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Diagnostics and mechanistic studies in plasma treatment of polyester textiles

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The effect of low pressure He/O_2 plasma treatment on the desizing of polyester textiles was studied. Namely, the effect of the total gas pressure and of the fraction of O_2 in the gas mixture with He on the surface hydrophilicity of sized polyester textiles was investigated by implementing plasma diagnostics as electrical measurements and optical emission spectroscopy. Plasma treatment was found to be effective in desizing and to significantly improve the surface hydrophilicity especially when it is done at low pressures and with oxygen fractions exceeding 60%.

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1. Introduction

Surface modification of textiles with plasma treatment has gained increasing importance over the last few years [1,2]. The reason for this lies in the possibility of modifying properties leading to better performance in an environmental friendly process. The changes in properties induced by plasma treatment are restricted to the surface and any damage to the interior of the textile is very unlikely. From the physical point of view, roughening of the surface is responsible for changes in the coefficient of friction, yarn strength etc. From the chemical point of view, oxidation of the fibre surface and interaction with polymeric materials are the main factors responsible for the improvement of various properties of the plasma treated materials.

Polyester textiles and fibres can be effectively modified by low pressure plasma treatment. For instance, wetting out properties of polyester can be achieved with plasma treatment. The fabric can be processed without the use of wetting out agent in an economical and environmentally friendly way [3-5].

In this direction, the present work discusses the effect of He/O_2 plasma parameters on the surface hydrophilicity of sized polyester textiles. The study is mainly focused on the optimum conditions concerning the rate of water droplet absorption. The mechanisms that may leed to the fabric surface modification are also discussed.

2. Experimental

The plasma treatment apparatus is a fully characterized cell from the electrical point of view [6]. Briefly, the chamber consists of a 160 mm diameter stainless steel chamber having two parallel round stainless steel electrodes with a diameter of 55 mm, equipped with four 50 mm quartz windows.

For these experiments, the electrode distance was kept constant at 25 mm for all the experiments presented here.

Polyester textiles were mounted on the grounded electrode surface. The size of the fabric was amylase (figure 1). Polyester textile treatments were performed using as variables the fraction of O_2 in the gas mixture with He, the total gas pressure (150, 250, 400, 750 and 1000 Torr), the total RF power and the treatment time. In this paper the results of the first two parameters are presented. The radiofrequency (rf) power provided by a 13.56 MHz generator was fed to the discharge through a wattmeter (Diamond, SX200) via a proper impedance matching network. Voltage (Hameg, model AZ92) and current (FCC model F-35-1) probes were used to acquire entire waveforms to a digital storage oscilloscope (Lecroy 9361) in order to determine the exact power consumed in the discharge [7]. Water droplet absorption was measured as follows: a drop of distilled water (0.04 ml) was placed on the fabric surface and the spread of the droplet is observed until the disappearance of water. The environmental conditions during the test were 20 ± 2 ^oC and 65 ± 2 RH. Optical emission spectra and 2D images of emitted lines were obtained using the setup described in Ref. [8].

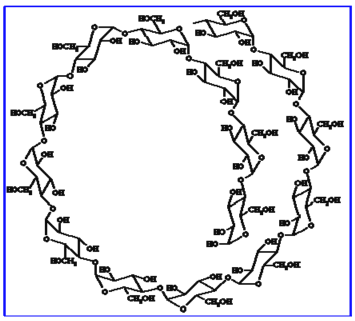


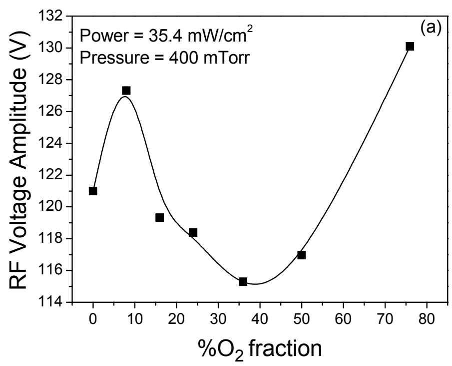
Fig. 1. Helical structure of amylase

3. Results and discussion

a. % O_2 fraction effect

Different sets of electrical measurements were initially carried out at different fractions of O_2 in He in order to determine the appropriate electrical conditions required to maintain constant power dissipation in the discharge (35.4 mW/cm^2) as the gas composition is varied. The total gas pressure in the reactor for these experiments was set at 400 mTorr. This approach permits to isolate as much as possible the effect of O_2 fraction on the surface hydrophilicity and helps to avoid power variations due to changes in the power coupling. Figure 2 (a) and (b) summarize the results of the electrical measurements. As one can observe, there is a minimum of both voltage and current for O_2 fractions in the range of 35 to 50 %. In these conditions the discharge demonstrates the less capacitive character as the phase difference between voltage and current reaches values of about -56^o (fig. 2b, right axis). In fact, the increase of the mole fraction of O_2 in the mixture results in a change of the discharge character from electropositive (He) to an electronegative (O_2). This has a direct effect on the discharge phase impedance (fig. 2(b) right axis). Thus, in He-rich discharges (>80% in He) the capacitance of the RF sheath is very high and this gives rather negative phase difference values. In this case higher voltage is required to maintain the same RF power dissipation (fig.1). As the fraction of O_2 increases, there is a significant enhancement of the inductive character of the discharge that is due to the presence of heavy negative ions trapped in the bulk of the plasma. This leads to less negative phase differences and to a subsequent drop of the voltage and current required for maintaining the same power level. Finally, in the case of O_2 rich discharges (>50% O_2) there are significant electron losses due to electron – molecule attachment; the low electron density plasma in this case raises again a highly capacitive RF sheath in order to be sustained. In this case the applied voltage has to be increased again in order to maintain the same power level.

