Simple Preparation of Multifunctional Luminescent Textile for Smart Packaging

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ABSTRACT: Linen has been a significant material for textile packaging. Thus, the application of the simple spray-coating method to coat linen fibers with a flame-retardant, antimicrobial, hydrophobic, and anticounterfeiting luminescent nanocomposite is an innovative technique. In this new approach, the ecologically benign room-temperature vulcanizing (RTV) silicone rubber was employed to immobilize the environmentally friendly Exolit AP 422 (Ex) and lanthanide-doped strontium aluminum oxide (RESAO) nanoscale particles onto the linen fibrous surface. Both morphological properties and elemental compositions of RESAO and treated fabrics were examined by transmission electron microscopy (TEM), scanning electron microscopy (SEM), wavelength-dispersive X-ray fluorescence (WD-XRF), Fourier transform infrared (FTIR) spectroscopy, and energy-dispersive X-ray spectroscopy (EDX). In the fire resistance test, the treated linen fabrics produced a char layer, giving them the property of self-extinguishing. Furthermore, the coated linen samples’ fire-retardant efficacy remained intact after 35 washing cycles. As the concentration of RESAO increased, so did the treated linen superhydrophobicity. Upon excitation at 366 nm, an emission band of 519 nm was generated from a colorless luminescent film deposited onto the linen surface. The coated linen displayed a luminescent activity by changing color from off-white beneath daylight to green beneath UV source, which was proved by CIE Lab parameters and photoluminescence spectral analysis. The photoluminescence effect was identified in the treated linen as reported by emission, excitation, and decay time spectral analysis. The comfort properties of coated linen fabrics were measured to assess their mechanical and comfort features. The treated linen exhibited excellent UV shielding and improved antimicrobial performance. The current simple strategy could be useful for large-scale production of multifunctional smart textiles such as packaging textiles.

1. INTRODUCTION

Smart clothing, particularly protective technical textiles represent enormous commercial possibilities for smart products. The antibacterial, superhydrophobic, and flame-retardant textiles are examples of functional textiles that were developed for nonaesthetic goals.1−3 Textiles that respond to external stimuli like luminous and thermochromic textile materials are also known as smart textiles. Long-lasting phosphorescence has been a desirable phenomenon for the preparation of smart luminescent textiles.4−9 The long-lasting phosphorescence compounds work by absorbing light into their crystal constituents. The phosphorescence compound’s trap ingredient subsequently captures the light energy. These captured light photons eventually release out of the traps. For example, strontium aluminates activated with rare earths have been used for phosphorescence in many applications including emergency signs, protective textiles, and luminescent inks.10−12 The potential to produce numerous products with varying emission colors has long been associated with phosphorescent pigments, such as the greenish emissive SrAl2O4:Eu2+,Nd3+;13 the bluish emissive CaAl2O4:Eu2+,Pr3+;14 and the reddish Y2O2S:Mg2+,Ti4+;15 RESAO has long been recognized as important long-lasting phosphors because of their high quantum efficiency, high recyclability, nonradioactivity; as well as strong thermal, chemical, and photostabilities.16 It is possible to reduce the potential harm caused during combustion by performing flame-retardant treatment on combustible materials. It is possible for firefighters to carry out rescue operations and enable people who have been caught in wildfires to escape by delaying and limiting the blazing process.17 Various compounds designed to enhance flame resistance in a variety of items have been produced. Despite their widely used effectiveness, halogen-based flame-retardant...
chemicals have been shown to produce toxic fumes that are dangerous for humans and surrounding environment. The lack of binding capacity between the matrices of inorganic boron-based fire retardants prevents their continued production on a broad scale. In contrast, phosphorus-bearing compounds have long been recognized as ecologically benign flame retardants. Phosphorus-based flame-retardant agents do not produce hazardous compounds during the blazing process, which is in accordance with the environmental rules. Two or more kinds of organophosphorus and organophosphorus/nitrogen-based materials can be loaded into a product matrix to improve its flame-retardant effectiveness.

