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Research Article

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Antibacterial and Multifunctional Polyester Textile Using Plant-Based Cinnamaldehyde

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Abstract

Most of antibacterial agents used for functionalizing textile materials are based on silver nanoparticles and QACs - quaternary ammonium compounds, which are being targeted due to environmental concerns. In the present work, natural cinnamaldehyde was used to produce an antibacterial multifunctional polyester woven fabric using a one-shot diffusion process without the use of any other added chemicals/solvents.

Experiments were based on theoretical assumptions on basis of the total solubility parameter ‘ δ ’ value calculated from Hansen solubility parameters of cinnamaldehyde, which was δ = 24.1 MPa1/2 which is close to that of polyester-PET (poly(ethylene terephthalate): δ =21.9 MPa1/2). Hence using a diffusion process similar to dyeing, cinnamaldehyde may diffuse in the polyester fiber above its glass transition temperature. Experimental work confirmed this hypothesis, and the diffusion process of the yellowish cinnamaldehyde imparted a pale yellowish coloration to the polyester fiber. The color performance and durability of the dyed fabric were analysed and the functionalized fabric was then characterized with respect to ultraviolet (UV) protection ability and antibacterial activity against Staphylococcus aureus and Klebsiella pneumoniae. The cinnamaldehyde functionalized polyester fabric showed pale yellow coloration with excellent UV protection performance. In addition to the pleasant fragrance of the fabric, the functionalized textile showed antibacterial activity. With 10% on weight fabric (owf) of cinnamaldehyde, antibacterial activity of the dyed fabric against Klebsiella pneumoniae (gram-) was excellent. Diffusion method using cinnamaldehyde, allows to obtain a multifunctional woven polyester fabric with fragrance, colour, UV protection and antibacterial properties

Keywords: Cinnamaldehyde; Diffusion; Multifunctional; Textile; Antibacterial; Ultraviolet protection ability

Introduction

Textiles today are mainly made antibacterial using antimicrobial products such as silver nanoparticles [1,2] and QACs - Quaternary ammonium compounds [3], which are being targeted due to environmental concerns. Alternatives such as bio-based antibacterial compounds are renewable and are in most cases, biodegradable. Many bio-based compounds, such as natural dyes [4,5] have already shown to impart several functionalities to textiles in addition to coloration, for example antibacterial or UV protection.

Essential oils are natural bio-based multicomponent products containing active agent(s) responsible for fragrances [6] but also antibacterial activity [7]. In literature, different essential oils have been shown to exhibit antibacterial activity against a broad range of microorganisms [8,9]. Cinnamaldehyde, one of the major constituents of cinnamon bark oil (~ 60-90 %) [10,11] is a phenolic terpenoid classified as GRAS (Generally Recognized as Safe) by the FDA (Food and Drug Administration), with high antibacterial, antifungal, anti-inflammatory and antioxidant activity. It is widely used in cosmetic, food and pharmaceutical industries since it is a natural antimicrobial substance [12]. Different studies analysed the effects of cinnamaldehyde on the bacterial membranes, showing that it permeabilizes the internal membranes of different bacteria, such as *Escherichia coli* and *Staphylococcus aureus*, [13] or *Staphylococcus epidermidis* and *Enterococcus*, altering their structure.

Different polymers have been used as carriers of cinnamaldehyde: cellulose [14], pectin [15], PLA [16], starch [17], proteins [18] or alginates [19]. Most often, casting method such as melt-spinning

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[15], nanoencapsulation (Makwana, 2014), were used to functionalize the polymer films with cinnamaldehyde. Nevertheless, the antimicrobial action depended not only on the active compound and microorganism, but also on the effective release of antimicrobial compounds onto the contaminated product where microbial growth occurs.

Polyester fiber (poly(ethylene terephthalate))-PET, is an important synthetic fiber holding the highest market share (>50%) in textile industry, used for apparel, medical and architectural and food applications. Bio-based PET is under development, too [20]. Functionalization of such fabrics with bio-based renewable antibacterial molecules will help to improve environmental issues. Microcapsules [21], nano capsules such as cyclodextrins [22] and textile chemical finishing's [23] have been used to functionalize textiles with essential oils, to yield textiles with fragrance [24]. The functionality of such textiles is non-lasting as the microcapsules/nano capsules and the finishing's lose all their contents after few washes. Moreover, additional chemicals are required to achieve the textile functionalization.

