Chapter 9

Plasma Surface Treatments of Nonwovens

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Additional information is available at the end of the chapter

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Abstract

Plasma treatment has been used for surface activation and modification of textiles. The ionized, highly reactive species, such as ions, electrons, and radicals, in plasma modify the surface of the substrate material, and the composition of plasma depends on the gas used. Plasma technology is an environmentally friendly process and resource-efficient in nature. There is no solvent emission or wastewater in the process and drying processes with high energy and time consumption are not required. The textile applications of plasma include sterilization, wettability and hydrophobicity, dyeability enhancement, flame-retardant finishing, and antimicrobial properties. Plasma surface modification applied to fiber is a way to add value to a nonwoven fabric and enhance the functional performance of the final product. This chapter provides an overview of the plasma treatments of nonwovens that enhance their surface-related properties.

Keywords: nonwoven, plasma, surface modification, coating, functionalization

1. Introduction

1.1. Surface functionalization of nonwovens by plasma techniques

Nonwoven fabrics are produced by bonding or interlocking of fibers by different means such as mechanical, chemical, thermal, or physical. These fabrics may be designed to have a limited lifetime or as single-use fabrics, or very durable ones. The technologies enable the production from simple roll-goods to micro- and nanofiber webs. The structure of a typical nonwoven made from polypropylene fibers by melt-extrusion technologies is shown in Fig. 1. Nonwovens have been used in a wide range of products such as wipes, baby diapers, filtration media, garment interlinings, furniture padding, and many others. The end-use properties of nonwovens are determined by the properties of the fibers they are made from, the fabric structure, and other functionalities, such as absorbency, hydrophobicity, wettability, and antimicrobial property, imparted by potential modifications. Surface modification applied to the fiber is a way to add value to the nonwoven fabric and enhance the functional performance of the final product.

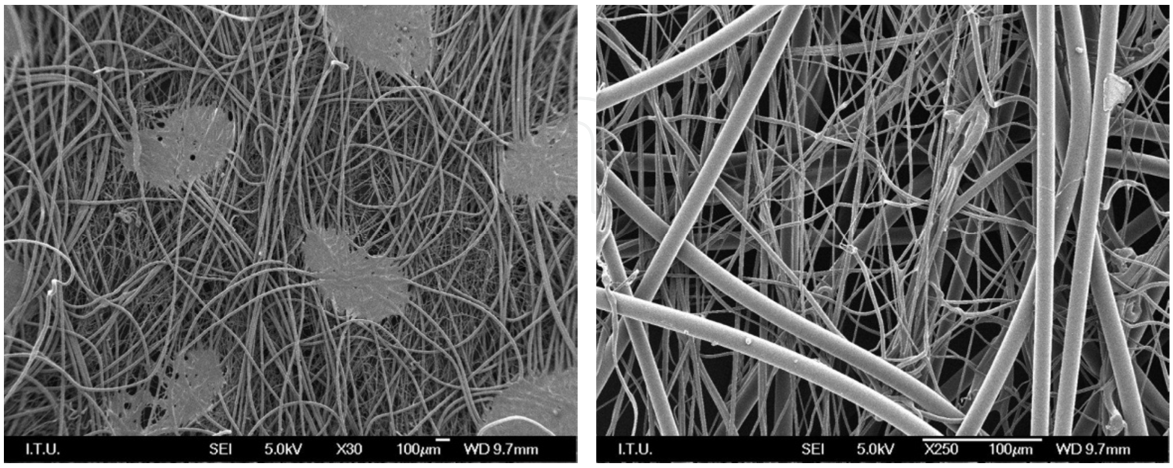

Figure 1. Scanning electron microscopy images showing the surface texture of a nonwoven fabric

Plasma is the mixture of partially and fully ionized gas, photons, free electrons, and chemically reactive atoms and radicals [1]. Plasma is generated by raising the energy content of matter through different methods such as dielectric barrier discharge, glow discharge, corona discharge, or the application of a high electric field.

The gas molecules become ionized when the kinetic energy of gas molecules exceeds their ionization energy. The active species in plasma only interact with the outermost surface of the substrate, approximately 10 nm in depth, without altering the bulk properties of the substrate [2].

Plasma treatment has been recognized as an alternative ecological surface treatment to conventional liquid-based textile-coating treatments, which use solvents, additives, and organic reagents and produce large volumes of liquid waste.

Plasma treatment can be used to modify the surface properties of nonwovens for:

- Enhancement of surface energy, thereby improving wettability and adhesion properties of nonwovens
- Improvement of the surface properties of natural and synthetic fibers to improve wettability, printability, and dyeability of nonwovens
- Hydrophobization, oleophobization (dirt- and oil-repellent effect), and functional and bioactive coatings (antibacterial, fungicidal)

With plasma, different reactions such as surface cleaning, etching, deposition, cross-linking, surface roughening, and grafting are possible [2]. Polymer surfaces can be cleaned or etched, mostly using nonpolymerizable gases such as O_2, N_2, H_2, the noble gases, or gas mixtures [3].

