Research Article

Superhydrophobic cotton fabrics based on ZnO nanoparticles functionalization

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Abstract
In the present work, 100% cotton knitted fabrics were functionalized with ZnO nanoparticles, in order to enhance the hydrophobic properties of the fibers surface. The incorporation methods of ZnO NPs and different types of cotton samples (including polymer coated, and uncoated with and without a nonionic pretreatment) were evaluated, in order to understand the influence in the hydrophobicity values. Cotton fabrics with and without NPs were characterized by ground-state diffuse reflectance, field emission scanning electron microscopy and energy-dispersive spectroscopy, attenuated total reflectance–Fourier transform infrared spectroscopy and water contact angle (WCA). The best results were obtained when the polymer-coated fabric was functionalized using a precursor concentration of 0.2 M, exhibiting superhydrophobic behavior with static WACs of more than 150°. Although pretreated and untreated ZnO-functionalized cotton fabrics had a slightly lower wettability, they showed interesting results, with improvements in WCA from 116° to 143°. In summary, this work demonstrates that the ZnO NPs have a huge potential to be used as surface finishings for the development of easy cleaning fibrous structures.

Keywords Cotton fabrics · Zinc oxide nanoparticles · Dip-pad-dry method · Superhydrophobicity

Abbreviations

ZnO NPs	Zinc oxide nanoparticles
GSDR	Ground-state diffuse reflectance
FESEM	Field emission scanning electron microscopy
EDAX	Energy-dispersive spectroscopy
ATR–FTIR	Attenuated total reflectance–Fourier transform infrared spectroscopy
WCA	Water contact angle
M	Molar
UV	Ultraviolet
C	Carbon
O	Oxygen
Zn	Zinc
µm	Micrometer

1 Introduction

Cotton is one of the most abundant natural fibers in the world. Owing to its excellent properties such as biodegradability, low cost, high mechanical stability, strong absorption capability and flexibility, the use of this natural fiber has been received exhaustive attention for applications in technical textiles [1, 2].

In the last few years, the innovation on fiber-based smart materials has increased substantiality. These materials, that are capable to respond and to adapt to external stimuli, can be applied in different areas such as healthcare, sports, electronics, military and aerospace [2–4]. The search for materials with superhydrophobic surfaces has become the center of attention to many research projects where, by mimicking nature organisms such as lotus leaves, butterfly and insect wings, superhydrophobic

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surfaces are obtained [5–8]. Applications of these liquid repelling surfaces, such as self-cleaning, low resistance, oil/water separation and antifouling [9–11], have been in high demand by many research groups.

The behavior of a fiber-based material when in contact with water or any liquid is one of the most important properties of textiles. In close to human skin applications, any fabric should present good moisture, wetting and wicking management capacity to make the wearer feel comfortable [12]. It is possible to control the wettability of the textiles by measuring the static contact angle of the water with the sample surface. When the contact angle (CA) between the water drop and the surface is greater than 150° and the CA hysteresis is lower, this surface is considered superhydrophobic [13]. In general, the increase in WCA is attained by adding roughness to the surface of the material or by introducing hydrophobic chemical groups to the structure [6, 14]. A variety of new products and processes, including nanomaterials, has been developed to obtain superhydrophobic and oleophobic surfaces. The superhydrophobicity can be obtained by the use of hydrogels, nanocomposites and other nanostructures [15]. Among these additives, zinc oxide nanoparticles (ZnO NPs) are of particular interest, due to their low cost, biocompatibility and biodegradability for environmental applications and to their hexagonal prism shape, which allows an increase in the surface roughness [16, 17]. Therefore, this nanomaterial can be beneficial to obtained multifunctional materials due to its numerous properties, such as water resistance, antibacterial effect, UV blocking, flame retardancy, corrosion inhibitor and electrical conductivity [18–22]. Gao et al. [23] described the preparation of a superhydrophobic coating based on SiO_2-coated ZnO nanorod and obtained a WCA greater than 150° (as high as 157°). In 2017, Singh et al. [24] suited the effect of using of zinc oxide (ZnO) nanoneedles embedded with cement composites and found that the surface hydrophobicity of the composites increased with the increase in the ZnO concentration, from 46.90° to 88.03°. In 2018, Shaban et al. [25] used ZnO NPs to modify the cotton surface, in order to obtain a hydrophobic fiber. After treatment with these nanoparticles and for the 0.5 M concentration, a superhydrophobic surface was obtained, with a WCA of 154°.

