An Update on the Technology and Application of Plasma Treatment for Textiles

A dissertation work submitted to School of Textiles, University of Borås, in Partial Fulfillment of the Requirements for the Degree of MSc. in Mechanical Engineering with specialization in Textile Technology

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An Update on the Technology and Application of Plasma Treatment for Textiles

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**Synopsis**

The thesis treatise can be divided in three major parts as A, B and C respectively.

Part A constitutes the concept and objectives of the plasma treatment for textiles. It includes a technical overview, the principle and the recent developments in plasma types for textiles.

Part B, provides an overview on plasma technology to its interaction with the substrate. The part describes the surface modification phenomenon i.e. physical and chemical interaction and the highlights the effects yields from it.

Part C, constitute a bibliographic analysis on the application of this technology to textiles. Various paper and patents and cited to provide an overview on key aspects of the scientific research, commercial technology, and information on manufacturers which are being taken place to date. The section followed then critical dichotomy appraisal on the plasma technology in the field.

All the citation, are thoroughly questioned and evaluated before own use of the material in this study. Also, the citation has been taken from reliable sources. In order to increase the reliability of the material comparison between sources is done

**Background**

At the outset, the main aim of this treatise is to build a theoretical background on the potential of Plasma Technology for Textiles. The study is critically elaborated to understand the possibility and the capability of the technology to discern on real time practice. As it parts, later, if there is an opportunity would came to exist. The design of experiment would be easy to setup in agreement of academia and industrial partner to obtain the experimental results and further implementation.
1 Introduction

Plasma is an ionized form of gas. It contains electrons, ions and neutral atoms and molecules. The application area is huge and diverse, found application in niche applications in many industrial areas including, polymers, paper, metals, ceramics and in-organics, biomaterials (Shishoo, Introduction – The potential of plasma technology in the textile industry, 2007), electronic equipments. And now plasma technology is finding its promising position in textile as well. Predominantly, Table 1 tabulates the perceived advantages of plasma processing over wet processing with respect to Atmospheric pressure plasma.

The potential of plasma technology for textiles has been widely described in scientific, technical and industrial literatures. The functionalities induce by plasma includes

- Improve physical properties of substrate (Cheng-Chi Chen, 2010)
- Improved hydrophilic and wicking characteristics (Carneiro, et al., 2001) (Marija Gorensek, 2010)
- Increased chemical reactivity of the fiber surface (Goto, 1991)
- Adhesion enhancement (Marija Gorensek, 2010)
- Improve polymer matrices (Gakkaishi, 2009)
- Sterilization (Negulescu, et al., 2000),
- Plasma induced hydrophobic properties
- Fiber surface cleaning, removal of thin films of organic impurities (Radetić, et al., 2008) (Köchler & Fritzshe, 2007)
- As a precursor leading to other surface modification techniques (John & Anandjiwala, 2009)
- In smart applications as (Herbert, 2007)
  - Smart/responsive surfaces, e.g. F + PEG – in air, stain-repellent F on surface, in water, stain removing PEG on surface
  - Trapped active coatings
  - Conductive coatings (Herbert, 2007) with improved crocking fastness
Table 1 Perceived advantages of plasma processing over wet processing, particularly with respect to Atmospheric Pressure Plasma (Herbert, 2007)

<table>
<thead>
<tr>
<th>Manufacturing operation</th>
<th>Conventional</th>
<th>Plasma processing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Handling and storage of bulk chemicals</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Mixing of chemicals, formulation of baths</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Use of water</td>
<td>Heavy</td>
<td>None or very low</td>
</tr>
<tr>
<td>Raw materials consumption</td>
<td>High</td>
<td>Low</td>
</tr>
<tr>
<td>Drying ovens and curing operations</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Need for solvents, surfactants, acids</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Number of process steps</td>
<td>Multiple</td>
<td>Single</td>
</tr>
<tr>
<td>Energy consumption</td>
<td>High</td>
<td>Very low</td>
</tr>
<tr>
<td>Waste disposal/recycling needs</td>
<td>High</td>
<td>Negligible</td>
</tr>
<tr>
<td>Environmentally costly</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Equipment footprint</td>
<td>Large</td>
<td>Small</td>
</tr>
<tr>
<td>Manufacturing versatility from single kit</td>
<td>Limited to single or few process options</td>
<td>Depending on kit, can be highly flexible with wide range of available processes</td>
</tr>
<tr>
<td>Innovation potential</td>
<td>Moderate</td>
<td>Very high</td>
</tr>
</tbody>
</table>

2 The principle
A gas is in general is an electric insulator. However, when a high voltage is applied across a gap containing a gas or gas mixture, it breaks down and conducts electricity. (Bryant, 2007). As a consequence of this, the gas becomes ionize, and splits into electrons, ions, neutral atoms and molecules.

Also, in (Shishoo, 2007) describes as, when the coupling of electromagnetic power into a process gas volume generates the plasma medium which comprises a dynamic mix of ions, electrons, neutrons, photons, free radicals, meta-stable excited species and molecular and polymeric fragments, rather affecting their bulk properties. These species move under electromagnetic fields, diffusion gradients, etc. on the textile substrates placed in or passed through the plasma. This enables a variety of generic surface processes including surface activation by bond breaking to create reactive sites, grafting of chemical moieties and functional groups, material volatilization and removal (etching), dissociation of surface contaminants/layers (cleaning/ scouring) and deposition of conformal coatings can be achieved.

3 Types of Plasma
Though, the plasma exists in many forms. But, not are applicable to textiles. However, the typical route to textile applicable plasma is being described in later section(s). For easy understanding, the types of plasma can be illustrated as shown in figure 1.
3.1 Thermal Plasma

It is a type of plasma that has a exceptionally high temperature. That is, temperature of several thousand degrees. This plasma is characterized by a condition of thermal equilibrium between all the different species contained in the gas. In fact, if the gas density is sufficiently high, the frequency of collisions between electrons, ions, and neutral species composing the plasma is such that an efficient energy exchange is possible.