Various methods, such as lithography, plasma, sol–gel, chemical etching, and nanofibers, have all lately been used to create hydrophobic surfaces. However, those approaches are often costly, time-consuming, and need a wide range of complicated equipment. Consequently, they are not widely used. To create functional apparel, an efficient spray-coating process has been employed as an easy and cost-effective approach to coat textiles. Spray coating has been presented as a facile, noncontact, and economical technology for surface coating at a high rate and negligible agglomeration, using the minimal quantity of the coating aerosol. The hydrophobic properties of textiles tend to limit their usefulness, especially in transportation and packaging. Superhydrophobic materials must have static contact angles of more than 150° and sliding angles of less than 10°. Several disciplines have benefited from the use of superhydrophobic materials, including anticorrosion, antifouling, marine industry, and water–oil separation. Superhydrophobic substrates with high surface roughness can be prepared using micro/nanoscale hierarchical materials. To create hydrophobic materials, fluorine-based compounds have been used. However, these chemicals have been shown to be expensive and hazardous. Research has been focusing on ecologically friendly chemicals for superhydrophobic fabrics in recent years. Silicone rubber that can be vulcanized at room temperature using a butynorate catalyst is an ecologically friendly polymeric material. Silicone rubber is resistant to a wide range of bases, acids, chemicals, heat, and aging. Low viscosity, high hardness and little shrinkage characterize silicone rubber. It has been used in aerospace, three-dimensional (3D) printing, optics, and electronics.

Linen fibers have high strength, low heat conductivity, and high water absorption and dry quicker than cotton fibers. Various products have been produced from linen, such as aprons, towels, wears, bags, bed and table cloths, napkins, and chair covers. Nowadays, linen is expensive and usually made in relatively small amounts. It possesses a long staple compared to cotton fibers. Currently, linen is one of the most favored textiles for bed clothing owing to its durability and hypoallergenic properties. In spite of this, the inherent flammability, poor water resistance, and microbial infection of linen substrates have restricted their use. There are several functional textile products for both clothing and high-performance applications, which can be created by incorporating superhydrophobic, photoluminescence, and flame-retardant agents into the textile material. However, linen products with flame-resistant, superhydrophobic, and photoluminescent features have not been reported in the literature. Since the treated linen is nonwettable, the flame resistance performance can be held for long time periods, allowing the linen materials to provide more protection value. Thus, it is potential to increase the durability of treated linen textiles by combining photoluminescence, superhydrophobic, and fire resistance properties.

In this context, a simple dip-coating procedure is used to manufacture flame-retardant, photoluminescent, and superhydrophobic linen textiles. A mixture of silicone rubber, RESAO nanoparticles, and Exolit AP 422 was used to cushion the linen substrate. Superhydrophobic and photoluminescence characteristics were shown by the as-coated linen fibers and the ability of linen to generate a char film in the duration of the burn test demonstrating self-extinguishing property. The superhydrophobic properties of the treated linen samples have been maintained after 35 washing cycles. The linen fibers served as a host for RESAO as a photoluminescence agent, room-temperature vulcanizing (RTV) as a hydrophobic and cross-linking agent, and Exolit AP 422 served as a flame-resistant agent. Nanoscale phosphor particles were investigated by transmission electron microscopy (TEM) to examine their shape and diameter. X-ray fluorescence (XRF), energy-dispersive X-ray spectroscopy (EDX), scanning electron microscopy (SEM), and Fourier transform infrared (FTIR) spectroscopy were utilized to study the treated linen morphologies and elemental contents. The amount of RESAO nanoparticles applied into the treated linen substrates resulted in significantly different properties. Upon varying the concentration of the phosphor nanopowder, both static contact and slide angles were measured to establish the water-repellent characteristics. CIE Lab measurements, as well as excitation, emission, and decaying spectra, were utilized to assess the luminescence features of the dip-coated linen fibers. Both bend length and air permeability were measured to assess the level of comfort. The present approach is simple and economical, and it can be used to finish textiles on a wide scale without the requirement of complicated machinery, making it suitable for the mass production of multifunctional apparel.

