Surface activation of polyester fabric using ammonia dielectric barrier discharge and improvement in colour depth

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Received 8 May 2013; revised received 30 September 2013; accepted 14 October 2013

The effect of atmospheric pressure ammonia dielectric barrier discharge on the surface of polyester fabric has been studied using contact angle goniometer, ATR–FTIR spectroscopy and SEM. It is observed that the surface of plasma treated polyester is more hydrophilic due to incorporation of nitrogenous polar functional groups and shows different conformational composition and crystallinity. The ATR–FTIR results clearly indicate the conversion of trans ethylene glycol residue to gauche one after plasma treatment and thus reduction in crystallinity. Plasma treatment has shown synergetic effect on dye uptake. Natural dyeing of plasma treated polyester with '*Rubia cordifolia*' has shown 65% improvement in colour depth in comparison to untreated polyester fabric.

Keywords: Ammonia plasma, Colour depth, Crystallinity, ATR–FTIR, Hydrophilicity, Natural dyeing, Polyester, Surface modification

1 Introduction

Polyester is the most consumed synthetic fibre¹. It has a highly compact and crystalline structure having small amorphous regions as dye sorption sites, and is chemically inert in nature. Due to this its dyeing is carried out by synthetic disperse dyes under high temperature (around 130°C) and high pressure which needs lot of energy and special equipment^{2,3}. In addition, dyeing with synthetic dyes is not environment friendly process as synthetic dyes produce pollution throughout its lifecycle. Its production not only contributes to the depletion of global supplies of non-renewable fossil fuels but also causes water pollution due to the synthetic dyes and other dyeing auxiliaries; effluent treatment continues to be a problem as bio-treatability of many of these is $poor^4$.

The recent trends of the textile industry are aimed to improve the environmental performance by reducing consumption of energy, water and chemicals used in processing, dyeing and finishing as well as development of a new dyeing method to accelerate dyeing rate, improve dye uptake and lower dyeing temperature⁵. In this regard, surface modification by non-thermal plasma treatment has opened up

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new possibilities in textile processing. Compared with conventional chemical treatments, non-thermal plasma techniques have the advantages such as eco-friendliness and bulk properties of textile remain unchanged and it can replace some wet chemical process^{6,7}. In the past decades, the use of non-thermal plasma for selective surface modification of textiles has been a rapidly growing research field⁸⁻¹⁰. It has also been observed that in the last decade there is significant revival of interest in application of natural dyes for coloration of textile material all over the world as a consequence of increasing awareness about environment, ecology and implementation of strict regulation of pollution control¹¹. Surface modifications by non-thermal plasma treatment can advantageously improve dyeing properties of various natural and synthetic textiles with natural dyes¹². However, there have been very few references that report the application of plasma for natural dveing of polyester; thus use of plasma technology to increase dye-uptake of polyester fabric with natural dyes is still an unexplored field of research. Previously, improvement in dye ability with natural dyes has been reported for polyester fabric coated with nanolayer of platinum using DC magnetron sputtering¹³. Application of atmospheric pressure air plasma has recently been studied to improve dyeing of polyester with curcumin¹⁴.

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The present paper therefore reports the studies on the surface modification of polyester fabric using dielectric barrier discharge (DBD) in ammonia gas at atmospheric pressure. The effect of plasma treatment on wetting behaviour is characterized using dynamic wetting measurements. The changes in surface morphology is examined using scanning electron microscopy (SEM) while Fourier transform infrared spectroscopy is used to provide a (FTIR) comprehensive view of changes in chemical and conformational structure of polyester due to plasma treatment. The dyeing properties of untreated and ammonia DBD treated polyester fabric is studied using natural dye derived from 'Rubia cordifolia'. Powdered roots of the plant is used as dye in this study which contains anthraquinones derivatives as active colouring constituents which are similar to the ordinary disperse dyes in structure and solubility and have been previously explored for dyeing of polvester^{4,15,16}.