Cinnamaldehyde which is a small aromatic compound, has the potential to diffuse inside the polyester fiber. Indeed, theoretical assumption shows that the solubility parameter value (calculated using Hansen solubility parameter [25]) of cinnamaldehyde (δ= 24.1 $(J.cm^{-3})^{1/2}$)is close to that of polyester-PET (poly(ethylene terephthalate) (δ = 21.9 $(J.cm^{-3})^{1/2}$). Hence using a diffusion process similar to dyeing, cinnamaldehyde can potentially be transported inside the polyester fiber. Indeed, concerning transport of molecules through PET fibers, Slark et al. [26] specified that this transport is a function of both diffusivity and solubility of the molecule inside the PET fiber. The diffusion process requires thus a temperature above the glass transition temperature of PET fiber, which can induce segmental movement of PET polymer chains in the amorphous regions of a semi-crystalline fiber (Figure 1).

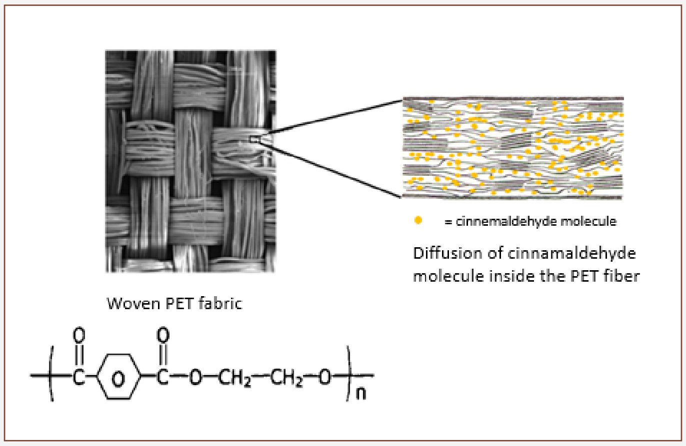


Figure 1: Diffusion of cinnamaldehyde molecules inside the semi-crystalline PET fiber.

Previous works published by our team showed the effectiveness of diffusion method to obtain antibacterial polyester textiles, using curcumin [4] and madder dye [5]. The challenge is to achieve relevant results namely an antibacterial multifunctional PET fabric, using the cinnamaldehyde, without using other chemical agents. Multifunctional PET fabric using cinnamaldehyde via diffusion process has, to our knowledge, not been addressed in literature before this study. The present work is the first of its kind by characterizing not only the color, but also antibacterial activity and UV protection ability.

Cinnamaldehyde was solubilized in aqueous medium before being used for PET fiber functionalization by diffusion method at 130 °C under high pressure conditions (HTHP).

In the first part of the study the color performance and the durability of the dyed fabric were evaluated. The dyed fabric was then characterized with respect to ultraviolet (UV) protection ability and antibacterial activity against *Staphylococcus aureus* and *Klebsiella pneumonia* in particular via quantitative antibacterial absorption method and agar diffusion tests.

Materials and Methods

PET Fabric

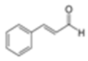
A 100% polyester (PET- poly(ethylene terephtalate) twill woven fabric of density 180 g/m^2 was used (see figure 1). The polyester fabric was cleaned to remove impurities using Soxhlet method with petrol ether, then with ethanol. Then the samples were rinsed in three different water-baths with distilled water, before being dried and ready for use.

Bio-based cinnamaldehyde

Indeed, cinnamaldehyde is the principal component (90%) of cinnamon oil. Pure cis-cinnamaldehyde was purchased from Sigma Aldrich. It is a yellowish liquid.

The chemical formula, the CAS number, the color in aqueous solution, and the Hansen Solubility of cinnamaldehyde, are given in Table 1. The chemical formula, the CAS number, its vapor pressure at 25 °C, and other data were obtained from an open chemistry database (Pubchem web site). Hansen solubility parameters were obtained from the web-based solubility parameter data base which considers the HSP values see Table 1.

Table 1: Cinnamaldehyde properties.

Chemical product	Natural origin	Chemical structure	Color	Hildebrand solubility parameter	Molecular weight	Vapor Pressure
Cinnamaldehyde CAS number: 104-55-22	Cinnamon 		Yellow liquid 	23.4 $MPa^{1/2}$	132 g/mol	2.89 10^{-2} mm Hg at 25°C

The Hildebrand solubility parameter δ_t was calculated using equation (1).

$$\delta_{t^2} = \delta_{d^2} + \delta_{p^2} + \delta_{h^2}$$

Qualitative antibacterial test carried on agar-plate showed that all active agents used alone, showed antibacterial behavior against *Staphyloccoccus aureus*, with the appearance of more or less important inhibition zones (Figure 2a).