2. EXPERIMENTAL SECTION

2.1. Materials. Misr-Helwan Spin and Weave Co. (Egypt) provided linen substrates. The provided linen was first bleached and scoured using an earlier procedure. The scouring process was performed at a liquor ratio of 1:50 using 2 g L−1 Na2CO3 at 50 °C and 2 g L−1 detergent (nonionic; Hostapal, Swiss) for 30 min to eliminate impurities and waxes. The fabrics were exposed to rinsing with tap water and left to dry under ambient conditions. Exolit AP 422 (ammonium polyphosphate; n > 1000) was supplied by Shandong Shi’an Chemical Co. (China). ADM Chemical Industries (ADMICO; Egypt) supplied us with Decoseal 2540 silicone rubber. Merck supplied the petroleum ether (pether; 60–80 °C). Boric acid (H3BO3), dysprosium oxide (Dy2O3), europium oxide (Eu2O3), strontium carbonate (SrCO3), and aluminum oxide (Al2O3) were all provided by Aldrich (Egypt) and Merck (Egypt).

2.2. Preparation of RESAO. The previously established solid-state high-temperature synthesis procedure was utilized to make the alkaline earth-activated aluminum strontium oxide.

Al2O3 (2 mol), SrCO3 (1 mol), dysprosium oxide (0.03 mol), boric acid (0.2 mol), and europium oxide (0.02 mol) were mixed in EtOH (100%; 400 mL). Once homogenized (25 kHz; 60 min), the suspension was dried at 90 °C overnight for 23 h. The provided powder was milled for 2 h and exposed to sintering for 3 h at 1300 °C in an environment of reductive carbon. To get microparticles of phosphor, the produced residue was subjected to a grinding process.
and sieving procedure. To prepare the phosphor nanoparticles, the top-down approach was applied by charging the previously prepared phosphor micro-scale powder (10 g) into a stainless steel ball milling tube (20 cm) mounted on an oscillating disk, where RESAO nanoparticles were produced utilizing Triple Roll Mill ES80. Throughout 24 h, a 0.1 cm silicon carbide ball was used to repeatedly collide with the ball mill vial containing the phosphor particles and the vibrating disk for 23 h to produce the desired RESAO nanoparticles.

2.3. Preparation of Multifunctional Linen. A solution of RTV (15%; w/v) in pether (petroleum ether) was exposed to stirring for 45 min. Exolit AP 422 (15%; w/v) was added, and the provided solution was stirred for 45 min. RESAO nanoparticles were added at various contents: 0, 0.05, 0.1, 0.25, 0.5, 0.75, 1, 1.25, 1.5, 1.75, and 2% w/w. The provided mixtures were represented by RESAO-0, RESAO-1, RESAO-2, RESAO-3, RESAO-4, RESAO-5, RESAO-6, RESAO-7, RESAO-8, RESAO-9, and RESAO-10, respectively. The pristine uncoated linen is represented by the blank sample, whereas the phosphor-free coated linen is represented by RESAO-0. Each mixture was homogenized (25 kHz) for 30 min. Linen fabrics (10 × 15 cm²) were spray-coated with the synthesized nanocomposites at room temperature. The spray-coating process was performed utilizing Lumina automated spraying gun (STA-6R; Fuso Seiki Co. Ltd; Tokyo, Japan) equipped with an orifice-sized spraying nozzle (1 mm) placed at 20 cm from the linen fabric. Pressurized air (250 kPa) was employed as a carrier gas. The spraying nozzle was pushed back and forth over the linen fabric at approximately 3 cm s⁻¹ while spraying at a 10 mL min⁻¹ flow rate. An air-drying process (30 min) was performed to completely evaporate the solvent from the treated linen. Figure 1 shows a schematic representation of the preparation procedures of functional linen.

