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Plasma-assisted deposition of microcapsule containing *Aloe vera* extract for cosmeto-textiles

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Abstract. Dielectric Barrier Discharge (DBD) atmospheric-pressure plasma was employed to enhance the deposition of commercial microcapsules (MCs) containing *Aloe vera* extract onto a cotton/polyester (50:50) fabric. DBD conditions were optimized in term of energy dosage and contact angle. The MCs were applied by padding and printing methods and the coatings were characterized in terms of SEM and FTIR. MCs display a spherical shape with size between 2 and 8 μm with an average wall thickness of 0.5 μm . The MCs applied by printing and pre-treated with a plasma dosage of 1.6 $\text{kW m}^2 \text{min}^{-1}$ showed the best results with an increased adhesion of 200% and significant penetration of MCs into the fibres network. Plasma printed fabric retained 230% more MCs than untreated fabric after 10 washing cycles. However, the coating resistance between unwashed and washed samples was only improved by 5%. Considering the fact that no binder or crosslinking agents were used, the DBD plasma-assisted deposition of MCs revealed to be a promising environmental safe and low cost coating technology.

1. Introduction

The use of active-substances incorporated onto structure or surface of cosmeto-textiles are increasingly used by cosmetics and pharmaceutical industries in order to provide cosmetic effects such as pleasant feeling, energising, slimming, refreshing, vitalising, skin glowing, anti-ageing, body care, fitness and health [1]. Textile manufacturers are demonstrating particularly interest in the application of durable fragrances to textile as well as skin softeners [2]. In this context, microencapsulation technology is a growing area in textile industry [3]. Microencapsulation is a technique of surrounding solid, liquid or gas particles with a continuous film or polymeric material ranging in size from 1 μm to 1 mm. Microencapsulation can prolong the shelf life of various volatile and non-volatile cosmetic ingredients by delaying oxidation, heat degradation and evaporation. MCs can be applied to textile fibres as dispersion with a binder, using padding, spraying, impregnation, and exhaust or screen-printing techniques [4]. However, the current methods for applying the MCs are not satisfactory. The main disadvantage of using film-forming binders in the application of MCs onto textiles is hindrance of the active substances to be release. To overcome this issue MCs can be covalently linked onto textile substrate by using chemical (e.g. crosslinking reagents) or physical methods (e.g. ultrasound, plasma, microwave) [5]. Challenges as to be overcome such as storage stability, washing resistance, skin transfer while textiles are used, produce real and perceived effects. Additionally, wet methods are substrate specific, not environmental friendly, expensive, and have to be adapted to treat textiles [6]. In recent years plasma technology has assumed a great importance to improve the fibre-matrix adhesion by introducing polar groups, by deposition of a new layer of the same polymer or by



changing the surface roughness of the substrate [7]. It is a dry, environmentally- and worker-friendly method to achieve surface alteration without modifies the bulk properties of different materials [8]. These characteristics may favour the formation of strong bonds between the fibre and polymeric matrix [9]. In particular, atmospheric plasma is an alternative and cost-competitive method to low-pressure plasma and wet chemical treatments, avoiding the need of expensive vacuum equipment and allowing continuous and uniform processing of fibres surfaces [10]. The dielectric barrier discharge technology (DBD) is one of the most effective non-thermal atmospheric plasma sources and has been attracting increasing interest for industrial textile applications [11, 12]. Recently, DBD plasma was successfully employed to improve the adhesion of MCs on wool fabric, PET and cork [13-15]. The main objective of this study is to investigate the adhesion of MCs containing *Aloe vera* extract applied by padding and printing methods in a cotton/polyester (50:50) fabric pre-treated with a DBD plasma discharge. Plasma treatment onto the fabric was optimized and analysed by contact angle, SEM and FTIR analysis. The printing and padding methods was compared in term of coating efficiency and the washing fastness of the MCs deposited onto the textile substrates was evaluated up to 10 washing cycles.

