Surface treated PET fibers for enhanced functionalization with lavender oil microcapsules

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ABSTRACT

The objective of this work was to functionalize with microcapsules of untreated and treated polyester fabrics with plasma and ozone. Plasma treatment was carried out using a corona discharge at 200 W, 400 w and 800 W with a constant gap between electrode and roll of 4 mm. For ozone treatment, polyester samples were exposed for 20, 30, and 45 min to ozone production by low-pressure mercury lamps. After plasma or ozone, lavender oil microcapsules were immobilized onto fabric by a conventional pad–dry–cure process, using citric acid as a crosslinking agent. The microcapsules were produced by complex coacervation between chitosan and arabic gum, with lavender oil in emulsion. The release showed that the ozone on the surface of PET (polyester) increased the affinity for the microcapsules, resulting in a greater adhesion of the microcapsules and, as consequence, more amount of oil to be liberated. On the other hand, the effect of the plasma treatment was related to physical etching, without a chemical change, and does not interfere with the affinity of the microcapsule, whose external layer is a polar polymer. The delay in the oil release from PET treated with plasma can also be related to the pore formation, which could retain the microcapsules and difficult the release of the oil. Ozone proved to be a good surface treatment to enhance the functionalization of the textile substrate, so that the oil release with time can be greatly improved.

Keywords: PET; lavender, release; fabrics.

1. INTRODUCTION

The term nanotechnology refers to the science and engineering of materials classified in sizes ranging from 1 to 100 nm and concerns the understanding, manipulation and control of matter at this scale so that physical, chemical and biological properties can be designed, synthesized or altered. with the aim of developing new materials (National Nanotechnology Initiative, 2023). In recent decades, there has been a growth in advances in nanotechnology and with it the possibility of various applications in the textile industry. With this, the advancement in the use of nanotechnology by the textile industry is increasingly frequent due to its unlimited potential being used to improve textile attributes such as softness, durability, water repellency, fire retardancy, antimicrobial properties and fiber-like properties, yarns and fabrics using active microencapsulation techniques, providing controlled transfer of these compounds under different conditions. (Sawhney *et al*., 2008; Bezerra *et al*., 2019). Even with the emergence of the corona virus, which causes the acute respiratory syndrome (SARS)-CoV-2, the need to reduce transmissibility using textile materials that can kill or inactivate the virus is evident (Layne *et al*., 2020). Polyester is the most widely used fiber today due to its excellent mechanical properties, resistance to moisture, temperature, light and good chemical resistance, but it has some disadvantages such as high crystallinity, low surface energy and low wettability, which makes it difficult to the functionalization of these materials (Jaffe, Easts and Feng, 2020). One way to add functionalization to polyester fibers is through surface modifications along the fiber. Surface modifications involving ozone gas and plasma treatment are effective not only for improving surface wettability, but also changes in the internal fine structure (crystalline and amorphous regions) leading to an improvement in moisture recovery, water absorption and even enabling active sites for further reactions (Gengenbach and Griesser, 1999; Costa *et al*., 2006; Lee *et al*., 2006). The use of microcapsules in textiles protects the encapsulated assets from evaporation and chemical degradation, controlling the release (Perinelli *et al*., 2020). The functionalization of textiles can be done through biopolymers. Biopolymers are biocompatible, biodegradable and renewable (Arif *et al*., 2022). Many polymers can be applied, but chitosan can associate with anionic chemical groups forming complexes due to its polycationic nature in an acid medium (Massella *et al*., 2019). Another biopolymer that can be used together with chitosan to produce microcapsules involving actives is gum arabic, characterized as a polysaccharide with high solubility in water, good emulsification capacity and film-forming properties, protecting the sensitive core material (Tupuna *et al*., 2018). Essential oils (EOs) are generally complex mixtures of natural compounds and are known for their antiseptic and medicinal (antimicrobial) properties. They are liquid and volatile mixtures obtained from all parts of plants (Nazzaro *et al*., 2017). Lavender essential oil has a composition of more than 100 different molecules, conferring a pleasant fragrance and antimicrobial activity (Lesage-Meessen *et al*., 2015; Ghayempour and Montazer, 2016). Thus, this oil has become important for studies in textile applications. Based on this, the objective of this study is to evaluate the functionalization of polyester fabric modified with gum arabic/chitosan microcapsules containing lavender oil (Valle *et al*., 2021). For this study, unmodified polyester was used using CD plasma and ozone.

2. MATERIALS AND METHODS

2.1. Materials

Plain woven PET, polyester poplin, glycerol, lavender oil, chitosan, Tween 20, and arabic gum are kindly supplied by CARINSA Group (Spain).

