Journal of Fluorescence (2019) 29:703–710
https://doi.org/10.1007/s10895-019-02384-2

ORIGINAL ARTICLE

Development of Illuminant Glow-in-the-Dark Cotton Fabric Coated by Luminescent Composite with Antimicrobial Activity and Ultraviolet Protection

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Received: 21 February 2019 / Accepted: 17 April 2019 / Published online: 30 April 2019

Abstract

The main objective of technical protective clothing is to enhance people safety at work, which may save their life or keep them healthy away against some hazards. We developed a warning cotton fabric with a traffic safety warning photoluminescence character that continues emitting light for a long period of time after the removal of the illuminant source. Rare earth-doped strontium aluminate was dispersed in an aqueous medium of a polyacrylic-based binder to give a cross-linkable photoluminescent formula to be applied onto cotton substrate employing spray-coat approach. To introduce a transparent photoluminescent film, the Rare earth pigment must be fully dispersed to prevent aggregation. The long-persistent photoluminescent layer was deposited on cotton surface employing different concentrations of the rare earth pigment phosphor. The excitation wavelength maximum band of the spray-coated film on cotton fabric was found to occur at 365 nm, while the emission was recorded at 515 nm. Yellowish-green emissive color was monitored by CIE color data under the ultraviolet excitation source. The spray-coated fabric was characterized by wavelength dispersive X-ray fluorescence (WD-XRF), phosphorescence and excitation spectra, elements mapping, scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX). The comfort measurements were studied by exploring both of fabric stiffness and air-permeability. Furthermore, the spray-coated textile substrates displayed good fastness properties and a reversible luminescent glow in the dark.

Keywords Lanthanide · Spray-coat · Cotton · Luminescence · Glow in the dark · Protective fabric · Cotton

Introduction

Photoluminescent products have been employed in different applications, such as safety guidance marks to direct public to buildings exits to help evacuation to safe sites during fire incidents [1, 2]. Long-persistent photoluminescent phosphors, such as lanthanide-doped strontium aluminate ($SrAl_2O_4$:Eu^{2+}, Dy^{3+}), are characterized by their ability on long time glow in the dark due to their excitation and ability to store light energy when exposed to an external light source [3–8]. The rare earth doped strontium aluminum oxide is characterized by good chemical, physical, thermal and photostability, as well as excellent reversibility, nontoxicity, extended emission time (about 10 h), brightness, recyclability, high quantum efficiency, non-radioactivity, and durability [9–14].

Textile industries and high performance technical textiles in particular are as diverse as their explored products [15–21]. The main objective of protective textiles is to protect people from hazards particularly at work, such as textiles with flame-retardant, water-repellent and antimicrobial performance [22–34]. Reflective ribbons usually used for traffic warning textile manufacturing to introduce fluorescent clothing. However, those warning fabrics are usually harsh, stiff, and less permeable to air. The comfortability of warning clothing

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is a necessary quality criterion, while maintaining their colorfastness and mechanical performance, photo- and thermal properties [35–42]. Spray-coat has been introduced as a facile, non-contact, and low cost technique to coat objects at high rate, very low agglomeration, while using the lowest amount of aerosol [43–51]. An aqueous phosphor-adhesive spray-coat technology can be considered a simple process to create traffic warning clothing with a glow in the dark performance.

To the best of our knowledge, the facile preparation of traffic warning clothing from long-persistent photoluminescent cotton fabrics via spray-coat of a binder with strontium aluminum oxide phosphor in a water environment has not been reported. We studied the breathability, flexibility and comfortablility of the coated cotton fabrics. This long-lasting luminescent cotton fabric is to present safety and protection to human as it has the added advantage of being readily visible to the naked-eye and can be located in the dark. Morphology, surface and elemental composition, as well as colorfastness and luminescence properties of the spray-coated cotton substrates were explored. Both bending length and air-permeability can be enhanced by reducing the thickness of the luminescent film while maintaining its glow in the dark efficiency. Low film thickness will increase both air-permeability and flexibility of the spray-coated film.