Such type of plasma can be observed in stars, sun and other celestial bodies. It can also be seen on earth as flash lightening, since, no material can withstand against such
intrinsic destructive nature (Marcandalli & Riccardi, 2007), particularly if addressing Textile. Thus, it is not the topic of our discussion.

Besides, another fascinating type of plasma can be observed in Polar zones known as Aurora borealis and Aurora australis. Occurs, when the solar wind is get more attractive to the poles due to high polarity. So, the particles reach the atmosphere at high altitude excite the molecules, ions and atoms, which they end up in a more energy-rich state. When they return to their normal condition and emits the extra energy is emitted plasma in different colors.

### 3.2 Low Temperature Plasma (Cold Plasma)

There are two types of plasma which can be used for application on textiles, namely vacuum pressure and atmospheric pressure. Since plasma cannot be generated in a complete vacuum the name vacuum pressure is somewhat misleading and only refers to the low working pressures of such systems. Many authors, however, choose to classify vacuum pressure plasmas into sub categories of low and medium pressures (Li & Hsueh, 2005); (Einagar, Kh.; et al, 2006); (De Geyter, Morent, & Leys, 2006). Table 2 gives an idea of the working pressures of vacuum and atmospheric plasmas.

<table>
<thead>
<tr>
<th>Pressure</th>
<th>Atmosphere</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low</td>
<td>0 – 0.29</td>
</tr>
<tr>
<td></td>
<td>0 – 2.175</td>
</tr>
<tr>
<td></td>
<td>0 0.003</td>
</tr>
<tr>
<td></td>
<td>0 0.0029</td>
</tr>
<tr>
<td>Vacuum</td>
<td>0.3 – 7</td>
</tr>
<tr>
<td>Medium</td>
<td>2.25 – 52.5</td>
</tr>
<tr>
<td></td>
<td>0.003 0.069</td>
</tr>
<tr>
<td>Atmospheric</td>
<td>101.3</td>
</tr>
<tr>
<td></td>
<td>760</td>
</tr>
<tr>
<td></td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>1.013</td>
</tr>
</tbody>
</table>
Figure 2 provides an idea of the variation in the existence of plasma as the current is increased. The regions marked as dark and glow discharge are normally suitable for surface modification. The corona region of dark discharges is used in atmospheric plasmas while vacuum pressure plasmas usually lie in the glow discharge region. Arc discharges, due to heavy bombardment of the cathode at high currents attain temperatures which are too high for safe surface modification techniques (Reichel, 2001). The section will thus discuss vacuum and atmospheric plasmas which have been realized as suitable for application on textile substrate.

![Figure 2 Voltage current characteristics of the classical DC intermediate pressure electrical discharge tube (Roth, 2001)](image)

### 3.2.1 Vacuum pressure plasmas

If a voltage is applied across a nearly evacuated gas chamber, under appropriate conditions, plasma will ignite (Reichel, 2001). Changes in these conditions vary the effect and appearance of the plasma.

Vacuum pressure treatments are generally used to achieve varying outcomes of textile substrate. These plasmas will either etch or form radicals on the surface of the processed material.
Vacuum pressure plasma systems have certain limitations adhered with them in terms of commercial application. The vacuum creating equipment adds to the cost of treatment and is expensive to run. Also, the operating pressure range allows only for batch processing of material to be possible.

There are certain advantages in terms of application such as etching and coating which can be performed better under low pressure plasmas.

### 3.2.2 Atmospheric pressure plasmas

As the name suggests, these systems process materials at atmospheric pressures thereby increasing the processing capabilities of the machine while reducing processing costs and loading times.

Different types of cold plasma can be described as: (Marcandalli & Riccardi, 2007)

#### 3.2.2.1 Dielectric barrier discharge

DBD is an atmospheric-pressure plasma source. Figure 3 shows a schematic of DBD. In this case a symmetrical electrode arrangement is set up comprising two parallel conducting plates placed in opposition, separated by a gap of ~10 mm, and a high voltage, 1–20 kV, is applied, the gas between the plates can be electrically broken down and a plasma discharge generated (Herbert, 2007).

![Dielectric barrier discharge](image)

*Figure 3 Dielectric barrier discharge (Mathews, 2005)*
Consequently, a pulsed high voltage is applied between electrodes, one or both of which is covered by a dielectric layer. The purpose of the dielectric layer is to terminate rapidly the arcs that form in the region between electrodes. The discharge consists of series of rapid micro discharges.

### 3.2.2.2 Atmospheric Pressure Glow discharge

This is obtained at low pressures (~200 V), typically less than 10 mbar. The plasma is generated by antennas, fed with electromagnetic fields at frequencies of 40 kHz or 13.56 MHz or microwaves (2.45 GHz). Figure 4 illustrate a schematic of Glow Discharge Equipment. The APGD is denser than the DBD, with typical free electron densities of 10^{11}–10^{12} electrons/cm³, but the free electrons are slightly cooler at temperatures of 10 000 to 20 000 K. Textile treatment temperatures can run at 25–50°C.

![Figure 4 Glow discharge plasma (Mathews, 2005)](image)

### 3.2.2.3 Corona discharge

This is obtained at atmospheric pressure by applying D.C., low frequency or pulsed high voltage between two electrodes of very different sizes. The corona consists of a series of rapid, non-uniform, non-arcing discharges. Plasma density drops off rapidly
with increasing distance from the electrode. The basic configuration of corona discharge is shown in figure 5.

![Schematic diagram of Corona Discharge (Mathews, 2005)](image)

**Figure 5 Schematic diagram of Corona Discharge (Mathews, 2005)**

It has one limitation in corona systems that it affects only in loose fibers and cannot penetrate deeply into yarn or woven fabric so that their effects on textiles are limited and short-lived. Essentially, the corona plasma type is too weak. Corona systems also rely upon very small inter-electrode spacing (-1 mm) and accurate web positioning, which are incompatible with ‘thick’ materials and rapid, uniform treatment (Shishoo, Introduction – The potential of plasma technology in the textile industry, 2007)

### 3.2.2.4 **Atmospheric pressure plasma jet (APPJ)**

This a non-thermal, atmospheric pressure, glow discharge plasma produced in continuously flowing gases, as referred from (Marcandalli & Riccardi, 2007). This technology enables plasma to be applied to textile fabrics in the *in-situ*¹ mode in which the fabric is passed through the plasma generation region between electrodes (Herbert, 2007)

---

¹ Being in the original position
The chain of major characteristic applications by atmosphere plasma techniques is illustrated in figure 6, while table 3 tabulated lists the surface engineering processes delivered by each of the treatments (Herbert, 2007)

The table 3, emphatically does not state that the treatments are equivalent. It simply says that each treatment can, to certain materials and given the right conditions, carry out the generic process to some degree. The power and sophistication of the treatments for each APP type is, in general, listed in ascending order in the table.

