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Screen Printing of Cotton Fabric with Hydrochromic Paste: Evaluation of Color Uniformity, Reversibility and Fastness Properties

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ABSTRACT
Smart textiles are promising for the future of the textile industry, providing natural fibers with attractive and interactive features. Within this context, it is possible to emphasize the chromic materials, as, for example, the hydrochromic, which alter their visual properties (color) through an external stimulus (water). The purpose of this work is to functionalize and characterize cotton knitted fabric with hydrochromic agent studying the reversibility and fastness properties of the print. It was observed that the particles are well dispersed and adhered to the natural fiber surface and did not display significant changes in the color after subsequent washing and rubbing fastness cycles.

摘要
智能纺织品为纺织工业的未来带来了希望, 它提供了具有吸引力和互动功能的天然纤维. 在这种情况下, 可以强调铬材料, 例如水变色材料, 通过外部刺激（水）改变其视觉特性（颜色）. 本研究的目的是利用水致变色剂对棉针织物进行功能化和表征, 研究印花的可逆性和牢度性能. 经观察, 这些颗粒在天然纤维表面分散良好, 粘附在纤维表面, 在随后的洗涤和摩擦牢度循环后, 颜色没有明显变化.

KEYWORDS
Smart textile; chromic materials; natural fiber; knitted fabric; functionalization; colorimetry

关键词
智能纺织品; 铬材料; 天然纤维; 针织物; 功能化; 比色法

Introduction

Textiles can be divided mainly by four groups: conventional, technical, functional and smart textile. Specifically, the smart textiles appeared to recreate the user's interaction with clothing. They are substrates that respond to stimuli, either by varying light, touch, temperature, moisture; and when not exposed to external factors, behave normally, without showing any changes. The response to the stimulus is given by changes in physical, mechanical, chemical, magnetic, electrical or thermal properties of the material (Sengupta and Behera 2014), being seen, for example, through the change of the color, shape, volume, viscosity, among others (Syduzzaman et al. 2015).

According to the report released in March 2019 by Grand View Research, Inc., in 2018 the global market for smart textile was valued in 878,9 USD million. For the years from 2019 to 2025, an expansion of 30.4% is expected and a prospect of reaching 5.55 USD billion by 2025. These numbers confirm the importance of the smart and functional textiles, which can be applied in different areas, such as, for example, related to design, sports, medical, transportation, protection, military and architecture (Grand View Research, Inc 2019).

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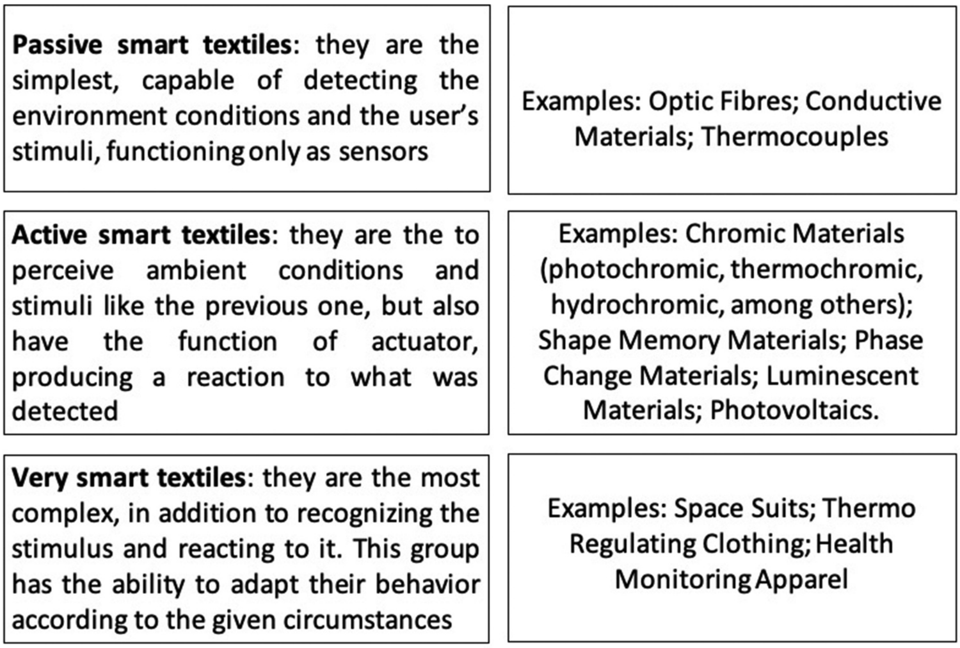

Figure 1. Subdivision of the smart textiles.

As examples of smart textile, there are wearables (Picard and Healey 1997), activity-inducing materials, phase change materials, shape memory alloys (Mattila 2006) and also, the chromic materials (Syduzzaman et al. 2015). The smart textiles can also be classified in subgroups, as illustrated in Figure 1.