In the present study, the potential of ZnO NPs as agent to obtain superhydrophobic surfaces was evaluated. Polymer-coated and uncoated cotton knitted fabrics with and without pretreatment were functionalized with ZnO NPs by two different methods, in order to achieve the best method for functionalization. All functionalized samples were characterized and evaluated by FESEM, EDAX, GSDR, ATR–FTIR and WCA.

2 Materials and methods

2.1 Materials

The cotton fabrics, a JERSEY 100% CO with a mass per unit area of 145 g/m^2, were supplied by Tintex Textiles SA (Vila Nova de Cerveira, Viana do Castelo), as well as the polymeric coating protocol. Zinc acetate dehydrated (M = 219.49 g/mol) (Zn $(CH_3COO)_2.2H_2O$) and the precursor sodium hydroxide (M = 40.00 g/mol) (NaOH) were obtained from Akzo Nobel (Table 1).

2.2 Sample preparation

2.2.1 Cotton fabrics pretreatment

In order to test, the influence of the pretreatment in the fabric functionalization, three replicates of the T_CoF sample were pretreated with 5% (v/v) of nonionic detergent at 40 °C for 30 min to remove impurities (fats, waxes, etc.). After this, the fabrics were cleaned in running water and before in distilled water in the same conditions (40 °C for 30 min). Finally, the pretreated cotton fabrics were dried at room temperature.

Table 1 Samples descriptions

Code	Description	Polymeric coating	ZnO (M)	Dip-pad-dry	Pretreatment
Co	Dark blue cotton	–	–	–	–
CCo	Coated cotton	Yes	–	–	–
$CCoF_{0.2}$	Coated cotton functionalized	Yes	0.2	2	–
$CCoF_{0.05}$	Coated cotton functionalized	Yes	0.05	2	–
2CoF_*insitu*	Uncoated cotton in situ functionalized	–	0.2	2	–
3CoF_*insitu*	Uncoated cotton in situ functionalized	–	0.2	3	–
T_CoF	Pretreated cotton functionalized	–	0.2	2	Yes
CoF	Untreated cotton functionalized	–	0.2	2	–

2.2.2 Coating preparation

The samples $CCoF_{0.2}$ and $CCoF_{0.05}$ present a polymeric coating that was provided by Tintex Textiles S.A. The coating applied to these fabrics is pink and was further functionalized using the solution of $Zn(CH_3COO)_2.2H_2O$. For this purpose, the fabric was immersed in the $Zn(CH_3COO)_2.2H_2O$ solution, passed by the dip-pad-dry equipment (*Foulard Werner Mathis*) and dried, in order to obtain a superhydrophobic surface.

2.2.3 Synthesis and deposition of ZnO NPs onto cotton fabrics

The ZnO NPs were synthetized accordingly to the method described in [3], with few modifications. The solution of $Zn(CH_3COO)_2.2H_2O$ (CAS [5970-45-6]) (0.05 M and 0.2 M) was heated to 50 °C under constant stirring for 1 h, and 10 mL of NaOH (CAS [1310-73-2]) (1 M) was added dropwise. Different approaches were tested, in order to select the best method for ZnO NPs deposition. In this way, two methods were tested: (1) the fabrics were immersed in the aqueous solutions of $Zn(CH_3COO)_2.2H_2O$ overnight (in situ) under magnetic stirring and (2) the aqueous solution of $Zn(CH_3COO)_2.2H_2O$ was firstly prepared and the fabrics were immersed rapidly in the solution only two times followed by dip-pad-dry method. In all methods, after the immersion in the solution, the samples were impregnated by dip-pad-dry method and dried at 100 °C for 3 min.