After this procedure, samples of sized polyester textiles were treated at the specific conditions for 15 sec. Measurements of water droplet absorption on the treated samples are shown in fig. 3. In fact, a minimum time of water absorption is reached at the highest O_2 fraction (76% O_2). A satisfactory explanation for this result is that a large amount of oxygen is needed for removing the starch from the textile surface [9, 10]. It is worth noting that for the untreated textile the observed rate of absorption is negligible i.e. the textile practically does not absorb water.



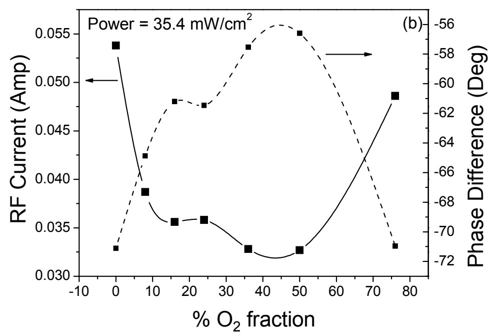


Fig. 2. Variation of (a) RF voltage and (b) current (left axis)) and phase impedance (right axis) as a function of O_2 fraction in He for a constant power of 35 mW/cm^2.

b. Total gas pressure

In order to further improve the surface hydrophilicity of the polyester textiles an investigation of the effect of the total pressure on the water droplet absorption was performed.

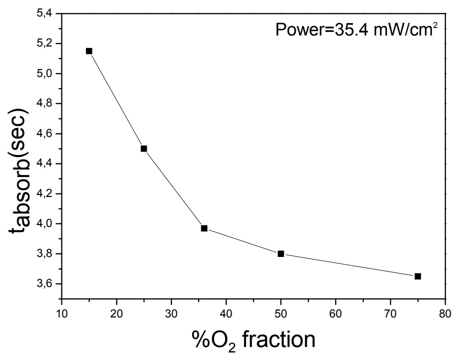


Fig. 3. Measurements of water droplet absorption time on the plasma treated surfaces as a function of O_2% in the mixture.

As in the case of O_2 fraction, a set of electrical measurements was carried out prior to the treatments in order to find the electrical conditions that ensure constant power dissipation in the discharge at different gas pressures. For these experiments, the fraction of O_2 in the gas mixture was set at 76 % i.e. the fraction we have achieved the best results in terms of surface hydrophilicity.

Figure 4 (a) and (b) presents the variation of applied voltage, discharge current and phase impedance required to achieve constant power dissipation. The discharge power for this set of experiments was set at 176 mW/cm^2. It is observed that a drop of the applied RF voltage is required as pressure increases due to the higher electron molecule collision frequency and the reduction of charged species losses towards the reactor walls. In addition, the discharge current increases with pressure indicating an enhancement of electron density. Finally, the discharge phase impedance reaches its lowest value at the lower pressure used in this study indicating a strong resistive character of the discharge at this pressure. This rather unexpected result is probably caused by the lower electron density and the enhancement of high energy electron molecule collision processes in the low pressure conditions. Above 400 mTorr the phase impedance remains constant mainly due to the simultaneous increase of sheath capacitance and electron–molecule collision frequency.

Samples of polyester fabrics were treated at the specific conditions and their surface hydrophilicity was estimated using water droplet absorption as shown in figure 5. The minimum water droplet absorption time is reached at the lowest pressure (150 mTorr).

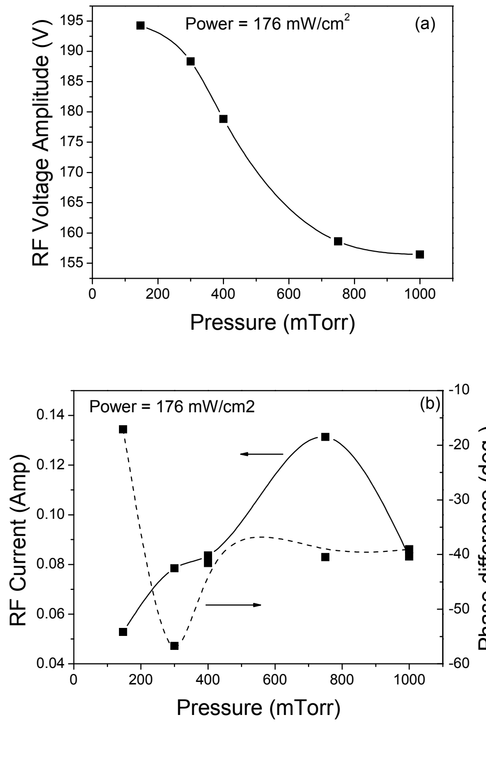


Fig. 4. Variation of (a)RF voltage, (b) current (left axis)) and phase impedance (right axis) as a function of total gas pressure for a constant power of 176 mW/cm^2