Chromic materials are part of the active smart textiles' subgroup, a very promising class of materials when it comes to the future of textile products (Stoppa and Chiolerio 2014). Chromic substrates are those that change their visual properties, reflecting different colors due to its induction by external stimuli (Chowdhury, Joshi, and Butola 2014). They are also described as "chameleon" materials, due to its changes according to the place conditions or induced stimuli (Ferrara and Bengisu 2014). The classification of the color change phenomenon is shown in Table 1, correlating each phenomenon to its respective stimulus.

The preponderance of applications for chromic materials in the textile area is in fashion and design segment. But it is also a huge opportunity to apply in other areas, such as workwear, protection and medical textiles (Sengupta and Behera 2014).

Table 1. Phenomenon and stimulus for color change.

Phenomenon	Stimulus
Photochromic	Light
Thermochromic	Temperature
Electrochromic	Electricity
Halochromic	pH
Hydrochromic	Moisture
Piezochromic	Pressure
Vapochromic	Steam
Chemochromic	Chemical reaction
Touchchromic	Touch
Biochromic	Phatogens
Mechanochromic	Mechanical forces

Hygro-Hydrochromic materials are getting even more popular in the textile area (Mal and Iqbal 2014; Vik and Periyasamy 2018). They are materials that suffer visual property changes (color changes) with the moisture presence (Mal and Iqbal 2014). They form a category of a most embracing group called "solvatochromic," which changes its own properties by being sensitive to a certain solvent (liquid or gas), related to its polarity. Therefore, solvatochromism is a color change brought about by a change in solvent polarity (Ferrara and Bengisu 2014).

Hydrochromatic agents can be reversible or irreversible. The irreversible ones are usually found in several colors such as blue, black, red, yellow and green colors. It depends on the chemist who defines the chemical composition and the absorption ranges of the compounds (Đurđević et al. 2015; Huang 2017). The reversible ones are more common, and they normally change their color from white to transparent (when wet) and return to white when they get dry again (Yates 2012). Figure 2 represents schematically the hydrochromic reversible material: the fabric is screen printed (a); after its functionalization, the print is covered by the hydrochromic material, making the fabric white (b); when the fabric gets wet, it shows the initial impression (c); when the fabric gets dry, it returns to the hydrochromic material white color, hiding the initial pattern design (d).

The best results with hydrochromic agents with regard to applicability and durability are achieved when the paste is applied on smooth and dry surfaces. In fact, the surface on which the hydrochromic paste is applied to affect the results as well, although paper, plastic, wood, metal and glass can be covered by the ink, the surfaces must all be flat. A non-uniform surface may affect the transparency of the wetted state because the depressions or holes in the surfaces can get clogged by the paste in a way that prevents the absorption of water (Tarhan 2019). For this reason, the hydrochromic paste application in textile structure to obtain a good uniformity and reversibility is not an easy task. Examples of materials that can be used: soft sheet vinyl, paper, coated paper, styrene sheet, among others. The techniques used to apply the hydrochromic are screen printing or spray coating, followed by passing it through a forced hot air tunnel (Ferrara and Bengisu 2014). Specifically, in textile substrates, studies using hydrochromic materials were printed onto polyester, cotton, polyamide and blended; elastane and polyester (Ferrara and Bengisu 2014; Toffanetti 2019). However, the study of the reversibility using different liquids, the washing and rubbing fastness were not presented in the literature aforementioned.

When hydrochromic agents are included in textile substrates, the most diverse products can be suggested, especially where the liquid introduction interacts with the material, such as furniture covering, umbrellas, tablecloths, swimwear, sportswear, dressing, among others (Vik and Periyasamy 2018; Yates 2012).

Nowadays, due to the current pandemic related to COVID-19, hydrochromic materials can be a good suggestion when applied, for example, in face masks or other hospital textile materials. The ink or coating with hydrochromic properties, when printed and fully dry, becomes opaque such that it will preferably mask a previously printed image or legend. However, when a liquid or humidity gets in touch with the functionalized coating it becomes translucent allowing the underlying the image or legend to be seen. In this case can appear a message indicating that the mask must be discarded or changed, creating a smart textile material to be used in medicine area.

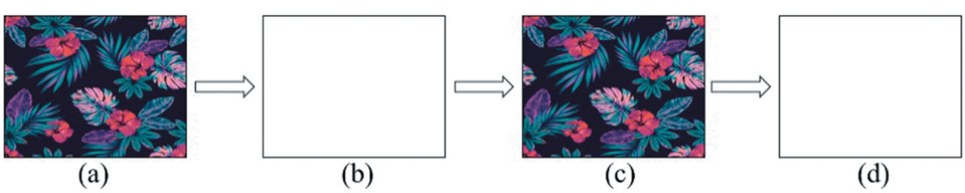

Figure 2. Operation scheme of reversible hydrochromic material: screen printed fabric (a); functionalization by the hydrochromic material (b); wetted fabric (c); dried fabric (d).