2.3 Characterization of functionalized samples

2.3.1 Ground-state diffuse reflectance (GSDR)

Diffuse reflectance spectra were obtained using a Shimadzu spectrophotometer UV 2600, equipped with an integrating sphere. All samples were recorded in the 290–600 nm wavelength range. The remission function F(R) was generated by using Kubelka–Munk equation [26, 27]:

$$F(R) = \frac{(1-R)^2}{2R} = \frac{K}{S'} \quad (1)$$

where R is the reflectance, K is the absorption coefficient, and S is the dispersion coefficient.

2.3.2 Field emission scanning electron microscopy (FESEM) and energy-dispersive spectroscopy (EDAX)

Surface morphology of the samples was made by FESEM using a NOVA 200 Nano SEM from FEI Company (Hillsboro, OR, USA). For the analysis, the samples were coated with a thin film (20 nm) of gold–palladium (Au–Pd), before the experiments. The EDAX technique coupled to FESEM was performed in order to evaluate the chemical composition of the samples, using EDAX Si(Li) detector.

2.3.3 Attenuated total reflectance–Fourier transform infrared spectroscopy (ATR–FTIR)

The evaluation of chemical composition of ZnO functionalized cotton fabrics was made by ATR–FTIR, using an IRAffinity-1S, SHIMADZU equipment, in the range between 400 and 4000 cm^{-1}. The crystal present in the equipment was diamond, and the analysis was done in the transmittance mode, by the accumulation of 45 scans with a resolution of 16 cm^{-1}.

2.3.4 Water contact Angle measurement (WCA)

The wettability of the samples was obtained by the measurement of WCA using contact angle system with high-resolution camera attached. The contact angle (θ_f) of the liquid droplet on a solid surface can be expressed by Young's equation:

$$\cos\theta_f = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}} \quad (2)$$

where γ_{SV}, γ_{SL} and γ_{LV} are the interfacial surface tensions with solid (S), liquid (L) and vapor (V), respectively [28, 29]. A 5 µL droplet of distilled water was dispensed onto the sample's surface and measured the WCA. The samples were analyzed in 10 different places to ensure the homogeneity of the measurement, and the mean and standard deviation were calculated.

3 Results and discussion

3.1 Synthesis and characterization of the functionalized cotton fabrics

The synthesis of ZnO NPs was performed using a sol–gel method, where the NaOH acted as the reducing agent of zinc acetate (precursor), in order to produce the ZnO NPs. The synthesis used is simple and low cost and occurs accordingly with the following reactions [3, 30]:

$$Zn(CH_3COO)_2 \cdot 2H_2O + 2NaOH \rightarrow Zn(OH)_2 + 2CH_3COONa + 2H_2O$$

$$Zn(OH)_2 + 2H_2O \rightarrow Zn(OH)_4^{2-} + 2H^+$$

$$Zn(OH)_4^{2-} \rightarrow ZnO + H_2O + 2OH^-.$$

Initially, the functionalization was performed only for polymer-coated cotton fabrics, and later, when the

concentration was optimized, uncoated cotton with and without pretreatment was tested. Thus, the concentration of precursor (zinc acetate) was varied between 0.05 and 0.2 M, in order to infer about its influence in the final property (superhydrophobicity). The concentration with better hydrophobicity results was 0.2 M, and for this reason was chosen for the following tests. After functionalization with ZnO solution, the appearance of the coated fabric did not change, but in the case of uncoated fabrics, the surface became white.

3.1.1 Ground-state diffuse reflectance (GSDR)

In order to evaluate the functionalization of the fabrics with ZnO NPs, GSDR was used. The results obtained for Kubelka–Munk remission function are shown in Fig. 1.