Experimental Details

Materials and Chemicals

Cotton fabric (100%) was obtained from Misr Spinning and Weaving El-Mahalla El-Kobra Company, Egypt. The cotton fabric was 96 × 52 plain weave (110.27 g m^{-2} varieties); tensile strength warp 55 kg f; warp and fill yarns were 34^S and 32^S respectively; bending length warp 3 cm; bending length weft 2 cm; DCRA weft 60°; DCRA warp 55°; CRA warp 56°, and WCRA weft 54°. The fabric was further desized, scoured and bleached depending on previously reported literature procedure [9]. A binder additive was obtained from Dystar, Egypt. The lanthanide-doped strontium aluminum oxide ($SrAl_2O_4$:Eu^{2+},Dy^{3+}) was synthesized depending on literature method [4].

Production of Traffic Warning Cotton Fabrics

The spray-coat photoluminescent formula solution was prepared according to previously described work [4]. A mixture of diammonium phosphate (0.05 wt%), ammonium hydroxide (1 wt%), pigment phosphor and binder (10 wt%) was well dispersed in distilled water using magnetic stirrer. Different photoluminescent formula solutions were prepared at different concentrations of the lanthanide-doped strontium aluminate phosphor (1 wt% (Cot-1), 2 wt% (Cot-2), 4 wt% (Cot-4), 6 wt% (Cot-6), 8 wt% (Cot-8) and 10 wt% (Cot-10)). The prepared solutions were spray-coated on cotton substrates and samples were then left to be air-dried for 30 min followed by curing at 150 °C for 5 min. The spray-coated cotton substrates were rinsed at 50 °C, subjected to rinsing by cold water and finally dried under atmospheric conditions.

Apparatus and Methods

Scanning electron microscope images were obtained on Czech Republic Quanta FEG-250. Elelemtal composition was explored by energy-dispersive X-ray (TEAM-EDX) and wavelength-dispersive X-ray fluorescence spectrometer (Axios advanced, Sequential WDXRF). Emission and excitation spectra were assessed by JASCO spectrofluorometer FP-8300. The colorfastness performance of the sprayed cotton was explored according to ISO standards. The coloration measurements were (including color coordinates (L^*, a^*, b^*) and color strength (*K/S*) recorded on Chroma meter Konica Minolta CR-400 with a D65 illuminant, 2° standard observer function and 8 mm diameter illumination area.

Antimicrobial Activity

The antibacterial/antifungal activity of both pristine and spray-coated cotton fabrics were studied against two gram negative and gram positive bacteria including *E. coli* and *S. aureus* respectively, and *Candida albican* fungus. The antimicrobial examination was applied in a quantitative manner via standard methodology (AATCC 100–1999) [4].

Ultraviolet Protection

The ultraviolet shielding was explored by measuring the ultraviolet protection factor (UPF) of the spray-coated samples. The assessment of ultraviolet protection factor of sun shield can be defined in AS/NZS 4399:1996 employing ultraviolet protection factor computation of UV/Vis spectrophotometer (AATCC 183:2010-UVA Transmittance) [9].

Results and Discussion

Characterization and Morphology

It was indicated by both surface morphology and elemental content from scanning electron microscope image and energy dispersive X-ray diagram respectively, that the treated cotton was effectively spray-coated with irregular clusters of nano/microsized particles from the strontium aluminum oxide pigment doped with dysprosium and europium as shown in Fig. 1a. The distribution of phosphor particles size on the cotton fabric surface occurred from 180 nm to 20 μm, while,

