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Review Article

REVOLUTION OF NOVEL AND ECO-FRIENDLY PLASMA TREATMENT TECHNOLOGY IN THE FIELD OF TEXTILES; FUTURE PERSPECTIVES IN TECHNICAL TEXTILES OF DEFENCE APPLICATION

M S Yogendra Kumar* and Francis V Varghese

Defence Bioengineering and Electromedical Laboratory, Defence Research and Development Organization, C V Raman Nagar, Bengaluru – 560093 India

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ABSTRACT

Textile industry is searching for innovative and eco-friendly production and finishing techniques to improve the product quality. Plasma surface modification does not require the use of water and chemicals, resulting in a more economical and ecological process. In addition, a large variety of chemically active functional groups can be incorporated or generated on textile surface without affecting its bulk properties. Possible aims of this are improved hydrophilicity, antimicrobial agents/dye up taking, coating, hydrophobicity and/or oleophobicity, etc.

This article is reporting the current status of atmospheric plasma treatment on the textiles, its effect on physico-chemical properties, characterization of treated textiles by measurement of contact angle, wicking height, SEM, FTIR, XPS and future perspectives of plasma in technical textiles of Defence Application.

INTRODUCTION

The concept of plasma as "fourth state of the matter" was suggested by Sir William Crooks in 1879 and in 1926 Irving Langmuir used the term plasma first time[1, 2]. Plasma is defined as partially ionized gas, composed of highly excited atomic, molecular, ionic and radical species with free electrons and photons. This mixture of reactive species makes it a unique and diverse media for surface modification. Plasma treatment technology is a dry and clean process which offers numerous advantages over conventional chemical process. This technology has been successfully used in various areas such as electronics, automobiles, medical, biomedical, plastic industry, space application, textile industry, nanotechnology, waste management, etc.

In developed countries especially in the field of textile industry there is an increasing global competition in the world market. This aspect forced to exploit their high technical skills in the development of textile materials for high quality and technical performances[3]. Increasing concerns on environmental and health issues due to the usage of large quantities of water and hazardous chemicals in the conventional textile finishing techniques lead to the development of new eco-friendly technologies. Plasma treatment process has been reported as superior among all the available processes for surface modification on the textiles.

Corresponding author:* **M S Yogendra Kumar
Defence Bioengineering and Electromedical Laboratory, Defence Research and Development Organization, C V Raman Nagar, Bengaluru – 560093 India

It is a dry and environmental friendly method to achieve surface alteration without modifying the bulk properties of the materials[4]. In particular, non-thermal plasmas are especially suited because most textile materials are heat sensitive polymers[5,6]. Atmospheric pressure plasma (APP) equipment is an alternative and cost competitive method among plasma technologies to wet chemical treatments, thus avoiding the need of expensive vacuum plasma equipment and allowing continuous and uniform processing of fibers surfaces[7,8]. APP technology offers advantages over low pressure systems, such as working in the atmospheric pressure, possibility of integration with the existing textile processing set up, etc.

Plasma surface treatment process is being used to enhance the quality of textile products in fabric preparation, dyeing and finishing methods for improving the functional requirement as per specific requirements. In this article, recent developments in the plasma treatment on textile surfaces are presented. In the first part a brief discussion on plasma then followed by some case studies and the characterization of the treated materials by measurement of contact angle, surface energy, SEM, FTIR and XPS is reported. The last part contains a description on the future perspectives of plasma technology in the field of technical textiles specifically of Defence application.

Plasma technology[2,9]

Plasma medium was generated by coupling of electromagnetic power into a process gas volume containing dynamic mixture of neutrons, electrons, ions, photons, meta-stable excited species, free radicals of molecules and fragments of polymer at room temperature (Figure 1 and Figure 2). This allows the surface fictionalization of fibers and textiles without affecting their bulk properties. These generated species move in the diffusion gradients, electromagnetic fields, etc. on the placed textile substrates or passed through the plasma. This enables surface modification such as surface activation by bond breaking to create reactive sites, grafting of chemical moieties, generation of functional groups, material surface etching, dissociation of surface contaminants/layers and deposition of conformal coatings. In these entire processes a highly surface specific region of the material (□1000 Å) is given new, desirable properties without negatively affecting the bulk properties of the constituent fibers/fabrics.