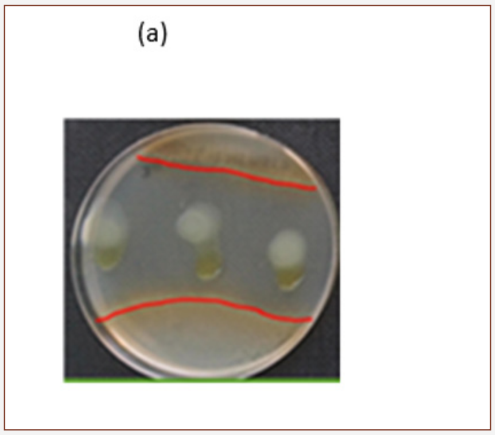


Figure 2: (a) Antibacterial qualitative test of pure cinnamaldehyde performed on agar dish plated with Staphylococcus Aureus bacteria after 24 hours.

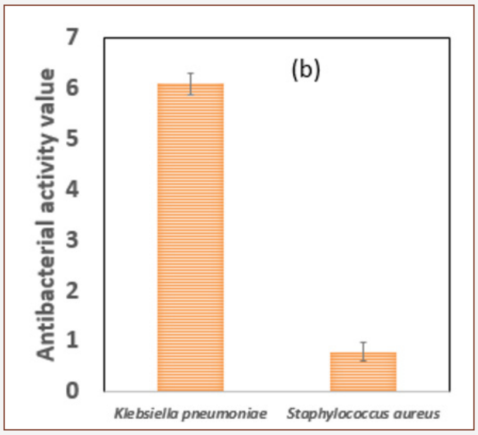


Figure 2: (b) Quantitative antibacterial activity of polyester fabric functionalized with 10% cinnamaldehyde, against Klebsiella pneumoniae and *Staphylococcus aureus.*

Thermal stability analysis

As the diffusion process was carried at high temperature-130 °C, there is possibility of thermal degradation of the cinnamaldehyde [27,28]. The thermal stability was tested using Thermogravimetric Analysis (TGA) which was carried out on a TA 2050 Instrument under atmospheric air. This method has already been used by researchers to assess the thermal stability of vanillin [29]. For each experiment, a sample of approximately 10 mg was used. A heating rate of 10°C min^{-1} was applied, and the temperature was raised from 20 to 700 °C. Figure 3 shows the TGA thermogram. Maximum stability temperature, determined at 5% loss of initial mass was 136 °C, indicating that cinnamaldehyde has quite good stability in the temperature range (130 °C) used for the diffusion process.

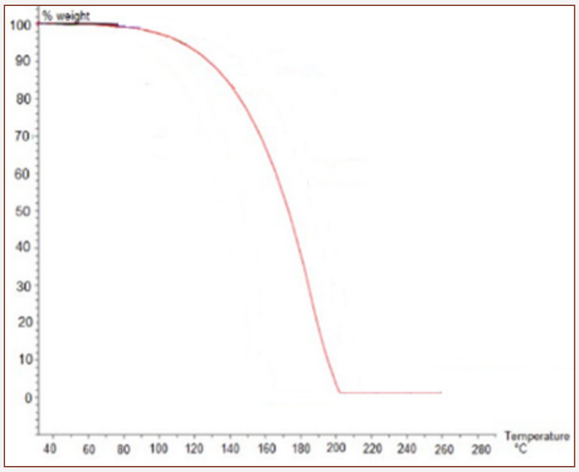


Figure 3: TGA Thermograph of cinnamaldehyde.

Functionalization using diffusion method by exhaustion procedure

The procedures were performed in accordance with the general dyeing method using the diffusion method in a HTHP (High Pressure and High Temperature/Beaker Dyeing Machines, at 130 °C) [30] using 1 % ethanol-water solution and a liquor ratio of 1:20. The samples, weighing 5g, were treated in 200ml beakers (Labomat machine) with 1, 3, 5 and 10% o.w.f cinnamaldehyde.

To summarize 5g of polyester fabric were placed in different aqueous bath containing respectively

of 3.78 x 10^{-3} M, 1.13 x 10^{-2} M, 1.89 x 10^{-2} M, and 3.78 x 10^{-2} M cinnamaldehyde.