Figure 6 The chain of Atmospheric Plasma to Surface Engineering (Herbert, 2007)

Table 3 The surface engineering processes that can be delivered by each of the three APP types useful for textile processing (Herbert, 2007)

<table>
<thead>
<tr>
<th>APP types</th>
<th>Treatments</th>
<th>Surface engineering processes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corona</td>
<td>Ambient air treatment</td>
<td>Cleaning Activation</td>
</tr>
<tr>
<td></td>
<td>Controlled atmosphere</td>
<td>Etching</td>
</tr>
<tr>
<td></td>
<td>treatment</td>
<td>Cleaning Activation</td>
</tr>
<tr>
<td></td>
<td>Gas precursor coating</td>
<td>Coating</td>
</tr>
<tr>
<td></td>
<td>Liquid precursor coating</td>
<td>Coating</td>
</tr>
</tbody>
</table>
4 Plasma surface modification routes

Two major routes can be considered as surface engineering of textile substrate through plasma:

- **Plasma – Physical Surface modification route**
- **Plasma – Chemical Surface modification route**

The interaction of plasma with substrate occurs, when the reactive species (positive and negative ions, atoms, neutrals, meta-stables and free radicals) are generated by ionization, fragmentation, and excitation. These species lead to chemical and physical interactions between the plasma and the substrate surface depending on plasma conditions such as gas, power, pressure, frequency, and exposure time. The depth of interaction and modification, however, is independent of gas type and is limited. (Mathews, 2005) (Rakowski, Okoneiwski, Bartos, & Zawadzki, 1982)

As mentioned earlier, plasma is used in no. of ways to synthesis and modifications the substrate surface as, removal of thin films of organic impurities (Radetić, et al., 2008) (Köchler & Fritzshe, 2007), selective etching of composites (Wang, Ren, & Qiu, 2007) (Akishev, Grushin, Monich, Napartovich, & Trushkin, 2003), sterilization (Negulescu, et al., 2000), passivation of metals (Costa, Feitor, Alves, & Freire, 2006), ashing of biological materials (Park, 2008), etching of photo-resists (A., Raffaele-Addamo;, 2006), functionalization of polymers (Vesel, 2008), and
conditioning of tokamaks with carbon walls (Makabe, 2006). The choice of discharge parameters is determined by the requirements of each particular application.

Though there are number of opportunities exist for plasma treatment besides an only few have been graphically modeled in figure 8, and in figure 7 the possible mechanism of interaction of Plasma to substrate.

Figure 7 Mechanisms of Plasma-Substrate Interaction (Mathews, 2005)
4.1 Plasma – Physical Surface modification route

4.1.1 Etching

Surface Etching is most common phenomenon/technique in Plasma – Physical Surface modification route. Plasma etching is the key process for the removal of surface material from a given substrate. This process relies on the chemical combination of the solid surface being etched and the active gaseous species produced in the discharge. The resulting etched material will have a lower molecular weight and the topmost layer will be stripped. In previous methods, such as chemical wet processing, plasma has shown much more controllability and a much finer resolution (Mathews, 2005) (Chapman, 1980). The etching process conditions configure changes in the frictional properties of fibers. The mechanical properties change via an increase in tensile strength, bursting strength and wear resistance (Morent R. D., 2008).

However, the four basic plasma processes commonly used for surface removal are shown in Figure 9

![Figure 9](image.png)

Figure 9 The Four Basic Plasma Etching Processes: (a) sputtering, (b) pure chemical etching, (c) reactive ion etching, and (d) ion inhibitor etching. (Dennis M. Manos and Daniel L. Flamm, 1989)
The first process, sputtering, is a purely physical, unselective process. Energetic ions crossing the sheath transfer large amounts of energy to the substrate, resulting in the ejection of surface material. This mechanical process is sensitive only to the magnitude of bonding forces and structure of the surface, rather than its chemical nature (Tsai, 2005).

The second process, chemical etching, involves gas-phase etchant atoms or molecules formed through collisions between energetic free electrons and gas molecules, which stimulate dissociation and reaction of the feed gas. These etchants chemically react with the surface to form volatile products (Tsai, 2005). Chemical etching is the most selective kind of process because it is inherently sensitive to differences in bonds and the chemical consistency of substrate (Kan & Yuen, Plasma Technology in Wool, 2007). This process is invariably isotropic or non-directional (which is sometimes a disadvantage), since the gas-phase etchants arrive at the substrate with near uniform angular distribution. The etch rate for pure chemical etching can be quite large due to a high flux of etchants to the substrate (Ferreira, 2007).

The third is the combine process of both first and second process. Thus, have greater effect on the substrate. Reactive ion etching is characterized by a combination of physical sputtering and chemical activity of reactive species. In most situations, the chemistry in this process is provided by the neutral species. It is a directional etching mechanism where the impinging ions damage the surface of substrate and increase its reactivity. Whatever the microscopic details are (which undoubtedly can vary greatly from one surface/etchant system to another), the generic mechanism is one in which ions impart energy to the surface, which serves to modify it and to render the impact zone as well as is environment more reactive (Kan & Yuen, Plasma Technology in Wool, 2007). However, a basic reactive ion etch system proceeds by (i) generation active species in the plasma, (ii) transport of reactive intermediates from the plasma bulk to the substrate surface, (iii) absorption of reactive radicals and “active site”
formation, concluding with (iv) chemical reaction and desorption of volatile reaction products (Drenik, 2005).