2 Materials and Methods

2.1 Materials

The plain woven grey stage poly(ethylene terephthalate) (PET) fabric (density 29 ends/cm and 21 picks/cm) supplied by Reliance Industry, India was used for the study in its as received form. Roots of Indian madder plant (*Rubia cordifolia*) in powdered form (commercial brand name Eco-alizarin), purchased from Sam Vegetables Colours (P) Ltd., Moradabad, U.P., India, was used as natural dye. Methanol (GR grade) used in experiments was purchased from Merck Specialities Private Limited, Mumbai, India.

2.2 Methods

2.2.1 Plasma Treatment

The schematic configuration of plasma reactor used is represented in Fig. 1. It is composed of the two rectangular wire mesh (grade 325) electrodes $(40 \times 50 \text{cm})$ which are placed within a rectangular stainless steel chamber $(70 \times 70 \times 30 \text{cm})$ connected to rotary pump. Both electrodes were covered with dielectric layer of PET film with thickness of 125 microns and separated 3 mm apart from each other using Teflon spacers $(300 \times 20 \times 3 \text{mm})$. The pressure in the experimental system was measured with a digital high pressure pirani gauge (VT–DHP–11). Polyester fabric $(20 \times 20 \text{ cm})$ to be treated was placed on lower electrode. To generate the ammonia plasma the ambient air within the chamber was evacuated using rotary pump. Once the pressure in chamber dropped down to 0.05 mbar, rotary pump was disconnected from the chamber and chamber was filled with commercially available ammonia gas using needle valve at top of chamber. Plasma was produced in ammonia gas at atmospheric pressure applying voltage of 7kV rms using 10kVs power source operated at 50 Hz AC and the polyester fabric was treated with plasma for 15 min time duration. The voltage applied to the electrodes was measured using a high voltage probe (Tektronix-P6015A), whereas the discharge current was measured with a current transformer (1V/1A) and resulting waveform recorded using four channel digital Oscilloscope (Tektronix-TDS2014B). The current-voltage waveforms of DBD operating at applied voltage of 7kV are shown in Fig. 2.



Fig. 1-Schematic diagram of experimental set-up



Fig. 2–Voltage current waveform of NH_3 dielectric barrier discharge

2.2.2 Characterization

The wettability of the PET fabric surfaces before and after plasma treatment was estimated by means of dynamic wetting measurements¹⁷, which were carried out using video based optical contact angle goniometer (OCA15, Dataphysics, Germany) as per the sessile drop method. This instrument consists of a high speed CCD video camera (resolution of 768×576 pixel and up to 50 images per second) and an integrated software-operated high precision liquid dispenser micro-syringe unit which can dispense precisely control drop size of the used liquid on surface of test specimen. The instrument was located in a temperature controlled laboratory maintained at $25 \pm 1^{\circ}$ C with a constant relative humidity. The fabric test piece was placed on a movable platform in front of CCD camera and deionized water droplet (Millipore water having a surface tension of $\gamma_{1v} = 72.8$ mN/m at 23°C, 5µL volume) was placed on the fabric surface through a computer-controlled process. Through CCD video camera the entire process was recorded as digital movie showing changes in water droplet as a function of time and was then stored and processed using a PC-based acquisition and data processing software (SCA20, Dataphysics). The drop images were processed by the software, which calculates both the left and right contact angles from the shape of the drop with an accuracy of $\pm 0.1^{\circ}$. Each reported contact angle is the mean value of at least three measurements taken at different positions on the sample. The initial contact angle of water and the water droplet absorption time (i.e. time needed for a complete penetration of 5µL drop into the PET fabric surface) are used to evaluate the wettability of the untreated and ammonia DBD treated polyester fabrics¹⁷. ATR-FTIR spectra of the untreated and plasma treated polyester fabric are recorded by FTIR spectrometer (Nicolet 6700, Thermo Scientific, MA, USA) at resolution of 4 cm⁻¹. Morphological changes of polyester fabric surface are investigated using SEM (LEO s440i). The tensile properties of untreated and 15 min ammonia DBD treated polyester fabric were measured as strip strength as per standard method ASTM D 5035.