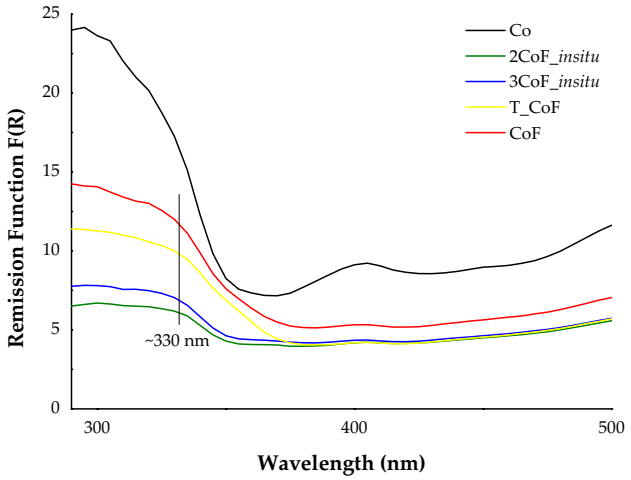

Fig. 1 Ground-state diffuse reflectance (GSDR) spectra of Co, 2CoF_insitu, 3CoF_insitu, T_CoF and CoF

As example, Fig. 1 exhibits the remission function corresponding to uncoated samples. In the cotton fabrics spectra treated with ZnO NPs, the appearance of a new absorption band in the UV region (≈330 nm) is clearly visible, which can be attributed to the ZnO [31, 32].

Furthermore, it is also possible to observe that although the wavelengths of all absorption bands are similar, the F(R) values differ with the incorporation method. Thus, the CoF and T_CoF bands, red and yellow, respectively, have higher values than the 2CoF_insitu and 3CoF_insitu samples. Once again, the pretreatment does not appear to interfere with fabrics functionalization.

3.1.2 Field emission scanning electron microscopy (FESEM) and energy-dispersive spectroscopy (EDAX)

For the coated cotton fabrics, the impregnation of ZnO NPs was made by the immersion of the samples into the solution and passed in the dip-pad-dry equipment. The results obtained by FESEM for these samples are presented in Fig. 2.

For this functionalization, different concentrations (0.05 M and 0.2 M) of the initial solution were tested, in order to understand the influence of the precursor (zinc acetate) on the hydrophobic properties of the samples. After functionalization, the samples were characterized by FESEM analysis, where the appearance of the ZnO NPs in the functionalized samples ($CCoF_{0.05}$ and $CCoF_{0.2}$) is clearly visible presented in Fig. 2. Moreover, it is also possible to see that the morphology presented by ZnO NPs is platelets, according to other authors [33].

Furthermore, by the observation of FESEM images, it was also possible to verify that the amount of NPs present in sample $CCoF_{0.05}$ is lower than in sample $CCoF_{0.2}$.

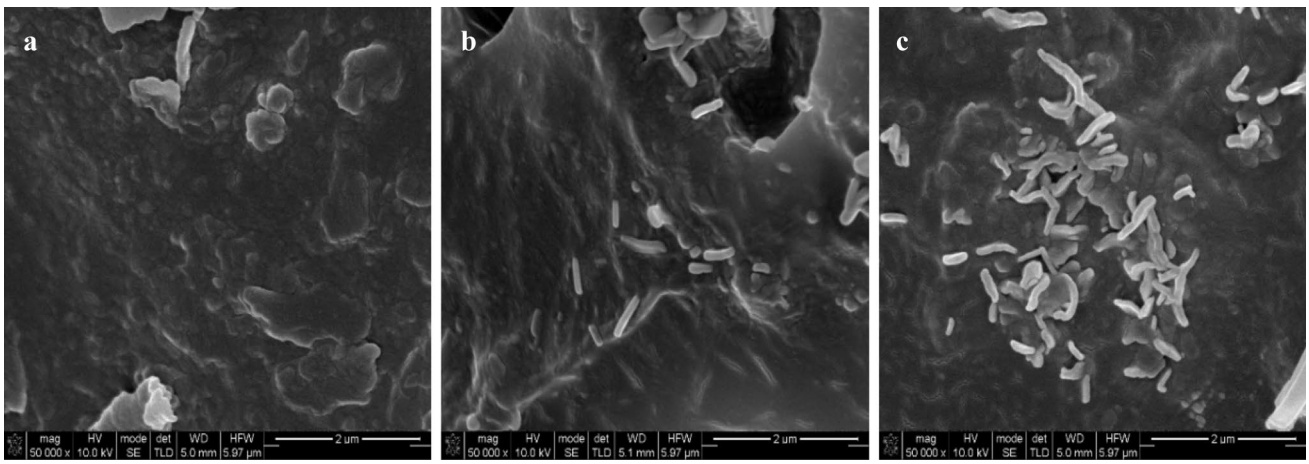

Fig. 2 FESEM images of coated cotton **a** CCo **b** CCoF0.05 and **c** CCoF0.2. The scale of the presented FESEM images is 2 µm and the magnification ×50,000, for all samples