2. Materials and Methods

2.1. Materials

Commercial cotton/polyester (50/50) fabric (CO/PES) with a warp density of 16 threads cm^{-1} , a weft density of 14 threads cm^{-1} , a 20 tex yarn count in both yarns and a surface density of 114.70 g m^{-2} was pre-washed with a 1 g L^{-1} of non-ionic detergent solution at $30 \text{ }^\circ\text{C}$ for 30 min and then rinsed with water for another 15 min. Commercial polyurethane-based MCs of liposoluble *Aloe vera* essence in aqueous dispersion (Bayscent® *Aloe vera*, Tanatex Chemicals B.V., Netherland) was used. All the other reagents were analytical grade purchased from Sigma–Aldrich, St. Louis, MO, USA.

2.2. Plasma treatment

The DBD plasma treatment was conducted in a semi-industrial prototype machine (Softal Electronics GmbH/University of Minho) working at RT and atmospheric pressure in air, using a system of metal electrode coated with ceramic and counter electrodes coated with silicon with gap distance of 3 mm and producing the discharge at high voltage 10 kV and low frequency 40 kHz. The machine was operated at the optimized fixed parameters of 1 kW of power and velocity of 5 m min^{-1} . The dosage ($\text{kW m}^2 \text{ min}^{-1}$) applied in each sample, is defined by the equation: $\text{Dosage} = N P / v l$, where: N, number of passages; P, power (W); v, velocity (m min^{-1}); and l, width of treatment (0.5 m).

2.3. Contact angle measurement

The contact angles of treated and untreated samples with plasma were characterized with Dataphysics equipment (Filderstadt, Germany) using OCA20 software (Germany), with video system for the capturing of images in static mode. Fifteen measurements were carried out for each sample.

2.4. MCs application methods

All samples were cut into $10 \times 5 \text{ cm}$ pieces and stored for up to 30 days indoors at $25 \pm 1 \text{ }^\circ\text{C}$ and $65 \pm 5\%$ relative humidity. CO/PES fabrics with and without plasma treatment were padded in a mini-fouldard (pressure $\frac{1}{4}$ 4 bar, $v \frac{1}{4}$ 6 m min^{-1}), twice through an aqueous finish bath containing 80 g l^{-1} Bayscent *Aloe Vera* MCs nipped to obtain a wet pickup of 70%, and dried in the tensor frame at $140 \text{ }^\circ\text{C}$ for 3 minutes. In the application of MCs by print-screen process frameworks, the same number of padded MCs was prepared in the folder and dry for 3 minutes at $140 \text{ }^\circ\text{C}$.

2.5. Fourier transform infrared spectroscopy (FTIR)

A Nicolet Avatar 360 FTIR spectrophotometer (Madison, USA) with an attenuated total reflectance accessory was used to record the FTIR spectra of the fabric samples. The spectra were collected in the region of 4000–650 cm^{-1} and at a resolution of 16 cm^{-1} with 60 scans at room temperature.

2.6. Scanning electron microscopy (SEM)

SEM analyses were carried out with an ultra-high resolution Field Emission Gun Scanning Electron Microscope (FEG-SEM), NOVA 2000 Nano, SEM, FEI Company. Secondary electron images were performed with an acceleration voltage between 5 and 10 kV. Backscattering Electron Images were made with an acceleration voltage of 15 kV. Samples were covered with a film of Au–Pd (80-20 wt%). SEM images were analysed by ImageJ software to determine size and number distribution of the MCs coated onto the fabric assuming MCs have perfect spherical shape.,

2.7. Washing fastness

The washing fastness (5 and 10 washing cycles) was evaluated according to the standard ISO 105 C06, A1S method at a temperature of 40 °C.

3. Results and Discussion

The surface properties of CO/PES fabrics were analysed by static contact angle measurement to evaluate the effect of different plasma dosages (Table 1). The reduction of the surface contact angle is proportional to the number of plasma treatments. The contact angle continues to reduce until reaching a saturation point between samples 4 and 5 with a reduction of about 70% ($\sim 16^\circ$). The best hydrophilization effect was obtained with a plasma dosage of 1.6 $\text{kW m}^2 \text{min}^{-1}$. Further plasma treatments did not significantly improve the contact angle. CO/PES samples used in this work retained their characteristics for 36 hours after plasma treatment. With time, migration and reorientation of oxidized groups occur driven by diffusion of the high-energy surface radical [16]. These changes could lead in time to significant reduction in properties and loss of surface oxidation [17]. Improved plasma effect durability can be achieved with care in storage, humidity and temperature [18].