Plasma etching has been used to remove contaminations such as lubricants, oils, surfactants from textile fibers and also to increase the wettability of the surface by changing the surface roughness [3].

1.2. Plasma reactions

1.2.1. Plasma grafting

Surface properties of polymer materials such as nonwovens can be tailored by wet chemical treatments or physical techniques such as glow discharge plasma treatment [2, 3, 4, 5].

Grafting is a method to modify and functionalize fibrous surfaces and it leads to a thin film coating on the substrates (Fig. 2). In surface grafting, a second polymer is attached to the polymer backbone of the fibrous surface through covalent bonding. Properties of the surface, such as wettability, hydrophobicity, adhesion, and friction, can be dramatically changed by this method [2, 3].

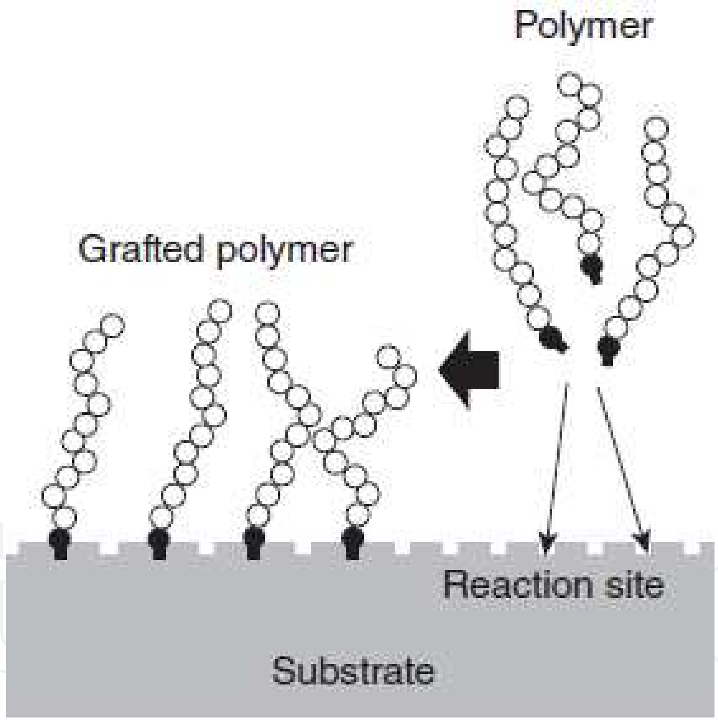

Figure 2. Schematic of surface grafting of a polymer on a substrate [2]

Plasma glow discharge is a method used for surface grafting reaction, where radio frequency energy is applied to an electrode (Fig. 3) pair in order to excite a gas such as oxygen, air, helium, or argon at low pressure (0.1 - 1.0 torr). Free radicals are generated at high energy levels through stripping of electrons from the gas particles. Then polymerization starts with the introduction of one or more types of monomer gases such as acrylic acid, carboxyl and amino groups into the plasma treatment chamber where the radicals react with monomer gases [2, 3, 6].

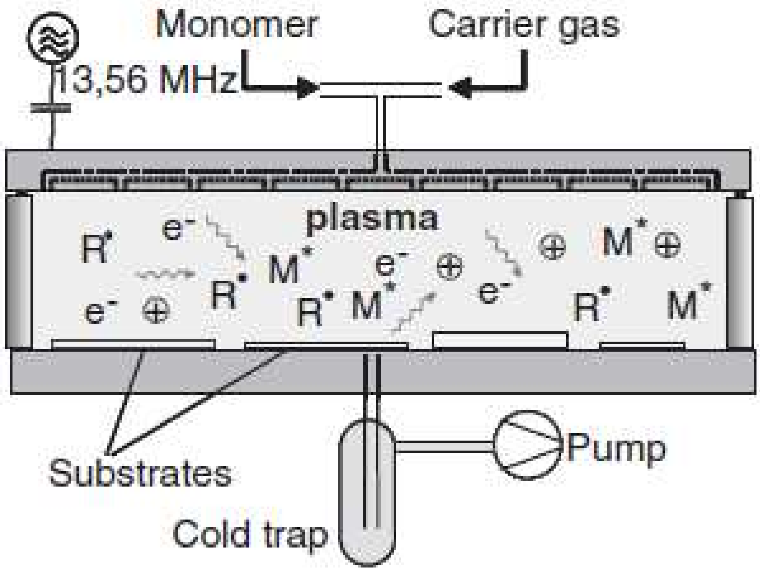

Figure 3. Schematic of plasma treatment reactor for surface modification and deposition [3]

Plasma conditions are controlled by plasma parameters such as plasma power, frequency, duration, carrier gas, gas pressure, flow rate, and monomer types [3].

Corona discharge is commonly used for surface treatment of polyethylene and polypropylene having low surface energy. In this method, an electrode at a high electric potential (of 15 kV at 20 kHz) ionizes the surrounding gas which generates a corona discharge (Fig. 4). While the fabric passes between the high-potential electrode and a grounded electrode, chemical reactions occur between some of the ionized gas particles and the surface of the substrate. During this process, surface roughening and addition of functional groups such as carbonyls, hydroxyls, carboxylic acids, and unsaturated bonds to the fabric surface take place. Oxidation of the substrate surface occurs when corona discharge is performed at atmospheric pressure in air, which oxidizes the surface [6].

