary, Conclusions and Future Recommendations

Plasma treatment can significantly modify polymer surface chemistry and morphology. While typically thought of as a surface treatment, plasma can significantly affect fiber modulus and tenacity by way of chain scission or surface cross-linking. Depending on the type of gas used, plasma treatment can produce different surface chemistry combinations. The ultimate properties of a plasma treated web are dependent upon the specific plasma substrate interactions for each treatment regime.

This study demonstrates that atmospheric plasma pretreatment of carded webs has an effect on the hydroentangling process which can be significant depending on the various processing parameters such as feed gas, fiber material etc. For all substrates tested, changes in web and fiber tensile properties, as well as changes in filtration efficiency are evident. These changes have a strong dependence on aging time post-plasma treatment, prior to hydroentangling. In most cases, the best results are seen at maximum aging times compared to no aging. It is apparent that surface changes brought about by plasma which are known to decrease with aging such as wettability/ hydrophilicity or introduction of reactive species on the surface such as radicals, ions and functional groups, do play an important role in hydroentangling process. From the variety of tests performed, the role of fiber-to-fiber friction is apparent in affecting the overall properties of hydroentangled webs.

The most critical conclusions drawn from this study are:

1. The effect of plasma on the ultimate web properties depends on the type of fiber.
2. Fiber surface roughness affects entangling and the ultimate web properties.
3. Excessive hydrophilicity may enhance secondary bonding and hence, enhance fiber-to-fiber cohesion and fiber-to-carrier mesh adhesion.
4. Ultimate web properties are a function of hydrophilicity and fiber surface roughness.

This project has opened many avenues for research on fiber-to-fiber and fiber-to-wire mesh interactions. The role of plasma is not completely modeled and requires future consideration. The author suggests quantification of fiber-to fiber frictional force and its role in altering hydroentangling efficiency and the final web properties. A study on the effect of concentration of reactive gases may also be useful in explaining the effects of particular plasma on specific fibers and their combined effect on hydroentangling efficiency.