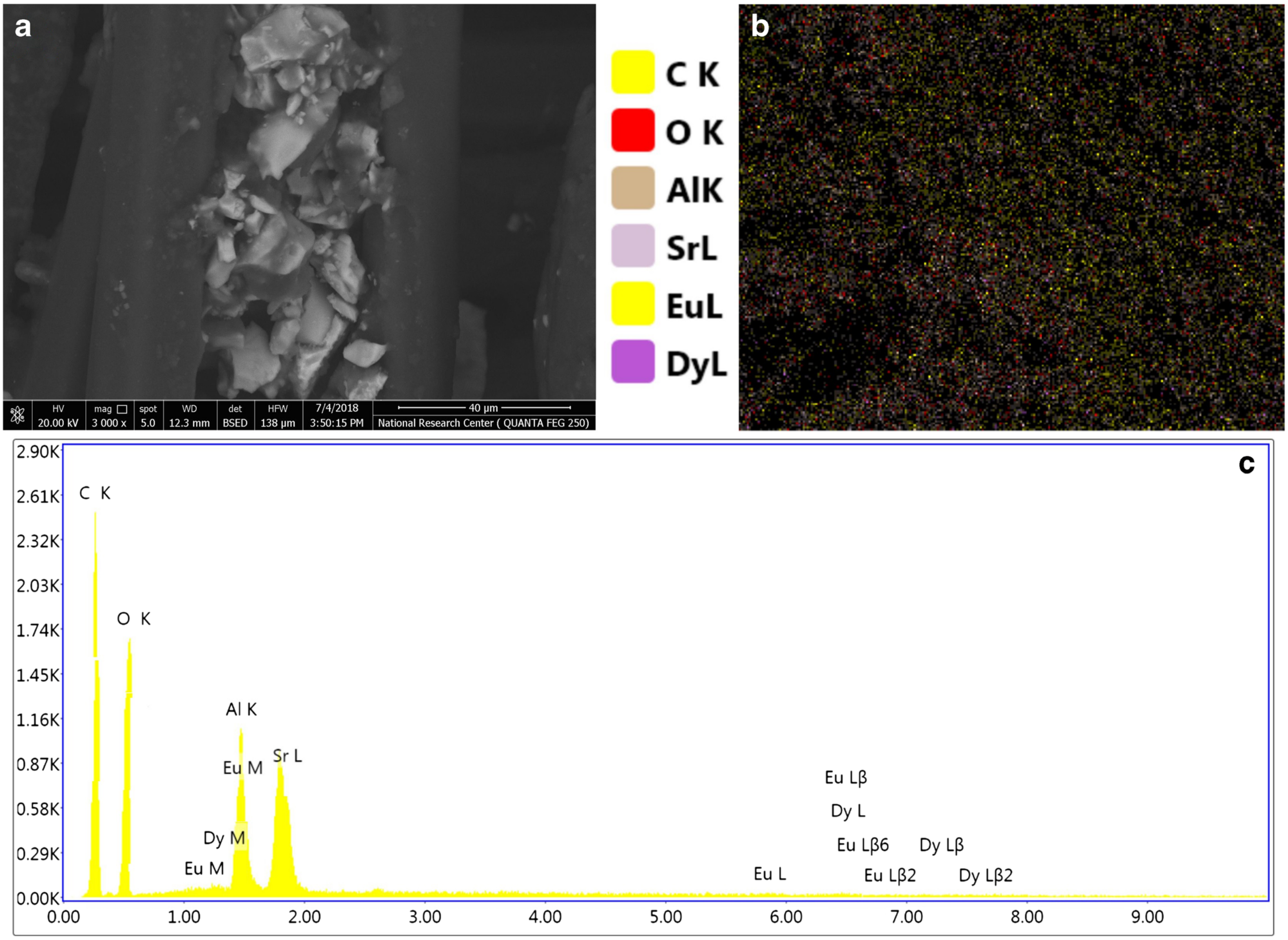

Fig. 1 Scanning electron microscopic image (**a**), Elements mapping (**b**) and energy dispersive X-ray diagram (**c**) of the spray-coated cotton substrate (Cot-6)

the major phosphor particles size average was between 2 μm. It was found that this nano/microparticle phosphor had a tendency to agglomerate leading to a slight heterogeneous distribution of the pigment phosphor particles on the cotton substrate surface, which can be ascribed to the character of the physical interaction between phosphor molecules each other's or between pigment molecules and cotton fibers.