Despite checking the literature on several works related to the smart textile topic, specifically, the study of application and fastness properties with the hydrochromic materials in natural fibrous substrates is still very little explored scientifically. Thus, the objective of this work is the study of cotton knitted fabric functionalization with hydrochromic agents, its washing and rubbing fastness, reversibility process with subsequent morphological and structural characterization.

Materials and methods

Materials

In the present work 100% cotton (CO) jersey knitted fabric (beige color) was used with a density of 46.8 courses/inch and 36.0 wales/inch and a weight of 148 g/m2.

The hydrochromic paste utilized is a commercial water based (white color) and is named Water Based Wet & Reveal Ink, SFXC HC dispersion.

Methods

Functionalization of the samples

The application of hydrochromic materials on the textile surface was performed by using the flat screen printing technique. The polyester screen frame used in the printing process (20 cm × 20 cm) was manufactured with a 55 mesh (holes per cm) fixed on a rectangular wood frame. The emulsion thickness was 0.20 mm and it was exposed on a screen mesh through an indirect screening process and a drying time of 2 hours at 35°C. The fabric to be printed is put under the printing screen and the hydrochromic paste is applied on the technical right of the knitted cotton fabric by using a wooden applicator with a rubber edge.

To verify the ideal amount of hydrochromic agent and uniformity, paste application was made with 1, 2, 3 and 4 passages of the squeegee over the frame. After the samples were placed immediately in the lab oven at approximately 130°C for 3 minutes. The fabrics were then washed with cold water, hot water for 4 min and finally at 70°C for 2 min with a solution containing 1 g/L nonionic detergent. The printed fabrics were rinsed in cold water and air dried.

Subsequently, with the use of the reflectance spectrophotometer, the uniformity parameter was determined, through the quantity of squeegee passages that got the lowest variation coefficient. The functionalization of the samples was realized in triplicate.

Scanning electron microscopy

The morphological analysis of the textile surfaces before and after the functionalization, just as with the distribution of the hydrochromic material on the substrates, and the sample analysis after the fastness tests were realized with the scanning electron microscope JEOL, model JSM-6390LV, 2007. The samples were, initially, deposited in a stub and covered with gold, using the sputtering technique in a LEICA model EM SCD 500 coating machine. Samples were enlarged 50, 500, 1000 and 2000 times.

Reflectance spectrophotometer

The equipment utilized was the DATACOLOR 500, 2017, spectrophotometer. For all the measurements, the system used was CIEL*a*b* (Coordinates L*: luminosity, a*: red/green and b*: yellow/blue), illuminating D65, observer of 10° and an opening of 6.6 mm. The reflectance spectrophotometer was employed to characterize the uniformity, the washing and rubbing fastness of functionalized samples. Besides that, it was also used to simulate the reversibility behavior of the hydrochromic material on the knitted fabric, through the drying curve of the wet fabric, in this case using the color difference (ΔE^*) reported as

$$\Delta E^* = \left((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right)^{1/2}$$

For that, each sample was wetted with three drops of the respective liquid to be tested (deionized water and urine), each drop averaged 0.0361 g. The color change was spectrophotometrically measured over the time: 0, 3, 6, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, 60, 65, 80, 100, 120 and 180 minutes. For this analysis, the coordinate L*, which represents the luminosity, was verified. The measurement parameters used to simulate the reversibility behavior of the hydrochromic material on the knitted fabric were water and urine with environmental conditions of temperature and humidity controlled 22.0°C and 40.0%, respectively.

The cited liquids were applied because the water is the universal solvent, and the urine is due to a specific application in the area of diapers hygiene, which is normally produced from cotton fiber.

Washing fastness test

The HT-IR DYER equipment, TC-2200 model, TEXCONTROL, 2014, was employed to realize the washing of the samples according to the standard Norm ISO 105-C06:2010. For each sample, it was simulated 1, 5 and 10 washing cycles. After 10 cycles, specimens were analyzed in the reflectance spectrophotometer and the SEM technique was used to verify the hydrochromic agents that remained in the textile surface.

Rubbing fastness

The wet and dry friction resistance test was performed by using the crockmeter equipment, KIMAK, CA-11 model, 2015, according to the standard Norm ISO 105-X12:2016. After the experiments, the functionalized samples were evaluated by reflectance spectrophotometer and SEM images.