The fourth technique of etching mechanism can be classified as inhibitor ion-enhanced requiring two conceptually different species that is etchants and inhibitor. The substrates and etchants in this mechanism will react spontaneously and etch isotropically, if it was not for the inhibitor species. The inhibitors form very thin film on surfaces that cease little or no ion bombardment. The film acts as a barrier to etchant and prevents the attacks of the feature sidewalls, thereby making the process anisotropic. (Kan & Yuen, Plasma Technology in Wool, 2007)

4.2 Plasma - Chemical Surface modification route (Mathews, 2005)

4.2.1 Radical formation

Formation of Radical sites occurs through ionization or excitation of the polymers through electrostatic when there is the interaction between fast moving electrons and the orbital electrons in the polymers.

The consequent ionization leads to molecular fragmentation (Separating into fine particles) and the formation of a free radical. Similarly, excitation leads to dissociation (i.e. removing from association) of the excited polymers, also forming free radicals.

\[
e^{-} + AB \rightarrow AB^{+} + e^{-} \quad \text{(Ionization)}
\]

\[
e^{-} + AB \rightarrow A^{+} + B + e^{-}
\]

\[
AB \rightarrow AB^{*} \quad \text{(Excitation)}
\]

\[
AB^{*} \rightarrow A^{-} + B^{-} \quad \text{(Dissociation)}
\]

If the free radicals formed are unstable, they will rapidly undergo recombination. Stable radicals, however, will remain in the polymer as living radicals. These radical sites can then “catalyze” the next steps for further chemical processing such as initiation of grafting, cross-linking, or functional group attachment.
4.2.2 Grafting

Plasma grafting, often referred to as plasma graft-copolymerization, can occur through either of the following two mechanisms [from (Mathews, 2005)]

1. The creation of active species on the polymer surface, followed by contact with the monomer:

   ![Diagram 1](image1.png)

   In this mechanism, free radicals are formed on the polymer surface as a result of inert gas plasma treatment. These radicals can either directly initiate grafting or be converted into peroxide or hydro-peroxides by the inclusion of an oxidative gas. These activated peroxides will also initiate grafting in the presence of the monomer species.

2. Direct grafting of the polymer with common or unconventional monomers under “monomer”-plasma conditions:

   ![Diagram 2](image2.png)

   Unlike the previous method, this involves a combined plasma and monomer exposure in one step by the use of gaseous monomers in the working gas mixture. Both of these techniques have shown great advantages over conventional grafting by offering a large
range of chemical compounds to be used as monomers, varying thickness of monomer layers, and limited destruction.

4.2.3 Polymerization

Plasma-induced polymerization can be defined as a film-forming process by which thin films are deposited directly onto the surface of a given substrate without any fabrication. The elemental reactions occurring during this process include fragmentation of monomer molecules, the formation of reactive sites (radicals), and recombination of the activated fragments. This mechanism follows similar steps to that of traditional radical polymerization with the inclusion of a possible re-initiation step. Apart, from typical polymerization describe above, table 4 lists some examples showing various application of plasma in polymer surface engineering.

Table 4 Examples of Polymer surface modifications via plasma polymerization (Kan & Yuen, Plasma Technology in Wool, 2007)

<table>
<thead>
<tr>
<th>Applications</th>
<th>Substrate</th>
<th>Monomer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesion</td>
<td>Polyamide</td>
<td>Allyl amine, propane epoxy, hexamethyldisiloxane</td>
</tr>
<tr>
<td>Adhesion</td>
<td>Polyethylene, poly(vinyl fluoride), polytetrafluoroethylene, poly(vinyl chloride)</td>
<td>Acetylene</td>
</tr>
<tr>
<td>Adhesion</td>
<td>Polyethylene, polycarbonate, poly(methyl methacrylate), polytetrafluoroethylene, polypropylene, ABS rubber</td>
<td>Tetramethylsilane, tetramethyltin</td>
</tr>
<tr>
<td>Adhesion</td>
<td>Polyethylene, polycarbonate, polytetrafluoroethylene</td>
<td>Tetramethylsilane + O₂, tetramethoxysilane</td>
</tr>
<tr>
<td>Surface hardening</td>
<td>Polyethylene sheet</td>
<td>Tetramethylsilane</td>
</tr>
<tr>
<td>Blood compatibility</td>
<td>Poly(ethylene teraphthalate)s</td>
<td>Acetone, ethylene oxide, glutaraldehyde, formic acid, allyl alcohol</td>
</tr>
</tbody>
</table>
4.2.4 Cross-linking

Cross-linking occurs when two polymer molecules join to form one large molecule/network. This occurs when radical sites are created in the polymer, resulting in the formation of H or Y-links. Cross linking can result in improved mechanical properties, decreased solubility, elimination of the melting point, and increased resistance to corrosive attack, all of which are desirable properties. During plasma exposure of polymeric materials, both chain-scission and cross-linking occur randomly and simultaneously. The predominance of one process over the other will depend on the polymer structure, crystallinity, temperature, and gas composition. If scission/etching is the dominating process, then degradation of the physical properties will occur, and the polymer may become unusable. For this reason, an exact balance must be obtained to control the competing processes.

5 Plasma applications

The application to plasma surface modification routes (which have been described above) are being described in the sections followed:

5.1 Hydrophobic Functionalization

Deposition of hydrophobic coatings on various materials has many important applications such as protective garments, water repellent textiles, corrosion prevention, and some other multilateral applications as micro-device lubrication, microfluidics, barrier coatings in biomedical systems, etc (Xie, et al., 2004); (Thieme, et al., 2001); (Shiu, Kuo, Chen, & Mou, 2004); (Riekerink, Engbers, Wessling, & Fei, 2002); (Ibnabddjalil, Loh, Chu, Blumenthal, Alexander, & Turner, 1994); (Zhai, Cebeci, Cohen, & Rubner, 2004); (Lau, et al., 2003). Among many techniques that can be utilized for this purpose, a plasma-based process is very attractive in many aspects. Plasma applications offer an alternative to conventional
processing/ modification techniques. That is surface modification of the substrate without changing/effecting on the bulk (Mukhopadhayay, Joshi, Datta, & Macdaniel, 20022), (Rickette, Wallis, Whitehead, & Zhang, 2004), (Cai Z. S., Qiu, Zhang, Hwang, & McCord, 2003) characteristics of the substrate/polymer. However, the lasting effect of the treatment is yet a big limitation.