The temperature of the exhaustion bath was then gradually raised (about 2 °C/min) to 130 °C and was kept at this temperature for about 45min. The bath was then cooled to 60 °C; then the fabric was squeezed, rinsed thoroughly with hot water and air dried. No surfactant was used in addition in the diffusion method bath.

The diffusion induced notable pale-yellow coloration of the fabric which was characterized spectrophotometric analysis.

Spectrophotometric analysis of fabric samples

Reflectance of the functionalized samples "R" was measured with a Konica-Minolta CM3610A spectrophotometer for wavelength-λ varying from 360nm to 700nm. Relative color strengths "K/S" were automatically calculated from the reflectance values by the software using the Kubelka-Munk equation (2) [4]. K/S value is directly related to the color yield of the fabrics:

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

where, K refers to coefficient of absorption, S is the coefficient of scatter, and R is fractional reflectance.

UV protection

The UV protection capacity of the samples was evaluated according to EN 13758 (determination of the sun protection coefficient). This European Standard specifies a method for the determination of the erythemally weighted UV radiation transmittance of standard conditioned apparel fabrics to assess their solar UV protective properties. The Ultraviolet Protection Factor (UPF) is the expression of the level of protection as attained by the method described in EN 13758. The UPF of a textile material is determined from the total spectral transmittance T (λ) as follows:

$$UPF = \frac{\sum_{\lambda=290}^{\lambda=400} E(\lambda)\varepsilon(\lambda)\Delta\lambda}{\sum_{\lambda=290}^{\lambda=400} E(\lambda)T(\lambda)\varepsilon(\lambda)\Delta\lambda} \quad (3)$$

E (λ): the solar irradiance;

ε (λ) : the erythema action spectrum;

Δλ : the wavelength interval of the measurements;

T(λ): the spectral transmittance at wavelength λ.

The total spectral transmittance is measured by irradiating the sample with polychromatic UV radiation and collecting the total (diffuse and direct) transmitted radiation.

Antibacterial tests

Two different antibacterial tests were carried out. The agar diffusion test (ISO20645:2004) and the absorption method (ISO20743) were performed using two different bacteria: a gram positive (*Staphyloccoccus aureus* - ATCC 6538) and a gram-negative bacterium (*Klebsiella pneumoniae* -ATCC4352) [31].

Agar diffusion test (**ISO20645:2004**): The level of antibacterial activity is assessed by examining the extent of bacterial growth in the contact zone between the agar and the fabric specimen and, if present, the extent of the inhibition zone around the specimen. 10 (± 1) mL of nutritive agar medium were poured on Petri dishes. Inoculums of bacteria (0.5 ± 0.1) mL with a bacterial culture of 1-5 x10^8 CFU/mL were then poured on the agar media. Circular textiles sample of 10cm^2 were then placed on the surface. To maintain good contact, if necessary, a sterilized inox ring was placed on the surface of the textile sample to guarantee good contact between the fabric and the agar. Immediately after placing textile samples on the agar, petri dishes were placed in incubation for 24h at 37 (± 1) °C. The inhibition zone was measured. The halo is the zone free from bacteria near the sample edges. Contact zone under the tested textile sample was analyzed visually to check whether bacteria growth occurred or not.

Absorption method (ISO20743:2003): This test is used to quantitatively measure the antimicrobial activity of textile samples. The treated and untreated PET samples were cut in small pieces (of 0.4g) and placed in a glass vial. Six test samples in individual vials plus six separate vials of control samples (untreated samples) constitute one test. Each sample and each control sample were inoculated with 200µL of bacterial suspension adjusted to 1-3x10^5 CFU/mL.

Directly after inoculation (0 contact time), an extraction of the bacteria present on three of the six samples of each series was performed, and bacterial count was determined using the plate count method. Then, other vials were incubated at 37 °C for 24 hours, after which the bacterial count was performed. For each trial the number of "viable" active bacteria was calculated and then expressed in "log".

The growth value and the activity values were then computed. We proceed as follows:

$$F = C_t - C_0 \quad (4)$$

Where, F is the growth value on the control sample, Ct is the common logarithm of the arithmetic average of the numbers of bacteria obtained from three test control specimens after 24-hour incubation. C_0 is the common logarithm of the arithmetic average of the numbers of bacteria obtained from three control specimens immediately after inoculation (0 contact time).