The elements content in weight percent at three locations of the spray-coated cotton (Cot-6) are shown in Table 1. The elements content at three different scanned locations were nearly the same confirming the homogenous distribution of pigment phosphor on cotton surface. Furthermore, the major elemental composition of pigment nano/microparticles monitored by EDX spectrum was found to follow the elemental molar ratio employed in phosphor synthesis. The elemental content can be studied by different spectroscopic methods, such as energy dispersive X-ray (EDX; Fig. 1c) and wavelength dispersive X-ray fluorescence (WDXRF; Table 2). However, the WDXRF generally detects a few elements of interest with detection limit equal to or higher than 10 mg kg^{-1} [6], while the EDX method introduces an excellent detection limit for all elements with a small error [52–55]. The WDXRF was performed for sample Cot-6 (surface area was 12.75 cm^2) to affirm the availability of Sr and Al elements in the oxide form obeying their molar ratio during the preparation process. Nonetheless, both Dy and Eu elements were not detected by WDXRF because of their very low concentration. The elements mapping proved the uniform dispersion of pigment on the fabric surface (Fig. 1b).

Table 1 The elements content in weight percent at three locations on the fabric (Cot-6) surface

Location on fabric surface	C (Wt%)	O (Wt%)	Sr (Wt%)	Al (Wt%)	Dy (Wt%)	Eu (Wt%)
1	52.92	32.69	6.12	6.26	1.03	0.98
2	52.09	32.63	6.80	6.29	1.17	1.02
3	52.45	32.87	6.24	6.12	1.13	1.19

Table 2 Elements analysis by WDXRF

Element (oxide form)	wt%
Al_2O_3	59.76
CaO	1.83
SrO	33.98
Na_2O	1.56
K_2O	1.15
P_2O_5	0.31
Cl	1.41

Luminescence Properties

The lanthanide-doped strontium aluminum oxide pigment was incorporated onto the surface of cotton fabrics via spray-coating using polyacrylic-based binder as an organic trapping layer keeping the pigment on cotton surface via coordinative bond [4]. The thermal treatment was applied to crosslink the binder polymer chains to afford three dimensional layer attached to cotton surface while trapping the strontium aluminate pigment inside this film. All spray-coated fabrics showed reversible phosphorescence upon UV excitation. However, directly after UV excitation, the phosphorescent emission bands were only detected for cotton fabrics Cot-4, Cot-6, Cot-8 and Cot-10; whilst Cot-1 and Cot-2 demonstrated only weak emission bands. Therefore, only cotton substrates treated with pigment concentration higher than 4 wt% can develop long-lasting luminescence. After irradiation and removal of UV source, the maximum phosphorescence band ($\lambda_{em} = 526$ nm) was monitored. The long-persistent phosphorescence effect was proved by monitoring phosphorescence emission intensity decreasing over a period of time. The emission spectra showed a broad and strong emission band in the visible region of electromagnetic radiation as shown in Fig. 2. The emission intensity was found to decrease slowly with time in the dark. The spray-coated cotton substrates exhibited green-yellow phosphorescence.

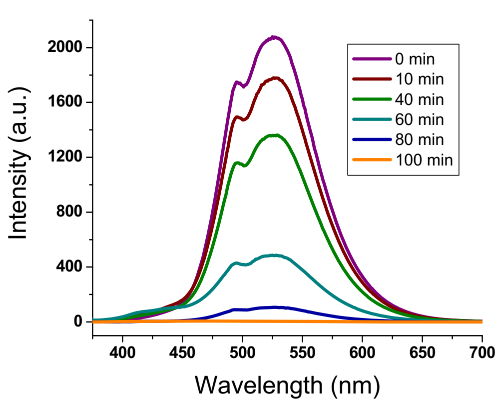

Fig. 2 Phosphorescence emission spectra of the spray-coated cotton substrate (Cot-6) at different periods of time in the dark after being excited under ultraviolet light ($\lambda = 365$ nm)

