

Study of the chemical grafting of polyester textile material with chitosan biopolymer after its pre-activation with atmospheric plasma treatment

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Abstract. The current work investigated the study of the influence of the pretreatment of polyester textile material with atmospheric pressure oxygen plasma on its grafting behavior with chitosan biopolymer as direct grafting procedure without the use of crosslinking agents, which generally modifies the original surface properties of textiles. Firstly, polyester textile material was pre-activated with oxygen plasma treatment under referred conditions. Then chitosan polymer was grafted to pretreated polyester using pad-dry-cure simple method. The different grafting parameters such as time and curing temperature were studied. To evaluate the chemical grafting performance, FT-IR analysis was performed via ATR mode. Mechanical characterization was then conducted showing no degrading of the original mechanical properties of polyester material. The number of functional carboxylic groups formed at different steps of functionalization was evaluated using toluidine blue cationic dye. Finally, antibacterial effect of chitosan grafted polyester biomaterial was evaluated against gram-positive and gram-negative bacteria cultures. The promising results revealed the effectiveness of the plasma pretreatment of polyester process in the improvement of its grafting with chitosan biopolymer.

Keywords: Plasma treatment; Polyester Textile; Chemical Grafting; Chitosan; Characterization; Antibacterial

1. Introduction

The efficient functionalization of textile biomaterials via bioactive polymers has become a great challenge to bring several new efficient properties which find their application in various applied fields [1-6]. Different surface functionalization techniques have been applied in the literature [7-10]. We find chemical functionalization by the creation of covalent chemical bonds between the polymer and the surface of the textile biomaterial [11-14]. This method is effective but remains limited especially if the surface of the material does not contain functional chemical groups in addition to the hydrophobic character of these materials. The physico-chemical functionalization was shown to be effective for inert and hydrophobic materials. But this method involved a functionalization with a low stability and permanence in view of the weak bonds produced between the polymers and the surfaces of the materials [15,16]. Recently, new techniques based on the pretreatment of textile surfaces before functionalization, with the aim of providing new surface properties to subsequently improve the functionalization. Among these pretreatments we find chemical pretreatments which are effective but pollute the environment via the discharges and generally modify the original surface properties of textile biomaterials [17,18]. Other eco-friendly techniques that are effective have been studied, such as UV irradiation treatment [19] and plasma treatment. These methods create active chemical groups by surface activation, allowing permanent grafting of bioactive polymers and molecules [20,21].

Polyester textile is the most used material in the field of textile biomaterials for various medical applications. It is found in masks, sutures, cardio-vascular prostheses, belly prostheses and others. Atmospheric air plasma induces physical treatment by oxidizing textile surfaces. The activation of the material surface allows different functional groups, generally without degrading the original mechanical characteristics of the treated textile [22-24]. The plasma technology involves the use of reactive charged particles

(ions, electrons, excited atoms, and photons) to break covalent bonds by the collision phenomenon [25] and the creation of free radicals and functional groups on the treated surface [26]. Consequently, plasma treatment allows the achievement of different desired properties such as wettability [27], adhesion improvement [28], printing and dyeing, indeed, it promotes further grafting via polymerization process [29].

The present work aimed to study the grafting of chitosan biopolymer onto polyester textile material after its pre-activation with atmospheric pressure oxygen plasma treatment. The modification of polyester surface and the creation of new active groups could promote the chemical grafting and thus bringing the polyester textile biomaterial of the biological properties provided by chitosan.

Different characterization techniques were performed to evaluate the influence of the plasma pretreatment on the grafting of polyethylene terephthalate (PET) textile material with chitosan biopolymer (FT-IR, wettability behavior, mechanical analysis). The number of functional carboxylic groups formed at different steps of functionalization was assessed using toluidine blue cationic dye. Finally, antibacterial activity of functionalized polyester textile materials was evaluated against gram-positive and gram-negative bacterial strains.