The test is judged to be effective, when the growth value is ≥1 and when the difference in extremes for the three controls immediately after inoculation as well as after incubation is ≤ log10.

The calculation of the activity values is obtained according to the following formula:

$$A = (C_t - C_0) - (T_t - T_0) = F - G \quad (5)$$

Where, A is the antibacterial activity value, F is the growth value on the control fabric (F= C_t–C_0), G is the growth value on the antibacterial treated sample (G= T_t–T_0), Tt is the common logarithm of the arithmetic average of the numbers of bacteria obtained from three antibacterial testing specimens after 24 hour incubation, and finally T_0 is the common logarithm of the arithmetic average of the numbers of bacteria obtained from three antibacterial testing specimens immediately after inoculation.

The obtained value for the antibacterial activity (A) can be exploited by the following way:

- If A>3, the antibacterial activity is strong
- If 2<A<3, a significant antibacterial activity is detected
- If A<2, the antibacterial activity is insufficient

Experimental Results

Visual and spectral analysis of the functionalized polyester fabrics

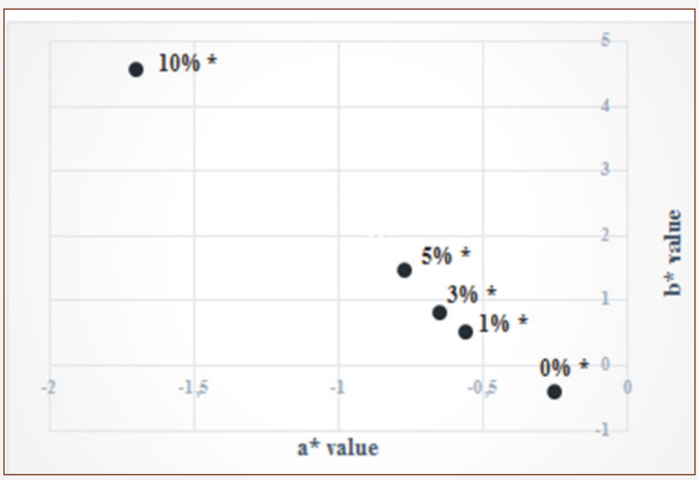


Figure 4: CIE Color space coordinates of the dyed polyester fabrics at varying owf % of cinnamaldehyde.

figure 4 represents the a* and b* coordinates in the CIELab color space, of the samples functionalized with 1, 3, 5 and 10% o.w.f cinnamaldehyde. These were obtained using spectrophotometric analysis.

The a* axis represents the green–red component, with negative values for the green component.

The b* axis represents the blue–yellow component, with positive values for the yellow component.

Cinnamaldehyde induces only a very pale-yellow coloration of the fabric. The higher the concentration of cinnamaldehyde used, the higher is the color depth seen by the naked eye.

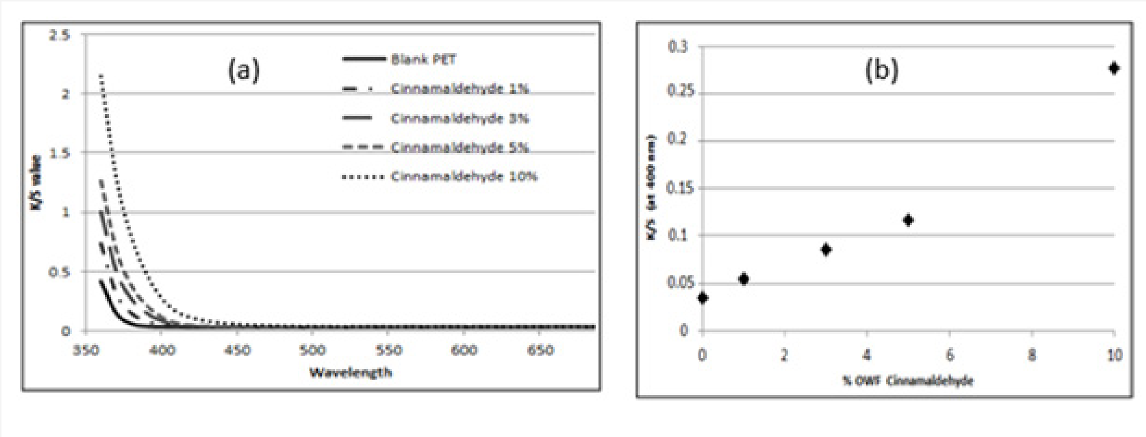


Figure 5: K/S (at varying wavelength) (a); and K/S (at λ = 400 nm) of treated PET with varying owf % of cinnamaldehyde(b).