2.4. Analysis Methods. 2.4.1. Morphologies and Elemental Compositions. Both shape and size of RESAO were investigated by JEOL1230 (Japan). An ultrasonic bath at 35 kHz homogenized the phosphor powder for 15 min before being put onto a Cu-grid to create a suspension for the TEM study. The morphology and chemical compositions of linen fabrics were examined by Quanta FEG250 SEM and TEAM EDS (Czech Republic), respectively. ImageJ software loaded on SEM was used to measure the diameters of nanoparticles on the linen fabric surface. In addition, AXIOS XRF was used to evaluate the chemical composition of the spray-coated textile samples. A Nicolet Nexus 670 spectrophotometer was used to record FTIR spectra (400–4000 cm⁻¹) in transmission mode.

2.4.2. Hydrophobicity Screening. Dataphysics OCA15SEC (GmbH, Germany) was employed to determine contact angle, slide angle, and wettability time. The ASTM D-7334 standard protocol was used to take all measurements. An adhesive tape (double-sided) was employed to connect the cloth sample to a glass slip to create a flat surface.

2.4.3. Photoluminescence Spectra. The photoluminescence spectral analysis of linen was carried out by the JASCO FP6500 (Japan). Initial exposure of the luminous linen fabric to UV light for 15 min was done using a 150 W xenon arc lamp, which was used to determine the decay time. Once the ultraviolet light had been blocked, the data were collected in complete darkness. A UV light (6 W; 365 nm) positioned 5 cm above the cloth was used to examine the fabric luminescence reversibility. To restore the fabric to its original state, the light supply was removed and the sample was placed in a dark box for 10 min. Some cycles of the above procedure were undertaken, and after each cycle, the emission spectra were recorded.

2.4.4. Coloration Measurements. CIE Lab and color strength (K/S) were employed to investigate changes in the color of the spray-coated linen fabrics, where lightness from blackest (0) to whitest (100) was represented by L*, color ratio of green(−) to red(+) was represented by a*, and color ratio of blue(−) to yellow(+1) was denoted by b*. Images of luminescent linen were captured using a Canon A710IS under visible daylight, ultraviolet rays, and complete darkness.

2.4.5. Comfort Evaluation. Measurements of uncoated and coated linen bend lengths were made using a Shirley Stiffness apparatus under British specification 3356:1961. A Textest FX3300 was used to measure air permeability at 100 Pa in accordance with Standard ASTM D737.

2.4.6. Flammability Testing. The char length was determined using the standard BS 5438 (1989) protocol. The char length is defined as the linen fabric length (mm) destroyed by flame.

2.4.7. Durability Assessment. The linen fabrics (10 × 15 cm²) were subjected to different laundering cycles, including 5, 10, 15, 20, 25, 30, 35, and 40 cycles, according to AATCC 61 (1989). At 40 °C, the samples were washed in an aqueous solution of detergent (66 g; AATCC standard) in a launder-meter appliance. Every laundry cycle lasted 45 min. After each cycle, the char length was reported as a measure of durability.

2.4.8. Antimicrobial Activity. The coated linen was tested for antibacterial characteristics against Escherichia coli, Staphylococcus aureus, and Candida albicans microorganisms. The AATCC 100:1999 standardized method was used for microbial counting for antimicrobial testing, which is a quantitative process.

2.4.9. Ultraviolet Protection. To verify the ultraviolet shielding capabilities of the produced fabrics, the ultraviolet protection factor (UPF) was determined. It was recorded using the AATCC (Transmittance) 183 (2010) UVA standard technique.
3. RESULTS AND DISCUSSION

3.1. Preparation of Smart Linen. Photoluminescent smart linen fabrics with flame-resistant and hydrophobic qualities were developed utilizing a silicone rubber composite containing Exolit AP 422 as a flame-resistant substance and RESAO as a luminescent substance. As a superhydrophobic layer, the TV matrix held both RESAO nanoparticles and Exolit AP 422 to the surface of the linen cloth. As shown in Figure 2, the size of the obtained RESAO particles was reduced to yield nanoscale powder with a diameter range of 25–55 nm. After synthesis of RESAO microparticles using the solid-state high-temperature method,43,53,54 the RESAO nanoparticles were produced using the top-down approach.44 When phosphor nanoparticles were included in the RTV multifunctional film that was applied to linen, the efficient dispersion of those nanoparticles must be ensured throughout the matrix. This is beneficial to provide a transparent film on the linen surface. Silicone rubber and Exolit AP 422 nanoparticles were

Figure 2. TEM images of RESAO nanoparticles.

