

Fabrication of Electronic Silk Fabrics via RGO Adhesion Incorporating Oxygen Plasma Treatment [†]

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Abstract: Plasma Technology has proven to be the most effective eco-friendly method for the textile industry in improving surface adhesion. Two different silk fabrics, raw and degummed silk are treated by low-pressure glow discharge oxygen plasma to improve hydrophilic properties for better adhesion and coating process. Oxygen plasma can produce etching and formation of polar functional groups on the surface of the fabrics. The plasma conditions like voltage and working pressure are maintained constant with different exposure times. The plasma-exposed fabrics are characterized by SEM, XPS and adsorption tests. SEM reveals that the changes in the fabric surface are prominent for higher treatment time. According to the results of XPS, the oxygen-containing functional groups are increased after plasma treatment. The GO adsorption test indicates the enhancement of GO on the plasma-treated fabrics than untreated silk. The GO is prepared by the Modified Hummers method. The GO is coated on the plasma-treated silk fabrics by a dip coating method. The GO-coated silk fabrics are converted into RGO fabric by ascorbic acid as a reducing agent. Electrical conductivity measurement of the fabricated silk reveals that adequate current flows through it to glow an LED bulb.



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Keywords: silk; surface modifications; oxygen plasma treatment; adhesion; electrical conductivity

1. Introduction

Silk is polymer fibre produced from the silkworm. It is a protein fiber and it is biodegradable [1,2]. Two types of domestic silks are available in the market, raw silk and degummed silk [3]. Hydrophilic and wettability are important properties of the fabrics because they enhance the uptake of the liquids during the coating and dyeing process. Hence, the textile industries are deemed to be essential for their sustainable growth [4]. Textile companies are searching for innovative surface modification technologies that are efficient, adaptable, flexible and versatile to make novel products with high-added values [5]. Surface modification of textile materials can be done primarily by using conventional chemical methods. This technique consumes a large quantity of water, chemicals, and energy. The disposal of chemicals would cause environmental issues. Physical methods overcome the limitations of conventional chemical methods. Among physical methods, plasma technology is green and eco-friendly which can change the surface without affecting the bulk properties [6]. When a substrate surface is exposed to plasma, different reactive species such as ions, electrons, radicals, and excited particles can interact with the substrate surface. Depending on the plasma gas, various effects such as etching or cleaning, activation, grafting, polymerization and coating can occur [7]. Oxygen plasma

treatment enhances to generation of oxygen functional groups and radicals on the surface of the materials.

This study investigates the effect of low-pressure glow discharge oxygen plasma to enhance the hydrophilicity and wettability of raw and degummed fabrics. SEM analysis provides information about the surface morphology of the fabrics. AFM gives information about the surface roughness of the treated fabrics. XPS results present the surface chemical composition of the fabrics. GO adsorption test is used to check the hydrophilic properties of the treated fabrics. Finally, the electrical properties of the RGO-coated silk fabrics are checked by the I-V characterization.

2. Experimental

2.1. Materials

Raw silk (R silk) and degummed silk (D silk) are purchased from Central Silk Board Bangalore. The thickness of the R and D silk are 0.14 mm and 0.8 mm respectively.

2.2. Methods

2.2.1. Silk Fabric Plasma Treatment

The raw and degummed silk fabrics are cut into $5 \times 5 \text{ cm}^2$ and mounted on the substrate holder inside the chamber. Initially, the chamber is evacuated using a rotary pump up to 2×10^{-2} mbar and is noted as base pressure. After evacuation, the treatment gas oxygen is allowed into the chamber. The working pressure inside the chamber is maintained at 2×10^{-1} mbar. Discharge occurs between the constricted anode and the cylindrical mesh grid. The stainless-steel mesh grid acts as the cathode with a height of 20 cm and 60 cm in diameter. Fabrics are exposed to different treatment times 5 min, 10 min, 20 min and 60 min at an output voltage of 500 V. The sample codes of plasma-treated raw silk are R5 min, R10 min, R20 min and R60 min and for degummed silk are D5 min, D10 min, D20 min and D60 min. Untreated raw and degummed silk fabrics are noted as UR and UD. The photograph (a) and schematic diagram (b) of the plasma reactor is shown in Figure 1.