2.2. Methods

2.2.1. Production of microcapsules

Microcapsules coated with chitosan and gum arabic were prepared by complex coacervation. The emulsion was carried out at 3000 rpms for 1 minute with oil and surfactant, after which 1% insoluble chitosan and acacia gum (2%) with citric acid (0.5%) were added and then the mixture was carried out again at 8000 rpms for 1 minute. Afterwards, there was cooling to 5 ºC and tannic acid and hypophosphite were added and then the reaction was maintained for 3 hours.

2.2.2. Chemical modification of samples

CD plasma pre-treatments were performed with the Corona plus Type TF-415 (Vet- 91 aphone, Denmark) with a constant gap between electrode and roll (4 mm). The sample was passed once or twice or five times through the discharge area at treatment power 200 W and 400 W and 800W. The measurement of changes in the surface of the polyester fabric after plasma treatment is carried out using the XPS technique with the K- Alpha TM X-Ray Photoelectron spectrometer (Thermo Fisher Scientific, EEUU) equipment. The analysis was done on the same day that glycerol treatment is carried out performed according to Valle et al. (2021). The elemental and superficial content of C and O in the 1s orbital is analyzed. Electron counting is done with a total of 5 scans for each sample. All fabrics were conditioned under standard atmospheric pressure at 20 °C and 65 % relative humidity (ISO 554-1976) for 24 h in a climatic chamber before characterization and this process was performed by triplicate on each sample. After the treatment, all samples were rinsed with deionized water at a specific bath ratio (BR 1/50) and let dry to room temperature. The procedure used was applied following previous works (Gonçalves *et al*., 2021).

2.2.3. Polyester modification by O3

The treatment of substrates with Ozone has been developed using the procedure established by Gabardo *et al*. (2021). Summarizing, ozone modification was performed using a UV-SURF X4 (UV-Consulting Peschl España, Spain) equipment, 17 W power, and an emission spectrum varying from 185 to 254 nm. Polyester samples of 10 cm 20 cm were inserted into the equipment's chamber and exposed for 20, 30, and 45 min to ozone production by low-pressure mercury lamps.

2.2.4. Polyester fabric functionalization

The microcapsules were immobilized by a conventional pad–dry–cure process using citric acid as a crosslinking agent (Arias *et al*., 2021). A aqueous solution was prepared with each of the samples containing 10 % (w/v) of the solution of the microcapsules, 3 % (w/v) citric acid, and 3 % (w/v) Monobasic Sodium Phosphate monohydrate (catalyst). The textiles were immersed into the above finishing liquid (bath ratio = 1:20) for 10 min and, with the help of a foulard, the samples have impregnated a pressure between rolls of 1 bar. Drying was carried out at 80 °C for 3 min.

2.2.5. In vitro” release behavior
The release of lavender oil microencapsulated incorporated into the textiles was done using the technique introduced by Ghaheh *et al*. (2017) and Carreras *et al*. (2013). Samples of 2 x 2 cm of the polyester fabrics were placed in Erlenmeyer with 24 mL of distilled water and 1 mL de ethanol (WEt) maintained in a thermo-stabilized of 37.0 ± 0.5 °C in a static bath. Aliquots of 1.2 mL were extracted at predetermined times during 310 min. Their absorbance was determined by spectroscopy (Shimadzu - UV-240LPC) in the ultraviolet region, between 700 e 200 nm. Oil corresponding to the maximum peak of absorption. A calibration curve for the oil concentration was constructed to determine the amount of active principal ginger compound released into the medium from the microcapsules. After each sample withdrawal, the same volume of fresh WEt was added to the receptor medium to keep constant in the receptor solution during the experiment (Carreras *et al*., 2013).

2.2.6. Scanning electron and optical microscopy
The morphology, size and polydispersibility of the microcapsules were recorded using an optical microscopy Olympus BX43F, coupled to Schott KL300 LED digital camera and Micron Measurement video recorder capture software. Superficial morphology and topography analysis of the polyester fabrics were carried out with scanning electron microscope JEOL, JS-5610 and the gold overlay made in the SCD 005, Bal-Tec equipment.

3. RESULTS

3.1. Microcapsules
The microcapsules obtained presented a circular form as shown in Figure 1. The average diameter of the microcapsules was 15.62 µm. The optical microscopy revealed that the lavender oil is in the center. The difference in the coloration indicates that the surfactant is on the inner edge of the shell. There are two layers visibly formatted, internally composed of chitosan and the external layer is of Arabic gum.

Figure 1 – Optical micrographs of chitosan microcapsules.

The spherical shape of the produced microcapsules is also presented in the SEM micrographs obtained for polyester fabrics impreganted with microcapsules (Figure 2, 3 e 4). Comparing the untreated and plasma or ozone treated samples, a higher amount of microcapsules was impregnated in the polyester fabrics with plasma or ozone treatment. The generation of physical voids through the plasma treatment has increased the absorption capability of the textile substrate.