Results and discussions

Uniformity of the paste on the cotton fabric

To determine the uniformity of the hydrochromic paste over the fabric, it was used as a spectrophotometer to measure the colorimetric coordinates L*, a* and b* of each sample. The color of the light reflected by the screen printed layer can give a qualitative indication of the particle distribution on the textile substrate. The colorimetric coordinates obtained after 1, 2 and 3 passages of the hydrochromic paste over the CO knitted fabric are presented in Table 2. With four passages, the higher amount of hydrochromic paste gave a high increase in stiffness to the printed fabric, presented also an irregular paste deposition. From the numbers obtained from four measurements the average, standard deviation and coefficient of variation of the aforementioned colorimetric coordinates were calculated.

Through the numbers presented in Table 2, it can be seen that the standard deviation and coefficient of variation (CV) numbers from the L*, a* and b* coordinates are lower for samples with three passages, indicating a more uniform coating. Therefore, this is the number of passages chosen to develop the work.

Table 2. Evaluation of the paste uniformity over the CO fabric.

	Passage 1			Passage 2			Passage 3		
	L*	a*	b*	L*	a*	b*	L*	a*	b*
Point 1	82.79	3.42	3.93	77.39	5.28	5.90	78.18	5.25	5.78
Point 2	82.60	3.56	4.12	76.66	5.47	5.87	77.22	5.61	6.02
Point 3	81.29	3.99	4.57	78.21	4.95	5.49	78.42	5.09	5.75
Point 4	80.43	4.32	4.86	77.65	4.96	5.79	78.69	5.20	5.93
Average	81.78	3.82	4.37	77.48	5.17	5.76	78.13	5.29	5.87
Standard deviation	1.12	0.41	0.42	0.64	0.25	0.9	0.64	0.23	0.13
CV [%]	1.37	10.75	9.67	0.83	4.93	3.25	0.82	4.26	2.17

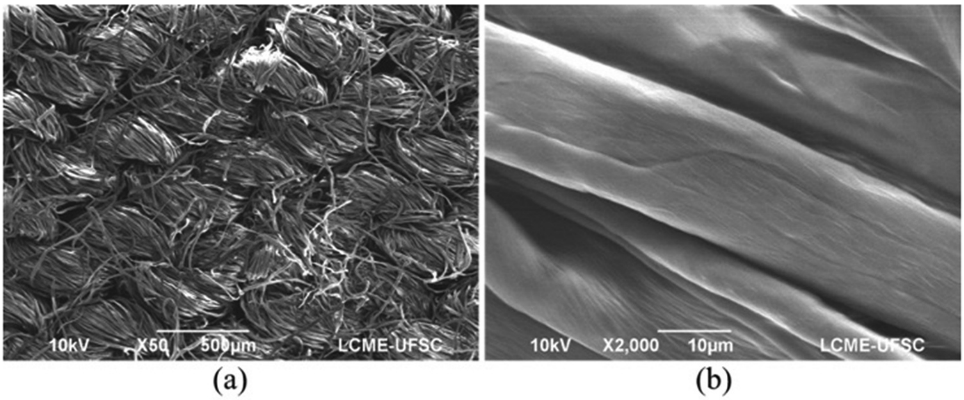

Figure 3. SEM micrographs of CO knitted fabric without functionalization: 50x (a); 2000x (b) magnification.

Scanning electron microscopy

SEM analysis allows the comparison between the samples' surface with and without the functionalized agent. Figure 3a presents the morphological structure of the CO in the jersey knitted fabric, showing visible fibrils outline. Figure 3b shows the convolutions of CO fiber. Both fibrils and convolutions are properties of cotton fiber and those aspects can influence directly in the paste's adhesion on the substrate surface.

The hydrochromic paste incorporation on the CO knitted fabric is observed in Figure 4 in different magnifications, from which is possible to analyze the distribution on the surface and the adhesion of the agent among the fibers. It can be seen that the hydrochromic paste forms a thick layer on the textile surface. Moreover, it is possible to observe that the paste covered completely the cotton knitted fabric surface and are able to penetrate into the fibers. It is noteworthy that cotton is usually provided from spun yarns, which can originate the fibrils. This is important, because it contributes to a longer durability of the applied functionalization, since the hydrochromic materials that are among the fibers and not just superficially tend to present greater fastness.

Drying behavior of hydrochromic material

Wetting tests were performed using water and urine, and the luminosity colorimetric coordinate (L*) was analyzed through the drying curve of the fabric in function of time. Coordinate L* represents clarity (L* = 0 black and L* = 100 white) and it was used because the hydrochromic past color is white when dry.

The drying curves of the liquids in the CO knitted fabric are presented in Figure 5 in the range of 0 to 180 minutes. These data are presented through the average of the values obtained for the L* coordinate. For all samples, the coordinate L* decreases when is wetted and along the time, this value gradually increases. This effect is awaited, because the coordinate L* indicates lightness of the color space. During the drying experiment, the samples with hydrochromic agent come back to standard color (white).