K/S values appear to be higher for the functionalized PET compared to the untreated blank PET, in the wavelength region from 360nm to 400nm, which is the UV A region (Figure 5a).

Figure 5b shows the K/S (at λ= 400 nm-yellow) values of each functionalized PET using the different concentrations of cinnamaldehyde. Higher K/S values of the functionalized PET fabric with increased cinnamaldehyde (owf %) confirmed an increase uptake of the cinnamaldehyde as its concentration in the water bath increases.

The spectral curves of PET fabric functionalized with cinnamaldehyde were compared with spectral curves of cinnamaldehyde in aqueous medium (water) and in an organic solvent (toluene) obtained using a UV/visible spectrophotometer (Figure 6).The spectral curve of the functionalized fabric did not seem to be correlated to that of cinnamaldehyde in the organic or aqueous medium. Interaction between PET macromolecules bearing aromatic ring and the cinnamaldehyde would perhaps be responsible for the shift in the K/S spectral curve in the cinnamaldehyde functionalized PET fabric.

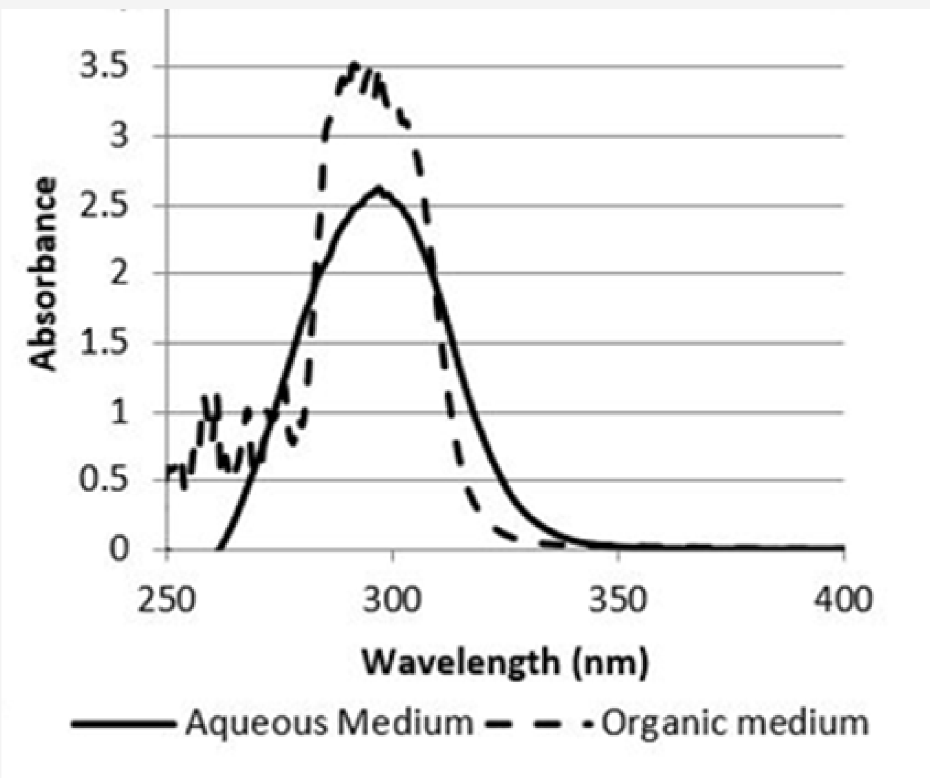


Figure 6: UV/Visible spectral curves of 2×10^{-4} mol/L cinnamaldehyde in aqueous (water) and organic medium (toluene).

Durability of the coloration

The functionalized samples were subjected to washing in ethanol and water baths respectively. The textile was rinsed with ethanol during three minutes at 30 °C to remove all particles physiosorbed at the textile surface. Washing with water was carried during 3 hours at 30 °C. The yellow coloration of the cinnamaldehyde functionalized polyester fabrics present a good durability to these wet treatments.