1.2.2. Thin film deposition

Plasma polymerization includes the formation and deposition of thin polymeric films on the surface of substrates with the influence of plasma generated by some kind of electrical discharge [6].

Plasma polymerization process includes fragmentation of vapor-type organic, organosilicone, or organometallic monomer molecules, the formation of reactive radicals, and recombination of the activated fragments in which the treatment time influences the thickness of the applied coatings [6].

Thin films of metals and metal oxides have been applied to nonwoven substrates using different thin film deposition techniques such as ionized gas treatments, plasma treatment,

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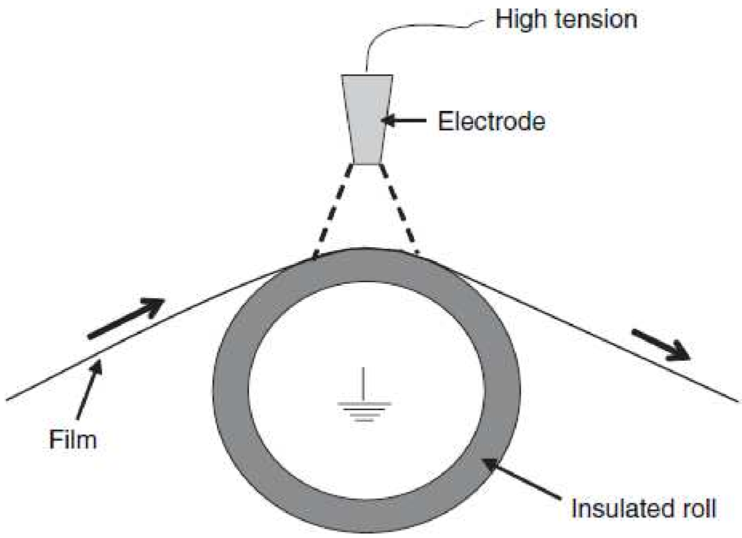

Figure 4. Schematic of corona discharge treatment process [6]

chemical vapor deposition (CVD), physical vapor deposition (PVD), surface grafting, and layer-by-layer deposition [2, 7, 8, 9, 10, 11, 12, 13].

In physical vapor deposition (PVD), vaporized material is deposited on the substrate surface uniformly as a thin film in a vacuum environment where the use of different coating materials is possible [14]. Coatings on polymeric materials by PVD methods have been found to improve the surface properties without altering the bulk properties [15, 16, 17, 18]. PVD techniques offer advantages over conventional textile coating [10, 12, 16, 19, 20, 21], since it is an environmentally friendly, solvent-free process without any need to dispose of any liquid waste. Moreover, a strong bonding is achieved between the fibrous substrate and coating layer.

PVD techniques are commonly used for coating very thin metallic or ceramic films on different substrates. Sputtering is a PVD technology in which atoms are ejected from a solid target material through bombarding the target by energetic ions in a plasma confinement. These atoms condense on the substrate and form a thin film. Sputtering techniques include direct current (DC) sputtering, radio frequency (RF) sputtering, reactive sputtering, and magnetron sputtering [12].

Wei et al. [22] studied the interfacial bonding between polypropylene (PP) fibrous nonwoven substrate and sputter-coated copper. Adhesion of the coating layer to the PP fibrous substrate was found to be affected from plasma treatment and heat during the sputtering process. PP nonwoven showed the lowest abrasion resistance among the tested materials, whereas the sputtered copper coating significantly improved its abrasion resistance further.

Deposition of layers onto the substrates by chemical vapor deposition (CVD) is achieved by chemical reactions in a gaseous medium [23]. A thin film is deposited on the substrate surface through decomposing and/or reacting one or more volatile precursor materials using plasma energy [2]. Different monomers such as methane, hexamethyldisiloxane, tetramethylsilane, hexafluoropropylene ethylene, butadiene, hydroxyethylmethacrylate, and *N*-vinylpyrrolidone have been used in plasma-enhanced chemical vapor deposition processes to obtain surface coatings with different functionalities such as oleophobic, hydrophobic, and corrosion-resistant [24].

1.2.2.1. Industrial-scale production-line application

Atmospheric pressure plasma CVD (APCVD) offers a continuous processing opportunity in textile production. The disadvantages such as costs and operating difficulties related to high vacuum equipment are eliminated and the need to interrupt the line is avoided since a continuous coating process is possible instead of a batch process. An atmospheric pressure plasma CVD system is schematically shown in Fig. 5.