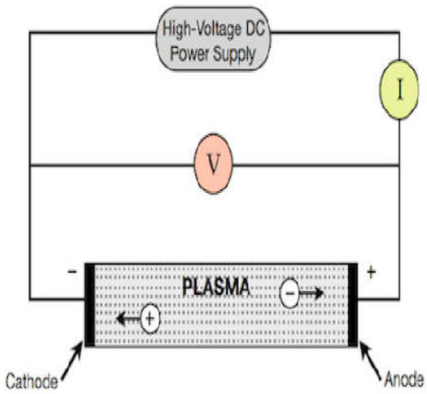

Figure 1 Generation of Plasma[2]

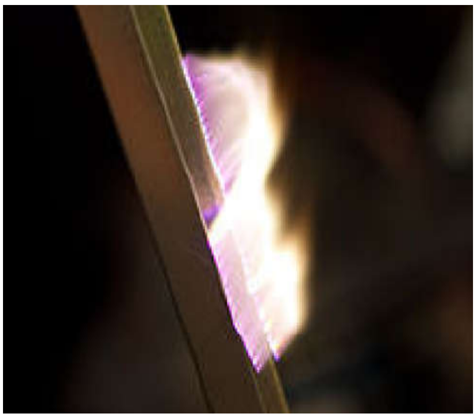

Figure 2 Artificial plasma produced in air[2]

Plasma reactors

Types of power supply required for the generation of plasma: a) Low-frequency (LF, 50–450 kHz), b) Radio-frequency (RF, 13.56 or 27.12 MHz) and c) Microwave (MW, 915 MHz or 2.45 GHz). The requirement of power ranges from 10 to 5000 watts, depending on the size of the reactor and the desired treatment.

Low-pressure plasmas

This is one of the highly mature technologies in the field of microelectronics industry. However, the requirements of microelectronics fabrication are not compatible with textile processing, and many companies have developed technology of low pressure reactors to achieve an effective and economically viable batch fictionalization of fibrous products and flexible web materials. Vacuum systems requirement for the development of low-pressure plasmas is the burden for textile industry; hence atmospheric pressure plasma will be more appropriate technology.

Atmospheric pressure plasmas

The most common forms of atmospheric pressure plasmas are;

Corona treatment

Corona discharge is characterized by bright filaments extending from a sharp, high-voltage electrode towards the substrate. This process has the advantages of operating at atmospheric pressure and generally using ambient air as reagent gas. In principle, these systems do have the manufacturing requirements of the textile industry, but the type of plasma produced cannot achieve the desired spectrum of surface modification especially on textiles and nonwovens. These systems have an effect only on the loose fibers hence its effects in the textiles are limited.

Glow discharge

This is defined as a homogeneous, uniform and stable discharge generally generated in inert gasses such as helium, argon (also some in nitrogen). This type of plasma will be generated by applying radio frequency voltage between two parallel plate electrodes. Atmospheric Pressure Glow Discharge (APGD) offers an alternative homogeneous cold-plasma source, which has many of the benefits of the vacuum, cold-plasma method, while operating at atmospheric pressure.

Dielectric barrier discharge (Silent discharge)

It is a broad class of plasma source that has an insulating cover over one or on both of the electrodes and it operates with high voltage power ranging from low frequency AC to 100 kHz. These results in a non-thermal plasma and a multitude of random, numerous arcs form between the electrodes. These micro discharges are non uniform and causes uneven treatment on the surface. Among the available three different kinds of atmospheric pressure plasma technologies dielectric barrier discharge technology (DBD) is one of the most effective non-thermal atmospheric plasma sources and has been attracting increasing interest for industrial textile applications[10,11].

Plasma processes[2,9,12]

Plasma processes performed to modify fibers and polymer surfaces and are classified into four overall processes: Cleaning, activation, grafting and deposition

Cleaning process: In this process Ar, He and oxygen plasmas are used. The plasma-cleaning process removes, via ablation, organic contaminates such as oils and other production releases on the surface of most industrial materials. Surface contaminants undergo abstraction of hydrogen leading to free radical formation and chain scissions with the influence of electrons, ions and free radicals as shown in Figure 3.