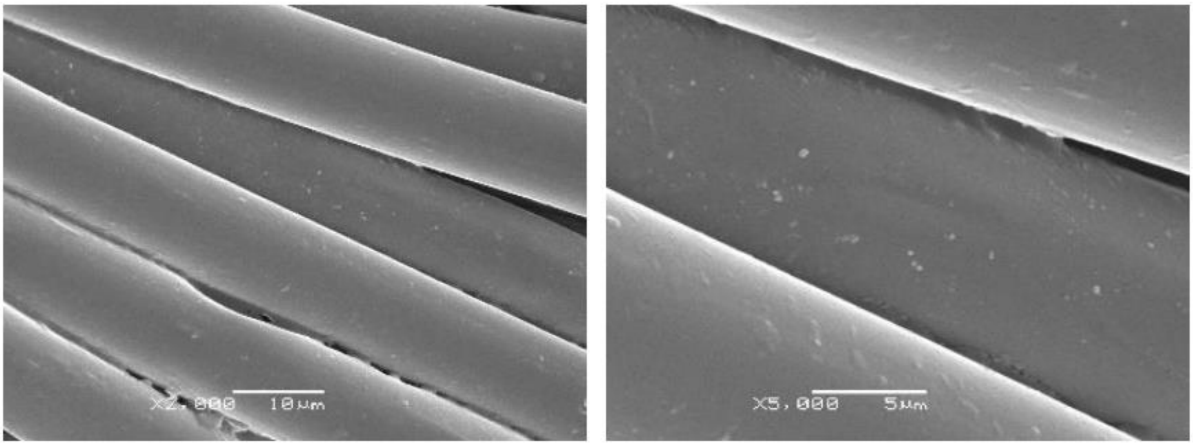

Figure 2 – Morphology of untreated polyester impregnated with lavender oil microcapsules. Magnification of 2000 and 5000x.

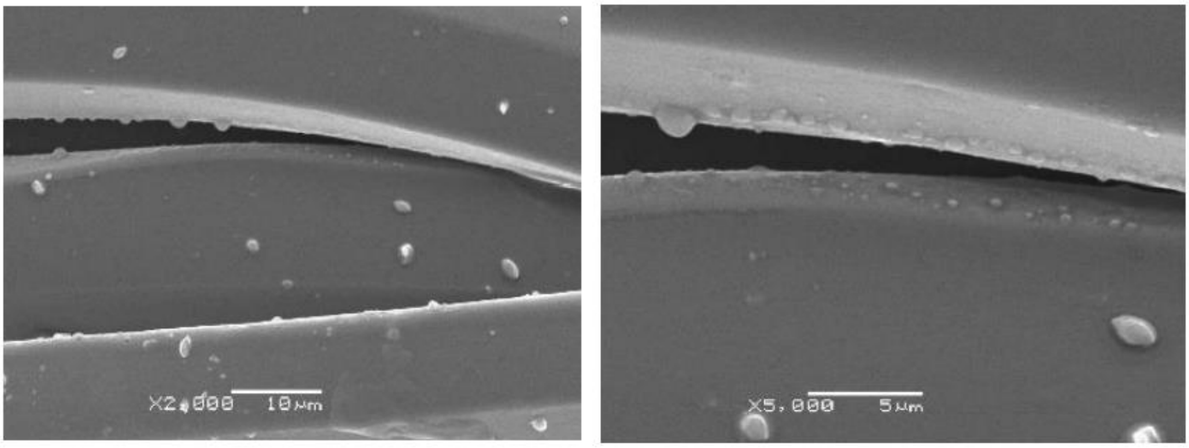

Figure 3 – Morphology of polyester treated with plasma, impregnated samples of lavender oil microcapsules. Magnification of 2000 and 5000x.

