Long-persistent night glow fabric for traffic safety warning protective clothing

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Abstract

In the present paper, we focused on developing novel smart cotton fabric exhibiting warning photoluminescence glow-in-the-dark character that continue emitting light for a long time period. This long-persistent night glow cotton fabric brought added value for people safety improvement. We present a cotton fabric printed with a glow-in-the-dark transparent film. Strontium aluminum oxide pigment doped with rare earth elements was added to a mixture of a thickener, a polyacrylate-based binder and distilled water to introduce phosphor-loaded viscous paste which can then be applied directly onto cotton substrate using screen-printing technology followed by thermofixation. The transparency of the printed film can be achieved easily by the well dispersion of pigment via physical immobilization within the printing paste to avoid the pigment aggregation. Results indicated that the optimum absorption band of the printed cotton substrates was at 365nm and emission band was monitored at 516nm. A long-lasting photoluminescent homogenous film was deposited on cotton fabric relying on the lanthanide-doped strontium aluminate concentration present in the phosphor-loaded printing paste. This printed luminescent film developed a green-yellow emission color as demonstrated by CIE lab color coordinates under UV excitation source. Both decay-time and life-time was explored. The energy-dispersive X-ray spectra (EDAX), excitation and emission photoluminescence spectroscopy, wavelength-dispersive X-ray fluorescence (WD-XRF), elemental mapping and scanning electron microscopy (SEM) were explored. The comfort features of printed cotton substrates were explored by investigating their air permeability and stiffness. The printed cotton samples demonstrated a reversible and fast photoluminescent response without fatigue during UV irradiation. The fastness characteristics including rubbing, light, perspiration, and washing were explored.

Keywords: Cotton fabric; $SrAl_2O_4:Eu^{2+}$, $Dy3^{+}$; Screen-print; Long-lasting phosphorescence; Warning fabric.

1. Introduction

Technical protective textiles are usually designed for non-aesthetic purposes, but to improve workers safety [1-3]. Optical warning textiles demonstrating illuminant color shades and is not printed by plastic materials are much desired for public service and traffic employees, as well as workers of road construction. These warning textiles were usually coated by fluorescent dyes in order to build the fabric easy to see and to function as protective clothing for the wearer from the risk of probable injury arising from traffic incident [4-7]. This kind of protective garments, which is visible to the naked-eye, is consequently well-known as warning protective textiles. However, there are many disadvantages became apparent from these coated warning garments. One of the major disadvantages is their low air-permeability and high stiffness due to the high thickness caused by the coated film. This resulted in decreased skinny respiration and consequently to difficulties in the work performance. Moreover, low air-permeability of clothing may lead to skin diseases and irritations [8, 9]. Another drawback of the traditional protective textile finishing for this particular warning clothing is the unsatisfactory colorfastness of the warning color against washing, light, perspiration or rubbing. Even subsequent to a short period of time, the fabric efficiency deceased due to the low colorfastness to light. The cloth accordingly became virtually ineffective as warning clothing due to this low stability to light [10-17].

Strontium aluminate doped with lanthanides is very beneficial to impart glow-in-the-dark photoluminescent property to textiles while keeping their original physicochemical properties, such as whiteness, handle, appearance, and touch [18, 19]. Long-lasing photoluminescent pigments have been proved to be an excellent long-persistent phosphors (>10 hours) owing to their non-toxicity, non-radioactivity, high brightness, ability to be recycled, as well as their photo, physical and chemical stability. Thus, the immobilization of such long-lasting luminescent phosphor in a binder/thickener printing matrix to be screen-printed on a cotton fabric surface can be considered as a promising approach for the production of long-lasting photoluminescent comfortable cotton fabric with an improvement of illuminant color-exchange character, high durability, good handle, pigment stability and improved fastness properties [20-27].

Both stiffness and air-permeability can be improved without incurring additional disadvantages, by using a low thickness luminescent film with low stiffness to be coated on cotton fabric. Cotton fabrics are particularly characterized by high breathability compared to other textile fibers [28-41]. Compared to conventionally coated warning clothing, reducing the thickness of the printed illuminant film will consequently reduce the stiffness and increase air-permeability of the printed film while maintaining the printed film warning efficiency. To the best of our knowledge, the easy assembly of glow-in-the-dark cotton fabrics via screen-printing of a thickener/binder aqueous composite and europium and dysprosium-doped strontium aluminum oxide pigment has not been reported yet. We developed long-persistent photolumiescent traffic warning cotton fabrics which acted as excellent hosts for the photoluminescent phosphor. Such long-persistent photoluminescent cotton fabrics are capable to glow-in-the-dark and consequently visible to the naked-eye at night darkness. Morphological colorfastness, luminescence and mechanical properties, and elemental content of the screen-printed cotton fabric were explored.

