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Plasma techniques for enhancing silk fabric properties

Amina L. Mohamed [a] and S. Abd El-Moez [b]*

[a] *National Research Centre (Scopus affiliation ID 60014618), Textile Industries Research Division, Pre-treatment, and Finishing of Cellulose-based Textiles Department, 33 El-Behouth St. (former El-Tahrir str.), Dokki, P.O. 12622, Giza, Egypt*

[b] *Liner conture Makeup GmbH, Tal 18 80331 Munich, Germany*

Abstract

Modern environmental finishing processes, like plasma, are necessary for society, and the textile sector is on the lookout for cutting-edge production technology to improve fabric quality. Corona discharge at atmospheric pressure (CDAP) and atmospheric-pressure dielectric barrier discharge (APDBD) are two treatments that are quickly becoming the norm in the textile industry as a result of their many benefits over more conventional wet processing techniques. It is possible to initiate plasma using air or standard industrial gases like hydrogen (H2), nitrogen (N2), and oxygen (O2) at room temperature and pressure. After plasma treatment, materials like silk and surface fabrics get desirable features including antibacterial, flame retardant, antistatic, and UV protection thanks to the surface groups introduced by plasma.

Keywords: plasma techniques; silk fabric properties

Introduction

In textile industry, in order to meet the consumer requirements and reducing the costs, the innovation methods have been used. In this context, the plasma ultrasonic and microwave applications can be defined as innovative methods in the textile industry. In textile industry the plasma treatment is used as a pre-treatment before the dyeing process.

Plasma In textile industry

Plasma known as the materials 4th state and described as an ionized gas consists of electrons neutrons, ions and radicals. Generation of plasma can be created by applying an electric field to a low-pressure gas, as in neon or fluorescent tubes. Plasma can also be created by heating a neutral gas to very high temperatures.

Plasma technology is well known for imparting functional finishes to textile materials without the use of harmful chemicals or water, an environment-friendly surface modification. By plasma pre-treatment, the functionalities achieved include altered moisture relations (absorbance or repellence) and fiber capillarity faster dye or ink uptake), antimicrobial, soil repellence, stain resistance, soft handle and improved dyeing. [1-10]

This variety is possible by altering process parameters such as supply frequency, discharge power, treatment time, degree of ionization and type and pressure of gas. For example oxygen or helium plasma increases moisture absorbance while fluorocarbon increases water repellence. Several surface phenomena such as adsorption, desorption, etching, cleaning, surface activation and cross-linking occur singly or in combination on exposure to plasma. [11]

There has a number of report show the use of plasma treatment on modifying polyester, acrylic, wool, polyethylene, nylon ,6 cotton, silk, blended cotton-polyester and polyacrylonitrile with the use of different gases such as oxygen, nitrogen, argon, helium, carbon dioxide fluorine, CF4, SF6, air, or water vapor. Among the different plasma gases, oxygen is a good medium for introducing hydrophilic groups to the materials surface with easy operation criteria. Many researchers have improved the hydrophilicity of natural and synthetic fibers (cotton, wool, flax, linen, silk, PP, PET and PA6) by using plasma treatment. [1, 12-53]

Classification of plasma In textile industry

In textile industry, the plasma application is classified as thermal and cold plasma. Generation of plasma can be created by applying an electric field

*Corresponding author: Heba Ghazal, E-mail: drheba_ghazal@yahoo.com
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to a low-pressure gas. Plasma can also be created by heating a neutral gas to very high temperatures. Since the thermal plasma energy cause faster ignition on the textile materials, the cold plasma application become in use. The cold plasma application can be classified as low and atmospheric pressure plasma treatments depending the used pressure.