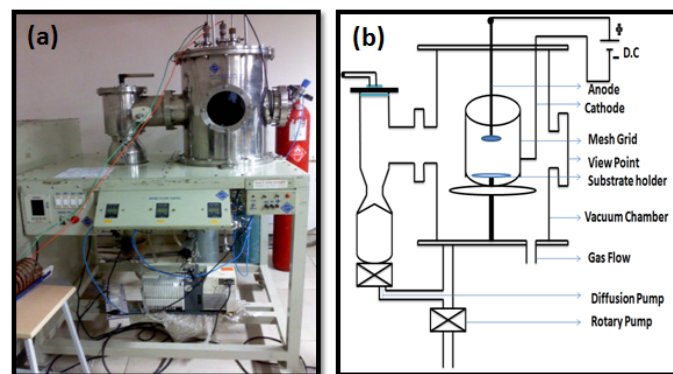


Figure 1. (a) Photograph and (b) schematic of the plasma reactor.

2.2.2. Fabrication of RGO on Silk Fabrics

The graphene oxide (GO) has been synthesized according to the modified Hummers method, and the results are previously reported [8]. GO is coated on the fabrics by the dip coating method. GO-coated fabrics are converted into RGO-coated fabrics with the help of ascorbic acid as a reducing agent.

3. Results and Discussion

3.1. Results of Plasma Treated Silk Fabrics

3.1.1. SEM Studies

The SEM of UR and UD silk fabric is shown in Figures 2a and 3a. It shows that the weft and warp direction of the woven fibre structure of UR silk and gaps are observed between the fibres. Figures 2 and 3b–f represent the SEM images of untreated and plasma-treated fabrics. Figures 2 and 3b indicate the typical longitudinal fibril structure of fibre and it shows a clean and smooth surface, while slight longitudinal flutes appeared on the surface of R5 min fabric (Figures 2 and 3c). Increasing the plasma treatment time to 10 min, the surface of the fibres degraded, and cleavages and micro-cracks appear on its surface (Figures 2 and 3d–f) as the plasma treatment modifies the surface of the fabric by ion impingement. The etching of the sample surface by oxygen ions and oxygen functional groups such as C-O, C=O and O-C-O are formed on the surface of the fabrics. Therefore, R5 min and D5 min plasma treated samples are considered for further studies.

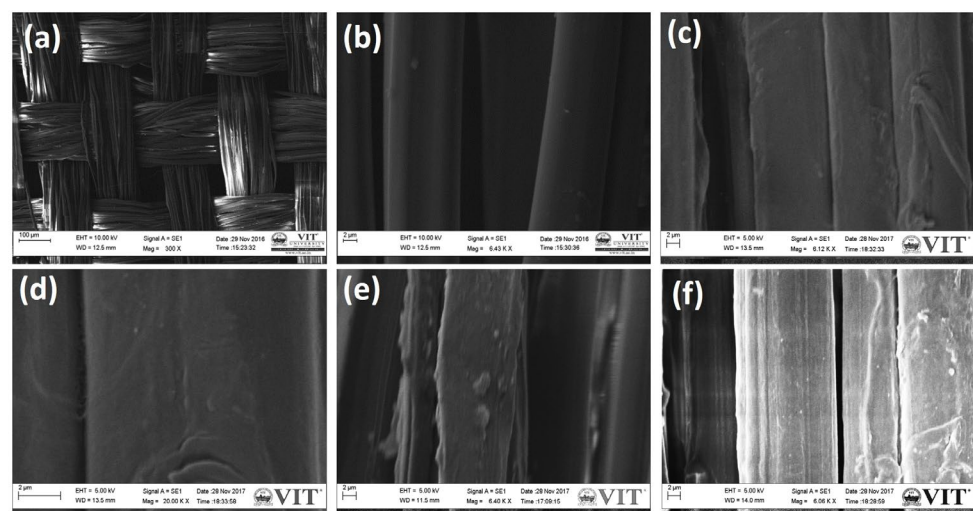


Figure 2. SEM of (a,b) UR silk, (c) R5 min, (d) R10 min, (e) R20 min and (f) R60 min plasma time.