2. Experimental details

2.1. Materials and reagents

Cotton fabric (100%; plain weave, weft 30 yarn cm^{-1}, warp 36 yarn cm^{-1}, 150 g m^{-2}, thickness 0.4 mm, micronaire 3.88 μg $inch^{-1}$) were used. The cotton substrates were desized, scoured, and bleached according to previously reported literature procedures [12]. Thickener Alcoprint-PTP and polyacrylate binder were obtained from Dystar. Chemicals including H_3BO_3, Al_2O_3, $SrCO_3$, Eu_2O_3 and Dy_2O_3 were obtained from Sinopharm-Chemical-Reagent Co. Ltd, China. The rare earth-doped strontium aluminum oxide pigment ($SrAl_2O_4$: Eu^{2+}, Dy^{3+}) was synthesized according to previously described literature method [6]. After preparation, the sintered pigment was milled and sieved to produce the phosphor at low particle size (10-30 μm).

2.2. Preparation of glow-in-the-dark cotton substrates

The printing composite was prepared by direct immobilization and full dispersion of NH_4OH (0.25 wt%), $(NH_4)_2HPO_4$ (0.25 wt%) and polyacrylate binder (5 wt%) were admixed in distilled water (92 wt%). The mixture was stirred for 15 minutes using a magnetic stirrer. Then, the synthetic thickener (2.5 wt%) was added and the paste was mixed by vigorous stirring employing a high shear-mixer for 30 minutes. The luminescent pigment (2, 4, 8, 12 and 15 wt%; replacing the water content) was added while stirring by a high shear-mixer for 1 hour. The composites prepared above were printed on cotton substrates employing the flat screen-printing, left on a flat dry clean surface to dry at ambient conditions, and s

samples were washed with hot water at 45°C and finally dried.

2.3. Characterization and measurements

Decay-time, life-time, excitation and emission spectra were explored on a JASCO spectrofluorometer FP-8300. Scanning electron microscope with a Quanta FEG-250 (Czech Republic) connected to energy-dispersive X-ray analysis (TEAM-EDAX) was applied to study both morphology and elemental content. The elemental analysis of the printed fabrics were further investigated by wavelength-dispersive X-ray fluorescence (Axios advanced, Sequential WD-XRF) Spectrometer.

2.4. Colorimetric and colorfastness measurements

The colors of the treated cotton samples were recorded before and after ultraviolet excitation employing a Chroma meter Konica Minolta CR-400 with D65 illuminant, 2° standard observer function and 8 mm diameter illumination area. The color data were explored by studying CIE L^*, a^*, and b^* coordinates. The color strength (*K/S*) was studied by the high reflectance method applying Kobelka Munk equation. The colorfastness properties were examined according to ISO standards; ISO 105-X12(1987) for rubbing; ISO 105-C02(1989) for washing, ISO 105-B02(1988) for light, and ISO 105-E04(1989) for perspiration.

3. Results and Discussion

3.1. Characterization and morphology measurements

The elemental composition and morphological properties of the cotton fabric surface screen printed by ***europium*** and ***dysprosium doped strontium aluminum oxide*** were explored as shown in **Figure 1**. The scanning electron microscope images of the treated samples demonstrated successful spray-coating of cotton surface with clusters of strontium aluminate pigment showing nano/microstructures of irregular shapes. The size distribution of the produced nano/microstructured phosphor on cotton fabric surface was in the range from ~350nm to ~25μm. The major size average of the strontium aluminate phosphor was about ~9μm. Such nano/microstructural pigment tended to agglomerate, and accordingly dispersed slightly heterogeneous onto the cotton fabric, which could be assigned to the type of chemical or physical interactions of the phosphor molecules, with the cotton fabric. Additionally, the SEM images demonstrated no physical variations happened to the surface of the cotton fabric upon spray-coating.