The material or the substrate with low surface energy is hydrophobic. The phenomenon can also be achieved using plasma treatment with specific conditions according to the substrate. The plasma often forms the deposition of film or layer on the surface of the substrate, thus enabling the hydrophobic characteristics without changing the mechanical properties of bulk material.

For instance, in a work (Nelvig, Engström, Hagström, & Walkenström, 200), developed water based electro spun PVA nano fibers for technical applications. Environmental approach was being behind to develop water soluble polymers. However, in coping such noble cause, when fibers are used for their applications in technical textiles, the fibers tend to dissolves when in contact with water, and even with humid air.

The group worked on one of the possibility to make the Water-soluble fibers hydrophobic. They treated the PVA nano-fibers with Plasma in presence of CF₃ to get the required characteristic. However, hydrophobic layer from the plasma treatment lasts for just couple of weeks.

A great versatile study reported by (Kim, Liu, & Kim, 2006), using atmospheric CH₄-He plasma process for treatment of both flat and rough surfaces to get the hydrophobic effects. The flat surfaces including metals and insulators, while rough surface includes textile such as cotton can be optimized to achieve the hydrophobic coating on the substrate. Means good hydrophobicity can be achieved regardless of the substrate.
The deposition of polymeric HC coatings (CH₂ and CH₃) gives quite smooth surface topography along with the contact angle of as high as of 150° on cotton. The water contact angles of these coating on different substrate including cotton are shown in real time picture in figure 10 while figure 11 illustrate the graphical plotting of the values obtained by contact angle measurement with respect to no. of cycles of treatment.

Figure 10 Optical images of water droplets placed on hydrophobic coatings deposited on (a) Si wafer, (b) Cu foil, (c) paper, and (d) cotton substrates (Kim, Liu, & Kim, 2006)
5.2 Hydrophilic Characteristics

Plasma treatment has been well known to increase the hydrophilicity, wet ability, wicking characteristics of the substrate. These characteristics can be induced on the substrate by introducing the polar groups (i.e. –OH–, –OOH–, –COOH– etc). The increases of the hydrophilic character of hydrophobic fibers such as PET, PA, PP, plays fundamental role in achieving various positive effects on wet processing and other technical effects. Recently (Píchal & Klenko, 2009) czech technical university conducted an experimental on PES sheets. The study based on DBD (plasma), reveals the substantial increase in hydrophilicity. Drop test was used to determine the result. Figure 12 illustrating the difference of both untreated and treated samples analyzed on SEM.
Figure 12 SEM image of (a) the untreated PET non-woven, (b) the PET non-woven after plasma treatment in air (energy density = 230 mJ/cm²) and (c) the PET non-woven after plasma treatment in air (energy density = 1.13 J/cm²) (Píchal & Klenko, 2009)

There are extensive academic literatures available to increase the hydrophilicity of PET. Shin et al., reported a remarkable PET surface functionalization in the presence of He/O₂ plasma.

The spunbond nonwoven PET surface with He/O₂ plasma at atmospheric pressure was treated. The experiment showed increased in crystallinity due to the reduction in amorphous region as due to effect of ablation of substrate due to plasma etching.

Also, the hydrophilic characteristics including wettability and moisture regain was increased. The typical moisture regain of PET is 0.4% which has been increased by 3 three times. On the other hand, the wettability has been noticed to increased by 10
times. Contrary, the effect on dye uptake has not been change significantly (Shin, et al., 2006).

The increase in crystallinity was greater at 30s exposure and it didn’t change significantly after 60 and 90s exposures. The increased hydrophilicity despite of the increase in crystallinity is observed to be the result of increase in polar functionality which is reflected by the increase of Oxygen/Carbon ratio, since, oxygen base functional groups on the surface of PET increase from 27 to 32% after prolong exposure of 90s. The data of the effect of plasma treatment to substrate interaction is tabulated in table 5. In addition, plasma etching opens up newly accessible surface to moisture, even though it depletes amorphous region.

<table>
<thead>
<tr>
<th>Exposure time (s)</th>
<th>Surface composition (%)</th>
<th>Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
<td>O</td>
</tr>
<tr>
<td>0</td>
<td>72.9</td>
<td>27.1</td>
</tr>
<tr>
<td>30</td>
<td>72.1</td>
<td>27.9</td>
</tr>
<tr>
<td>60</td>
<td>70.4</td>
<td>29.6</td>
</tr>
<tr>
<td>90</td>
<td>68.4</td>
<td>31.6</td>
</tr>
</tbody>
</table>

Similarly, in another work (Wei, Liua, Houb, & Huanga, 2007), treated PET fibers to enhance the wettability using Oxygen plasma. The treatments introduce the polar groups on fiber surfaces and so reduce the advancing and receding contact angles of the PET fibers. The contact angle hysteresis of plasma treated PET fibers is found to be altered by roughening of the fiber surface. The analytical tests showed; figure 11 surface morphology and figure 12 Surface chemistry of untreated and treated material.

The AFM image figure 13 (b, d) illustrated fibrils are oriented in the direction of fiber axis, while untreated figure (a) shows grooved structured on PET fibers. Since, the effect is not much visible in the fiber treated with 30s plasma but showed the ablation
on the surface. XPS results showed three main peaks of untreated PET, assigned as C-C, C-H, and O-C=O, Figure 14a. However, figure 14b confirms illustrate additional peak C=O in the spectra, confirms the formation of functional group on the fiber surface.
Figure 13 AFM images of PET fiber: (a) untreated; (b) plasma treated for 30 s; (c) plasma treated for 60 s not shown; (d) plasma treated for 90 s.

Figure 14 XPS spectra of PET fiber: (a) untreated; (b) plasma treated for 30 s.