For all the treatment conditions the absorption rate is below 1 sec and this demonstrates the effectiveness of the specific process in polyester textile desizing. Furthermore, in order to have a better understanding of the mechanism of surface modification as well as to identify the species actively involved in the treatment, optical emission spectroscopy was used. The obtained emission spectra in the spectral region from 300 nm to 700 nm are presented in figure 6. There is a large number of transitions in these spectra, corresponding to He excited species and O atomic lines, bands of CO and OH and also N_2 bands.

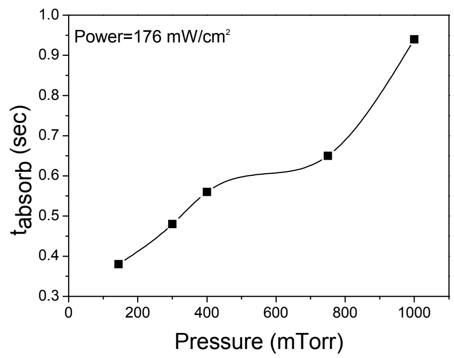


Fig. 5. Measurements of the water droplet absorption time as a function of pressure.

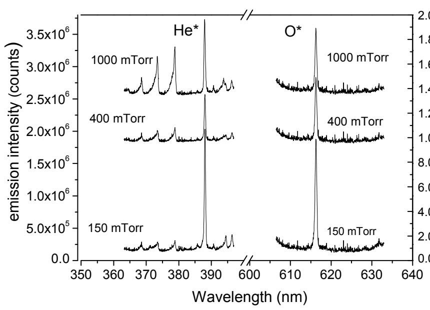


Fig. 6. Time–averaged emission spectra from 360 nm to 400 nm and 605 to 635 nm for 75% He/O_2 discharges at three different pressures

These fragments are produced either by electron impact dissociation of He and O_2 or from the treatment of the surface as well as from desorbing impurities. Figure 6 shows two parts of the emission spectra from 360 nm to 400 nm and from 605 nm to 635 nm for three total gas pressures. The first part includes the peak of the He (^{3}S-^{3}P^o) located at 389 nm, while the second part includes a characteristic line of excited O atoms located at 613 nm.

Figure 7 presents the ratio of the O* atoms (616 nm) to He* (389 nm) intensity as a function of the total gas pressure. We use this ratio in order to have an estimation of the processes that are favored as we change the total gas pressure. Actually, the I_{O*}/I_{He} ratio (fig.7) reflects the variation of electron population with energy higher than 13 eV relative to the electron population with energy higher than 23 eV. In fact, the lower pressure favors processes with high energy requirements (He ionization, excitation). In turn, we can estimate that for the effective desizing of the textile surface other species except of O atoms can have a role. The fact that we obtain the best result at lower pressures indicates that ion bombardment and UV may also have a significant role in the enhancement of surface hydrophilicity. The importance of these parameters is enhanced in the specific conditions as starch removal is also involved in the processing of the textiles.

4. Conclusions

The effect of O_2 fraction in the gas mixture and total gas pressure on the surface hydrophilicity of

He/O_2 plasma treated sized polyester textiles was investigated. The treatments were performed under constant power conditions in order to isolate as much as possible the effect of the plasma parameters on the effective textile desizing.

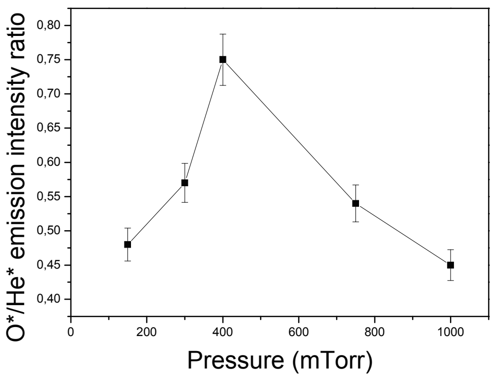


Fig. 7. Ratio of O to He* intensity.*

The best results according to measurements of the rate of water droplet absorption were achieved for high O_2 fractions (75%) and low pressures (150 mTorr). The optimum conditions were related to an effective production of O atoms but also to the important role of ion bombardment. The later is of particular importance when etching of the starch is required prior the treatment of the textile surface.

Acknowledgment

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