For the water's drying in controlled environment, the temperature and humidity were about 22°C and 40%, respectively, and the time for complete drying was about 30 minutes. For the urine, the drying was more prolonged (45 minutes), in the same conditions of temperature and humidity, and it could have been influenced by the composition of this liquid, which in the most part is constituted by water, nitrogen, phosphorous and potassium (Bethune, Chu, and Ryan 2015). The amount of energy needed for evaporation will vary according to intermolecular forces, density and molar mass of the liquid.

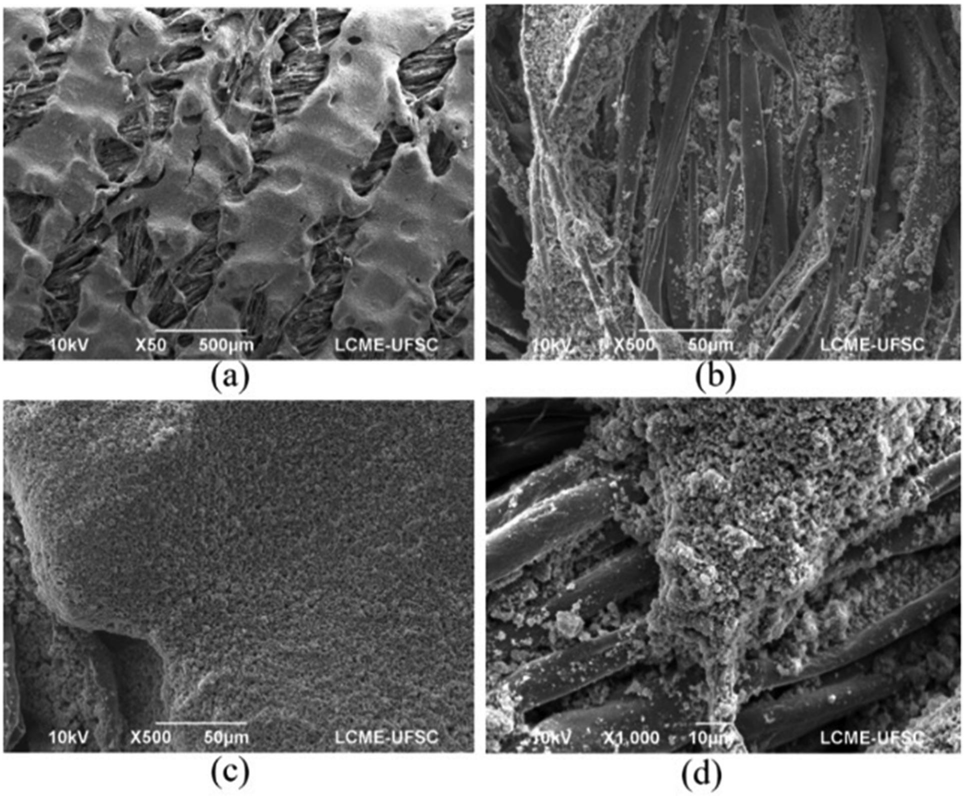

Figure 4. SEM analysis of the CO fabric functionalized with magnification of 50x (a); 500x (b) and (c); and 1000x (d).

In Table 3 it can be verified the coordinate values of L*, a*, b* and ΔE* to the studied samples with different liquids. These values were calculated by making a relation with the dry samples, before they had got in touch with the liquid. The ΔE* values show the change in the color of each sample at 0 time, which means the time that the cotton knitted fabric first got wet, and after 180 minutes. When the sample is wet, as expected, it can be verified a high value for ΔE*, as the color changes from white to the next original knitted fabric color. However, after 180 minutes, the ΔE* value in comparison to the wet sample presents a very low value, which means that the hydrochromic agent comes back to its original color (white). This behavior was observed for all the samples.

To illustrate the reversibility behavior of the hydrochromic paste, images of the CO specimen (Figure 6) can be observed during the study in the different intervals: dry state; in the instant that the knitted fabric got dry after the first drop (time 0); and over the time (until 180 minutes).

As observed in Figure 6, the functionality of the sample after wetting it with water, and dry in ambient temperature lasted about 30 minutes. It is also observed that the sample returned to the original paste color, white, after the evaluation time, without showing any type of staining. Still, the drops' margins remained with a subtle halo effect after drying, which showed to be reversible over time or after wetting again.

According to the literature, most of the situations in which the hydrochromic paste was applied occurred on a surface that already had a colored layer on it, thus making a thin white film that reflects the light waves, such as umbrellas, tablecloths, bathing suits and clothing like the "rainforest" showpiece. In these studies, the textile materials more used were fabrics made from polyester and elastane (Ferrara and Bengisu 2014; Huang 2017). This layer of paste prevents reaching the previous printed image. In the moment that the surface gets wet with a certain fluid, the film used to acquire a raise of the viscosity, creating "fissures" that makes it possible to the light waves to pass, which is known as diffraction effect.