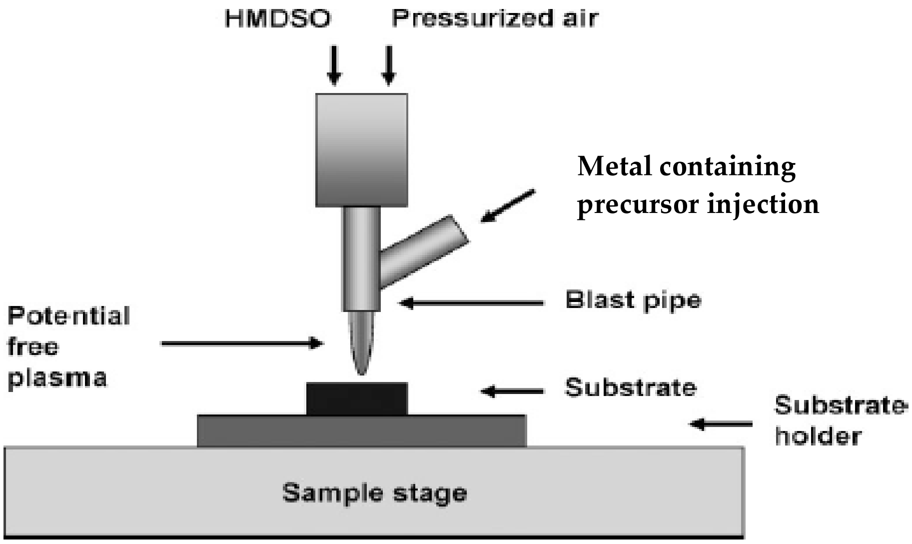

Figure 5. Schematic view of the APCVD system [25]

On a commercial scale, reactor geometry, gas feed, heat distributions are important for delivering coatings that are uniform across the substrate [12].

Oxidative chemical vapor deposition (OCVD) is a novel technique to obtain uniform polymer layers on a variety of flexible and rigid substrates that does not require any solvent to be processed while depositing uniform, thin polymer layers on various substrates [26].

ALD (atomic layer deposition) is another vapor phase vacuum film deposition technique that can generate thin conformal deposited layers by successive surface reactions of a precursor and a reactive gas [27, 28]. This technique has the advantages of precise thickness control and uniformity and conformability of the deposited layer on the substrate [27]. Metallic, nitride, or oxide films such as aluminum oxide can be deposited on nonwovens by this technique [28]. In comparison to the regular thermal ALD, in plasma-enhanced ALD, the reactant gas flows through a plasma source providing shorter deposition time, and lower deposition temperatures allow coating on heat-sensitive fibers and denser films can be produced [28].

2. Plasma treatment for improvement of wettability, printability, and dyeability of nonwovens

Plasma treatment, depending on the type of process gas, changes not only the surface morphology but also leads to a change in chemical composition of the surface. For instance, when oxygen is used as process gas, different oxygen-containing functional groups, such as –OH, –C=O, –COOH, are introduced onto the surface of the nonwoven [3, 29]. Therefore, oxygen plasma treatment was found to increase the dye-uptake and printability of textiles [3].

DuPont's Tyvek® is a flash-spun and calendered nonwoven substrate made of high-density polyethylene fibers. It is a paper-like, dense substrate providing a smooth medium for printing signs, banners, and other graphics. Tyvek® has a corona discharge treatment on one side. SEM cross-sectional image (Fig. 6) shows the micropores created on the treated side where the ink is wicked through [30]. Electronic charging during corona treatment of the substrate oxidizes the surface and increases the wettability of the substrate. This improves adhesion of ink, adhesive, and coating to Tyvek® [31].

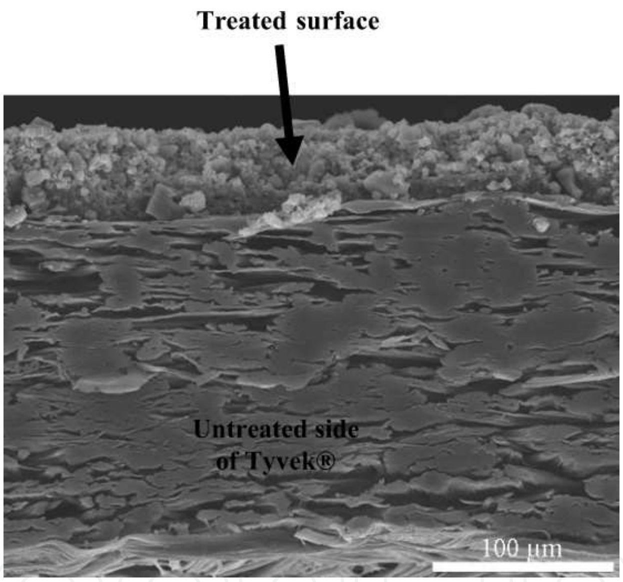

Figure 6. SEM cross-sectional image of plasma surface treated Tyvek® [30]

Enercon Industries Corp. [32] manufactures industrial atmospheric plasma treatment systems for nonwovens operating at low temperature and atmospheric pressure.

3. Plasma treatment for enhancement of adhesion properties of nonwovens

Surface modification of textiles by plasma treatment has been used for the adhesion enhancement of fibers and fabrics. Introduction of functional groups at the fiber surface helps to form affinity or chemical bonds with a coating material leading to an adhesion improvement [33, 34, 35]. For instance, Šimor et al. [36] used atmospheric-pressure nitrogen plasma as a pre-treatment to render the surface of polyester nonwoven fabric hydrophilic for subsequent electroless nickel plating.