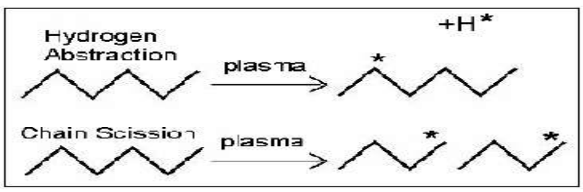

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Fig 3 Free radical formation, abstraction of hydrogen from the polymeric chain or can split chain[12].

3.2 Activation plasma processes: This process occurs when a surface is treated with a gas, such as oxygen, ammonia or nitrous oxide and others that does not contain carbon. The primary result is the incorporation of different moieties of the process gas onto the surface of the material under treatment. Polyethylene solely consists of carbon and hydrogen only. By plasma treatment surface can be activated with the incorporation of functional groups such as hydroxyl, carbonyl, peroxyl, carboxylic, amino and amines (Figure 4).

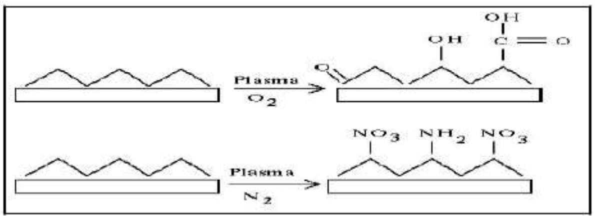

Figure 4 Surface activation by substituting hydrogen in a polymeric chain with other groups (O, OH, COOH, NO3, NH2, etc.)[12]

Almost all fiber or polymer surface may be modified to provide chemical functionality to specific adhesives or coatings, significantly enhancing the adhesion characteristics and permanency. Activated polymers will have enhanced adhesive strength as well as permanency and it will be a great improvement in the development of technical textiles.

Grafting: Inert gas such as argon is employed as process gas in the grafting. Free radicals generated on the surface of the material react with the monomer leading to grafting. This is a low-pressure plasma treatment proces for grafting and grafting can be also done by atmospheric plasma processing. Typical monomers are acrylic acid, allyl amine and allyl alcohol (Figure 5). Plasma treatment increases the surface energy, wettability and adhesion strength.

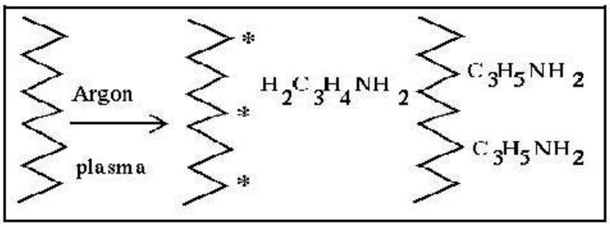

Figure 5 Grafting of a monomer on the surface[12]

Deposition: Plasma can also produce a material deposition when a more complex molecule is employed as the process gas, a process known as plasma-enhanced chemical vapor deposition (PECVD). Methane or carbon tetrafluoride gas undergoes fragmentations in the plasma, reacting with itself to combine into a polymer. By selecting the suitable process conditions, chemically unique films, can be deposited on the surfaces of materials in the plasma reactor. PECVD coating on the material permanently alters the material surface properties without affecting the bulk properties of the treated material.

Textile applications[9,12,13]

Research and development regarding the applications of plasma processes for textile treatments is very wide. Due to the great amount of reported literature, results of some exemplary applications with related plasma gases are summarized;