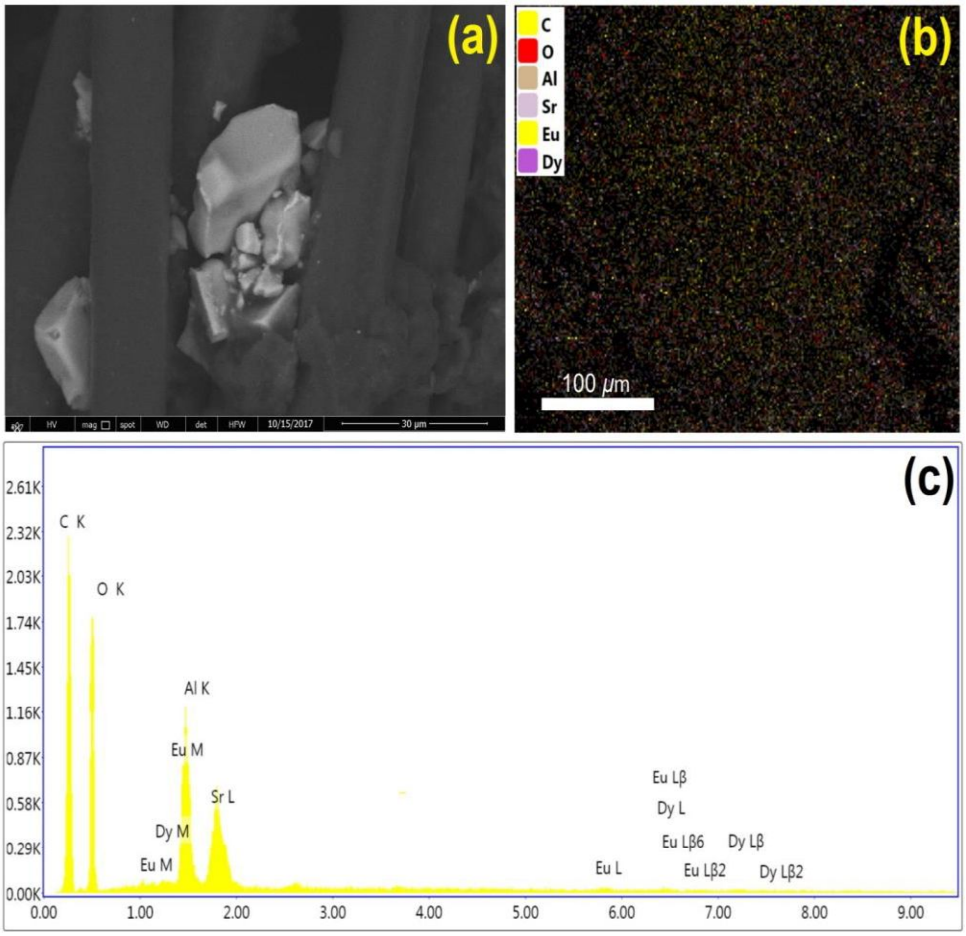

Figure 1: SEM image (a), elemental mapping (b) and EDAX diagram (C) of the screen printed cotton fabric (8 wt%)

Table 1: The elemental content (weight %) at two different spots of the treated cotton (8 wt%)

Samples	C	O	Al	Sr	Eu	Dy
Spot 1	46.90	30.89	4.44	4.09	0.71	0.70
Spot 2	47.05	31.07	5.49	5.64	0.78	0.74

The elemental composition of the screen-printed cotton was also explored by EDAX spectroscopy via exploring the elemental weight percent at two different spots on the surface of the printed fabric as shown in **Table 1**. The elemental contents picked at those two scanned spots were closely the same. This confirmed the homogenous immobilization of $SrAl_2O_4:Eu^{2+},Dy3^{+}$ on cotton surface. The wavelength-dispersive X-ray fluorescence (WD-XRF) was also applied to explore the elemental composition. The energy-dispersive X-ray spectroscopy (EDAX) is a good method to detect all elements with a very low error. However, the standards that applied WD-XRF commonly introduce detection limits higher than 10 mg/kg [6], therefore it analyzed only some elements of interest. the WD-XRF of sample 8 wt% with a surface area of 15.29 cm^2 proved the presence of strontium and aluminum elements as summarized in Table **2**. The other elements including europium and dysprosium were not detected by WD-XRF owing to their very low concentration. The major chemical composition of the different elemental content monitored by WD-XRF and EDAX was found to obey the molar ratio employed in the preparation of strontium aluminum oxide doped with europium and dysprosium. The mapping image of the key elements proved the uniform distribution of the pigment particles on the surface of the screen printed cotton (**Figure 1b**).