2.2.3 Dyeing and Analysis

Dyeing of polyester fabric was carried out using natural dye obtained by leaching of powdered root of the Indian madder plant (*Rubia cordifolia*) using methanol. To prepare dye bath, 2g of root powder was mixed with 100mL methanol and slurry is stirred continuously for 20 min. This solution was allowed to stay as such for some time and supernatant of the solution was collected and used as dye bath. Untreated and 15 min DBD ammonia plasma treated polyester fabrics were dyed using the liquor-to-fabric ratio (100:1) by keeping dye bath at temperature of 80°C for 1 h using constant temperature water bath. Dyed fabrics are thoroughly washed with distilled water and later dried with air.

To measure and compare colour intensities of dyed fabrics, the reflection of dyed fabric at wavelength corresponding to the maximum absorbance was measured using reflective Spectrophotometer (Data Colour Spectra flash SF 500). Reflection factor (R) was obtained as the average of four measurements. The relative colour strength (*K/S* value) was established according to the Kubelka–Munk equation¹⁸.

The percentage increase in colour depth (% *I*) was obtained from the difference between the (*K/S*) values measured for plasma treated and untreated samples dyed using same dyeing procedure according to the following equation¹⁸.

$$\% I = \frac{\left(\frac{K}{S}\right) \text{Treated} - \left(\frac{K}{S}\right) \text{Untreated}}{\left(\frac{K}{S}\right) \text{Untreated}} \times 100 \qquad \dots (1)$$

Percentage strength of colour was also calculated from spectroscopic data. The durability of dyed polyester fabrics was evaluated in terms of fastness towards washing and lighting, according to the standard methods IS: 3361 – 1984 (ISO – II) and IS: 2454 – 1984 respectively.

3 Results and Discussion

3.1 Improvement in Wettability

Static contact angle determination is a common method for quantification of wettability. However, to evaluate the wetting behaviour of heterogeneous surfaces such as textile fabrics, it is difficult to measure static contact angle because water could be able to penetrate in to the pores of fabrics by capillary forces. Therefore, we have used the time dependent contact angle measurements to study effectiveness of plasma treatment to improve wettability of polyester fabric¹⁷. Figure 3 shows changes in water contact angle of 5 μ L droplet placed on untreated and NH₃ DBD treated polyester fabric as function of time. It is clearly revealed from the figure that there is significant difference in wettability of untreated and 15 min NH₃ DBD treated polyester fabric. The untreated polyester fabric exhibits higher initial water contact angle of 85.56° while after15 min NH₃ DBD treatment of polyester fabric the initial water contact angle significantly decreases to 55.70°. Also, the droplet absorption is hindered for untreated polyester fabric which means that the untreated polyester fabric surface is hydrophobic, while wettability is greatly enhanced after ammonia plasma treatment and this results in rapid decrease in water contact angle due spreading and absorption. The spreading dynamics of water droplet on untreated and ammonia plasma treated polyester fabric is shown in Fig. 4 as image sequence for different time interval. In untreated polyester fabric, the time required for complete absorption of water droplet is higher than 1s, while in case of plasma treated polyester fabric the water droplet gets fully absorbed within 250 ms. This change in the wetting behavior can be attributed to the



Fig. 3–Variation in water contact angle as a function of time on surface of untreated and NH₃ DBD treated polyester fabric



Fig. 4–Image sequences of water droplet on surface of untreated and $NH_3 DBD$ treated polyester fabric

polar groups introduced onto the surface, which improves the wettability of the PET fabric due to resultant hydrophilic surface¹⁷.