1. Improvement of comfort properties: Softening of cotton and other cellulose-based polymers and reduction of felting in the wool by oxygen plasma treatment.
2. Wetting: Improvement of surface wetting in synthetic polymers (PA, PE, PP, PET PTFE) with treatment in O2, air, NH3 plasma. Hydrophilic treatment serves dirt-repellent and also as an antistatic finish.
3. Dyeing and printing: Improvement of capillarity in wool and cotton, with treatment in oxygen plasma. Improved dyeing reported in the polyester and polyamide with $SiCl_4$ and Ar plasma respectively.
4. Composites and Laminates: Good adhesion between layers in laminates depends upon the surface characteristics of fibres in layers and the interactions taking place at the interface. Higher surface energy of fibres is the prerequisite condition for good adhesion, it can be modified with the plasma treatment process.
5. Electrical Properties: Antistatic finish of rayon, with chloromethyl dimethylsilane in plasma.
6. Metal-Coated Organic Polymers: Metal-coated organic polymers are used for a variety of applications. If the metallised polymer is expected to fulfil its function, it is essential that metal strongly adheres to the polymer substrate. This property can be obtained by plasma pre-treatment of the polymer material.
7. Membrane and Environmental Technology: Gas separation to obtain oxygen enrichment. Solution-Diffusion Membranes to obtain alcohol enrichment. Ultra filtration membranes to improve the selectivity. Functionalized membranes such as affinity membranes, charged membranes, bipolar membranes are extensively used in the water treatment process.

Some case studies about the role of plasma treatment on the natural fiber cotton and other synthetic polymeric fabrics as follows;

Hydrophilicity

Plasma treatments for improved wettability have been done on all possible fibre/ fabric types, with varying success. The treatment aims at the generation of surface roughness and introduction of water compatible functional groups such as –COOH, –OH and –NH2. Polyester fabrics shown the improved wettability by the remote DC glow discharge stabilized by a fast airflow at atmospheric pressure and the treatment effect was stable for several days[14]. Similar remote reactor was used to treat cotton yarns. The results show that the wicking rate increases with treatment time and discharge power[15]. The efficiency of the air plasma treatment was tested by measuring the liquid wicking rate based on DIN 53 923 (EDANA 10.172).

The effect of 80 kHz dielectric barrier discharge (DBD) treatment on polyethylene terephthalate and nylon fabrics[16] in air, nitrogen or argon for the treatment of natural, synthetic and mixed fabrics was studied[11]. The enhanced wettability and wickability appeared within the first 0.1–0.2 s of treatment. Many kinds of plasma jets are being used for enhancing the hydrohilicity of the polytetrafluoroethylene (PTFE). In order to increase the production of OH products the ambient humidity was regulated instead of working gas to avoid the instability of the discharge. The results shown the improvement in hydrophilicity of PTFE treated by plasma jet[17].

Dyeing

Physico-chemical improvements by DBD plasma discharge in dyeing process of polyamide 6, 6 (PA66) fiber has been investigated. Analyses by various analytical techniques confirm the high polar functionalization of PA66 fibers due to plasma incorporation of oxygen atoms from atmospheric air[18]. Reactive species generated by DBD plasma does not break aliphatic C-C chain, but preferentially break the C-N bonds of PA66. Author expect the formation of low molecular weight molecules that act as dye “carrier” and creation of micro channels onto polyamide surface seems to favor dye diffusion into the fiber cores as shown in Figure 6.

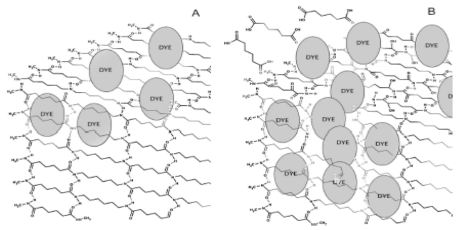

Figure 6 Dyeing mechanism of nylon 66 fiber before (A) and after (B) plasma treatment[18].

After plasma treatment, nylon 66 displays a significant amount of oxygen covalently bonded onto its surface and the presence of micro-channels that promote a deeper diffusion of the dye into the fibers at lower temperatures and shorter dyeing times than traditional dyeing methods[18].

Fourier Infra-red (FT IR) Analysis: Qualitative information about the change in the surface chemistry was determined by FTIR analysis. The attenuated total reflectance FTIR spectrum (Figure 7) of untreated PA66 sample shows the inherent bands of nylon at 3290 cm^{-1}, which may be attributed to NH stretching vibrations (Figure 7a). The peaks at 2930 and 2850 cm^{-1} may be attributed to the CH_2 asymmetric and symmetric stretching vibrations, respectively[19]. The absorption band at1630 cm^{-1}, often referred to as amide I band, may be assigned to the amide carbonyl C=O stretching vibrations. Instead, the amide II band at 1530 cm^{-1} may be attributed to N-H bending motion. As expected, the presence of the hydrogen-bonded secondary amide is confirmed by the in-plane N H deformation vibration of the peak shoulder at 720 cm^{-1}. The band at 680 $cm^{-1}$is attributed to the bending of O-C-N group[20].