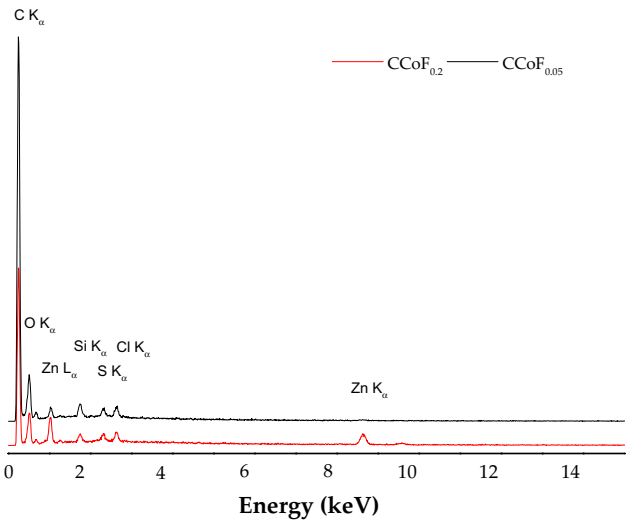

Fig. 3 EDAX analysis of the coated cotton samples functionalized with different concentrations of ZnO

Table 2 Chemical composition of polymer-coated cotton fabrics functionalized with 0.05 and 0.2 M of ZnO NPs

$CCoF_{0.05}$		$CCoF_{0.2}$	
Element	Mass (%)	Element	Mass (%)
C K	73.4	C K	67.1
O	20.8	O	21.7
Zn K	0.60	Zn K	6.70

These observations were corroborated by chemical analysis, presented in Fig. 3.

For the $CCoF_{0.05}$ sample, the ratio of Zn and O is 0.60% and 20.8%, respectively, which confirm the presence of ZnO (Table 2). However, the presence of higher percentage of Zn (6.70%) in the $CCoF_{0.2}$ sample supports the differences presented in the FESEM images. The existence of the C atom is due in part to the precursor zinc acetate, but also to the cellulosic groups present in cotton fabric [34, 35].

The surface morphology of uncoated samples was also studied by FESEM and EDAX analyses. The results obtained by FESEM are presented in Fig. 4.

After their synthesis, the ZnO NPs were incorporated onto the cotton fabrics, as can be seen in Fig. 4b–f. The presence of NPs on the surface of cotton fabrics is clearly visible when compared with the non-functionalized cotton fabric shown in Fig. 4a, d. It is also possible to analyze the NPs morphology at the different samples' surface. Comparing the NPs morphology of the samples with the polymer-coated samples (Fig. 2), in coated samples, the nanoparticles are in the form of platelets, while in these samples there is a coating of the cotton fibers.

As mentioned before in experimental section, three replicates of cotton fabrics were pretreated with a non-ionic detergent, in order to understand its influence in the adhesion of the NPs. The samples with pretreatment are presented in Fig. 4a (non-functionalized) and Fig. 4c (cotton fabric functionalized). Apparently, the presence of the pretreatment in the cotton fabrics surface influences the adhesion of the nanoparticles to the surface of the cotton fibers as shown in Fig. 4b, c. For the pretreated sample (Fig. 4c), the formation of a more uniform and less agglomerated NPs coating of the cotton fiber is notorious, when compared to the non-pretreated sample (Fig. 4b).

On the other hand, the number of passages in dip-pad-dry equipment was studied to optimize the process of the ZnO functionalization. Figure 4e, f clearly demonstrates the presence of ZnO NPs on the surface of cotton fabrics, when compared with the non-functionalized fabrics shown in Fig. 4d. Moreover, the deposition of NPs in the fabric surface appears to be different when passed two or three times by the dip-pad-dry equipment. When passed three times, the samples present a dense and agglomerated deposition, showing worse adhesion to the surface of the fiber.