The long-lasting (or long-persistence) luminescence (known as afterglow effect) is the continuation of light emission (for a period of time that may reach several minutes or several hours) from the treated cotton substrate after the removal of the irradiation light source (such as UV light source) [56–58]. This is different than fluorescence as the emission only occurs under the irradiation source and disappears after the removal of this irradiation source [59, 60]. In case of normal phosphorescence, it is a type of fluorescence which does not instantly re-emit the radiation it absorbed [61, 62]. However, it continues re-emit light for a very short period of time that could be a fraction of second after the removal of the irradiation source. Additionally, the photoluminescent europium and dysprosium-doped strontium aluminate pigment composed of two components: photonic traps and crystals of aggregated elements. Increasing the pigment concentration leads to increasing both crystals and photonic traps to result in higher long-lasting photoluminescent efficiency. The crystals are photoluminescent upon excitation or charging by an external light source. On the other hand, the photonic traps store those light photons leading to extended phosphorescence time [56–58]. Therefore, the crystals, such as strontium aluminum oxide, stay excited after the removal of the external light source and keep releasing light which is supported by photonic traps, such as divalent europium and trivalent dysprosium.

The lifetime was calculated to be 58,920.76 milliseconds. The maximum wavelength of the phosphorescent bands for the treated samples was monitored at 526 nm, which was nearly similar to that of the solid strontium aluminum oxide pigment at 520 nm [6]. Both of lifetime and decay curves of Cot-6 exhibited nonlinear correlation consisting of two regions, initially high and fast part and then slow decay as shown in Fig. 3. The major role of lanthanide doping was to function as trapping ions to extend the photons discharge time. Thus, the luminescent cotton fabrics kept glowing in the dark after the illumination light source was removed. To generate a transparent luminescent film on cotton fabric, the phosphor must be well dispersed to avoid agglomeration. Depending on changes in reflection spectra, both of color strength (*K/S*) and coloration coordinates (L^*, a^*, b^*) were applied to the photoluminescent character of the applied on cotton fabric (Table 3). All treated cotton substrates had a white color same as original untreated cotton fabric. In absence of UV, approximately no differences were monitored in color strength when increasing phosphor concentration from Cot-1 to Cot-10 to affirm the film transparency. After UV illumination, a substantial increment in color strength was observed to indicate an alteration from the less faded *K/S* to higher value with increasing pigment content. On the other hand, a negligible difference in color strength was detected at pigment content more than Cot-6 indicating that the best color data were attained

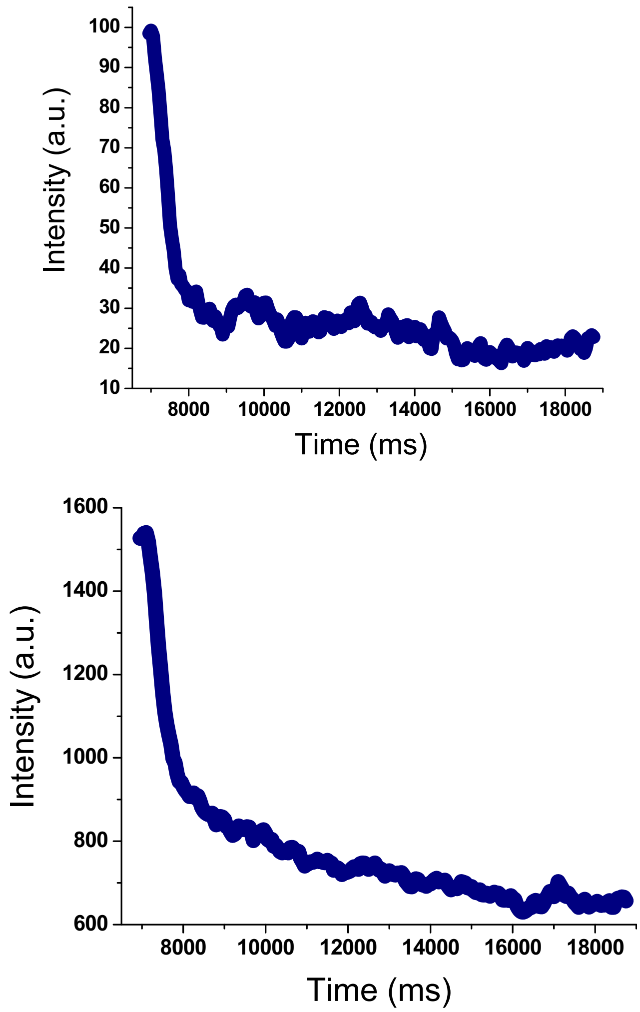

Fig. 3 Phosphorescence of Cot-6 demonstrating decay time (*top*) and lifetime (*bottom*)