The plasma application on the textile materials recently found more use since it causes various modifications on the fiber surface. there seen recovery on wettability, water repellence and dyeability behavior on the fiber. After different gas plasma application on the textile materials, the dyeability behaviors of the textile materials improve as a results of surface modifications and forming new functional groups. [54]

Silk Fabrics as an animal fabric

Silk has known traditionally as the 'queen of fibres'. It is the only fibre among all natural fibres that is spun. It has been used in textiles for more than 5000 years because it has unique lustre, tactile properties, durability, and dyeability. In order to improve the plasma effect, the sericin should be removed from the silk fiber. This treatment also increases the water absorption, dyeability and water repellency .In addition, silk fibres have good mechanical properties ،although they have the lowest weight among natural fibres .Silk is a filament fiber from the cocoon of silkworm. [12]

Silk is natural polymer consisting of repetitive hydrophobic and hydrophilic peptide sequences. Silk has poor wetting properties. Wettability is the initial behaviour of a fabric, yarn, or fiber when brought into contact with a liquid. It also describes the interaction between the liquid and the substrate prior to the wicking process. Wetting and wicking are important phenomena in processing and applications of fibrous materials. Fiber's behaviour in wetting and wicking is important to describe fluid transport in fibrous media. In most of the wet processes used on fibrous materials, uniform spreading and penetration of liquids into pores is essential for better performance of resulting products. Wetting of textiles involves several primary processes: immersion, capillary sorption, adhesion, and spreading. [12]

Wettability is important for characterizing liquid transport, fiber surfaces and adhesion with a polymer. Wicking is the capillary action of fluids that allows a liquid to flow through tight spaces, even against gravity. Wetting is a prerequisite for wicking. If a liquid does not wet fibers, it cannot wick into a fabric. Wicking and wetting are two interrelated processes. The ability of the fabric to wick depends on the surface properties of the constituent fibers, thickness, and their total surface area. Wetting and wicking are significant properties of textile and have a great industrial importance for many operations viz., scouring, dyeing, and finishing.

By changing the chemical composition of the fibrous material, it can modify its overall surface wetting properties. It has been reported that the hydrophilicity of the silk fabric is drastically improved by being pre-treated with amine solutions, such as serine, glycine, and aspartic acid.

Silk, however, is highly sensitive to concentrated alkalis and acids. Its lustre is affected and reduced by dilute acids and boiling in dilute alkalis also. This limits the chemicals and auxiliaries that can be used in the further processing of silk to improve its competitiveness. With the increasing popularity of natural products, the demand for silk not only for clothing use but also for personal care products has been increasing recently. Therefore, physical and chemical modification techniques are being developed for treating silk in order to increase its serviceability. Among the different treatments, most of the researchers have concentrated on low-pressure glow discharge plasma to improve the wicking properties of the silk fabric.

Plasma Treatment on Silk Fabrics

A number of researchers have studied the surface properties of silk fabrics at low-pressure plasma, treated the silk fabrics with different gasses such as O2, N2 and H2 plasmas for deep dyeing, they have improved the surface properties of silk fabric with oxygen low temperature plasma treatment. Among the different plasma gases used for improving wettability, oxygen is a good medium for introducing hydrophilic groups into the surface of the material with easy operation criteria. Plasma treatment on silk fabrics changes only the uppermost atomic surface layers of the fabric, while bulk properties are unaffected due to low range of penetrations.

Atmospheric pressure plasma is used for changing the surface functionalities like hydrophobic to hydrophilic. Introducing polar groups onto the fiber surface can increase the hydrophilicity. Plasma treatment modifies the physicochemical properties of the fabric surface.

It can be used to enhance the properties of fibres, such as their adhesion, wettability, and capillarity (faster dye or ink uptake), thus resulting in significant improvement in dyeing depth. has studied the surface morphology of silk fibroin fibers with low-temperature argon plasma which enhanced the surface roughness of the fabrics. has treated the silk fabrics with RF Ar plasma and improved the tensile strength at 5–10 min treatment time. [55] Since the surface modification under low-pressure plasma in textiles requires vacuum, longer treatment time for surface modification and plasma system should be operated in a batch mode as every time new sample needs to be loaded, Therefore, atmospheric pressure plasma is an alternative method for large scale production.