3.1.3 Attenuated total reflectance–Fourier transform infrared spectroscopy (ATR–FTIR)

The ATR–FTIR spectra of cotton, cotton pretreated functionalized with ZnO NPs, cotton non-pretreated functionalized with ZnO NPs and cotton functionalized with ZnO NPs passed in dip-pad-dry equipment two and three times are shown in Fig. 5.

As example, Fig. 5 exhibits the results obtained for the uncoated samples. All ATR–FTIR spectra show the appearance of two bands around 3330 and 2900 cm^{-1}, which, according to the literature, can be related to stretching vibrations of O–H and C–H groups corresponding to the cotton fabrics constituents: cellulose, lignin and hemicellulose [36].

By analyzing the spectra corresponding to functionalized samples, it is possible to observe the appearance of a peak at 1558 cm^{-1} and other at 1400 cm^{-1} relative to the stretching vibration of C=O group [37–39], which can be associated with ZnO NPs precursor (zinc acetate), proving the presence of ZnO on the fabric. In these spectra, it is also possible to observe two new small peaks at 667 cm^{-1} and 609 cm^{-1}, which are usually attributed to Zn–O stretching vibration, confirming the presence of ZnO NPs in the cotton fabrics [40]. The presence of ZnO in the structure is certain and can be attained by the alteration of the material by differentiation of new bands, which