Table 2: Elemental analysis by WD-XRF (8 wt%)

Constituent	(wt%) as sample 100%
SiO_2	2.12
Al_2O_3	63.76
MgO	1.67
CaO	1.46
SrO	27.02
Na_2O	1.62
K_2O	0.89
Cl	1.46

3.2. Colorimetric measurements

The three dimensional color coordinates L^*, a^*, b^*, as well as color strength *K/S* were summarized in **Table 3.** The lightness/darkness was represented by L^*, green/red was represented by a^*, and blue/yellow represented by b^*. The treated cotton fabrics possessed white color same as the blank untreated cotton (L^*= 95.83, a^*= 0.02, b^*= -1.05). No major changes were monitored in *K/S* when increasing the phosphor concentration of the printed cotton samples up to 15 wt%. This proved the transparent appearance of the printed glow-in-the-dark film due to the low pigment content. After exposing to UV, a significant increment in *K/S* was monitored to indicate a transformation from the less faded *K/S* to higher magnitudes when increasing pigment concentration. Nonetheless, negligible differences were observed in *K/S* for pigment concentration higher than 8 wt%. Additionally, *K/S* of UV excited cotton substrates were less than the corresponding un-excited ones. Thus, the best color data were explored at 8 wt%, at which no considerable changes were monitored in *K/S* at concentrations above 8 wt%. In absence of UV, all printed cotton substrates exhibited different values of L^*, a^* and b^* when raising the pigment concentration. Under UV, the negative a^* was found to increase with a decrease in the positive b^*, which caused a color change from white to green-yellow. During 90 minutes. in the dark, a higher increment in the negative a^* was monitored with higher reduction in the positive b^* proving an alteration in the shade from white to blue.

Table 3: Coloration data of the screen-printed substrates at several concentrations of $SrAl_2O_4:Eu^{2+}$, $Dy3^+$ before and directly after excitation by UV.

Phosphor wt%	**L^***		**a^***		**b^***		***K/S***	
	Before	***After***	***Before***	***After***	***Before***	***After***	***Before***	***After***
2	**92.28**	**89.12**	**- 1.89**	**- 1.41**	**17.25**	**18.32**	**1.15**	**0.38**
4	**92.63**	**88.19**	**- 1.91**	**- 1.30**	**16.78**	**17.09**	**1.33**	**0.76**
8	**91.08**	**88.24**	**- 1.86**	**- 1.41**	**17.33**	**18.67**	**1.19**	**1.15**
12	**91.27**	**89.13**	**- 1.77**	**- 1.42**	**17.65**	**18.55**	**1.14**	**0.92**
15	**90.53**	**87.44**	**- 1.96**	**- 1.39**	**16.24**	**17.74**	**1.07**	**0.98**

3.3. Photoluminescence properties

The screen printing stock paste was formulated using three key components including the strontium aluminate pigment at different concentrations (2, 4, 8, 12 and 15 wt%), thickener and binder. The thickener acted as film filler, while the polyacrylate binder acted as a trapping layer for the pigment on cotton fabric surface. All printed samples displayed a reversible luminescent character. However, the samples with pigment concentration above 4 wt% displayed slow reversibility. The normalized UV-Vis absorption and phosphorescence spectra of the screen-printed samples (8 wt%; emission wavelength 516nm) were studied. Both of life-time and decay-time were explored between 0-9000 ms, displaying a life-time at 72160.31 ms (error 0.00549217). The printed luminescent layer displayed long-lasting phosphorescence same as the solid phosphor powder. The emission peak of the printed cotton was at 516nm, which was just below that of the solid phosphor powder at

519nm [9]. The afterglow peak intensity profile of the printed cotton was initially high as the life-time of printed substrate 8 wt% displayed nonlinear relation as a function of time. It was demonstrated that the afterglow decay-time profile composed of two parts, firstly fast and then slows decaying progress. The dopants Dy^{3+} and Eu^{2+} are rare earth ions are generally applied as energy traps to extend the photons exhaustion time. Thus, the long-lasting effect depended mainly on the densities of the traps and the depth of the trapped photons.

3.4. Durability, comfortability, colorfastness and mechanical properties

The major reason of applying screen printing was to establish a smooth luminescent with low film thickness, low rough surface, while keeping the cotton fabric’s flexibility and air-permeability. Shirley Stiffness Tester was employed to report the bending length ***of the treated fabrics***(**Table4**).Mainly,the screen printing procedure did not affect on air-permeability, but a slight decrease in flexibility of the treated fabrics was detected in warp/weft with raising the phosphor concentration. The screen printed film possessed a surface free energy (39 mN m^{-1}) as determined on KRÜSS-DSA30S tensiometer. This proved that the treated cotton fabric surface has a high wettability. The durable performance of the treated cotton to light, perspiration, washing and rubbing was recorded. No changes were detected for the printed cotton after wash. The screen-printed substrates showed softness to touch. The color depth and colorfastness properties including photostability (colorfastness against light) were very good as displayed in **Table 5**.