However, bonding should be immediately performed following plasma treatment, since storage may result in a reduction or loss of adhesion to the treated surface [6].

Rombaldoni et al. [33] used low-temperature oxygen plasma treatment to enhance the adhesion between polypropylene nonwoven and poly(ethylene oxide) and polyamide-6 nanofibrous mats deposited onto polypropylene nonwoven. Improvement of adhesion between the nonwoven and the nanofibrous coatings was reported, which was attributed to the increased wettability of the supporting polypropylene fabric and polar functional groups introduced by plasma treatment allowing a stronger interaction between the treated fabric and coatings [33, 35].

Li et al. [37] fabricated a high-performance battery separator where they used polypropylene nonwoven as the support material. A fluorinated polymer, octafluoropentyl methacrylate, was grafted onto the surface of the polypropylene nonwoven by plasma treatment to improve the nonwoven's adhesion with poly(vinylidene fluoride-co-hexafluoropropylene).

The adhesion (peel bond) strength between two layers of polypropylene spun-bonded nonwovens, plasma treated and laminated by polyurethane-based adhesive, was improved by up to 150% compared to that of the untreated laminated samples [38, 39]. This was attributed to the increased surface roughness of the polypropylene fibers due to the etching effect of the plasma treatment (Fig. 7), leading to enhanced mechanical adhesion between the laminated layers.

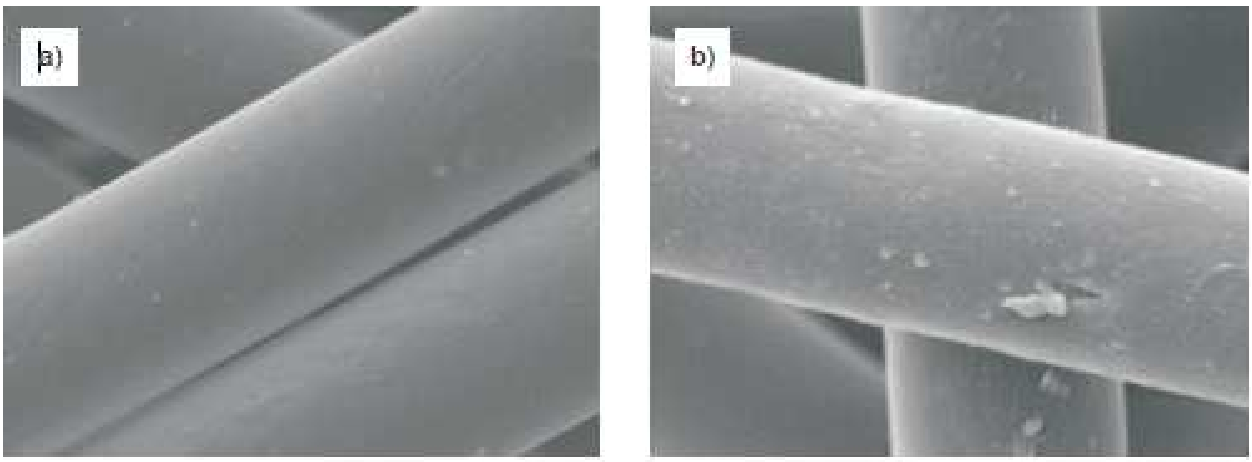

Figure 7. SEM images of PP fibers, (a) untreated (×5060) and (b) treated by argon plasma at 80 W for 10 min (×5490) [38]

The plasma-induced changes in surface morphology of polypropylene fibers of a spun-bonded nonwoven on the nanometer scale was demonstrated by [40] via AFM (atomic force microscopy) analysis (Fig. 8). It was found that plasma treatments increased the fiber surface area and surface roughness due to fiber etching by the bombardment of the fiber surface by plasma-generated energetic particles and reactive particles.

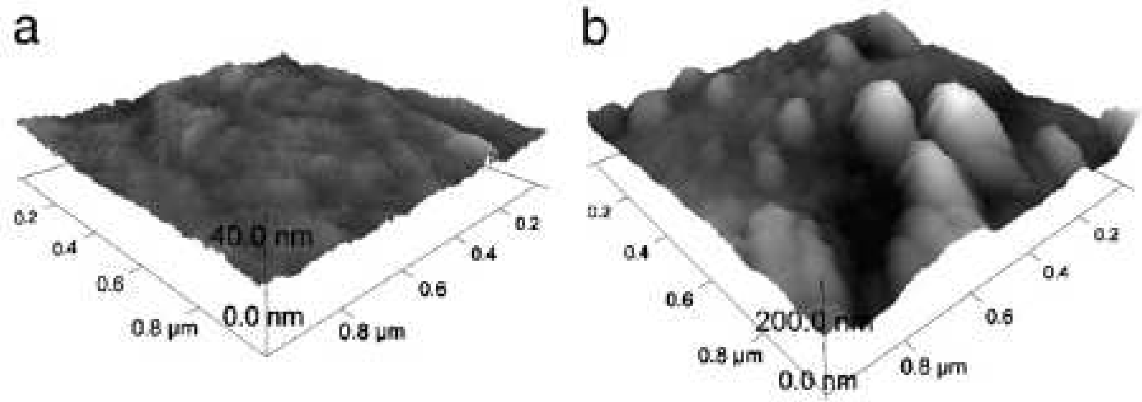

Figure 8. AFM images of (a) untreated and (b) dielectric barrier discharge plasma-treated spun-bonded polypropylene fabric [40]