Table 1. Number of passages, plasma dosage and contact angle of the cotton/polyester fabric (50:50).

Sample	Passages	Dosage ($\text{kW m}^2 \text{min}^{-1}$)	Contact angle ($^\circ$)
1	0	0	56.7 \pm 4.3
2	1	0.4	44.3 \pm 3.5
3	2	0.8	36.4 \pm 3.2
4	4	1.6	16.7 \pm 3.6
5	6	2.4	15.9 \pm 2.3

As expected the ATR-FTIR spectrum of untreated fabric exhibits peaks pertaining to polyester component of the blend at 1710 cm^{-1} assigned to stretching vibration of C=O group in ester, 1250 cm^{-1} assigned to asymmetric stretching of aromatic ester, 710 cm^{-1} attributed to aromatic C-H bending vibrations and 871 cm^{-1} attributed to C–C out of plane bending vibrations of the benzene rings (Figure 1-a) [19]. At the same time the spectra also displays the very strong bands at 1160, 1100 and 1020 cm^{-1} assigned to the vibrations of the C-O-C bond of the glycoside bridges of the cellulose structure [20]. The peaks at 2900 and 2850 cm^{-1} may be attributed to the C-H asymmetric and symmetric stretching vibrations of aliphatic -CH₂, respectively [21]. The broad and strong bands at 3340 and 3270 cm^{-1} can be attributed to the stretching vibration of the hydroxyl (O-H) group of the cellulose structure and to the intermolecular O-H bonded to C=O groups and O–H out of plane bending in terminal carboxylic groups in polyester chains [22]. After plasma treatment, the CO/PES sample shows a significant increase in the intensity and broadening of the C=O stretching band as well as of the vibration peaks of the C-O-C bond of the glycoside bridges were observed. This may be an indication of the plasma induced changes and oxygen addition onto the fibres surface. The addition of the printed paste without

MCs to the fabric surface (Figure 1-b) is evidenced by the presence of new peaks at 1370 cm^{-1} and 1450 cm^{-1} attributed to $-\text{CH}_3$ and $-\text{CH}_2$ hydrocarbons, respectively [23]. However, the intense peak at 1370 cm^{-1} as well as the new broad peaks appeared between $1000\text{--}1200\text{ cm}^{-1}$ may be also attributed to the C-O stretching of free and condensed C-OH groups since they show a significant increase in intensity after plasma treatment [24]. The introduction of the MCs in the printed paste show a remarkable increase in intensity of the peaks related with MCs presence onto the fabric surface (Figure 1-c). The spectra showed absorption bands of the polyurethane MCs shells at 1730 cm^{-1} for the C=O stretching of urethane, and shoulders at $1690\text{--}1650\text{ cm}^{-1}$ for urethane-urea formation [25]. However, there was not considerable change in IR spectrum of MCs printed fabric after plasma treatment suggesting that the concentration of free oxygen species is decreasing in favour of the formation of intermolecular O-H bonds between MCs and fabric surface. The MCs deposition by padding did not show significant difference for both untreated and plasma treated CO/PES fabric (Figure 1-d).