2. Experimental

2.1. Materials

The textile material investigated in this study was a 100% polyester woven fabric with a density of 160 g.m⁻², a thickness of 0.52 mm and a porosity of 71%. The plasma processing machine is a Corona / Dielectric Barrier Discharge hybrid type. This machine allows to obtain a homogeneous plasma at atmospheric pressure. The system operates with an electric frequency of 30 kHz and a voltage of 15 kV. An electrical power of 1000 W and a speed of the sample under the two ceramic electrodes (with an inter-electrode distance of 1.5 mm) of 2 m/min were kept constant throughout the various tests.

2.2. Grafting of plasma pretreated PET samples with chitosan

After bleaching and securing, the PET samples were pretreated under oxygen atmospheric plasma treatment. Different plasma pretreated PET samples were grafted with chitosan (solubilized in 2% acetic acid diluted solution) via

pad-dry-cure method [30,31]. Samples were firstly impregnated in a solution of chitosan (30 g/L) soluble in 100 mL of diluted acetic acid (10 mL/L) and completed with distilled water under continuous stirring (180 rpm) for 15 min at room temperature. After padding and roll-squeezing, they were dried in an oven at 105 °C for 30 minutes. Finally, they were thermo-fixed at different times and curing temperatures, washed with water and dried. The grafting rate (%Wt) representing the weight gain before and after weighing was determined according to following equation:

$$\%Wt = \frac{m_f - m_i}{m_i} \times 100 \quad (1)$$

Here, m_f and m_i are the sample weights before and after grafting process.

2.3. Characterization techniques

Washing tests after functionalization were conducted in the aim to evaluate the stability of the grafting. They were performed according to the standardized tests ISO1 (samples washed at 40°C during 30 min with the addition of 3 gL⁻¹ of detergent and 2 gL⁻¹ of sodium carbonate) and ISO3 (samples washed at 60°C during 30 min with 3 gL⁻¹ of detergent and 2 gL⁻¹ of sodium carbonate).

FT-IR analysis was performed on untreated and grafted PET samples. The ATR (attenuated total reflection) mode was used to evaluate the chemical grafting of chitosan biopolymer. Analyzes were realized with infrared spectrometer (Agilent Technologies, CA, USA, 600 Series FTIR Spectrometer). PET spectra were recorded from 4000 to 400 cm⁻¹ with a resolution of 2 cm⁻¹.

The surface wettability of virgin and grafted samples was evaluated via the measurement of contact angles with water liquid test having a surface tension of 72 mJ/m². A digidrop GBX with image analysis software was investigated for contact angles determination. Each value was the average of ten measurements.

2.4. Dosage of the carboxylic groups.

The density of carboxylic groups created on PET surfaces after plasma treatment, was evaluated according to a staining method [32] using an Ortho Toluidine Blue (TBO) cationic dye. The plasma treated PET samples were dipped into a prepared solution of TBO (pH 10) at 30°C for 5 hours. The

samples were then removed and washed with NaOH solution (pH 10) to eliminate any non-complexed molecule dye on the PET surface. The TBO dye was desorbed from the sample in a solution of acetic acid (50%) and the optical density of the desorption solution was determined via UV–Visible spectrophotometer at maximum absorption wavelength of 634 nm. The density of COOH functions was determined according to Beer Lambert Law.

2.5. Antibacterial analysis

Antibacterial activities of untreated and grafted PET samples were realized according to the Mueller-Hinton agar diffusion technique (method of disk). PET samples were prepared in the form of disks before being sterilized. The two different investigated bacteria were *Micrococcus luteus* (Gram-positive, NCIMB 8166) and *Pseudomonas aeruginosa* (Gram-negative, ATCC 33787). They are cultivated on nutrient agar at 37 °C for 24 hours. Physiological saline solution with a bacterial load of 10⁶ CFU /mL, was used for the preparation of the bacterial suspensions. 1 mL of each bacterial suspension was spread on the Mueller-Hinton agar and then incubated at 37 °C during 30 min. The untreated and grafted PET disk samples were then placed on the plates containing the Mueller-Hinton prepared agar. The petri dishes were left at 4 °C for 2 h, therefore the PET disks could diffuse into the agar. After that, they were incubated at 37 °C for 24 h. Finally, the petri dishes were examined, and the inhibition area appearing around PET samples was measured. This area corresponded to the zone of the antibacterial effect.