Dielectric barrier discharge (DBD) is a cold, non-equilibrium; atmospheric pressure plasma has been used in many industrial applications including the textiles department for surface modifications. DBD plasma can be used in the textile material to alter the surface properties of the fabrics. Plasma treatment has advantages when compared to the conventional wet chemical process in terms of reduction of waste and contamination problems and time. [54]

Mechanism of plasma treatment

The plasma treatment of silk fabrics was carried out in Atomflo 400 Series atmospheric plasma (Surfx Technologies, USA) in a conditioned environment (temperature 21 ± 1°C and relative humidity 65 ± 2%). Helium (99.95% purity) and oxygen (99.7% purity) were used as the carrier and reactive gases respectively. The treatment was carried out using a rectangular nozzle mounted vertically above the substrate (Figure 1).

The nozzle was heated to 100°C, and the helium flow rate was kept at 30 $l.min^{-1}$ during the treatment. The output powers used were 120, 130, and 150 W with respect to the corresponding oxygen flow rates of 0.1, 0.3, and 0.5 $l.min^{-1}$ respectively.

During the plasma treatment, the silk fabrics were placed firmly on the treatment platform. Three silk fabrics were treated under the same treatment conditions in order to ensure uniformity of treatment.

Different treatment conditions were combined with the following parameters: oxygen flow rate (0.1, 0.3, and 0.5 $l.min^{-1}$), treatment time (0.1, 0.5 and 1.0 s), and jetto- substrate distance (1, 2 and 3 mm); The plasma-treated fabrics were stored under standard conditions (i.e. a temperature of 21±1°C and a relative humidity of 65 ± 2%) for 24 h prior to further evaluation. [1]

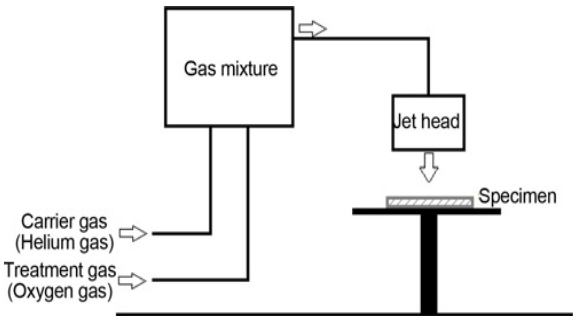
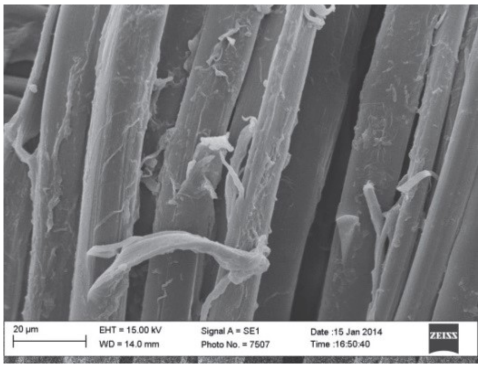


Figure 1. Schematic of the atmospheric plasma jet treatment system

Surface Analysis (SEM Analysis)

The scanning electron microscope micrographs of samples were given in Figure 2. According to the SEM micrographs, one can conclude that nitrogen and oxygen plasma treatment caused deformation on the surface of silk fabric, Plasma treatment causes microcracks and new functional groups in silk fibers. It was considered that the deformation of the plasma treatment increased in the capillarity of plasma treated sample. The amount of carbon reduced while the amount of oxygen and nitrogen increased on the surface of the oxygen and nitrogen plasma treated silk fabric.

It is concluded that, the reduced carbon ratio could be originated from the functional groups increase around the oxygen and nitrogen atoms. [12]



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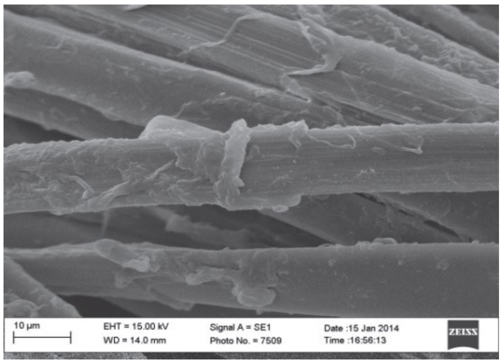