3.2 ATR-FTIR Analysis

FTIR spectroscopy shows the effect of plasma treatment on surface chemistry and molecular architecture of PET fabric, based on chemical and conformational origin of IR peaks^{1,19,20}. Figure 5 (a) represents ATR-FTIR spectra of polyester fabric showing typical absorbance bands of semi-crystalline PET similar to those reported in literature^{1,19,20}. The chain scission of the PET is reflected in the ATR-FTIR spectrum of ammonia DBD treated polyester fabric, as indicated by decrease in absorption intensity of IR bands corresponding to the carbonyl ester groups at 1711cm⁻¹ in spectrum of plasma treated fabric. Also new strong absorption is observed at 1530-1660 cm⁻¹ and at 3028 & 3214 cm⁻¹ in spectrum of ammonia plasma treated fabric, which are assigned to N-H bending vibration at 1549cm⁻¹, stretching vibration of amide at 1648cm⁻¹ and N-H stretch vibration in functional group region respectively²¹. This indicates formation of nitrogen containing functional groups such as amine and/or amide on the surface due to plasma treatment which is in accordance with previously reported work²². Electrons, ions, and H radicals in ammonia



Fig. 5–ATR-FTIR spectra of (a) untreated and NH_3 DBD treated polyester fabric, and that of normalized to 1410 cm⁻¹ peak (b)

plasma contribute to ester group destructions, generating carbon radicals at destroyed ester groups. In polymer chains of PET these carbon radicals are stabilized by existence of benzene ring, and as a result carbon radicals remain on polymer surface and can react with N or NH/NH₂ radicals present in plasma and thus the nitrogenous functional groups such as amino and/or amide groups are introduced after plasma treatment²².

The studies reported in literature have proved that FTIR spectroscopy is a powerful tool to probe conformational changes and to obtain information about crystal structure of PET^{1,19-21,23}. Since the ATR-FTIR spectroscopy investigates a surface depth of about 1µm, it could evidence the possible changes in surface crystallinity induced by ammonia plasma treatment. Semi-crystalline polyester is made up of two phase structure; it is the crystalline phase which is separated by disorder amorphous phase. PET in the crystalline phase is reported to be present in full trans conformation, meaning the glycol segments, the phthalates and the ester moieties are all arranged in trans conformation, while disorder amorphous phase is reported to be made up of mixture of trans and gauche conformations ^{20,23}. Thus, changes in crystalline structure of polyester after plasma treatment can be followed by IR conformational bands. The band at 1410cm⁻¹ (ring C-H in plane bending coupled with ring C-C stretching) has been considered to be insensitive to orientation and conformation is frequently used as reference band to normalize spectral intensity among materials which is a common practice to follow conformational changes ^{1,19-21,23}. Normalization of spectra to the 1410cm⁻¹ peak nullifies variation of intensity due to other optical effects. Therefore in this study, spectra are normalized before any data processing. Normalized spectra of untreated and 15 min NH₃ DBD plasma treated polyester fabrics are presented in Fig. 5 (b).

Changes in intensity of bands in normalized spectra are observed after plasma treatment which is due to conformational changes, indicating amorphization of polyester by plasma treatment. The bands at 1506, 1471, 1338, 1117, 968, and 842 cm⁻¹ are arising from vibration of the ethylene glycol in trans conformer and therefore represents crystalline structure of PET. Intensity of all these bands and also that of bands at 721, 1015, and 1235 cm⁻¹ decreases after plasma treatment which indicates amorphization of crystalline PET after plasma treatment^{1, 23}. On the other hand, bands at 1450, 1373, and 898 cm⁻¹ characterize gauche (amorphous) conformation. As seen from the Fig. 5 (b), these bands are more intense in spectrum of plasma treated fabric. Absorption bands due to wagging of glycol in gauche (1373 cm^{-1}) and in trans (1338 cm^{-1}) plus the band at 1410 cm^{-1} are particularly important to compare crystal volume fraction in polyester^{1,19-21,23}. As seen from Fig. 5 (b), intensity of band at 1338 cm^{-1} decreases after plasma treatment with increase in intensity of 1371 cm^{-1} band. Absorption ratios A_{1338}/A_{1410} can be used as quantitative assessment of degree of crystallinity; crystalline PET exhibits higher values of A_{1338}/A_{1410} is 0.96 for plasma treated fabric compared to 1.01 for untreated fabric^{1,23}.