3. Results and discussion

3.1. Pre-activation of PET with atmospheric pressure oxygen plasma

The free electrons present in the plasma will excite the atoms of the plasma gas and thus generate the appearance of ions, metastable species, and radicals. The radicals obtained in the plasma will react with the polymeric surface by cutting the weakest bonds of the macromolecular chains to react chemically, or at least to eliminate a side chain, or even to create a double bond. Note that on the surface of polymers, the effects obtained depend mainly on the nature of the plasma gas used. Indeed, a plasma gas such as air allows the modification of many polymers by adding not only oxidized groups such as -CO[·], -(C=O)[·], -O-(C=O)[·] and -C-OO[·] on their surface but also amino groups [33].

Regarding PET, the aromatic groups, being very stable, will be difficult to modify by plasma. However, on this type of polymer, the modifications will happen at the level of the much more fragile ester functions $-(C=O)OC-$ [34-36]. R. Zhao et al. [37] have proposed a mechanism for modifying PET by an air plasma based on a first step which is the cleavage of the weakest bonds (ester functions) followed by a second step which consists of oxidation radicals created at the end of the chain. This results in the production of carbonyl, carboxyl, and hydroxyl groups (Figure 1). These functional created groups on PET surface will be investigated as initiators for the reaction with chitosan biopolymer.

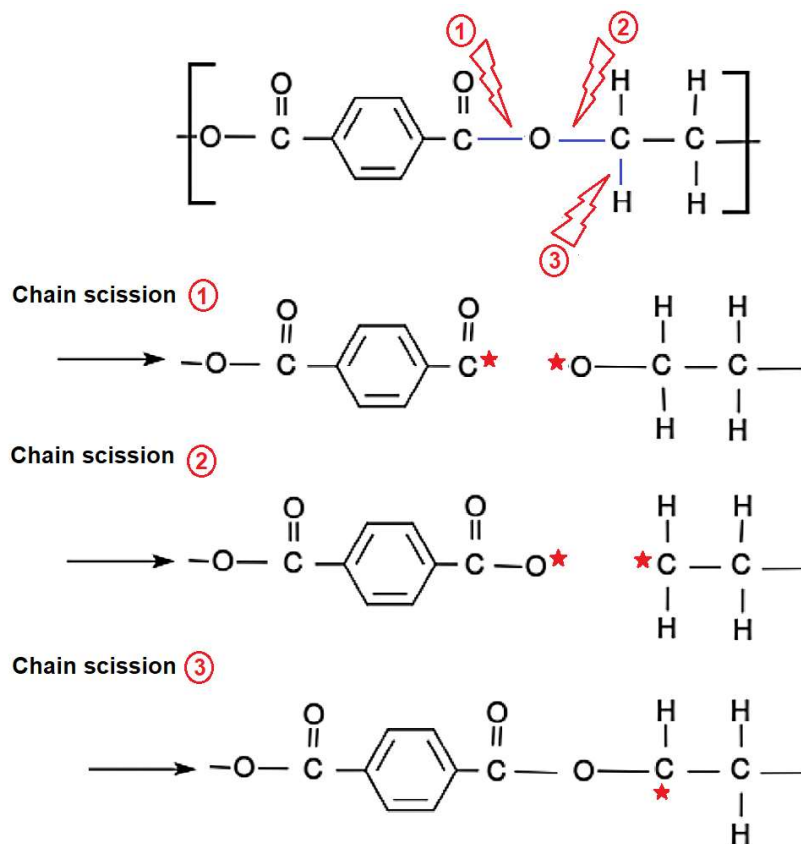


Fig 1. Different chain-scission reactions over atmospheric air-plasma treatment of PET macromolecule and formation of various functional groups on the PET surface

3.2. Grafting of plasma pretreated PET with chitosan

After activation of the PET surface with air plasma, they were functionalized with chitosan polymer. The grafting reaction was based on the creation of a poly-esterification reaction between created carboxylic groups on PET

surface and the hydroxyl groups of the chitosan. In addition, the amine functions of the chitosan could react with carboxylic groups of plasma activated PET via a polyamidification reaction. Untreated PET and plasma pretreated PET were grafted with chitosan according to the pad-dry-cure method previously described.