Table 4: Bending length and air-permeability

Pigment wt%	Bend length (cm)		Air-permeability ($cm^3/cm^2/s^1$)
	Weft	*Wrap*	
Blank	2.53	2.89	52.58
2	2.87	3.25	49.02
4	3.10	3.43	48.26
8	3.38	3.47	48.62
12	3.53	3.68	47.73
15	3.61	3.75	46.38

Table 5: Colorfastness properties

Pigment wt%	**Wash**		**Perspiration**				**Rubbing**		**Light**
	Alt.*	**St.***	**Acidic**		**Basic**		**Dry**	**Wet**	
			Alt.*	**St.***	**Alt.***	**St.***			
2	**4-5**	**4-5**	**4-5**	**4-5**	**4-5**	**4-5**	**3-4**	**3**	**6-7**
4	**4**	**4**	**4**	**4-5**	**4**	**4**	**3-4**	**3-4**	**6-7**
8	**4-5**	**4**	**4-5**	**4-5**	**4**	**4**	**3-4**	**3**	**6**
12	**4-5**	**4-5**	**4-5**	**4-5**	**4-5**	**4-5**	**3-4**	**3**	**6**
15	**4**	**4**	**4-5**	**4-5**	**4**	**4**	**4**	**3**	**6**

Alt. = alteration in color; St. = staining on cotton.

The reversibility was explored by reporting UV/Vis excitation after performing the standard processing of rubbing, light, wash and perspiration (Figure **2**). The reversibility was evaluated under UV light/dark mutual steps (excitation at 365nm; emission at 516nm). The printed cotton substrates displayed excellent reversibility and fatigue resistance under repeated glow-in-the-dark reversible cycles. The printed cotton sample was exposed to UV and then left in the dark for 90 minutes to discharge light returning to its original status. After each UV excite/fad cycle, the emission intensity was reported and compared to the value reported after the original UV irradiation to demonstrate high reversibility without fatigue.

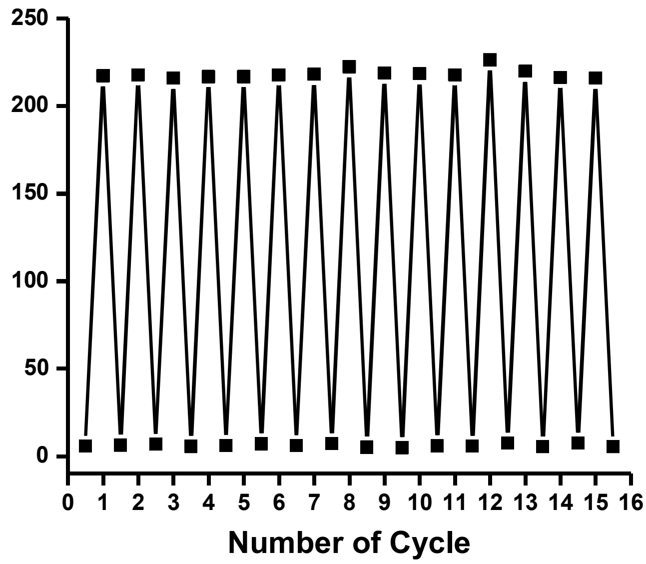

Figure 2: Variations in the emission intensity ratio at 516nm (8 wt%) after each UV excite/fad cycle (exc. 365nm).

4. Conclusion

Strontium aluminum oxide activated with europium and dysprosium was advantageous to produce long-lasting glow-in-the-dark photoluminescent cotton fabric while maintaining its original appearance, comfortability and air-permeability, as well as soft-handle. Simple screen printing method was developed to introduce smart warning cotton fabric for traffic safety with high durability and good colorfastness properties. The screen printing on cotton fabric was made employing an aqueous paste of lanthanide-doped strontium aluminate phosphor, a thickener and an adhesive polyacrylate binder. Only negligible variations were monitored in the printed fabric air-permeability and stiffness proving that the fabric maintained its breathability and flexibility. Different spectroscopic techniques were employed to characterize the printed cotton substrates including coloration measurements, decay-time and life-time, energy-dispersive X-ray, elemental mapping, wavelength-dispersive X-ray fluorescence, scanning electron microscopy. The excellent reversibility and fatigue resistance, and photostability of the printed cotton fabrics made them promising for traffic warning protective clothing.

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