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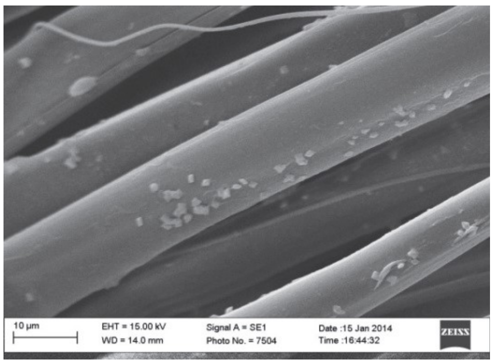

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Figure 2. SEM micrographs of samples
(a). The sample applied nitrogen plasma for 10 min,
(b). The sample applied oxygen plasma for10 min,
(c). The sample untreated plasma application

The XPS analysis of oxygen and nitrogen plasma applied silk fabric, the results shows that the amount of carboxyl, hydroxyl and amine groups increased. Besides, by the analysis of the results, it can be said that the nitrogen plasma occurred amid groups to some extend by making chemical reaction on with carboxyl groups.

The degradation rate (weight loss) of the plasma-treated fabric increased with increasing the exposure time due to etching of contaminant layer. This contaminant layer is responsible for hydrophobicity of the fabric. This etching process is predominant on the amorphous region of the surface than the crystalline region. Therefore, it is possible that the initial rate of the etching is more rapid. Once all the amorphous materials on the surface has been removed, the remaining crystalline and tightly bound amorphous regions could not be removed easily, causing decline in the etching rate. After the removal of this waxy layer, the fabric acquires hydrophilicity. [56]

The effect of plasma treatment on the dyeing properties of silk fabric.

Actually, studies have shown that plasma treatment is effective in enhancing the uptake of certain dyes owing to modification of the fibre properties. Comparing specimens treated under different oxygen flow rates, it was found that by the fact that an increase in oxygen flow rate results in an increased number of reactive species in the plasma and thus the formation of more polar groups As a result, silk fabrics treated with a higher oxygen flow rate more dye was absorbed.

Secondly, when comparing specimens treated under different exposure times, it was found that the accumulation of active species within the same area of silk fabrics. Therefore, more polar groups are formed, and thus hydrophilicity is increased and more dye is absorbed.

Lastly, when comparing specimens treated under different jet-to-substrate distances, it was found that with increase in the distance between the jet and the fabric surface, the velocity and the activity of the active species in the plasma jet decrease by the time it reaches the top side of the fabric. [1]

The higher the concentration of colourant in the substrate, the higher is the K/S value. It can be seen that the K/S values for plasma-treated degummed silk fabrics are higher than those for untreated degummed silk fabrics, indicating that the concentration of dye in the plasma-treated degummed silk fabrics is higher, i.e. the dyeability of the fabrics has been improved by the plasma treatment. The reason for this is probably the formation of polar groups on the fabric surface.

The surface roughness caused by etching also increases the surface area and aids wetting. The K/S values for raw silk fabrics are always higher than those for degummed silk fabric. This might be because raw silk fabrics are thicker, and therefore the amount of dye per unit surface area of fabrics (regardless of fabric thickness) is higher, thus giving higher K/S values. [1]

A number of researchers have studied silk fabrics were dyed with the natural dyes extracted from the fruits of Sambucus Ebulus L. by conventional and microwave methods. Before the dyeing process, silk fabrics were treated with oxygen plasma for 1 and 5 minutes in order to see if there is an increase in dyeing properties. After the dyeing process, the samples having deep shades were treated with three different fixing agent causing increase the fastness properties of samples. According to the results, the colour strength of the samples increased with increase of the duration of the plasma treatment. Furthermore, the results show that the microwave energy caused to increase the colour strength of samples. As known from the literature, microwave heating results in increasing the diffusion of organic molecules into polymers, which in turn increase the fixing rate of dyes into the polymeric textiles as a result the colour strength of samples increased with the microwave energy. [12]

Advantages and Disadvantages of Plasma Treatment of Textile Materials

Advantages

1. Endless chemical modifications are possible by choosing appropriate gasses or chemicals.
2. The dyeability behaviors of the textile materials improve as a results of surface modifications and forming new functional groups
3. By plasma pre-treatment, the functionalities achieved include altered moisture relations (absorbance or faster dye (repellence) and fiber capillarity or ink uptake), anti-microbial, soil repellence, stain resistance, soft handle and improved dyeing.
4. In most cases, it can be a dry process, reducing water consumption and energy to dry the treated materials.
5. Reduction for usage results in reducing the amount of wastewater and the waste water treatment cost.
6. It has an economical advantage over the conventional wet processing due to its low chemical consumption and reduction in chemical and water costs.