Results in figure 2, showed the absence of grafting rate in the case of the PET without plasma pretreatment. While for the pretreated PET sample, results revealed the presence of a grafting rate which increases with the time and temperature of curing. Successful grafting was induced without the use of a crosslinking agent. This is a very interesting result compared to other studies that used different crosslinking agents [38,39]. the use of crosslinking agents influences the surface properties of textiles especially the mechanical elongation and roughness.

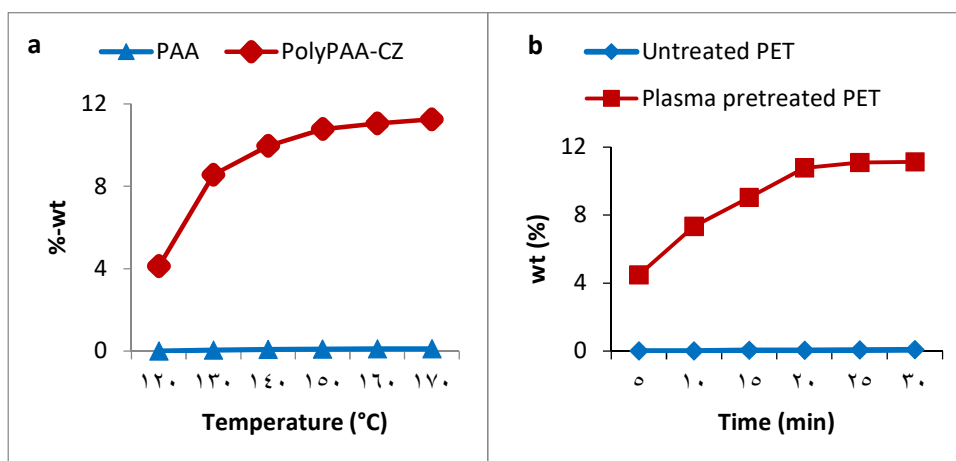


Fig. 2. Grafting rate of pretreated PET with chitosan polymer according to curing temperature (a) and time (b)

3.3. Washing standardized tests

Tests were performed in the aim to evaluate the permanence and the stability of the grafting of chitosan polymer on pretreated PET material. In Figure 3, we noticed no significant change in the grafting rates after the two applied standardized tests. This confirmed the permanence and the stability of chitosan grafting and the efficiency of the plasma pretreatment and the applied grafting process, which does not involve any toxic solvent. The stability of grafting against severe washings could confirm the presence of a

chemical covalent grafting between the pretreated PET and chitosan biopolymer, via poly-esterification or polyamidification reactions.