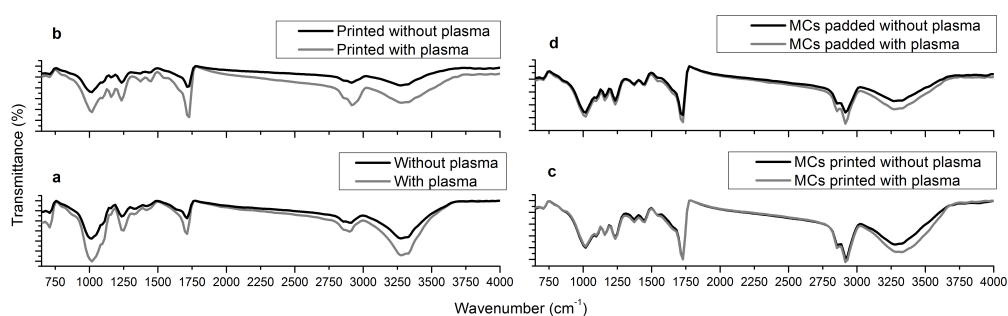


Figure 1. ATR-FTIR spectra of CO/PES without (black lines) and with plasma treatment (grey lines).

Figure 2 shows the SEM of the MCs deposition onto the untreated fabrics by padding. This is the method suggested by the producer to apply the Bayscent® MCs [1]. This MCs are prepared using interfacial polymerization and the release of the active ingredient is triggered by breaking the MCs. Figure 2a showed an efficient deposition of the dispersant but an uneven distribution of MCs on the fibre surfaces by padding. After 10 washing cycles (Figure 2b) the fibres remain covered by the dispersant but almost no MCs can be observed on the fabric surface. Fabric transversal cut of the washed samples (Figure 2c) reveals that the MCs are present in few numbers only in the inner part of the fabric. MCs display uniform spherical shapes but a wide range of sizes (Figure 2d). MCs dimensions vary from $2\text{ }\mu\text{m}$ to $8\text{ }\mu\text{m}$, and thickness is approximately $0.5\text{ }\mu\text{m}$ (Figure 2c). The application of MCs by printing method clearly shows a better adherence and dispersion of the MCs onto the fabric compared to padding process (Figure 2a'). A significant number of MCs remains on the fabric surface after 10 washing cycles (Figure 2b'). Transversal cuts of the washed fabrics show the presence of MCs on the surface of the fabric and confirm that most on the MCs remain confined on the surface without efficiently impregnating the fibres interspaces (Figure 2c'). No differences in size and shape were observed in the printing method compared to padding one (Figure 2d'). It is clear the printing is a more efficient MCs application method compared to padding. For these reason the application of MCs after plasma treatment was studied only using the printing method.

Figures 3 show respectively, the surfaces and transversal cuts of the fabric samples pre-treated with plasma at different dosages and impregnated with MCs by printing process after 10 washing cycles. Plasma treatment significantly enhances MCs adhesion showing a high number of MCs onto the fibres surface even after 10 washing cycles. Transversal cuts demonstrate the MCs greater penetration and adhesion compared to the untreated samples with a significant penetration of MCs into the fibres network. Four different plasma discharge dosages (0.4 , 0.8 , 1.6 and $2.4\text{ kW m}^{-2}\text{ min}^{-1}$) were tested in order to maximize the MCs deposition onto the fabrics' surface. It is well known that the DBD plasma treatment in air is able by chemical etching to create oxidized species on the surface of the fibres [26]. The higher the plasma dosage is, the higher the MCs deposited onto the fabric surface. However, as

can be seen in Table 2, the number of deposited MCs is not enhanced using the higher dosage ($2.4 \text{ kW m}^2 \text{ min}^{-1}$). The fabric treated with the optimal plasma dosage of $1.6 \text{ kW m}^2 \text{ min}^{-1}$ showed the best results and further plasma-energy dosages did not lead to significant differences.