7. Although for the above reasons all plasma processing is more environmentally friendly in comparison to the textile wet processing, the closed plasma treatment systems are an even more environmentally friendly process because the plasma by-products can be trapped rather than being released into the environment.

Disadvantages

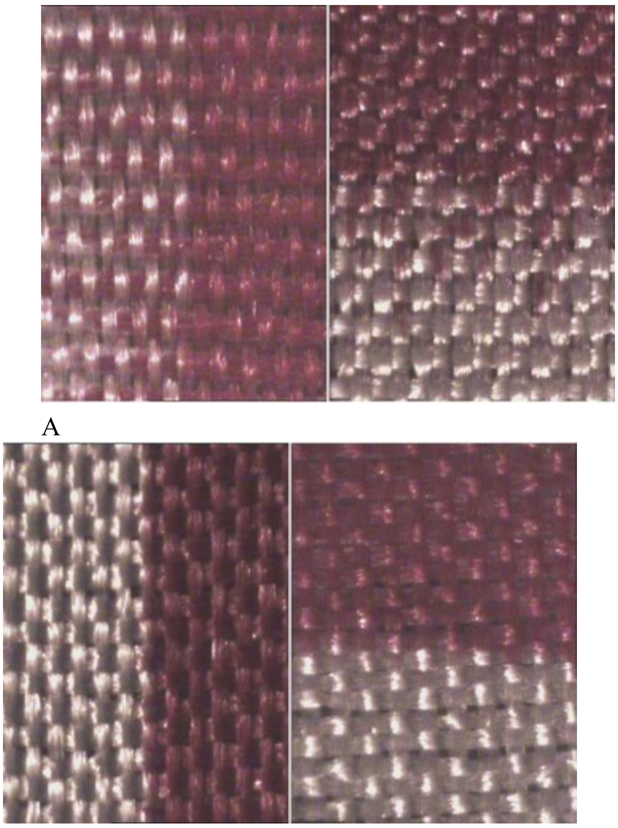
1. The degradation rate (weight loss) of the plasma-treated fabric increased with increasing the exposure time due to etching of contaminant layer. This contaminant layer is responsible for hydrophobicity of the fabric. This etching process is predominant on the amorphous region of the surface than the crystalline region.
2. System dependency is one of the most important disadvantages of the plasma treatment. This means that the same flow rate, gas pressure and power input may not produce the same level of the needed reacting species.
3. purchasing expensive plasma equipment and high vacuum pumps are considered to be limiting factors and could be considered as a disadvantage
4. It is harder to predict the exact structural characteristics of the plasma treated area for a more complex molecule. This is due to the fact that plasma causes the complex molecular structure to fragment into a multitude of coexisting active species which could react or deposit on the surface.

Atmospheric-pressure plasma method for improving the pigment-based ink-jet printing performance of silk fabrics

As an environmentally friendly technique, atmospheric-pressure plasma which is a secure dry-preprocessing method has been applied for textile surface modification more recently. the silk fabrics were processed with air plasma to improve surface strength and anti-bleeding performance of the treated surfaces.

According to literatures, the highly charged positive ions and photons which are the main species in plasma have a remarkable ability to break primary chemical bonds. According to the content change of C–C/C–H component, it is generally agreed that some of C–C bonds in silk surface were attacked and broken during the plasma treatments. The carbon radicals, formed by removal of hydrogen within the polymer chain will recombine with atomic oxygen and nitrogen generated by the electron impact dissociation in plasma. That reactions resulted in the formation of oxygen and nitrogen-containing polar groups which were introduced onto the fabric surface and promoted the wettability.