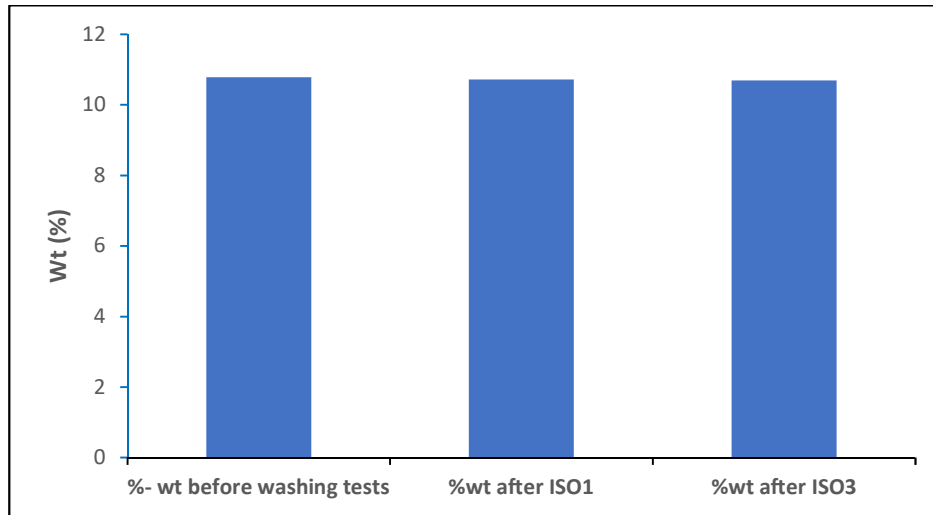


Fig. 3. Washing standardized tests ISO1 and ISO3 realized on plasma pretreated PET grafted with chitosan biopolymer.

3.4. Wettability study

The main impact of plasma treatments is the chemical modification of the surface of materials. This therefore leads to a change in the surface energy of treated samples and hence to a change in the hydrophilicity of the surfaces. Measurements of contact angles were performed on virgin PET, plasma pretreated PET and chitosan grafted pretreated PET. Results in Figure 4, showed the decrease of the contact angle of PET sample after its pretreatment with air plasma. This implies the increase in hydrophilicity after the plasma treatment. After the chitosan grafting onto the plasma pretreated PET, we noticed a significant increase in hydrophilicity, which can be explained by the hydrophilic character of chitosan via its amine and hydroxyl groups [4]. An interesting result for the possible application of our functionalized PET, as hydrophilicity is a required property in the various medical applications of PET biomaterial.

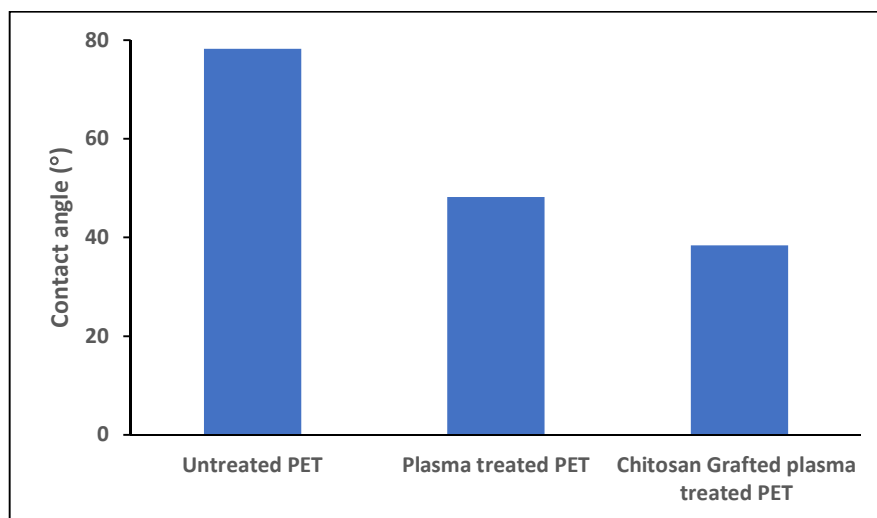


Fig. 4. Contact angles of untreated, plasma pre-activated and chitosan grafted PET textile materials.

3.5. Density of carboxylic groups

The spectrophotometric UV dosage of the complexed TBO was performed on the untreated PET, plasma pretreated PET and the chitosan grafted plasma- treated PET. Figure 5 shows the increase of the amount of TBO complexed dye which signified the increase of the amount of carboxylic acid groups after plasma pretreatment of the PET textile, while the quantity of free carboxylic acid groups decreased after grafting of chitosan on the plasma pretreated PET surface. This confirmed the evident reaction between the pre-activated PET fabric and the chitosan biopolymer via the poly esterification or polyamidification reactions, which significantly lowered the amount of free carboxylic acid function created on the surface of plasma treated PET.