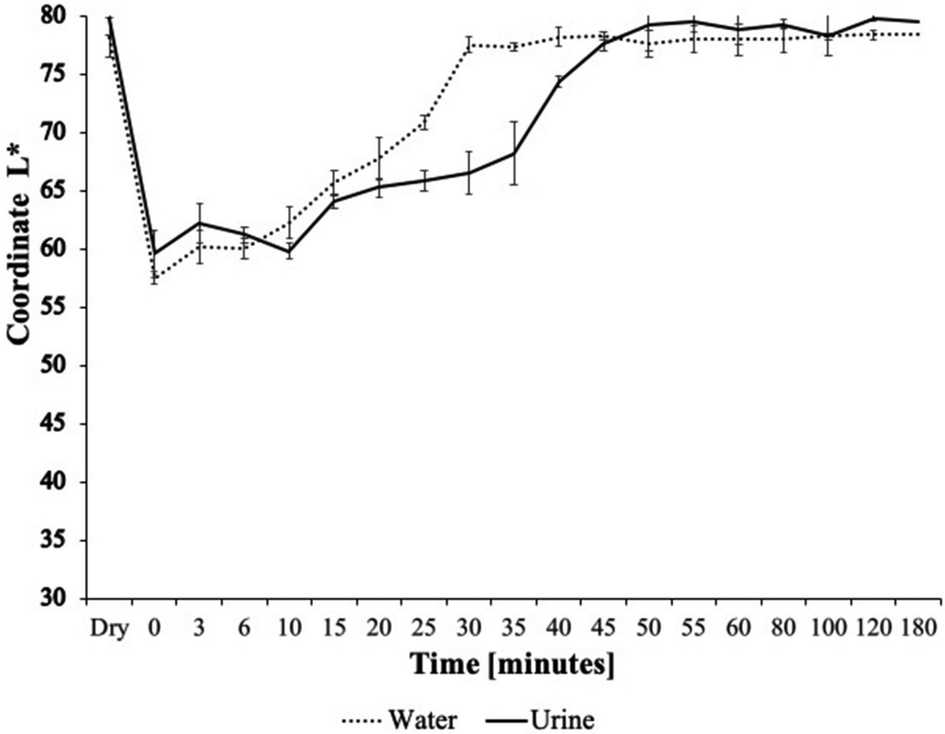

Figure 5. Drying behavior for functionalized CO fabric with different liquids.

Table 3. Colorimetric coordinates obtained during the drying process.

Time [min]	Liquid	Drying	L*	a*	b*	ΔE*
Dry			78.13	5.29	5.87	
0	Water	22.0°C and 40.0%RH	57.52	12.40	10.60	22.31
180			78.47	5.10	5.98	0.40
Dry			79.75	4.42	5.12	
0	Urine	22.0°C and 40.0%RH	59.56	10.98	10.70	21.95
180			79.43	4.36	6.29	1.22

This effect allows the light waves to get filtered, making the colored image underneath the paste to become visible (Ferrara and Bengisu 2014). This mechanism is represented in Figure 7.

Figure 7a represents the dry surface, blocking the visualization of the fabric print underneath the hydrochromic agent, where the light waves reflected are just white. Figure 7b illustrates the wet surface, allowing the visualization of the print underneath the paste, due to the change in the viscosity of the hydrochromic paste, allowing the light diffraction.

Other studies describe that the pigment employed in hydrochromic inks or paints can be a mixture of numerous inorganic compounds, including but not limited to silicates, bisulfates, carbonates, bicarbonates, hydroxides of alkali metals and alkaline earth metals (Kanakkanatt and Kanakkanatt 2013; Klofta et al. 2013; Lee et al. 2014). There can also be certain aluminum compounds, some chlorides, oxides and nitrates. These chemicals are mixed with other solid particles that either increase the stability and improve the adhesive properties of the resulting film, or act as porosigens that allow water to be transmitted throughout the film. Then, the pigment is dispersed in a medium and water is added until the desired consistency is achieved.