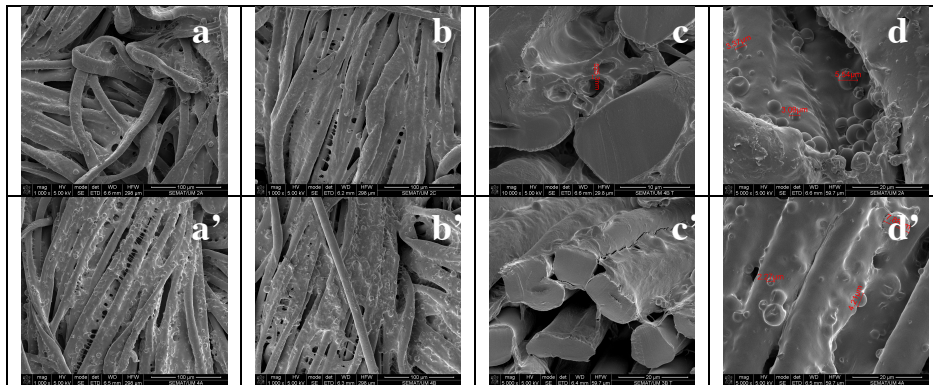


Figure 2. SEM of untreated CO/PES loaded with MCs applied by padding and printing without washing (a, a'), after 10 washing cycles (b, b'), transversal cut (c, c') and MCs size (d, d').

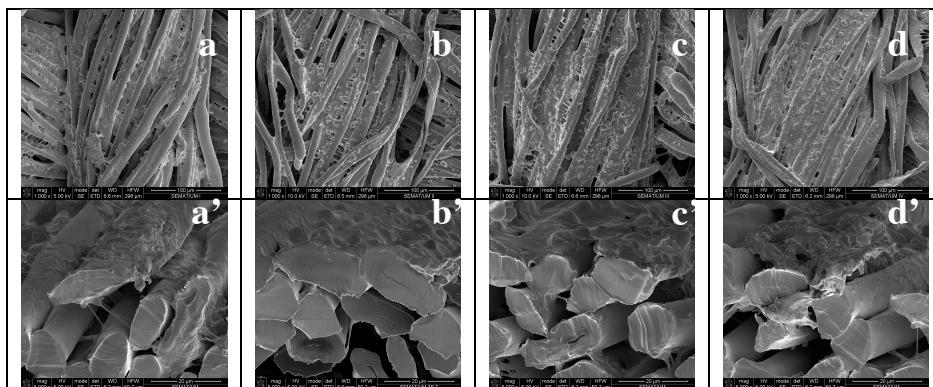


Figure 3. SEM of surface and transversal cut of plasma treated CO/PES loaded with MCs applied by printing at the plasma dosages of 0.4 (a, a'), 0.8 (b, b'), 1.6 (c, c') and 2.4 (d, d') $\text{kW m}^2 \text{ min}^{-1}$ after 10 washing cycles.

A deeper analysis of the deposited number of MCs showed in table 2 demonstrate the superior adhesion and washing fastness of the printing method over padding with a duplication of deposited MCs. Plasma treatment showed a MCs adhesion increasing of 200% at the best dosage of $1.6 \text{ kW m}^2 \text{ min}^{-1}$ and after 10 washing cycles the fabric retained 230% more MCs than the untreated one. However, the coating resistance between unwashed and washed samples was only improved by 5%.

Table 2. Quantification of coated MCs before and after washing (counting area 1 mm^2)

	Plasma dosage ($\text{kW m}^2 \text{ min}^{-1}$)					
	0	0	0.4	0.8	1.6	2.4
	Padding	Printing (number of MCs for 1 mm^2)				
Unwashed	246	466	654	772	939	950
5 washing cycles	120	225	339	392	479	470
10 washing cycles	67	160	259	320	370	367

4. Conclusion

This research demonstrates that the application of cosmeto-textile MCs by printing method using an atmospheric DBD plasma pre-treatment in air can overcome some of the current challenges found in the impregnation methods such as storage coating stabilization, washing fastness, uniform distribution and accessibility. The contact angle, FTIR and deposition tests show that at the optimize plasma dosage of $1.6 \text{ kW m}^{-2} \text{ min}^{-1}$ better hydrophilicity, adhesion and the polar groups concentration can be achieved on the fabric surface. Moreover, it was demonstrated that compared to the padding process the application of cosmeto-textile MCs by printing process provide greater adhesion, better resistance and also reduced costs, allowing the application of MCs in specific target area on the fabric. Overall, considering the fact that no binder or crosslinking agents were used, the DBD plasma-assisted deposition of MCs revealed to be a promising environmental safe and low cost coating technology.

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