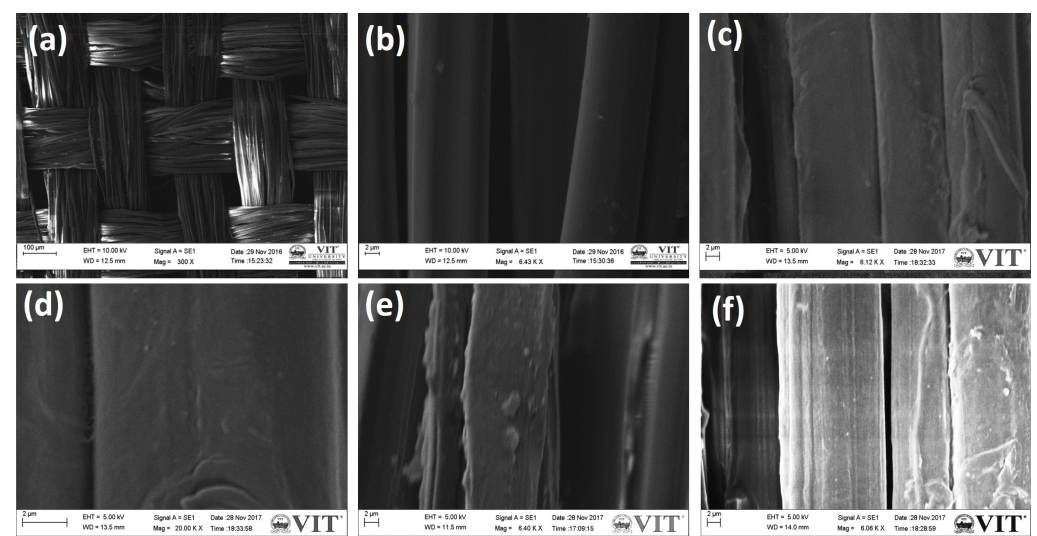


Figure 3. SEM of (a,b) UD silk, (c) D5 min, (d) D10 min, (e) D20 min and (f) D60 min plasma time.

3.1.2. XPS

The chemical nature of both the silk fabric surfaces is investigated by XPS and the results are presented in Figure 4. Figure 4a,c represents the survey scan spectra of UR,

and UD silk and Figure 4b,d shows the survey scans of 5 min plasma treated R and D silk. The XPS survey scan spectra of all the samples show strong peaks in the region of 200–600 eV. The peaks at 284 eV and 531.5 eV are assigned to C1s and O1s, respectively. For 5 min plasma treatment, carbon content on the R and D fabrics decreased from 87.2% to 79.7% and 82.5% to 74.5% respectively. The reduction of C1s is due to the removal or break of carbon bonds on the fabrics during oxygen plasma treatment. The XPS data indicates that the intensity of the O 1s peak is increased for all the plasma-treated samples. O1s originated from oxygen-containing functionalities responsible for making the fabrics hydrophilic. Therefore, it is clear from the XPS studies, that plasma treatment enhances the polar functional groups on the surface of the fabrics.

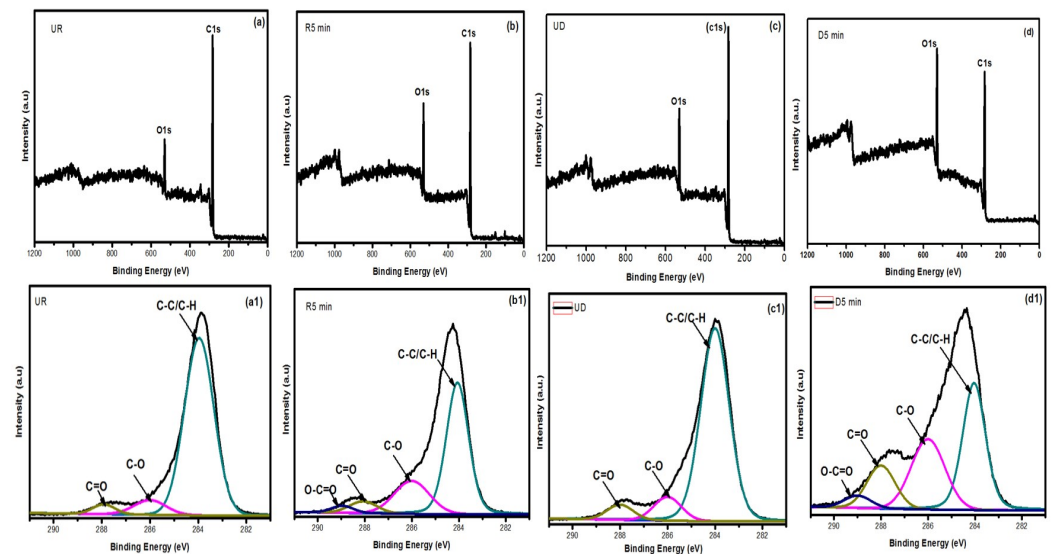


Figure 4. Survey scan (a–d) and C 1s scan (a1–d1) of untreated and oxygen plasma treated R and D Silk.

C1s high-resolution spectra were deconvoluted to find the changes in functional groups after plasma treatment. Untreated silk fabric contains three peaks at 284 eV (C-C/C-H), 286 eV (C-O) and 288 eV (C=O). It is well known that after oxygen plasma treatment, the concentration of the C-C/C-H bond of the silk is broken and the carbon radicals formed by the removal of hydrogen within the polymer chain will recombine with atomic oxygen generated by the electron impact dissociation in plasma. Theoretically, a higher oxygen concentration should be able to incorporate more polar functionalities on the fabric surfaces. This leads to the incorporation of oxygen-containing polar functional groups on the surface of silk fabrics, resulting in enhanced wettability.