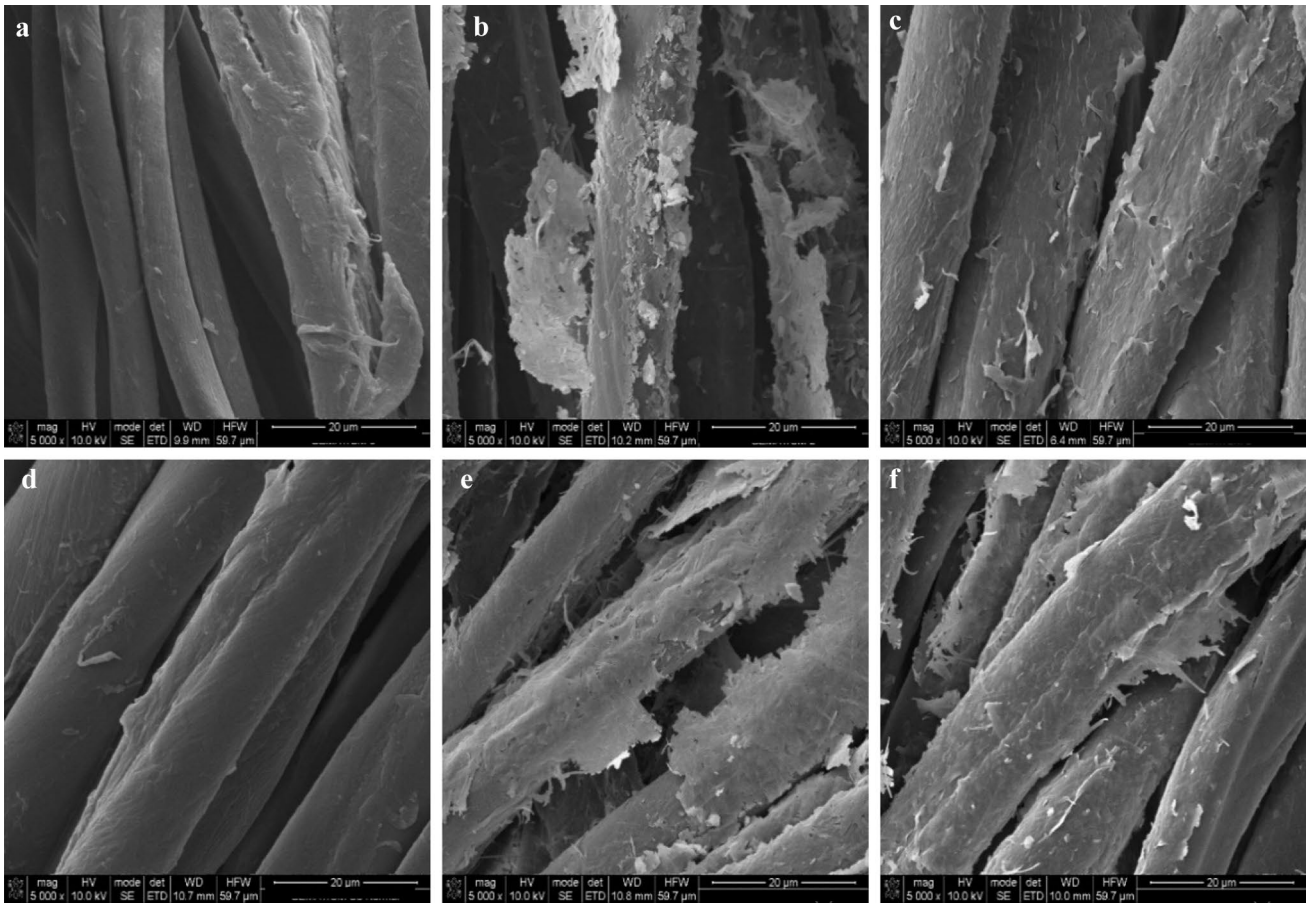

Fig. 4 FESEM images of uncoated cotton **a** T_Co **b** CoF **c** T_CoF, **d** Co, **e** 3CoF_insitu **f** 2CoF_insitu. The scale of the presented FESEM images is 20 µm and the magnification ×5000, for all samples

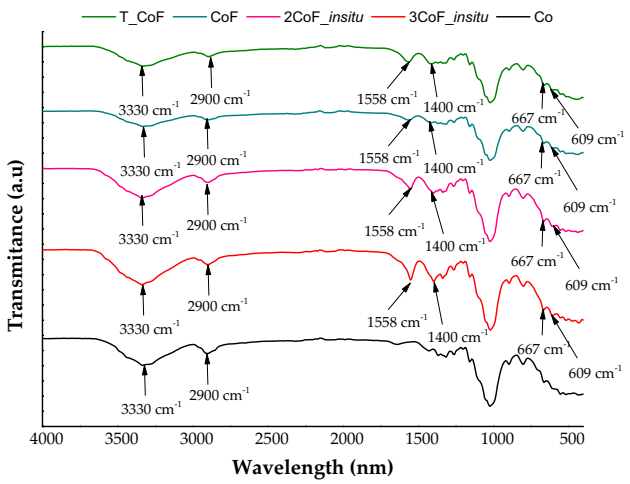

Fig. 5 Attenuated total reflectance–Fourier transform infrared spectroscopy (ATR–FTIR) spectra of T_CoF. CoF, 2CoF_insitu and 3CoF_insitu

previously did not exist. This spectrum also reveals that the pretreatment used before does not appear to influence the functionalization. This is because, in the untreated and pretreated samples the peaks presented were the same.

3.1.4 Water contact angle measurement

The wettability is one important property when it comes about superhydrophobicity. This property (dependent of the surface chemical composition and roughness) was evaluated by measuring the WCA in all samples in 10 different sites to ensure the accuracy of the results [41]. Figure 6 shows the WCA obtained for Co, CoF, T_CoF, 2CoF_insitu, 3CoF_in-situ and for CCo, $CCoF_{0.05}$ and $CCoF_{0.2}$. If the WCA is equal or higher than 150°, the surface is considered superhydrophobic, but if the WCA is between 90° and 120° the surface is considered hydrophobic [42].