Armağan et al. [29] reported about 28–60% improvement in the adhesion (peel-off) strength of oxygen plasma pretreated and laminated cotton/polypropylene fabrics using an acrylic-based adhesive compared to untreated laminated samples. After 40 washing cycles, an improvement in peel-off strength of plasma pretreated and laminated samples compared to that of untreated laminated samples was also reported. The improvement was attributed to the increased wettability of the surfaces with oxygen plasma treatment contributing to the adequate wetting of the surface by the adhesive. Functional groups introduced to the fiber surface resulted in better interaction between the adhesives and the plasma-pretreated fabric surface.

The change of the functional side groups on a polypropylene spun-bonded nonwoven after oxygen plasma treatment was determined by high-resolution XPS analysis of C1s peaks [29]. It was observed that the amount of C–C/C–H group decreased, while the amount of oxygen-related groups increased (Fig. 9) after the plasma treatment, rendering the fabric surface more wettable.

4. Plasma coating of nonwovens for functional and bioactive coatings

Plasma coating techniques have been utilized to obtain functional and bioactive coatings on nonwovens to improve surface hydrophilicity, hydrophobicity, electrical conductivity, UV and electromagnetic shielding, and antibacterial properties.

Polypropylene nonwoven fabrics are commonly used for hygienic and disposable absorbent products such as diapers, feminine care products, wound dressings, and wipes. In all these applications, nonwoven needs to be wettable by water or aqueous-based liquids [41, 42]. This is usually obtained by coating the fabric with a surfactant solution lowering surface tension of