Fig.3 shows the anti-bleeding property of silk fabrics before and after plasma modification. As shown in (a), the bleeding phenomenon of original fabric was severe along weft and warp edge of the printed color-block. By contrast, the bleeding resistance was dramatically improved with excellent edge definition after plasma treatment which could be seen at (b). As well-known, hydrophobic fabrics cause less ink absorption. Surface roughness caused by plasma etching increased fabric surface areas, captured more ink, and enhanced a larger ink color gamut and ink adhesion. It was also considered that the polar groups which were introduced onto the fabric surface improved the hydrophilicity of the fabric.



A

B

Figure3

The modified sample has deeper color (higher K/S value), darker shade (lower L* value) and higher chroma (C* value) than the untreated one. The surface wettability of the silk fabric had been remarkably enhanced which has been demonstrated by contact angle measurement. Furthermore, the ink-jet printing properties of silk were verified to be superior as shown by the higher K/S value and distinct printing edge. From the above, atmospheric-pressure plasma has been proved effective to improve the surface properties and actual printing performance of silk fabrics. The ink-jet printing is best to be carried out within 24 hours after the plasma processing. [54]

Plasma Pre-treatment on Tasar Silk Fabrics Coated with ZnO Nanoparticles against Antibacterial Activity

ZnO nanoparticles (ZnO NPs) play a significant role in antibacterial, cleaning and UV protection. ZnO NPs have been synthesized by the wet chemical method and the average crystallite size of the same is found to be 49 nm using XRD. Plasma treated silk fabrics are coated with ZnO NPs using vacuum thermal evaporation technique. It is found that the plasma pre-treated silk fabrics coated with ZnO NPs have better antibacterial activities against Escherichia Coli bacteria. [57]

Tasar silks are coarsest of the silk fibers. It is used in base materials for furnishing fabrics, handicrafts and wearing clothes. It is usually affected by strains, microbes, and sensitive to UV radiation in sunlight. The use of many antimicrobial agents is avoided because of their possible harmful or toxic effects. Application of nanotechnology in textiles is started by Nano-Tex to provide durable fabrics because nanoparticles have a large surface area-to-volume ratio and high surface energy.

Recently, the use of inorganic nanoparticles coating on textile fabrics works towards the fabrication of antibacterial textiles. Some metals and metal oxides at low concentrations are toxic to microbes. Silver (Ag), Zinc oxide (ZnO), Copper (Cu), Copper oxide (CuO), Titanium dioxide (TiO_2), Magnesium oxide (MgO), are commonly used as inorganic materials within the fabrication of antimicrobial coatings.

Out of which, ZnO NPs have its unique optical, chemical, electrical properties and it has a wide bandgap (3.3 eV) of the II-VI semiconductor groups. The ZnO NPs are used in solar cells, piezoelectric properties, gas sensors, photocatalytic, UV protection, microbes resistant, skin care applications and so on. The ZnO NPs are non-toxic to humans and animals. It inhibits the growth of bacteria due to the increased surface area as reduced particle size.

Many researchers have coated ZnO NPs on cotton, wool, polyester etc to improve UV protection, antibacterial effect, and hydrophobic properties. The ZnO nanomaterials can be prepared onto polyanilines to improve their hydrophobicity. The application of ZnO NPs on textile fabrics is aimed to produce antibacterial fabrics. To increase the adhesion of nanoparticles on the textile materials various pre-treatment has been done.

Plasma pre-treatment is offering an attractive path to enhance the surface properties of the textile material. Cold plasma (glow discharge) is an eco-friendly technology that has opened up many new possibilities for numerous industries including textiles.

The plasma treatment etches on the fabric surface in the nano range, which modifies the surface properties of the fabric to enhance the adhesion of the material. tasar silk fabrics have been pre-treated with argon glow discharge plasma with constant power and different time parameters (5, 10, and 30 min).

ZnO NPs have been synthesized and then deposited by a thermal evaporation method on the plasma treated silk fabrics to achieve a fresh glide path to develop high value-added silk fabrics which provide good adhesion of ZnO NPs and improved antibacterial performance.

The Tasar silk fabric is coated with ZnO NPs using a vacuum thermal evaporation system and schematic of the system is shown in Fig. 4. Control of coating thickness and uniform coating are the major advantages of the system. The physical method of coating can easily overcome the environmental pollution related to chemical treatments and at the same time, it can also increase the adhesion properties of the fiber.