Also, there are work reported on PET and PP. Morent et al. studied PET and PP non-wovens using DBD in air, helium and argon at medium pressure. Wanting et al., analysed the effect of cold plasma treatment of PP nonwoven fabric for hydrophilicity modification.

In the work by (Morent, Geyter, Leys, Gengembre, & Payen, 2007) showed the non-wovens, modified in air, helium and argon, points toward a major increase in liquid absorptive capacity due to the incorporation of oxygen containing groups, such carbonyl C=O. It was shown that an air plasma was more efficient in incorporating oxygen functionalities than an argon plasma, which was more efficient than a helium plasma.

(Wanting Ren, 2010) made the comparative analysis on the characteristic hydrophilicity retaining capability of cold plasma treated polypropylene non woven fabric with fibers of smooth surface v/s rough surface.

The group found out that PP non woven fabric made from fibers with smooth surface can only keep its hydrophilicity for a short time and then retrieve hydrophobicity quickly at room temperature. On the other hand, this hydrophilic property can last for a long time in the case of the polypropylene non woven fabric made from fibers with rough surfaces.
This difference in behavior of prolonged hydrophilicity was observed to be the result of two reasons; first is the irregular structure of PP fibers and second is the added polar/functional groups on fiber surface due to plasma treatment (figure 15). This observation strongly supports that the fiber surface morphology of PP non woven fabric is a critical factor for long-term hydrophilicity improvement after plasma.
treatment, which gives a positive indication to overcome the aging effect of hydrophilicity modification i.e. often found in this technique.

In another study, (Surakerk Onsuratoom, 2010), woven PET fabric was made hydrophilic by dielectric barrier discharge (DBD) plasma treatment; in addition it was then loaded with Ag particles to achieve antimicrobial characteristics. Surakerk et al treated the woven PET surface using DBD plasma technique in manipulating operating conditions (time, voltage, frequency, electrode gap distance, plasma treatment time, input voltage, and input frequency) and also under different gas environments (air, O2, N2, and Ar) to find the optimum conditions to improve its hydrophilicity. The trials with decrease in electrode gap distance and an increase in input voltage increased the electric field strength which in result leaded to higher hydrophilicity of the PET surface which was measured by wick-ability and contact angle measurements.

However, with respected to environmental gases, air is marked with highest hydrophilic chracterisite, being comparable to O2, while Ar and N2 showed lower hydrophilicity of the woven PET surface. An electrode gap distance of 4mm, plasma treatment of 10s, output voltage of 15kV at 350Hz frequency under the environment of air were found to be the optimum conditions for the maximum hydrophilicity of PET surface. It was then loaded with Ag particles using an aqueous solution of AgNO3 to acquire the antimicrobial property. The plasma treated woven PET loaded with Ag particles exhibited good antimicrobial activity against both E. coli (gram-negative bacteria) and S. aureus (gram-positive bacteria).

5.3 Bio scouring

The enzymatic removal of non-cellulosic impurities on the surfaces of the cellulosic fibers is known as Bio scouring. The process improves bleach ability and dye ability. To date, among all the enzymes utilized by different research groups, alkaline pectinases have proven to be the most effective for cotton bioscouring. However, the
cuticle of the cotton fiber, which contains the highest concentration of hydrophobic noncellululosics such as fats and waxes in cotton structure, forms a natural barrier for pectinase to contact its substrates (pectins beneath the cuticle). This results in an insufficient scouring of cotton fabrics, as referred (Wang et al., 2009).

Research on cotton bioscouring so far has focused on how to overcome this problem. Pretreatments before pectinase has been investigated and some positive results are reported. However, the said process has some disadvantages as energy consumption, processing cost, environmental concerns.

In a patent, (Seiji K, 2006) reported that corona pretreatment could cause an efficient removal of pectic substances in cotton fibers during subsequent pectinase incubation.

Two approaches of plasma based treatments were realized,

(i) DBD at atmospheric pressure

(ii) cold oxygen plasma at low pressure vacuum system,

as the pretreatments done before to cotton bioscouring, aimed to increase the accessibility of pectinases to the pectic substances on the cotton fiber. The effects of different processing parameters of DBD and oxygen plasmas on the wettability, whiteness and burst strength of pectinase-scoured cotton were determined and compared. The result showed that both of the pretreatment can improve cotton bio-scouring. Further, details have been describe in (Wang, Fan, Cui, Wang, Wu, & Chen, 2009). However, DBD might be more suitable for current bio-scouring due to its continuous processing mode and lower requirements to the equipment.

5.4 Desizing

The yarn is sized to form a protective sheath on the yarn surface. It provides extra strength, lubricating effect, so as make the single yarn to withstand during the high speed weaving process. PVA is usually used as coating material (Cacho, 1980). Though it is good for weaving efficiency but it should be taken out of the yarn.
(desizing) to make the nice hydrophilic fabric, ready to dye or for post finishing treatments. In general, the principle to require to remove the ‘size’ should have high degree of hydrolysis and less crystallinity, in addition it should have good water solubility to assure the ease in size removal. Starches and PVA are the sizing agents, used most commonly (Moreau, 1981).

Typically, sizing process comprises of several hot and cold water baths containing detergents, yet the size is not always removed completely through this method (Czerwin, 1966). The waste water containing the sizing material left behind from the cleaned fabric should also be taken care of before discharging or re-utilizing which is itself a major problem. Yet there are energy costs linked to the sizing with hot water and treatment of effluent despite of the fact that PVA is a material of low cost.

There are some techniques which can be used to improve the efficiency of desizing process using cold water, therein enables obvious saving on energy cost. However, Atmospheric pressure plasma treatment has been realized as one of the effective method for such process (Cai Z., Qiu, Zhang, Hwang, & McCord, 2003). (Matthews, McCord, & Bourham, 2005)

For instance, Mathew et al., investigated the of desizing of sized PVA films on fabric surface by APP. The films were exposed to Helium, Oxygenated-Helium and Carbon Tetra-Fluoride (CF₄) plasmas. The molecular weight, solubility surface ablation and weight loss of the treated films was assessed. An ablation pattern was observed through the measured figures. It suggested that ablation was increased with the increase in exposure time. Since the treatment was carried out in an enclosed arrangement, there was possibility of re-deposition. Means, there is a chance etched particles to get re-deposited on the material surface.