K/S (at λ= 400nm) values were measured by spectrophotometry. Only a small decrease in K/S values was noticed after rinsing 3 minutes with ethanol or 3 hours in water (Figure 7), indicating that the majority of cinnamaldehyde molecules were bound inside the PET fiber. The little amount of cinnamaldehyde that was physiosorbed to the textile fiber surface was washed away during the rinsing protocol

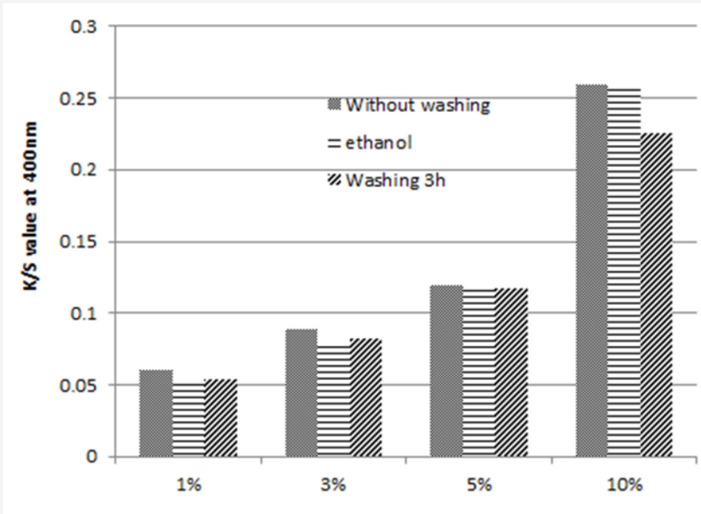


Figure 7:K/S at 400 nm, of cinnamaldehyde functionalized polyester before and after different wet treatments.

UV Protection

Ultraviolet radiation ranges between 100 nm and 400nm and is subdivided into UV-C (100-280nm) stopped in the stratosphere, UV-B (280-315nm) and UV-A (315nm and 400nm) [32]. It is known that over-exposure to UV-A and UV-B can cause harmful effects such as skin cancers or even the PET textile ageing [33,34], though the UV protection ability of textiles is also influenced by factors such as the fiber type, the fabric structure and its color.

In general, a UPF factor of 50 is considered as excellent protection of skin against sun by a textile material. Figure 6 shows the UPF factors measured and expressed in log values.

The presence of cinnamaldehyde improved the UV protective effect of the PET fabric. The blank undyed PET fabric showed a UPF value of 1620 (Table 2). This value is very high and may be explained by the presence of UV absorbers added during spinning of PET fiber. Nevertheless, when the fabric was functionalized with cinnamaldehyde (10%), the UV protection increased more than 30 times. This can be explained by the fact that cinnamaldehyde exhibits absorption (at 300nm) mainly in the UV region, as shown in Figure 8, when it is dissolved both in aqueous (water) solution or an organic medium (toluene). Specifically, UPF values of 14400 nm and 48000 were found for 5 % owf and 10 % owf cinnamaldehyde (Table 3) which also lead to K/S=1.2 and 2.2 respectively compared to original K/S=0.5 for the blank PET fabric, at a wavelength of 360nm. These results encourage the use of the cinnamaldehyde so as to simultaneously impart color and UV protective effect onto textiles.

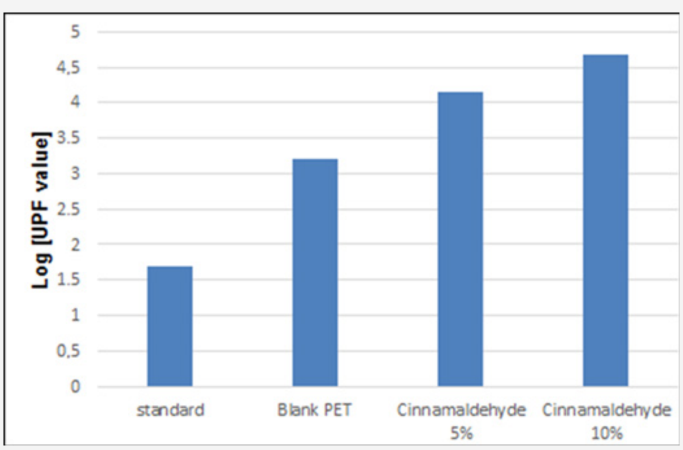


Figure 8: UPF values (expressed in logarithm) of polyester fabric without and with cinnamaldehyde (« standard » corresponds to textile with UPF of 50, giving optimal sun protection for the human skin.

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Table 2: Hansen solubility parameters.

Solubility parameters in $MPa^{1/2}$	δ_d	δ_p	δ_h	δ_t
Cinnamaldehyde	19.4	12.4	6.2	23.4
PET	18.2	7.3	7.9	21.4

Table 3: UPF values.