The thermal evaporation is a cylindrical system with 40 cm in height and 30 cm in width. The base pressure of the system is attained of the order of 10-5 mbar using a diffusion pump backed by a rotary pump. The plasma coated fabrics are placed in the substrate holder and the ZnO NPs are placed on the molybdenum boat under a vacuum of 10-5 mbar.

Applying the low tension current of the order of 6.0-7.0 A on the boat, ZnO nano-powder started to evaporate and deposited on the fabrics. The typical deposition time is kept for 2-3 minutes.

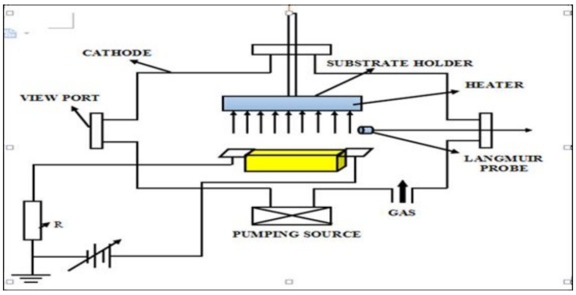


Fig. 4: Experimental set up of thermal evaporation unit

The surface morphologies of raw, untreated and plasma treated ZnO coated fabrics are shown in

Fig.5. From Fig.5 (a) shows the raw fabric at the magnification of 500 and Fig.5 (a1) shows the cross-section view of the fabrics at the magnification of 1k.

The raw fabrics have deep grooves on the surface. Fig. 5(b and b1) represents that the ZnO coated fabrics without plasma treatment have dendrite-like structure created on the surface which confirms the deposition of ZnO. It is clearly observed from Fig. 5 (c and c1), there is a drastic change in the fiber surface with the presence of a smooth coating of ZnO on the fibers. Similarly, Fig. 5(d and d1) show dense ZnO NPs covered on the fabrics. It could be seen that plasma treatment of the fiber could promote coating of ZnO nanoparticles on fibers when compared with that of untreated sample. Plasma treatment increases the adhesion properties of fibers. [57]

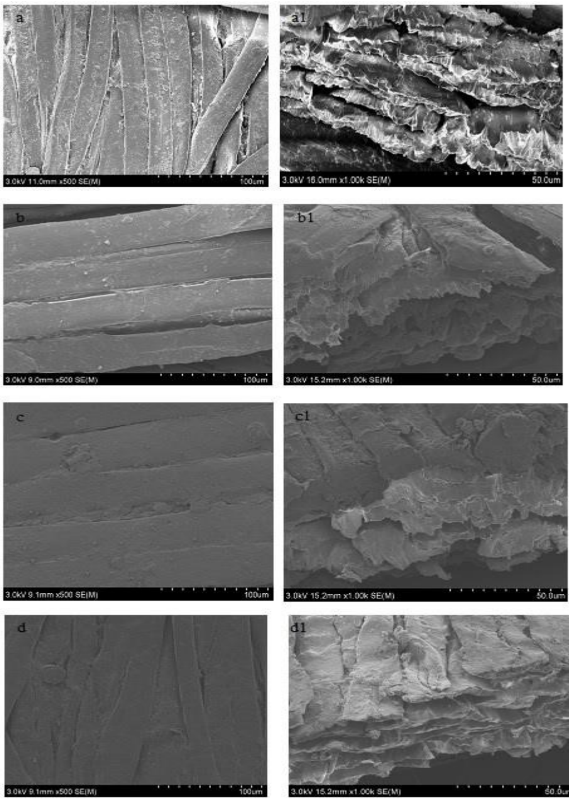


Fig.5. SEM micrograph of (a) raw, (b) untreated fabric coated with ZnO NPs, (c) 5 mins Plasma treated fabric coated with ZnO NPs, (d) 30 mins Plasma treated fabric coated with ZnO NPs and a1, b1, c1, d1 shows the cross-sectional view of untreated and plasma treated silk fabric coated with ZnO.