3.1.3. Wetting Property

The wetting properties of the R and D silk fabrics are shown in Figure 5. The dyeing of the plasma-treated fabrics is highly enhanced when compared to the untreated fabrics. D silk fabrics obtained better spreading results than R silk, which is due to the smoothness and thickness of the fabrics. On the other hand, prepared GO solution is placed on the silk fabrics by using a micropipette to check the adhesion of coatings. Figure 6 shows the photograph of untreated, and 5 min plasma treated R and D silk with GO droplet. From the image, it is understood that in UR and UD fabrics, the semi-circular shape of GO droplets is formed and it's stable for a long time without changing their shapes, but in plasma-treated R and D silk fabrics, the GO droplet adhered firmly all over the surface within a second. The incorporation of polar functional groups enhances the adhesion of coatings.

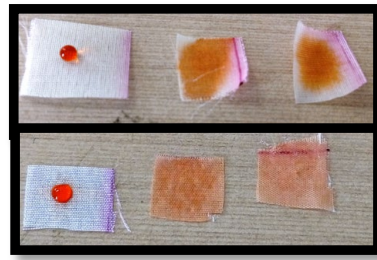


Figure 5. Dyeing drop test.

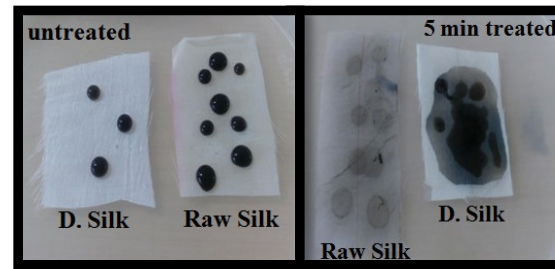


Figure 6. GO drop test.

3.1.4. I-V Electrical Studies

The electrical properties of RGO-coated fabrics with and without plasma treatment are examined from I-V graphs. The I-V curves of all the fabrics are represented in Figures 7 and 8. The fabrics are placed across the clip and -10 to $+10$ V is applied to it. It shows that plasma-treated RGO-coated fabrics result in significant changes in the I-V graph and it indicates successful conversion of RGO on the fabrics. The RGO-coated fabrics show the ohms behaviour of linearity. The electrical resistance of the fabrics is measured from the slope of the I-V graph of the different fabrics. The electrical resistance found for the R5 min and D5 min are $6.42 \times 10^{11} \Omega$ and $6.41 \times 10^{11} \Omega$ and it shows high electrical resistance. For RGO-coated fabrics, the electrical resistance has been reduced to $7.37 \times 10^5 \Omega$ (R.RGO) and $3.16 \times 10^5 \Omega$ (D.RGO). The electrical resistance decreased for all the RGO-coated fabrics due to the reduction of oxygen functional groups. The resistance value varies on the fabric's woven structure and materials. Therefore, it can be concluded that plasma pretreatment enhances the surface area and leads to improved electrical conductivity due to the conductive paths created by RGO.

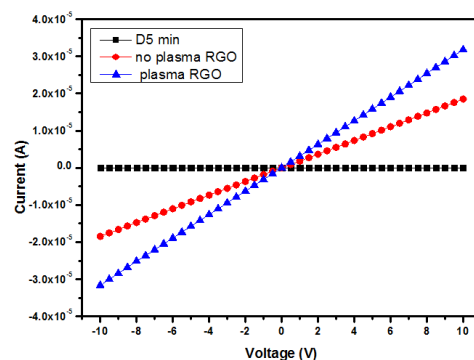


Figure 7. I-V plots of no plasma, RGO fabric, plasma-treated RGO D silk.

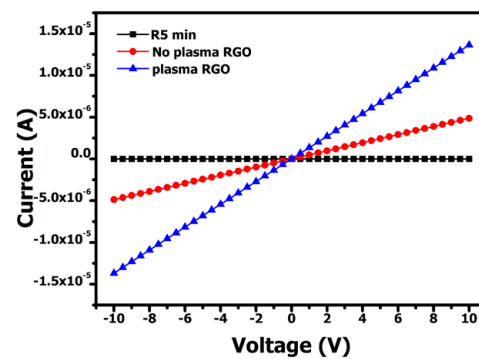


Figure 8. I-V plots of no plasma, RGO fabric, plasma treated RGO Rsilk.

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Conflicts of Interest: The authors declare no conflict of interest.

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