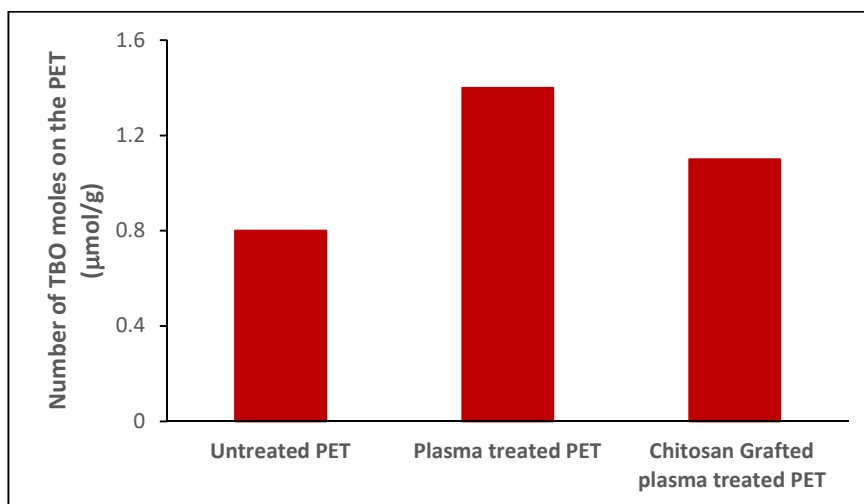


Figure 5. Density of carboxylic groups on untreated, plasma pre-activated and chitosan grafted PET textile materials.

3.6.FT-IR analysis

Infrared analysis was performed in the aim to evaluate the chemical modification of the PET after grafting. Figure 6 shows the different spectra of pretreated PET and chitosan grafted PET fabric. The appearance of a new wide peak close to 3400 cm^{-1} refers to hydroxyl and amine groups of chitosan polymer and could confirm the grafting. The ester or amide groups which could appear after grafting, existed but they are confused with the peak of carboxylic acid of pretreated PET at 1720 cm^{-1} .

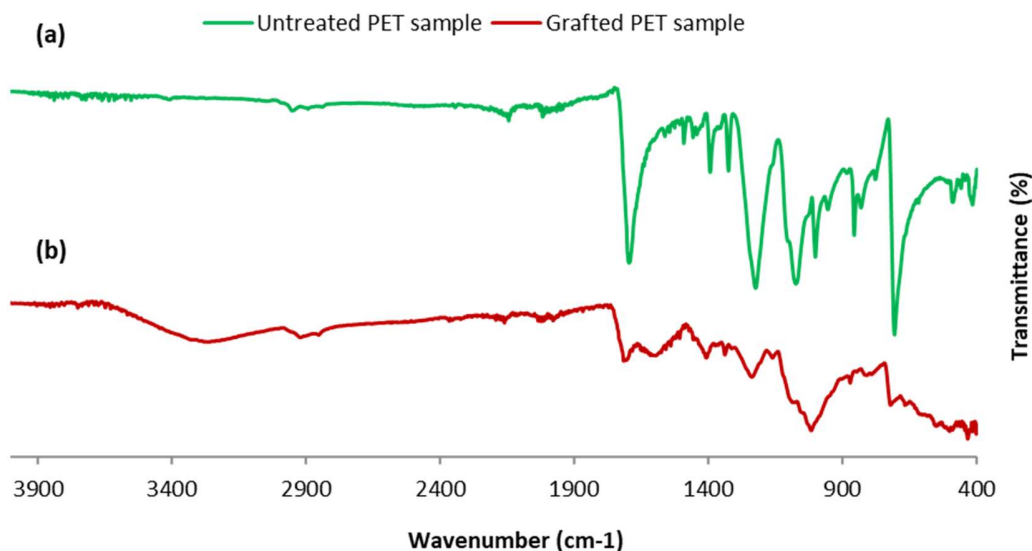


Fig. 6. FT-IR spectra upon attenuated total reflectance (ATR) mode of untreated PET (a) and grafted PET (b) textile materials

3.7. Antibacterial analysis

Two different pathogenic bacteria were selected for the bacteriological analysis, *Micrococcus luteus* (*Ml* ; gram positive) and *Pseudomonas aeruginosa* (*Pa*; gram negative). Antibacterial behavior of chitosan grafted PET fabric was compared to virgin PET sample via the disk diffusion method. The measurement of the diameter of inhibition area which appeared around disk samples was related to antibacterial behavior of the tested sample. Figure 7 reveals no antibacterial effect of the untreated PET samples against the two bacteria cultures. In the case of chitosan grafted PET fabric, we remarked the presence of a significant diameter of inhibition zone around samples for the two tested bacteria, the *Micrococcus luteus* (23 mm) and the *Pseudomonas aeruginosa* (21 mm). This confirmed the antibacterial effect of grafted PET biomaterial and the efficiency of the plasma pretreatment and the grafting processes, which allow a stable chitosan grafting and a preserved antibacterial effect after functionalization.