for Cot-6. The color data for untreated pristine cotton was white with L^* (96.81), a^* (−0.04) and b^* (0.66). The treated cotton fabrics showed only negligible differences in L^*, a^* and b^* with increasing pigment concentration and in absence of UV. On the other hand, Under UV irradiation, −ve a^* was increased and + ve b^* was reduced due to

Table 3 Color data of coated cotton fabrics reported at different phosphor content before and after exposure to ultraviolet light source

Phosphor content (wt%)		Cot-1	Cot-2	Cot-4	Cot-6	Cot-8	Cot-10
K/S	Before	1.06	1.28	1.37	1.72	1.98	2.17
	After	0.15	0.25	0.78	0.84	0.95	1.26
L^*	Before	93.57	91.86	90.81	89.36	89.82	88.25
	After	92.47	91.21	88.14	87.22	87.38	86.67
a^*	Before	- 1.56	- 1.82	- 2.02	- 1.78	- 2.07	- 2.21
	After	- 1.21	- 1.13	- 1.28	- 1.51	- 1.17	- 1.21
b^*	Before	17.89	17.13	17.29	16.77	16.61	16.32
	After	18.73	18.23	18.71	18.24	19.85	19.20

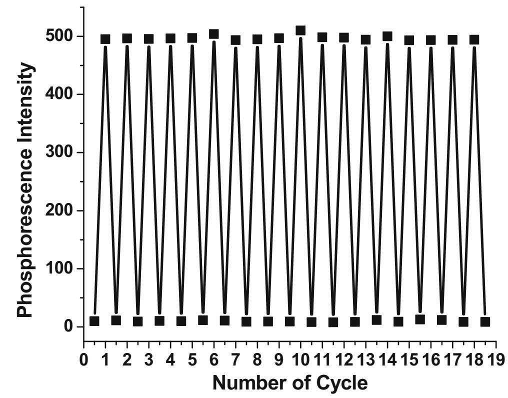

Fig. 4 Differences in emission intensity at 526 nm (Cot-6)

color change from white to green-yellow. After thirty minutes in the dark, a more increment in negative a^* and a more decrement in positive b^* was occurred.

Durability, Photostability and Reversibility

The durability of the treated fabrics to light was explored by studying the changes in UV-Vis absorption and emission intensity to show acceptable durable character and photostability. This proved the excellent bonding strength of the binder/pigment layer. The reversibility was tested under mutual ultraviolet excite/darken cycles (Fig. 4). The cotton fabric was subjected to ultraviolet and emission intensity was recorded then kept in the dark for 100 min. This cycle was repeated several times, while the emission was measured each time to indicate excellent reversibility.

The spray-coat technique is significant because it introduces a smooth film with low rough surface, while keeping the coated substrate flexibility and breathability. We determined the bend length and air permeability of the treated samples to show no differences in air permeability. However, only a slightly increment in fabric rigidity was monitored with increasing the strontium aluminate concentration (Table 4). Furthermore, the surface free energy (~41 mN m^{-1}) of the luminescent film was determined on Kruss-DSA30S meter

Table 4 Stiffness and air permeability properties

Cotton Sample	Bend length (cm)		Air permeability ($cm^3/cm^2/s^1$)
	Weft	Wrap	
Blank	2.23	2.66	53.93
Cot-1	2.95	3.09	47.25
Cot-2	3.26	3.45	47.42
Cot-4	3.58	3.86	46.17
Cot-6	3.92	3.96	45.54
Cot-8	3.98	4.17	43.37
Cot-10	4.57	4.92	43.38