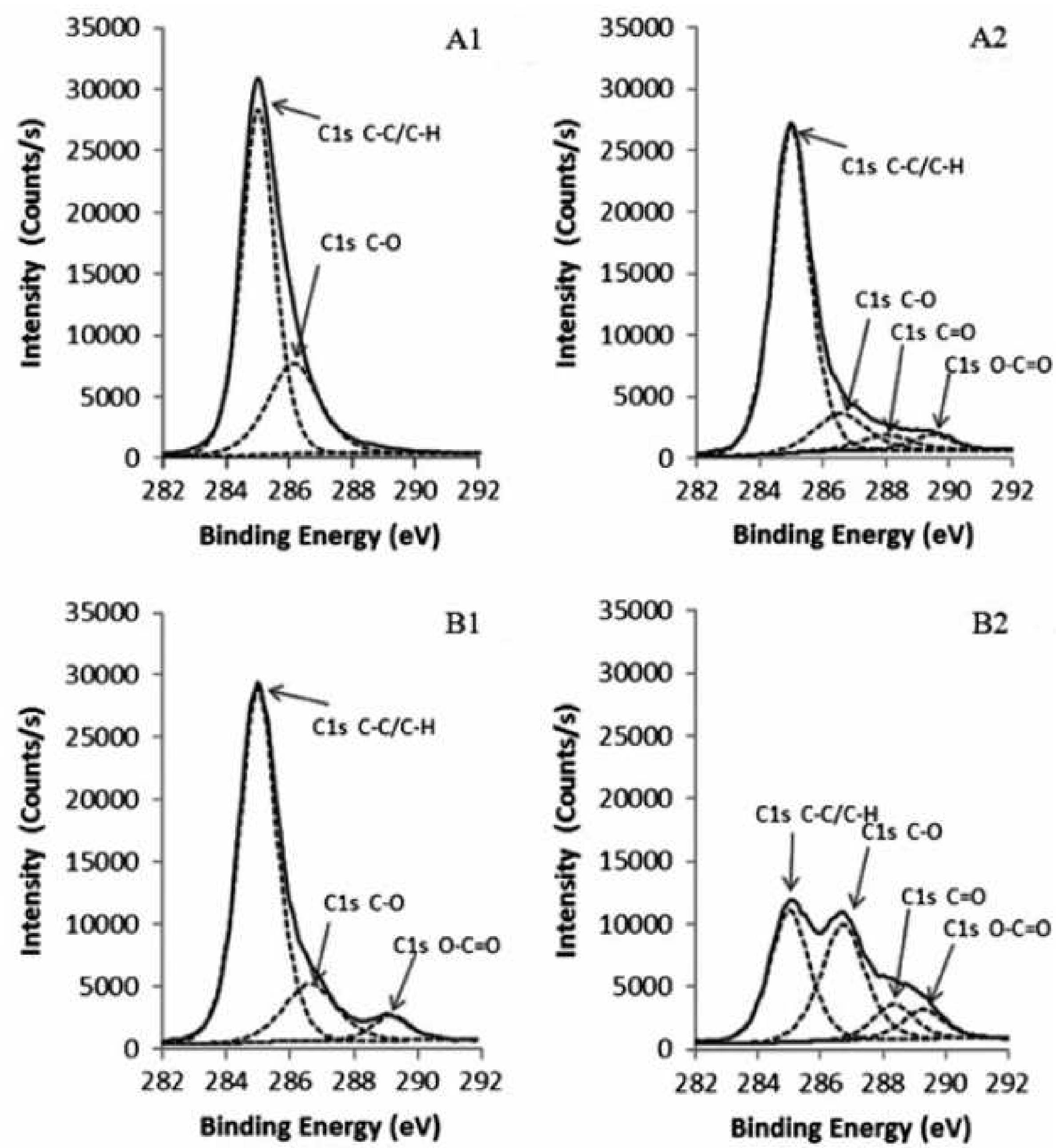

Figure 9. C1s peak of untreated and oxygen plasma treated sample at 80 watt and 10 minutes, (A1) PP untreated, (A2) PP oxygen plasma treated, (B1) cotton untreated, (B2) cotton oxygen plasma treated [29]

the aqueous liquid and subsequent drying of the fabric. In such case, the surfactants are effective in rendering the fabric wettable for a limited time during the use of the product. Surface treatment of nonwoven fabrics using plasma is an alternative method to improve their wettability [41].

Behary et al. [43] activated the surface of a carded and hydroentangled PET (polyethylene terephthalate) nonwoven by an air–dielectric barrier discharge atmospheric plasma. The wettability of the PET nonwoven was enhanced due to plasma treatment which was evident from considerable decrease in water contact angle results and increase in the proportion of polar groups, in particular carboxyl –O–C=O– groups.

On the other hand, super-hydrophobic, self-cleaning nonwoven surfaces have been produced by imparting of oxides such as TiO_2 and a polymethylsiloxane coating on cellulose via CVD technique [12]. Sobczyk-Guzenda et al. [44] used radio frequency plasma-enhanced chemical vapor deposition technique to deposit thin TiO_2 film (Fig. 10) on cotton fabric to impart a self-cleaning effect under UV illumination through a photooxidizing activity.

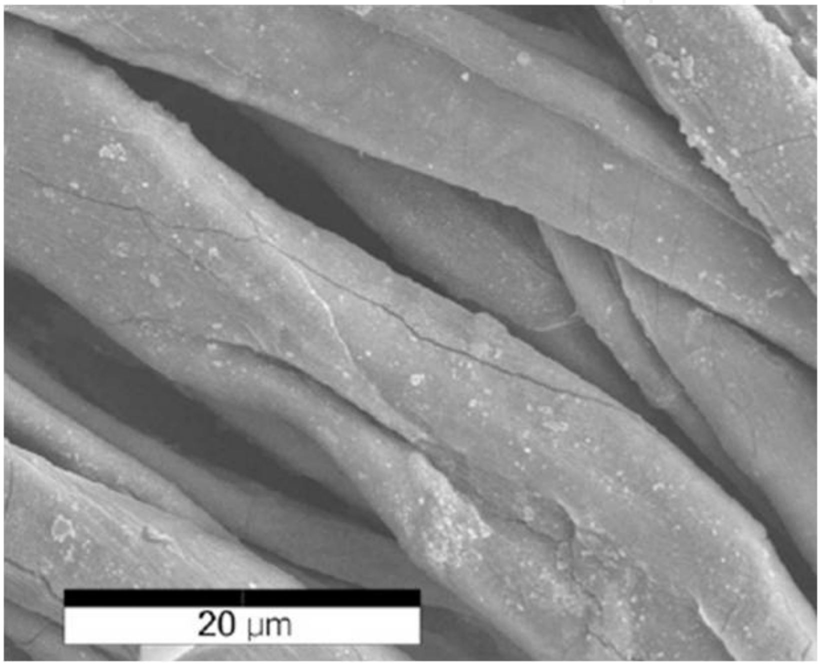

Figure 10. Scanning electron microscope picture of plasma deposited TiO_2 coating on cotton fabric [44]

The surface wetting of the nonwoven materials was changed by deposition of different materials through sputtering. Wei et al. [10] performed the deposition of copper, zinc oxide (ZnO), and polytetrafluoroethylene (PTFE) on the surface of polypropylene meltblown nonwoven through sputtering. While copper coating improved the surface conductivity of the material, ZnO coating significantly increased the UV absorption of the material necessary for UV shielding. Both Cu and ZnO coating rendered the nonwoven surface hydrophilic. Deposition of PTFE provided surface hydrophobicity on the nonwoven material due to increase in surface contact angle and roughness [10]. Sputter coatings of copper and silver on spun-bonded polypropylene nonwovens provided reduced transmittance both in UV and visible light ranges [20].

Deng et al. [45] and Wei et al. [46] reported a decrease in electrical resistance of aluminum and copper sputtered polypropylene spun-bonded nonwovens. Aluminum-doped zinc oxide (AZO) and doped indium oxide (ITO) films were deposited onto the polypropylene nonwovens by magnetron sputtering. For the same thickness, nonwoven materials coated with ITO showed a lower electrical resistance than those coated with AZO. The nanoscale AZO coating on nonwoven provided better UV protection than ITO coating for the same thickness [20]. Jianfeng et al. [47] studied the electromagnetic shielding efficiency of PET nonwovens by sputtering of nanoscaled Cu, Ag, Ag/Cu, and Ag/Cu/Ag films.