However, the trend in weight loss was directly related to the change in thickness as expected. The weight-average molecular weight of PVA chains decreased as the exposure duration increased as observed by gel permeation chromatography. This decrease was due to chain scission by exposure to plasma. Plasma exposed films
increasingly solubilized in methanol and swelling was decreased. These results correlate well with the chain-scission observed through GPC and weight loss trends. (Matthews, McCord, & Bourham, 2005)

In an another study, Zaisheng et al., Desized poly vinyl alcohol (PVA) from the cotton fabric using helium/oxygen/air plasma at atmospheric pressure and compared it with desizing by H₂O₂.

![Figure 16 Percent desizing ratio (PDR) vs. treatment (Plasma gas: air/helium)](image)

*Figure 16 Percent desizing ratio (PDR) vs. treatment (Plasma gas: air/helium) (Cai & Qiu, 2005)*

The graphical model illustrated in figure 16 of Percent desized ratio (PDR) vs plasma treatment conditions showed a linear increase with respect to the duration of plasma treatment. Thus, It’s found that the plasma treatments could directly lead to weight loss and enhance cold washing rate of PVA on cotton. The treatments also helps to a great extent of dissolving the PVA film too.

**5.5 Dye uptake**

There is extensive work reported and published on enhancing the dye ability, particularly on wool. W. Kan and C. W. M. Yuen has been published number of paper and monograph (Kan & Yuen, Plasma Technology in Wool, 2007); (Kan & Yuen, 2005) on wool.
In one of work, (Kan C. W., 2006) studied the effects of low temperature plasma (LTP) treatment on the dyeing properties of the wool fiber. The fibers were treated with oxygen plasma and then three types of dyes that commonly used for wool dyeing, namely: (i) acid dye, (ii) chrome dye and (iii) reactive dye, in the dyeing process.

The consequences for acid and chrome dyeing were not significant, further detail on the text has described in literature (Kan C. W., 2006). The result for reactive dyeing were found appreciated. The rate of dyeing rate Low Temp. Plasma treated wool fiber was greatly increased and also the final dyeing exhaustion equilibrium was increased significantly (Kan C. W., Dyeing behavior of low temperature plasma treated wool, 2006)

Studies have also been made on cotton to increase the dye ability. In fact, in the work (El-Shafei, Hauser, & Helmy) intended to meet two objectives 1) to achieve highly durable water and oil repellent finishes on cotton fabrics 2) to increase color yield of direct dyes on cotton. Graft polymerization of fluorocarbon containing acrylic monomers are used using APGD for water and oil repellency. Quaternary ammonium monomer are used for color yield.

5.6 Anti-bacterial characteristics

Yu-Bin et al evaluated the antibacterial properties of polyester fabric after activating its surface by atmospheric pressure plasma and then grafting it with chitosan oligomers/polymers. The antibacterial effect was most evident when the surface of fabrics was activated by atmospheric pressure plasma for 60 to 120 seconds and grafted with chitosan oligomers. The modified fabrics also exhibited good biocompatibility. This process can be applied to a large area and used to produce antibacterial polymer fibers. (Yu-Bin Chang, 2008)

There are successful work reported (Gorjanc, Bukosek, Gorensek, & Vesel, 2009) to make cotton (bleached and mercerized) fabric anti-bacterial using mild oxidizing plasma, by using a low-pressure inductively coupled radiofrequency (RF)
discharge/plasma. Reactive exhaust dyeing was used for loading of nano silver. Blank dyed cotton fabrics with nano silver were analyzed using optical emission spectrometry with inductive coupled plasma after microwave decomposition of the fabric sample. This method exploits the effect of the plasma which is formed when argon passes through a RF field in which gas particles become partly electrically discharged and emit light of characteristic wavelength the treatment enhanced nano silver adhesion to the fabric, which also contributed to antimicrobial characteristics without change in its mechanical properties Figure 17; morphology of (a) untreated-grooved surface, (b) treated fiber surface remains grooved and the macrofibrile structure has gained a much sharper outline.

![Figure 17 SEM of (a) untreated, (b) plasma treated bleached and mercerized cotton (Gorjanc, Bukosek, Gorensek, & Vesel, 2009)](image)

Some work has also been reported on Nylon. In the study, Hsiang-Jung et al observed the properties of nylon textiles after being activated by open air plasma and then grafted with chitosan oligomer and chitosan polymer. They observed that nylon textiles grafted with chitosan polymer had better antibacterial performances than those grafted with chitosan oligomer. Air plasma activation at a higher speed (26 m/min) for a few times assisted in the grafting of chitosan and critically determined the antibacterial activities. Further treatment with air plasma after grafting improved the antibacterial effect. Overall, chitosan-grafted nylon textiles showed good antibacterial potential as well as biocompatibility. (Hsiang-Jung Tseng, 2009)
6 Technical and Functional Applications

6.1 Stain Repellent Finishing

As mentioned earlier, often Plasma treatment used as pre/post treatment, in order to get the required characteristics. Stain repellent textiles has been a strong demand, particular for technical textiles applications such as tents, awnings, worker uniforms being as in medical applications (surgeon gowns, medical bed sheets) or slaughter wears. In the work (Dinkelmann, Lunk, Shakhatre, Stegmaier, & Vinogradov, 2004), Atmospheric plasmas have been investigated prior to the wet-chemical treatment on PET to get oleo phobic characteristics. The treatment revealed, oil repellency grade is enhanced by at least one grade. A maximum oil repellency grade between 6 and 7 was reported directly after applying ultra thin fluorocarbon films from C₄F₈ or C₃HF₇ in a dielectric barrier discharge (DBD).

However, some drawback can also be observed. In the treatment fluorocarbon films contain a high oxygen content, so aging effects are obtained, which remarkably reduces the oil repellency grade, whereas common wet-chemical treatments nowadays enable a stable oil repellency grade of around 6 without plasma processing.

Moreover, atmospheric plasma treatments also require a pretreatment to clean the textile samples and to obtain a suitable film adhesion. (Hegemann, 2005).