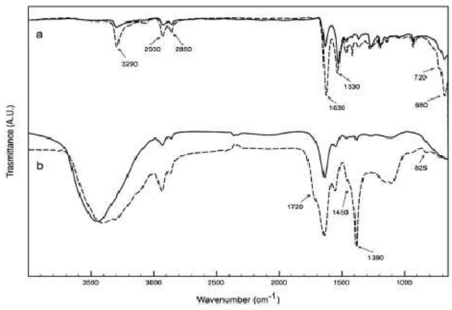

Figure 7 (a) ATR-FTIR spectra of PA66 fabric and (b) and FTIR spectra of the aqueous extraction residue after blank dyeing process. With plasma (dotted lines) and without plasma (solid lines)[18].

Plasma treated sample shows a significant increase in the intensity and broadening of the C=O stretching band as well as of the bending band of the O-C-N group were observed after the blind dyeing. This may be an indication of the micro-environmental changes and oxygen addition on the fiber surface. On the other hand, the significant increase in intensities for the N-H and asymmetric and symmetric C-H stretching vibration bands at 3290, 2930 and 2850 cm^{-1}, respectively, could also be assigned to the formation of low molecular weight etched material due to the air DBD treatment of the PA66fabric[21]. FTIR analysis of the aqueous extraction residues after a blank dyeing process (without dye) shows remarkable differences between untreated and plasma treated PA66 fabrics (Figure 7b). The new shoulder peak at 1720 cm^{-1}adjacent to the increased amide C=O band at 1630 $cm^{-1}$is a result of the additional contribution from carbonyl groups formed by air DBD treatment. The two characteristic IR peaks for CH_3 (intense peak at 1380 cm^{-1}) and for CH_2 (a shoulder peak at 1450 cm^{-1}) suggest that the hydrocarbon fragments from plasma treated PA66 are released in the aqueous medium[22].

X-Ray Photoelectron Spectrosopy (XPS) Analysis: XPS analyses were conducted to obtain a deeper understanding of the degree of chemical modification on the surface of PA66 fibers. The increased atomic ratio of O/C after DBD plasma treatment indicates a substantial incorporation of oxygen atoms onto the surface of fabric as shown in Table 1. Plasma etching may provoke chain scission in groups C-H, C-O, C-N, C-C, N-H present in the PA66 fiber promoting the formation of reactive species such as O^{-}, N, N^{+}, O, OH^{-},O_3, thus causing the decrease of carbon content and the increase of nitrogen and oxygen atoms[18].

Table 1 Chemical structure and relative chemical composition and atomic ratio plasma treated PA66 fabrics

Polyamide 6,6	Untreated	Treated
Carbon(C) at %	74.3	60.5
Oxygen(O) at %	16.3	28.5
Nitrogen(N) at %	9.4	11.0
Ratio O/C	0.22	0.47
Ratio N/C	0.13	0.18

Hydrophilicity and Oleophobicity

Shusen Peng and Yongcun Ma have reported[23] a special wetting surface with hydrophilic and oil-repellent characteristic preparation by treating hydrophilic PEGylated surface with CF_4 plasma. Wettability and oleophobicity of the modified PEGylated polymer surfaces are investigated by contact angle (CA) measurement as shown in Figure 8.

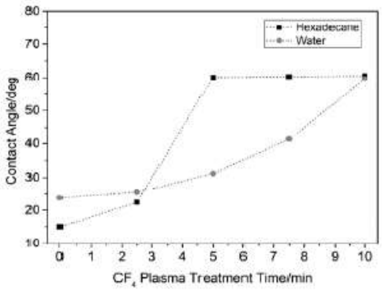

Figure 8 Contact angle measurements of water and hexadecane on the modified PEGylated surfaces with CF_4 plasma treatment time[23].