In another study, Baek et al. [48] used microwave-induced argon plasma to modify and sputter-coat the surface of nanofibrous silk fibroin scaffolds with gold/platinum to enhance the attachment and proliferation of the human articular chondrocyte cultures.

Nonwovens currently find applications in various industries such as medical and hygiene, home textiles such as mattresses, floor coverings, and shoe linings. A suitable environment for infection by microorganisms is created especially in nonwoven products used in hospitals, hotels, and the clothing of the personnel. Nonwovens made from natural fibers such as cotton are more susceptible to bacterial proliferation than synthetics due to the moisture content of natural fibers.

Antibacterial property has been imparted to nonwovens by the application of antibacterial finishes such as metallic ions of silver [40, 49], copper, and their compounds, also phenols, quaternary ammonium salts, and organosilicones. Nontoxicity of the antibacterial agent becomes critical depending on the end-use of the product.

Mazloumpour et al. [50] used atmospheric pressure glow discharge plasma to impart antimicrobial properties to a polypropylene spun-bond nonwoven. A durable antimicrobial property was achieved on the nonwoven by plasma grafting of diallyldimethylammonium chloride (DADMAC) in the presence of a cross-linker.

Several studies showed metal, that is, silver, sputtering onto polypropylene and polyester nonwovens and polyacrylonitrile electrospun nonwovens to impart antibacterial property [51, 52, 53]. Shahidi et al. [16] deposited copper onto the surface of cotton fabric samples by DC magnetron sputtering for antibacterial effect and found that duration of the application process was shorter compared to conventional application processes using nonionic detergent and metallic salts. The obtained antibacterial effect was found to be durable against 30 washing cycles.

Plasma deposition has also been used to impart flame retardancy to nonwovens. Acrylate monomers containing phosphorus have been plasma grafted on cotton and PET/cotton fabrics. Plasma enhanced chemical vapor deposition of an organosilicon thin film on polyamide 6 has been performed using the cold remote nitrogen plasma process [54].

5. Conclusion

Nonwoven fabrics have a wide variety of applications such as cleaning cloths, wipes, filters, disposable gowns and drapes, baby diapers, mattress coverings, shoe linings, insulating materials, and many others. According to EDANA [55], a trade organization in Europe, around

1,954 million tons of nonwovens (roll goods) were produced in 2012. There has been a significant growth for nonwovens market and between 2013 and 2018; a significant growth rate of 7.6% (tonnage) is predicted for global nonwovens market according to a market report by Smithers Apex [56].

Nonwoven technologies offer the ability to easily manipulate fabric properties such as porosity, weight, mechanical strength, and surface textures much more cost-effectively than their woven or knitted counterparts. Additional functionalities can be added to nonwovens by padding and coating treatments; therefore, functional performance of the final product can be enhanced. The conventional methods for coating application include wet chemical processes where there is use of large quantities of chemicals, solvents, water, and energy. There are major drawbacks for conventional wet processes such as use of toxic solvents that are harmful to human health and environment, large quantities of energy and water consumption, and disposal of chemicals.

Plasma surface treatments have been shown to have unique advantages including environment- friendly process, low production cost, and modification of only the upper molecular layers of the substrates without changing the material's bulk properties. Plasma treatment has been recognized as an alternative ecological surface treatment to conventional wet textile coating and finishing treatments.

Plasma-based techniques offer many opportunities to obtain different surface functionalizations on various substrates. Surface properties of nonwovens can be engineered from hydrophilic to super-hydrophobic with plasma treatments. Plasma techniques provide many surface treatment possibilities such as surface cleaning, surface activation, etching, surface roughening, thin film deposition at nanoscale and grafting. With plasma coating techniques, tailor-made surfaces can be created with specific functions such as flame retardancy, electrical conductivity, antibacterial, or self-cleaning effects.

The low surface energy fibers such as polypropylene and polyester are widely used in many nonwoven applications and inherently they are hydrophobic, so their wettability is generally achieved by a hydrophilic surface treatment such as a wet-chemical treatment. Plasma processes have been successfully applied to improve the wettability of inherently hydrophobic surfaces. Moreover, plasma treatment has been recognized as a pretreatment technique for enhancement of the adhesion of a nonwoven surface to another or a coating layer to a nonwoven substrate. The addition of oxygen-containing functional groups, such as –OH, –C=O, –COOH, on surfaces through plasma treatment was found to increase the dye-uptake and printability of nonwoven substrates.

Plasma technology continues to offer opportunities to tailor the surface properties of nonwovens and impart unique characteristics to the fabrics; yet there are still challenges in the integration of the plasma systems to industrial-scale roll-to-roll production lines. Although there are available industrial atmospheric plasma treatment systems, particularly the incorporation of plasma thin-film deposition to industrial roll-to-roll processes is still at an evolving stage.

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