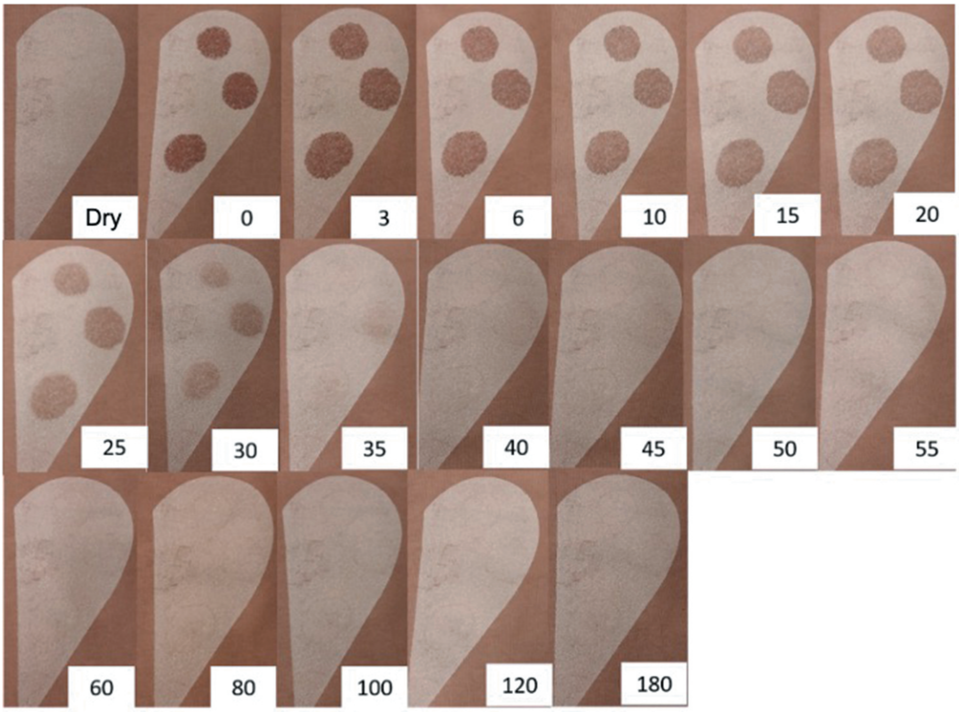

Figure 6. Evaluation of the reversibility behavior using water in different times (minutes).

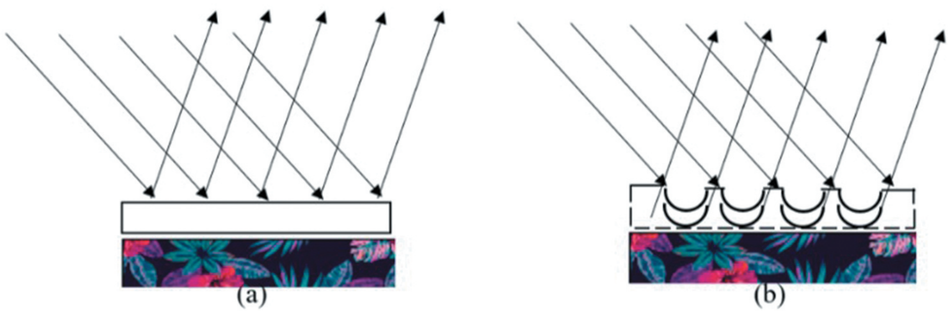

Figure 7. Scheme of changing the hydrochromic paste color: dried surface, where the light waves reflected are white (a); wetted surface, allowing the light diffraction (b).

Washing fastness

To the changing color analysis, specimens after the first, fifth and tenth washing cycles were compared to a standard sample, before washing. From this comparison, the spectrophotometer assigns value according to the variation of the obtained color, rating from 1 to 5, with 5 being 'excellent' and 1 being 'poor' (Table 4). From the obtained results is possible to verify that the samples presented an excellent washing fastness, even after 10 cycles, showing no significant color variation.

Table 4

According to literature, there are few scientific researches that studied the fastness of chromic materials in textile substrates. Cheng, applied photochromic dyes to natural fibers such as wool, cotton and silk, resulting in poor washing fastness (Cheng et al. 2007). In another study, Chowdhury and collaborators verified high values of color difference (ΔE^*) from 2.42 to 28.24 using green and yellow

Table 4. Analysis of color change after different washing cycles.

Functionalized samples	Cycles	L*	a*	b*	ΔE*	Grade
Before washing	0	79.51	4.58	5.21	-	-
01	First	80.11	4.41	4.65	0.14	5
02	Fifth	80.30	4.38	4.53	1.06	4–5
03	Tenth	80.69	4.19	4.66	1.36	4–5