The results of contact angle measurements shown in Figure 8 indicate that CA of water and hexadecane has different dependences with CF_4 plasma treatment time. After a 5 min treatment with CF_4 plasma, the difference between water and oil CA reaches a maximum value on the modified PEGylated surface, where in water and oil CA is 30.7° and 60.7°, respectively. These results indicate that the special wetting surface with hydrophilic and oil-repellent characteristic is successfully obtained by using CF_4 plasma treatment.

Scanning electron microscopy (SEM) analysis: SEM images of PEGylated surface is shown in Figure 9. The untreated surface is very smooth (Figure 9a). It can find an obvious surface morphology change for sample with 2.5(Figure 9b) and 5min (Figure 9c) CF_4 plasma, while the change of surface roughness is little. It is clear that the surface roughness of samples with 7.5(Figure 9d) and 10min (Figure 9e) CF_4 plasma, which is attributed to etching action of CF_4 plasma.

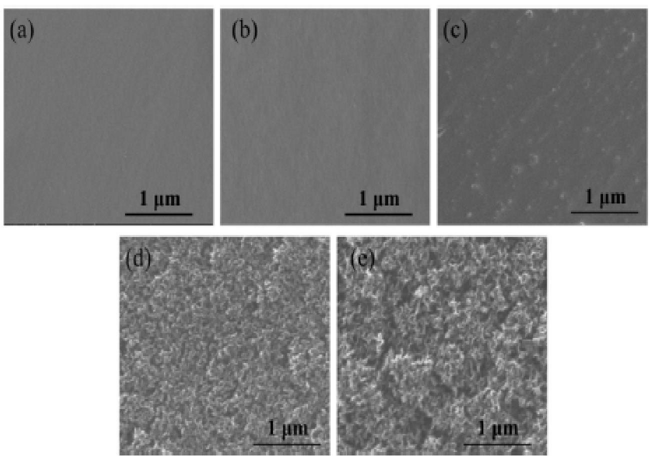

Figure 9 SEM images of un-modified (a), and modified PEGylated surfaces with different CF_4 plasma treatment time (b) 2.5 min, (c) 5 min, (d) 7.5 min and (e) 10 min[23]

The reaction mechanism of CF_4 plasma surface treatment is considered as two coexistence and interplay process of degradation and fluorination. Authors propose the changes taking place on PEGylated surface like as shown in the schematic representation of the wetting mechanism in Figure 10.

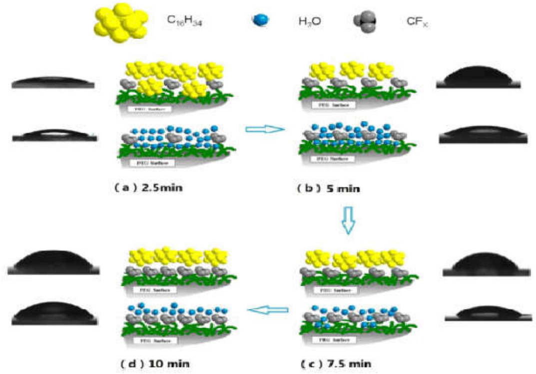

Figure 10 Schematic representation of wetting mechanism of the modified PEGylated Surfaces with different CF_4 plasma treatment[23]

Figure 10. (a) 2.5min, the modified PEGylated surface with scattered fluorocarbons, both water and hexadecane can pass through the fluorinated layer; (b) 5min, forming a porous fluorocarbons layer on the modified PEGylated surface,water can pass through the fluorocarbons layer but hexadecane droplet beads up on the fluorocarbons layer; (c) 7.5 min, water CA increases with the density of the fluorocarbons increasing; (d) 10 min, forming a dense fluorocarbons layer, water and hexadecane droplet both bead up on the fluorocarbons layer.

The authors Shusen Peng and Yongcun Ma expect this special wetting surface with hydrophilic and oil-repellent method is of great practical value for preparation a hydrophilic and oil-repellent surface[23].