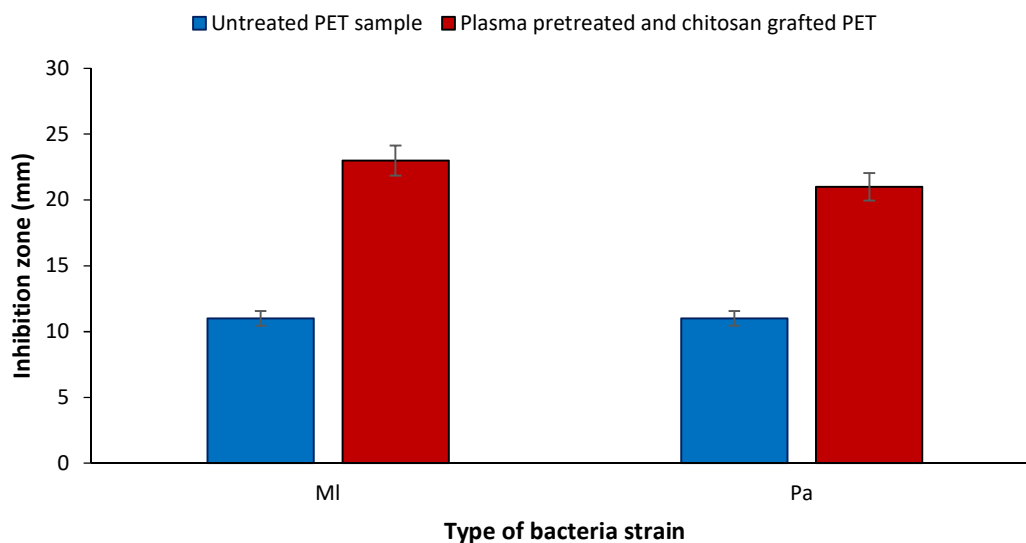


Fig 7. Antibacterial analysis of untreated and chitosan grafted plasma pre-activated PET textile materials.

Conclusion

The current study was an attempt to investigate the physicochemical, morphological, and antibacterial properties of new functionalized PET with chitosan biopolymer after its pre-activation with atmospheric oxygen plasma treatment.

An ecological pretreatment via atmospheric oxygen plasma process, was applied to the PET biomaterial textile. The increase of the hydrophilicity of treated surfaces and the augmentation of the amount of carboxylic acid groups after plasma pretreatment confirmed the successful surface activation of PET fabric. The chitosan biopolymer was then directly grafted to the pre-activated PET sample via pad-dry-cure process. Standardized washing tests have showed the stability of the chitosan grafting. Wettability tests via contact angle measurements revealed the significant increase of the hydrophilicity after chitosan grafting, which is a beneficial property for different medical applications of PET biomaterial. FT-IR analysis and the decrease of the amount of free carboxylic acids on PET surface, via a dosage with TBO dye, confirmed the chemical covalent grafting of chitosan to pretreated PET textile. Finally, bacteriological study revealed the

antibacterial effect of the chitosan grafted plasma pretreated PET biomaterial against two different bacteria strains.

The successful plasma pretreatment and the safe and simple grafting method developed in solid phase, with the use of non-toxic chemicals, could be considered as green and eco-friendly processes.

Knowing the many biological effects and medical uses of chitosan, the functionalization of PET biomaterial by this polymer without the use of crosslinking agent may offer promising potential, of PET and so other textile biomaterials for effective medical devices.

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