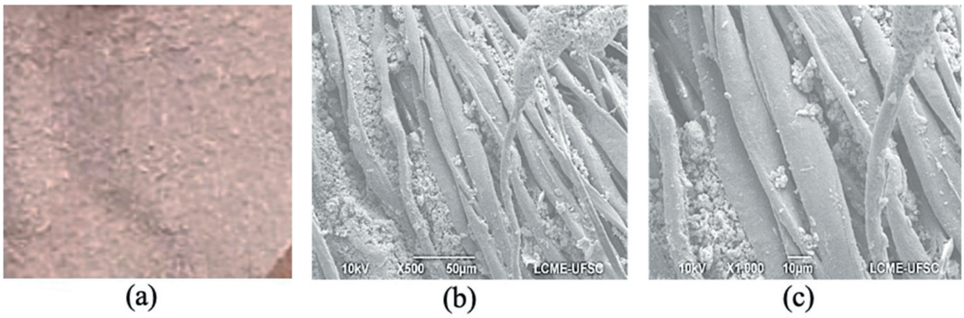

Figure 8. Functionalized knitted fabric after 10 washing cycles: 0x (a); 500x (b); 1000x (c).

thermochromic dyes, respectively, after 4 washing cycles (Chowdhury, Butola, and Joshi 2013). However, textiles screen printed with thermochromic and photochromic dyes have also been referred to as presenting reasonable washing fastness properties (Gulrajani 2013; Mattila 2006). Little and Christie (2011) researched factor affecting technical performance of textile screen printed with two photochromic types (naphthooxazine and naphthopyrans) and concluded that the binder has had a greater influence on the washing fastness results than the colorant types. When applied in natural textile substrate, any article was found studying fastness properties with hydrochromic paste. Thus, the obtained results in this study were, therefore, very satisfying, since the amount of the hydrochromic agent stayed practically unchanged after one (ΔE = 0.14), five (ΔE = 1.06) and ten (ΔE = 1.36) washing cycles performed on the studied cotton fabric, as can be also observed in micrographs in Figure 8.

Rubbing fastness

The results obtained after using the crockmeter to determine the dry and wet rubbing fastness can be visualized in Table 5.

After the friction test, the hydrochromic paste remained in the cotton knitted fabric, with good results, being 4 and 3–4 to dry and wet, respectively.

These results can be related to the interaction of the hydrochromic paste and the fiber surface, considering the morphologic and chemical aspects. The cotton fiber in the longitudinal section presents convolutions, and also the yarn produced with this fiber presents a high quantity of fibrils. Besides that, it is a cellulosic fiber that has many hydroxyl groups, which can contribute in a better interaction and consequently in the preservation of the hydrochromic material after the rubbing test.

Table 5. Analysis of the color changing after friction using the spectrophotometer.

Functionalized samples' test grade		
1	Dry	4
2	Wet	3–4

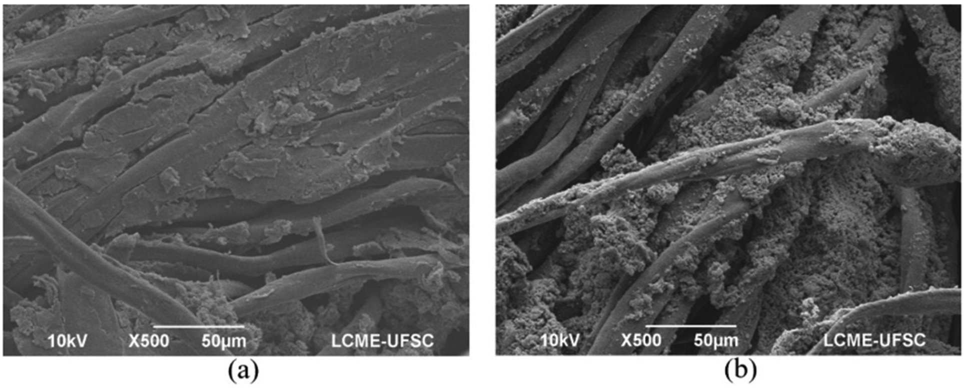

Figure 9. SEM analysis with 500x magnification after 10 cycles in the crockmeter of functionalized CO samples: dry (a); wet (b).

Figure 9 illustrates SEM analysis after the friction tests. Figure 9a,b confirm that after the realized procedure in the crockmeter equipment, most of the paste with hydrochromic agents remained on the surface of the CO knitted fabric.

Conclusion

Based on the result obtained in this study, it was observed that is possible to functionalize cotton knitted fabric with the screen printing method using hydrochromic agent. The hydrochromic paste was well distributed on the fiber surface. The color change occurred adequately for the two types of liquid studied: water and urine. To these liquids, the functionalized samples came back to its original color after drying, which demonstrate the color reversibility of the material. The results of washing and rubbing fastness were very satisfactory, obtaining, respectively, an evaluation with 4–5 and 4 grade in the spectrophotometer, which corroborate with the SEM images obtained. These results indicate that the hydrochromic paste displays good adhesion in the fiber surface and maintains their functionality after subsequent washing and rubbing cycles. Based in this context, it can be verified that a simple cotton knitted fabric can become a textile material that recognizes a certain stimulus and reacts to it, becoming intelligent which increase significantly its potential application.

Disclosure statement

The authors declare no conflicts of interest.

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