Such properties can also be achieved on nano scale using Sol-gel process (xerogel film²) based on hybrid (Metal/Mettaloid and Alkoxides) structures, which enables to get other multiple characteristics as high thermal stability, hardness of ceramic, elasticity of polymers and low concentration of solvent (Simoncik, 2010)

6.2 Adhesion Enhancement of polymer/metal matrices

² An organic polymer capable of swelling in suitable solvents to yield particles possessing a three-dimensional network of polymer chains (http://encyclopedia2.thefreedictionary.com/xerogel)
Plasma has been well known of its adhesion enhancement between two interfaces. Plasma Etching process of glow discharge with inert argon and reactive oxygen gas causes distinct weight decrease of polymer by volatility and removal of bombarded molecular to variability of gas pressure. Thus, it accelerates the anchoring effect which is composed of micro crater or micro pore, to say another word, irregular roughness.

This morphological phenomenon might be a cause of excellent adhesion between polymer interface. Simultaneously, this etching bombardment produces radicals and the other hydrophilic functional groups on fragmented molecular chain (Lee & Joeng, 2004).

Sen’I gakkaishi studies the effect of plasma treatment methods, between copper and high performance fiber PEEK, which has been known of its excellent thermal and chemical resistance at prolong time. As of his analysis on ordinary plasma treatment and pulsed plasma treatment, he reported the following three points of plasma interaction (Gakkaishi, 2009):

1. Oxygen plasma treatment enhances the adhesive strength at Cu\PEEK interfaces.
2. O plasma treatment introduces O functional group and produces an etching action at the interfaces. This etching action certainly contributed to the adhesive strength among these actions.
3. Introduction of O functional groups might also produce a synergetic effect with an etching action and this might turnout in stringer adhesion of the two interfaces.

7 Questioned?
This section is based on the dichotomy appraisal that ‘why not’, in a passive sense. Despite having all the potentials and the significance, yet ‘Plasma technology for
textiles’ is unable to make an expected commercial impact on textile production processes.

**Lasting effect**

Early Ageing has been crucial limitation of its application, though, it provides easy alternatives to get the required characteristics. The modification implemented doesn’t remain stable after treatment for prolong time. The concentration of functional group on a substrate may become extinct depending on the time and ambient conditions.

Since, polymer chains have greater mobility at the surface than in bulk, allowing the surface to reorient in response to different environments (Kan & Yuen, Plasma Technology in Wool, 2007). An example has been described above (section 5.1) on the plasma treatment lasting effect. The work conducted which has been conducted at Swerea to induce hydrophobic layer on the water soluble electro spun polymers. The treatment effect managed to last for couple of weeks. However, rate of ageing can be restricted by cross linking the surface, hereby no or lesser movement in the polymer chains.

**Large specific area**

The Textile assemblies have large surface area i.e. width of few meters to length of thousands of meters. The processing capabilities to date are not optimized to an industrially acceptable level to cover such large surface area at optimum speed.

**Penetration**

Plasma has been suffering penetrative difficulty to reach the Textile three dimensional pours structure. The penetration is found effective on the loosely assemble structures. However, new technological changes are being substantially overcoming such pitfall.

**Socio - Economic factors**
There are huge efforts and progressive research activities being conducted on plasma technology for textiles in developed countries, because of its potential and ability to conserve earth valuable resources. But, on the other hand, most of the textile processing industries are now based in developing countries. Such industries could significantly induct the errands of plasma technology. Though, the companies are now offering in-line atmospheric pressure plasma machines. However, the investments of such New equipments costs over 20 million Euros. Besides, the secondary factors such as, abrupt changes of working parameters, impinge advancements, skilled work force etc., also need to be considered.

Other competitive processing techniques

There are some other techniques such as Sol-gel technique which combines the number of multi functionalities in more competitive manner. For instance, the processing can be carried in conventional finishing systems and machines. So, no need of new investments to be made on systems, plus, the technique enable of less use of chemicals and less effluents to be discarded.

8 Conclusion and Recommendations

Again, coming to dialectal discussion, PRISTINE approach of the plasma processing embark it to spur of the moment. As the traditional processing and finishing methods in textiles usually consumes large amounts of chemical substances, frequently toxic or noxious, or use of organic solvents, as well as production of liquid and gaseous effluents which require expensive purification treatments.

Also, new technological changes in the field seem to provide cutting edge solutions to processing and finishing of textiles. The steps and ways are overcoming pitfalls which were being reported in old versions as the formation of harmful gases, ozone and nitrogen oxides. Surface treatments with non thermal high density plasma are pronounced and homogenized. Apart, there is a need to understand and analyze the effect on entire textile structure along the study the individual fiber, through
characterization techniques. The proposed general mathematical model would be a valuable basis to understand such needs. However, yet the challenges are at least as big as the potential benefits. A combine effort is required from different disciplines with the significant input from Textile Engineers. Progress is ongoing to observe the textiles during treatment to characterize plasma effect throughout the textile assemblies. The way to successful commercial development is long but many steps have already taken.

A facility on the setup of contemporary plasma equipments as atmospheric plasma systems, would definitely provide a better insight into the technology which has not been included in the treatise owing to the limited scope of the project and lack of post understanding of the subject, but is a very good area to look into after acquiring vital knowledge in the field. Though, it has been in pipeline for long at academic and research institutes based in Sweden. However, some rudimentary work has been initiated by research institute ‘Swerea IPF’ even in 80’s, as Sweden has always been a front liner in doing research, but, due to the lack of availability of technology of that time rather on ‘atmospheric’ to provide the viable outcomes, the project has not been taken the progressive interest.

9 Future Work

Plasma surface modification can have the enormous potential on the performance of textiles in various applications. Since, a bibliographic study has been undertaken on the plasma technology for textiles. The next step would like to move on materializing the concept on a more specific side. In reality, however, in-depth research is required to evaluate the surface engineering and characterization analysis to meet the specific performance specifications. A collaborative research project between the academia (University of Borås, Swerea IVF) and industry (FOV Fabrics) can be designed in a real time practice to exploit the opportunities and outcomes.
10 References