3.3 SEM Analysis

Figure 6 shows the SEM images of surface of untreated and ammonia DBD treated polyester fibres



Fig. 6–SEM micrographs of (a) untreated and (b) ammonia DBD plasma treated polyester fabric surface

which indicate that the surface of untreated fibre is smooth without any specific morphological features, while after plasma treatment surface is etched and roughed with appearance of some random and irregular protuberances throughout fibre surface. The observed variations in features may be due to breaking of chemical bonds by plasma etching which cause physical removal of molecules and differences in physical properties and crystallinity. The impinging plasma species can transfer enough energy to the polymer matrix to enable the destruction of crystalline domains, which might relax in a disordered amorphous form, causing volume differences at surface^{6,24}.

3.4 Tensile Strength Analysis

The effect of ammonia DBD treatment on tensile properties of polyester fabric is evaluated in warp and weft way. The tensile strength of untreated polyester fabric is 38.88kgf and 36.46kgf in warp and weft way respectively while tensile strength of ammonia DBD treated polyester fabric is 35.255kgf and 36.03kgf in warp and weft way respectively. This indicates that there is no significant variation in tensile strength of polyester fabric after treatment with ammonia DBD. It is observed from ATR-FTIR study that plasma treatment changes the surface conformational composition and crystalline structure of polyester fabric, however crystal structure affect the mechanical properties of polyester, if the bulk structure is affected. Since the plasma induced changes in crystalline structure evidenced by ATR-FTIR remain confined to the top most surface layers, and the bulk properties of PET fabric are not altered by plasma treatment¹⁸, the tensile properties remain unaffected.

3.5 Colour Measurement and Fastness Properties

Natural dyeing results using *Rubia cordifolia* extract on 15 min NH₃ DBD plasma treated sample to study the effect of plasma treatment on dye uptake (measured in terms of K/S value) are summarized in Table 1. It is found that dye uptake of polyester fabric increases after plasma treatment, resulting in significant improvement in K/S value with considerable increase in % strength. Light fastness

and wash fastness ratings of dyed fabric indicate that the fabric treated with ammonia DBD exhibits good fastness.

3.6 Effect Surface Modification on Dyeing Properties

In this study, dyeing of polyester is carried out using crude extract of R. cordifolia roots at lower temperature of 80°C without any axillaries to check effect of plasma treatment. R. cordifolia contains mainly alizarin as well as purpurin, pseudo-purpurin, munjistin, rubiadin, 1,3 dihydroxyanthraquinone belonging to anthraquinones class of dyes²⁵. Synthetic fibres are generally not dyed with natural dyes. Synthetic anthraquinonoid compounds constitute 32% of all commercial disperse dyes designed for use on polyester and other hydrophobic fibres. Hence, there is a potential for utilising natural anthraquinonoid dyes as disperse dyes for such fibres. The natural anthraquinones have very similar chemical properties to synthetic disperse dyes, but have hydrophilic functional groups which limits its substantivity for polyester and hence its direct utilization⁴. Previous results of dyeing carried out using natural anthraquinones as high-temperature disperse dyes for polyester indicates its usefulness as an alternative to the synthetic disperse dyes. However, good colour depth and colour fastness properties can only be obtained when it is used in purified form; this increases the material cost compared to synthetic dves, thus making use of natural anthraquinones uneconomical for textile dyeing^{4,15,16}.