When a drop of water is released onto a raw cotton surface, it is immediately absorbed, and its angle to the surface is considered to be 0° [41]. However, the cotton provided by Tintex has a water contact angle of 116° (Fig. 6a),

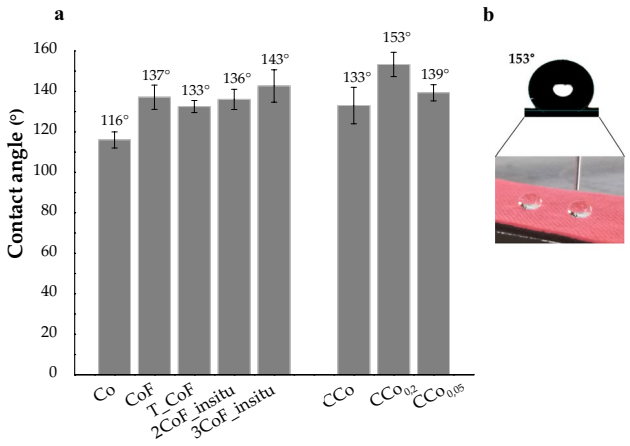

Fig. 6 Representative images of WCA analysis **a** values of WCA represented by mean ± SD and **b** value of WCA of the $CCoF_{0.2}$

which proves that it has some kind of finish and it is not raw cotton. Although raw cotton is already hydrophobic, this property has increased with the introduction of ZnO NPs.

With the addition of the ZnO NPs, the WCA was continuously increasing, as it can be seen in Fig. 6a. As previously mentioned, one of the factors that influence the WCA is the surface roughness of the sample. The non-functionalized fabrics have a smooth surface, as can been seen in Fig. 4a. In contrast, when the ZnO NPs are incorporated onto fabrics, the roughness increased (Fig. 4b and c) and, consequently, the WCA too. The CoF and T_CoF samples show an increase in WCA from 116° to 137° and 133°, respectively. This difference may be due to the pretreatment made before the experiment, which led to a removal of oils, lignin and pectin that have contributed to the hydrophobic character of this surface [3]. Besides that, the samples 2CoF_insitu and 3CoF_insitu also exhibit an enhancement of the hydrophobic properties. In this case, the number of the passages in the dip-pad-dry equipment had a significant impact, proving that the three passages are the best option to get a higher WCA. Although the incorporation of ZnO NPs improves the WCA of the uncoated samples compared to the initial cotton fabric, the superhydrophobicity level was not achieved.

When the polymeric coating was applied on the fabric, the roughness changed and, consequently, the WCA increases significantly to 133° (CCo sample), as shown in Fig. 6a. The addition of ZnO NPs to the polymeric coating induced a significant increase in WCA (higher than 150°), reaching the level of superhydrophobicity for the CCo0.2 sample, as can be seen in Fig. 6b. With these results, it was also possible to conclude that the precursor concentration (zinc acetate) used in synthesis influenced the hydrophobic properties of the samples.

Nevertheless, with exception of $CCoF_{0.2}$, all the samples exhibited a hydrophobic behavior, being in agreement with several studies that report the efficiency of ZnO NPs to produce hydrophobic structures [43–45].

4 Conclusions

In conclusion, the ZnO nanoparticles were incorporated in coated and uncoated cotton fabrics efficiently, and the most efficient method for samples functionalization was alternated immersion of the fabric in the solution and in the dip-pad-dry equipment, which enhanced the WCA results. Characterization analyses confirmed the synthesis and incorporation of nanoparticles onto the fabrics surface and allowed to observe that the 0.2 M concentration improved the WCA results, reaching to 153° for the CCoF0.2 sample, what proves that the precursor concentration (zinc acetate) used in synthesis influenced the hydrophobic properties of the samples. For the uncoated samples, it wasn't possible to achieve superhydrophobicity, although the improvements in hydrophobic properties were clearly visible, reaching WCA values of 133°. Due to the high WCA obtained for these samples, it might be possible to achieve even higher values of WCA, if the solution concentration was increased. The samples pretreated before the incorporation of the NPs showed no significant differences in GSDR, ATR–FTIR analysis, but in FESEM and WCA it was possible to verify some differences. GSDR study has shown an absorption band at UV region (≈ 330 nm), also proving the presence of ZnO NPs. Overall, this work demonstrated the development of superhydrophobic natural-based surfaces, obtained through the functionalization with zinc oxide nanoparticles, taking into account the sustainability, low cost and simplicity of the methodologies under use.

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Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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