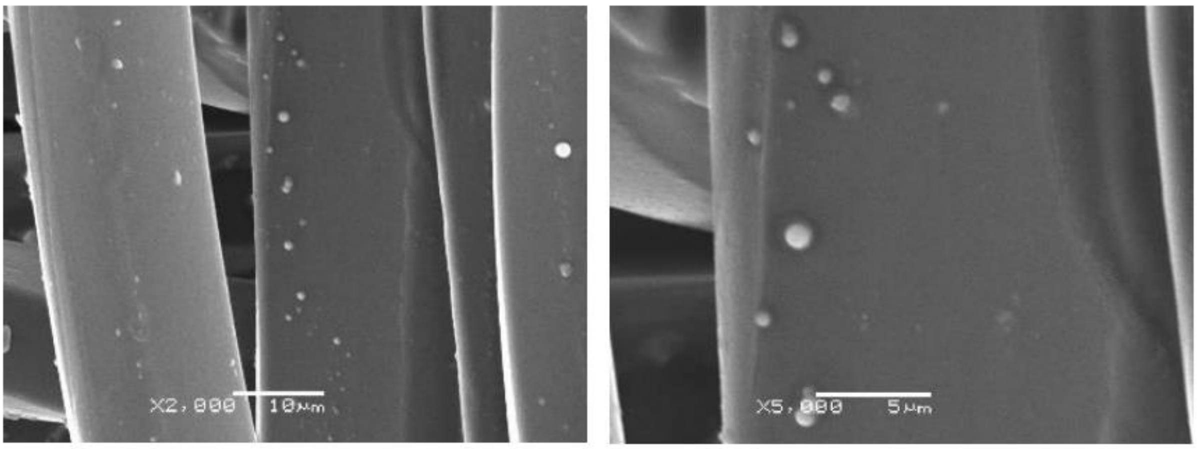

Figure 4 – Morphology of polyester treated with ozone, impregnated samples of lavender oil microcapsules. Magnification of 2000 and 5000x.

3.2. Lavender release

In this case, the UV-VIS was used to study the diffusion phenomena of lavender oil in water at 37 °C from the microcapsules produced for complex coacervation. The behavior of the release of the lavender oil (Figure 5) shows that the polyester treated with ozone liberated more quantity of the oil. This phenomenon can be explained due the polarization of the ozone on the surface of polyester increasing the affinity for the microcapsules. This affinity takes a major adhesion of the microcapsules and as consequence more disponibility of oil to be liberated. On the other hand, the effect of the plasma has a relationship with to formation of porous without a chemistry change, and does not interfere with the affinity of the microcapsule, whose external layer is a polar polymer. The complete release of the lavender oil to the medium lasted thereabout 20 min for the polyester without treatment and 60 min for the polyester treated with plasma and ozone.

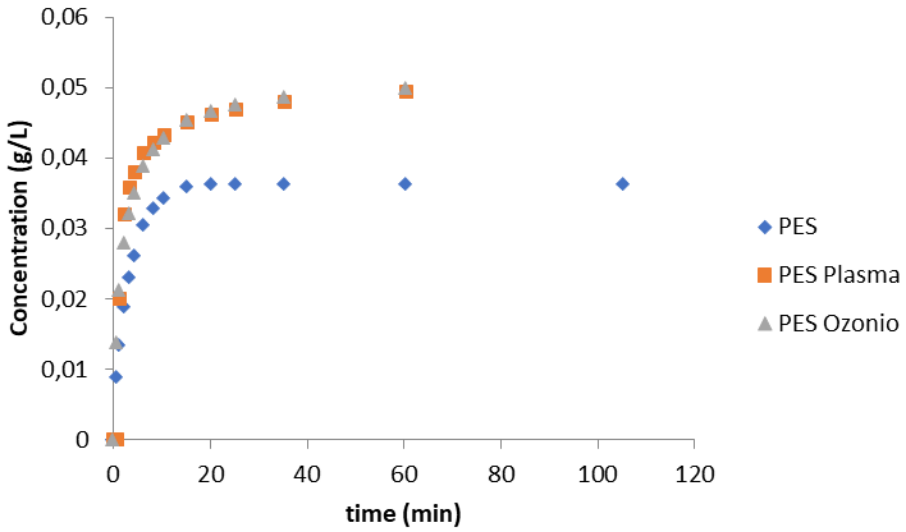

Figure 5 – Release of lavender oil from the microcapsules impregnated in polyester fabrics (PET).

As can be seen in the figure, the behavior can be divided into three different regions. First part, what can be considered as short times (around 4 minutes) where the bath remains, nearly infinite, the slope is related with the pure diffusion from the tissue. In this range of time, the rate of delivery of the essential oil is higher in polyester without treatment, than the observed by the ozone and plasma treated samples. In the case of plasma treated the rate is the lowest, while the plasma case shows an intermediate value. Retention power of microcapsules is higher when polyester has been treated with plasma than when treated with ozone. As it has been demonstrated previously, the essential oil shows a hydrophobic character and, therefore the slope corresponding to this range of times shows crossing lines between plasma and ozone treated samples, while polyester remains in a continuous delivery. After this second step on the delivery of the essential oil, the last part of the graph shows the behavior at very long time, nearly to equilibrium. Here, basically all samples reach the maximum possible amount of delivery at 60 minutes.

4. CONCLUSIONS

In the application of essential oils in textile substrates, the influence of the own substrate has been poorly study. In this case, using Lavender Oil as tracer, we have demonstrated that there is a strong influence on the surface of the substrate, especially when dealing with non-polar substrates. The external character of the shell in the microcapsules, influences, not only the amount of microcapsules to be applied, but also the way in which the active principle is being delivered. Previous treatments of the surface, plasma or ozone, can help to gain in power retention and to control the rate of delivery at different times. The influence can be detected in the study of the drug delivery kinetics.

From these results the join use of microcapsules and modified surfaces, can open a wide range of applications in the field of Bifunctional Textiles.

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