	UPF	Log (UPF)
Standard	50	1.6
Blank PET	1600	3.2
Dyed fabric Cinnamaldehyde 5%	14400	4.2
Dyed fabric Cinnamaldehyde 10%	47800	4.7

Antibacterial testsResults of the agar diffusion test (ISO20645:2004): None of the PET functionalized with cinnamaldehyde showed an inhibition zone against *Klebsiella pneumoniae* and *Staphylococcus aureus* bacteria. However, beneath the textile samples, no bacterial colony could be observed. We can conclude that bacteria which have a direct contact with the functionalized textile samples die. However, since no inhibition zone was seen, it can be concluded that the bio-active agent did not diffuse from the textile into the aqueous medium of the agar-plate away from the textile surface.

Results of the quantitative Antibacterial absorption method (ISO20743 -2003): The sample treated with 10% of cinnamaldehyde was tested to evaluate its efficiency against two different bacteria. This graph (Figure 2b) shows that functionalized sample had very good antibacterial properties against *Klebsiella pneumoniae* (A=6.1). However, the antibacterial activity against *S. Aureus* was low (A=0.8).

Discussion and Conclusion

It is known from literature that cinnamaldehyde is antibacterial, and as it is a bio-based renewable resource, it can potentially lead to reduced environmental impact and can serve as an interesting alternative to standard antibacterial or antifungal agents used to produce antimicrobial textiles. Table 4 summarizes the toxicity concerns obtained from ECHA web site for cinnamaldehyde compared to silver and QAC's used for antimicrobial textiles (European Chemicals Agency, http://echa.europa.eu/, 07/2018) [35]. Silver and QAC substance are very toxic and hazardous to aquatic life with long term (chronic) and short term (acute) effect, and certain QAC such as dodecyltrimethyl ammonium chloride induce additionally very high human toxicity.

Table 4: Toxicity issues of cinnamaldehyde compared to silver and QAC's.

				
	Corrosive to metals	Acute toxicity	Hazardous to the aquatic environment:	Very high toxicity
	Skin corrosion	Skin and eye irritation	- Acute Hazard	
	Serious eye damage	Skin sensitization	- Chronic Hazard	
Cinnamaldehyde		X		
Silver			X	
QAC's Dodecyltrimethyl ammonium chloride	X	X	X	X
QAC's Alkylbenzyldimethyl ammonium chloride	X	X	X	

Bio-based cinnamaldehyde seem to be nonhazardous for the environment but poses problems relative to handling of products, causing skin and eye irritation. Thus, precautions must be taken while handling cinnamaldehyde.

To functionalize polyester fabric with cinnamaldehyde, diffusion experiments were carried based on theoretical assumptions on basis of the solubility parameter value of cinnamaldehyde (δ = 21.9 $MPa^{1/2}$) which is close to that of polyester δ =24.1 $MPa^{1/2}$). Hence using a diffusion process similar to dyeing, cinnamaldehyde molecules were able to diffuse in the polyester fiber at high temperature (above glass transition temperature of PET). This resulted in a pale yellowish colored multifunctional polyester fabric, with excellent UV protection ability, and good antibacterial activity, using a one-shot diffusion process and without the use of additional chemical agents. The functionalized fabric also had a low nice fragrance, which was maintained over more than one year, as confirmed by a panel of voluntary students who did the odor evaluation .

Past studies showed that inclusion of cinnamaldehyde in cyclodextrins [36]or in hydrophobic polymer films by melt spinning [37] reduces the effectiveness of release of cinnamaldehyde. This may explain the low and extended fragrance of the cinnamaldehyde functionalized polyester fabric used in this study, since PET is also hydrophobic. Absence of inhibition zone during the qualitative agar-diffusion with the functionalized textile, and the reduced antibacterial activity against *S. Aureus*, may be also explained by the reduced release of cinnamaldehyde entrapped in between macromolecular PET chains, which reduces greatly their volatility, and their rate of diffusion in the gaseous form to the environment, at ambient temperature.

While allergenic issues are discussed for cinnamaldehyde in contact with skin [38], the antibacterial, multifunctional textile can find applications in fields such as food absorption pads or in outside architecture. Its excellent UV protection would potentially make it a good candidate against UV ageing of the PET fiber [39,40].

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Conflict of Interest

Authors declare no conflict of interest.

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