Future perspectives in the field of technical textiles of Defence application

Defence Bioengineering and Electromedical Laboratory (DEBEL) has been working in diversified areas such as Technical textiles, NBC protection, Biomedical Engineering and also develops various other systems and equipments for Army, Navy and Air Force. Wide variety of materials such as fabrics, fibers, polymers, adsorbents, special paints etc. are routinely used in many of these systems developed by the lab. The Fabrics developed for various purposes of the Defence Forces such as NBC suits, Flame Retardant uniforms and cold weather clothing has to be preferentially antimicrobial because most of the times these clothing are meant for continuous long duration usage. More over in many situations it is either impractical or impossible for washing these clothing. Thus it is very important and essential requirement for the development of self cleaning and antimicrobial finished of Technical textiles.

Flame Retardant overalls developed by DEBEL is already in use by the services and has multiple properties viz., flame retardant, antistatic, thermal stability apart from high tensile and tear strength. Plasma treatment improves surface energy, wettability, drape, air permeability and comfort properties of the fabrics. It is desirable to impart hydrophilicity / super hydrophilicity in this fabric for the improvement of the comfort level.

NBC suit is a product developed by DEBEL is also already in use by the services and has multiple properties viz., camouflage printed, oil and water repellent (contact angle of

120^0 and 144^0 respectively), flame retardant, antistatic, thermal stability apart from high tensile and tear strength. The surface free energy of the substrate should be lesser than the surface tension of water in order to introduce the hydrophobicity on the substrate. Pure water has a high surface tension of about 72 mN/m. Therefore, surface free energy of the surface must be lesser to 24-30 mN/m for the development of hydrophobic surface.

In the case of oleophobicity, the surface free energy of the substrate should be lower than 20 mN/m since the surface tension of oils is usually 20-30 mN/m. Surfaces with both oleophobic and superhydrophobic properties are of great importance. Therefore, nano-architectured surface coatings with low surface free energies are to be fabricated on surfaces in order to prepare superhydrophobic and super oleophobic surface finishes. An example for nano-architecture surface is inorganic nanorods such as zinc oxide and titanium dioxide have been grown on the surface of the product and then, liquid repellent molecules have been allowed to self-assemble on nanorods. The intermediate inorganic nanorods material should have an ability to bind with surface of product and also with the liquid repellent molecules. Therefore, nano-architectured superhydrophobic and oleophobic surfaces will cause a significant impact for the improvement of oil and water repellent property. It is technically and logistically important requirement to generate super hydrophobicicty and super olephobicity on the fabrics with the plasma treatment.

It has been a long standing effort of DEBEL to incorporate durable anti-microbial finish on the technical textiles used in the FR Overalls for the Armed Forces, without affecting the other functional properties of the fabric. After several investigations, DEBEL has now developed nano silver based anti-microbial finished fabric for FR Overalls. Antimicrobial activity of the finished fabric was assessed by the International standard method (JIS L 1902: 2008 Quantitative Absorption Method) against the AATCC recommended test species *S aureus* and *K pneumoniae*. Antimicrobial activity of the finished NIIIA fabric was found to be greater than 99.99% against both the test species. DEBEL is looking forward to improve the wash durability of nano finish and to impart self cleaning property through surface modification by plasma treatment and by nano materials loading.

CONCLUSION

Plasma is one among such treatments that is getting good attention these days for its application as a pre-treatment and finishing technique for textiles. Different kinds of plasma gases provide special functionality to textile materials such as to enhance the dyeing rates of polymers, improve color fastness, printability, comfort level, air permeability, wettability wash resistance of fabrics, UV-protection, antibacterial, flame retardancy, hydrophobic finishing and product quality without any alternation of the inherent properties of the textile materials.

Technical textiles developed for various purposes of the Defence Forces has to be preferentially self cleaning and antimicrobial with uniform shade and comfort finishes because most of the times these clothing are meant for continuous long duration usage. More over in many situations it is either impractical or impossible to wash these clothing. Thus there is an essential requirement to impart the special properties such as self cleaning, antimicrobial, hydrophobic, oleophobic, durable dye and improved comfort finishes on the technical textiles of Defence application by adopting the eco-friendly and unique plasma technology.

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