Table 5 Colorfastness properties of the treated cotton fabrics

Cotton Sample	Wash		Perspiration				Rubbing		Light
	Alt.*	St.*	Acidic		Basic		Dry	Wet	
			Alt.*	St.*	Alt.*	St.*			
Cot-1	4-5	4–5	4–5	4–5	4–5	4–5	3–4	3	6
Cot-2	4	4	4	4–5	4	4	3–4	3–4	6–7
Cot-4	4–5	4–5	4–5	4	4–5	4–5	3	3	6
Cot-6	4–5	4–5	4–5	4–5	4–5	4–5	3–4	3	6–7
Cot-8	4	4	4–5	4–5	4	4	4	3	6
Cot-10	4–5	4	4–5	4–5	4	4	3–4	3	6

* *Alt., alteration in color; St., staining on cotton*

affirming the fabric high wettability. Additionally, the spray-coated cotton fabrics showed soft-handle to touch and high stability to washing, light, perspiration and rubbing (Table 5).

Antimicrobial and UV-Shielding Properties

Both antibacterial and antifungal testing of spray-coated cotton substrates against two gram negative and gram positive bacteria including *E. coli* and *S. aureus* respectively, and *Candida albican* fungus, were carried out via applying the plate agar count methodology. The antibacterial/antifungal reduction percentage caused by the spray-coated cotton fabrics were tabulated in Table 6. The blank cotton fabric displayed no inhibition influence on the reduction percentage, while spray-coated substrates showed significant resistance to the above mentioned pathogens. This microbial resistance ranged from poor to excellent with increasing the concentration of strontium aluminate phosphor.

Ultraviolet protection factor (UPF) is commonly applied to assess the UV shielding effect of a certain substrate. The ultraviolet protection factor of the spray-coated fabrics was recorded and summarized in Table 7. The UPF of the spray-coated cotton substrates were ranging between good to excellent, which can be ascribed to the high ability of the lanthanide-doped strontium aluminum oxide to absorb ultraviolet light due to the electronic structure of such phosphor making it appropriate for ultraviolet shielding.

Table 6 Antimicrobial activity of the spray-coated fabrics

Cotton sample	Antibacterial (Reduction %)		Antifungal (Reduction %)
	*Escherichia coli (*Gram –ve)	*Staphylococcus aureus* (Gram + ve)	*Candida albican*
Cot-1	34 ± 1.2	31 ± 1.2	0.00
Cot-2	43 ± 1.2	40 ± 1.1	10 ± 1.0
Cot-4	55 ± 1.3	51 ± 1.4	12 ± 1.0
Cot-6	69 ± 1.1	64 ± 1.1	11 ± 1.0
Cot-8	78 ± 1.1	73 ± 1.0	10 ± 1.0
Cot-10	83 ± 1.2	81 ± 1.3	11 ± 1.0

Table 7 Ultraviolet shielding of of the spray-coated fabrics

Cotton sample	UPF
Cot-1	1256
Cot-2	1461
Cot-4	1610
Cot-6	1896
Cot-8	2936
Cot-10	3129

Conclusion

We developed a simple spray-coat approach toward the production of long-lasting luminescent functional film on cotton substrates and characterized by good comforting properties including appearance, softness, air permeability, flexibility and colorfastness. The main target was to introduce intelligent photoswitchable illuminating fabrics for traffic safety warning purposes with high durability. The spraying procedure on cotton was performed by an aqueous medium of the binder admixed with lanthanide-doped strontium aluminate phosphor. There were no major variations detected in the air permeability or mechanical properties of the sprayed cotton samples which proved the fabric breathability and flexibility.

Acknowledgements Technical support from National Research Centre, Cairo, Egypt; is gratefully acknowledged. This work was funded by the Deanship of Scientific Research at Princess Nourah bint Abdulrahman University through the Research Groups Program Grant no. (RGP-1440-0002).

Compliance with Ethical Standards

Competing Interests The authors declare that there is no conflict of interest.

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