Significant improvement of approximately 65% in colour depth is observed with the increase in wash and light fastness properties in case of plasma treated polyester dyed using crude extract of *R. cordifolia*. Plasma treatment with ammonia DBD modifies surface of polyester and results in more hydrophilic surface due to incorporation of nitrogenous polar functional groups as well as changes conformational composition and reduces crystalline fraction of surface. Surface properties are especially important in dyeing as dye molecules first get adsorbed on to the surface and then diffuse to the bulk of polymer. Incorporation of polar functional groups at surface

| Table 1—Result of polyester dyeing with natural dye from Rubia cordifolia | | | | | | | |
|---|----------------------|-----------|------------------|------------|--------------|-----------------------------|-----------------|
| Sample | $\lambda_{max} \ nm$ | %R | <i>K/S</i> value | % Strength | % Improvment | Wash fastness (Color loss)* | Light fastness* |
| Untreated | 440 | 8.00 | 5.29 | 30.17 | - | 2 | 2 |
| NH ₃ plasma treated for 15 min | 440 | 5.03 | 8.75 | 49.90 | 65.40 | 3 | 3 |
| *1-Very poor, 2-Fair, 3 | -Good, 4-V | Good, and | l 5–Excellent. | | | | |

may improve adsorption and diffusion of natural dye molecule containing polar substitutions. Also, it is well established in literature that dye sorption in polyester mainly takes place in the amorphous region because chain mobility is less restricted in the amorphous region, thus segmental motion can easily occur^{17,26-28}. Also, reorientation of non-crystalline phase due to effect of solvents/temperature at dyeing condition results in free volume or microvoids through which dye molecules diffuse in to polyester. The free volume of PET increases as a result of conformational changes from the gauche to the trans forms in amorphous region, thus dye uptake increases with the gauche content of the ethylene glycol unit of PET. Thus, quantitative and qualitative changes in amorphous region are main factor to govern dye uptake and a small change in amorphous content or structure can drastically change rate of dyeing²⁶⁻²⁸. In this study, the conversion of trans ethylene glycol residue to gauche one as a result of plasma treatment is clearly observed by ATR-FTIR spectroscopy, thus changes in amorphous content and structure induced by plasma treatment can improve dye uptake²⁹.

Therefore, the improvement in dye uptake with natural dye R. cordifolia reported in this paper is inferred as a consequence of polar group formation on the surface of polyester as well as amorphization of fabric surface. The surface activation with ammonia DBD can increase substantivity of natural anthraquinones and also results in increase in sites through which dyes can diffuse in to the bulk of polyester. Also natural anthraquinones containing carboxylic groups like pseudo-purpurin, munjistin may attach with polyester fibre forming bond with amine functional groups. Natural anthraquinones have strong antimicrobial properties, so dyeing of polyester with natural dye R. cordifolia dyes is predicted to have some healing and healthcare effect on the wearers^{13,16}.

4 Conclusion

The present study has clearly shown that the surface modification of polyester with ammonia dielectric barrier discharge improves wettability of polyester due to incorporation of nitrogen containing functional groups. Further, we observed that the plasma treatment results in amorphization of polyester. Improvement in dyeing properties of polyester is observed in terms of improvement in colour depth as well as fastness properties when plasma treated fabric is dyed using crude extract of *R. cordifolia.* It can be concluded that synergetic effect of plasma treatment on dyeing properties is due to wettability improvement and amorphization of polyester. This study has also demonstrated that plasma treatment can open up a new possibility for sustainable finishing of polyester with natural dyes by at least partial replacement of synthetic dyes as well as the technical aspects of dyeing. Both plasma treatment and natural dyeing offer environmental benefits thus their combination can suitably be used for making value-added environment-friendly apparel and other textile products.

Acknowledgement

The author(s) are thankful to the Department of Science and Technology, Government of India for financial support. The thanks are also due to Ms. Purvi Kikani (FCIPT) for carrying out ATR– FTIR, Mr. Narendrasinh Chauhan (FCIPT) for carrying out SEM, Ms. Sarika Singh, ATIRA, Ahmedabad for spectroscopic colour measurement of polyester fabric.

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