The antibacterial test of all the fabrics is carried out against E. Coli (ATCC 25922- American Type Culture Collection, Rockville, MD). The agar disc diffusion plate method is applied to the qualitative bacterial test. The bacteria membrane wall damage due to the direct interaction between Zn^{+2} ions and the cell wall or ZnO generates the active oxygen species which can destroy the cell wall. The zone of inhibition for all the fabrics is shown in Fig.6. There is a dense population of bacteria colonies around the raw fabric and no inhibition zone is formed (Fig. 6 (a)).

A distinct inhibition zone width of 10 mm is observed for without plasma treated fabric coated with ZnO NPs (Fig.6 (b)). Conversely, the plasma-treated silk fabric coated with ZnO NPs exhibits a clear inhibition zone of 12 mm around the sample, which clearly declares that the plasma treatment process gives a better antibacterial performance (Fig.6 (c)). Tasar silk combined with ZnO NPs is suitable for self-cleaning, antibacterial and medical applications such as surgical sutures, drug delivery, sensor etc. The human body without any harmful effects also accepts it. [57]

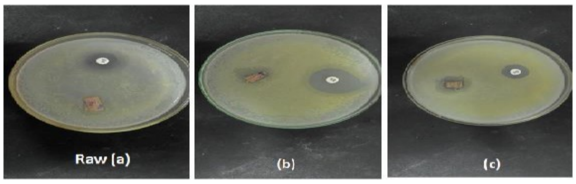


Fig. 6. Antibacterial activity E. Coli of (a) raw, (b) untreated fabric coated with ZnO NPs, (c) 5 mins Plasma treated fabric coated with ZnO NPs

Recommendation

ZnO Coated & plasma pre-treated silk fibers showed excellent antibacterial activity against E. Coli bacteria. Plasma pre-treatment of silk fabrics has been made using plasma unit under various experimental conditions. Plasma pre-treatment on silk fabrics improves the adhesion of ZnO nanoparticles.

The study indicates some hydrophilic polar groups, such as C－O, C=O and N－C=O, generated during plasma discharging were introduced onto fiber surface. Consequently, the surface wettability of the silk fabric had been remarkably enhanced.

Furthermore, the ink-jet printing properties of silk were verified to be superior as shown by the higher K/S value and distinct printing edge.

From the above, atmospheric pressure plasma has been proved effective to improve the surface properties and actual printing performance of silk fabrics.

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The authors declare that there is no funder

Conflict of Interest

There is no conflict of interest in the publication of this article.

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تقنيات البلازما لتعزيز خصائص أقمشة الحرير

أمينة لطفي محمد أ و ش. عبد المعز ب*

[أ] *المركز القومي للبحوث (معرف الانتساب إلى سكوبس 60014618)، معهد بحوث وتكنولوجيا النسيج، قسم التحضيرات والتجهيزات للالياف السليلوزية ، 33 شارع البحوث (شارع التحرير سابقًا)، الدقي، ص. ب. 12622، الجيزة، مصر*

[ب] **Liner conture Makeup GmbH**، *تال 18 80331 ميونيخ، ألمانيا*

المستخلص

إن عمليات التشطيب البيئي الحديثة، مثل البلازما، ضرورية للمجتمع، ويبحث قطاع المنسوجات عن أحدث تقنيات الإنتاج لتحسين جودة الأقمشة. يعد تفريغ الهالة عند الضغط الجوي (CDAP) وتفريغ الحاجز العازل عند الضغط الجوي (APPDB) معالجتين أصبحتا بسرعة القاعدة في صناعة المنسوجات نتيجة لفوائدهما العديدة مقارنة بتقنيات المعالجة الرطبة التقليدية. من الممكن بدء عملية البلازما باستخدام الهواء أو الغازات الصناعية القياسية مثل الهيدروجين (H2) والنيتروجين (N2) والأكسجين (O2) في درجة حرارة الغرفة والضغط. بعد معالجة البلازما، تحصل المواد مثل الحرير والأقمشة السطحية على ميزات مرغوبة بما في ذلك مقاومة البكتيريا ومقاومة اللهب ومضادة للكهرباء الساكنة والحماية من الأشعة فوق البنفسجية بفضل المجموعات السطحية التي أدخلتها البلازما.

الكلمات المفتاحية: تقنيات البلازما؛ خصائص الأقمشة الحريرية