Figure 3. SEM micrographs of spray-coated linen; RESAO-1 (a, b), and RESAO-10 (c, d).

Table 1. EDS Elemental Identification (wt %) of Coated Fabrics Inspected at Three Positions (P1, P2, and P3)

| sample   | C    | O    | P    | Si   | Sr   | Al   | Eu   | Dy   |
|----------|------|------|------|------|------|------|------|------|
| blank    | 60.76| 39.24| 0    | 0    | 0    | 0    | 0    | 0    |
| RESAO-1  |      |      |      |      |      |      |      |      |
| P1       | 41.73| 32.21| 6.11 | 17.29| 0.81 | 1.51 | 0.22 | 0.12 |
| P2       | 41.90| 32.54| 6.43 | 17.96| 0.73 | 1.32 | 0.23 | 0.07 |
| P3       | 41.27| 32.01| 6.04 | 17.73| 0.87 | 1.63 | 0.28 | 0.17 |
| RESAO-7  |      |      |      |      |      |      |      |      |
| P1       | 39.76| 34.70| 5.12 | 15.06| 1.80 | 2.64 | 0.61 | 0.31 |
| P2       | 39.91| 34.73| 5.73 | 15.74| 1.62 | 2.44 | 0.64 | 0.37 |
| P3       | 39.27| 34.24| 5.32 | 15.60| 1.88 | 2.72 | 0.59 | 0.38 |
| RESAO-10 |      |      |      |      |      |      |      |      |
| P1       | 38.65| 35.11| 4.65 | 14.53| 2.22 | 3.42 | 0.90 | 0.52 |
| P2       | 38.80| 35.84| 4.56 | 14.91| 2.04 | 3.62 | 0.83 | 0.58 |
| P3       | 38.47| 35.31| 4.25 | 14.70| 2.30 | 3.50 | 0.78 | 0.69 |
combined in petroleum ether, and then phosphor nanoparticles with predetermined total concentrations were added. During the spray-coating technique, each nanocomposite was applied to a linen surface under ambient circumstances. Nanocomposite comprising silicone rubber, Exolit AP 422, and aluminum strontium oxide nanoparticles doped with lanthanides was used in this study to impart the linen samples fire retardancy, photoluminescence, and hydrophobic activity.

3.2. Morphological Study of Linen. Various analytical methods were used to inspect the morphological properties of the coated fibers. The luminescent, flame-retardant, and superhydrophobic properties of sprayed fabrics were illustrated by studying their morphological structures. SEM images of RESAO-1 and RESAO-10 are shown in Figure 3. The sprayed coated linen fibers demonstrated a homogeneously distributed film of nanoscale hierarchical architectures that explains the promoted functional characteristics of the coated linen. An increase in roughness was achieved by achieving these nanohierarchical structures. This resulted in linen fibers that were more hydrophobic than blank linen.55 According to Table 1, EDX was employed to analyze the chemical composition of the sprayed textiles. The chemical contents investigated at three locations on the sample surface were identical, demonstrating homogeneous dispersion of RTV-RESAO-Ex nanocomposite on the linen surface. Since linen is made up of carbohydrate cellulose polymer, where oxygen (O) and carbon (C) are the primary elements. In addition, EDX detected silicone (Si) owing to RTV and phosphorus (P) owing to Exolit AP 422. Other elements including Al, Sr, Dy, and Eu were also detected owing to RESAO nanoparticles. XRF was used to establish the chemical compositions of the sprayed fabrics, as summarized in Table 2. A very accurate approach for evaluating the element contents at extremely low concentrations is EDX. WD-XRF, on the other hand, offers an elemental recognition technique with a detection limit larger than 10 mg kg$^{-1}$.56 Because certain elements (Eu and Dy) on the sprayed fabric surface exist in extremely low concentrations, XRF provides partial elemental identification, including Si, Sr, and Al. WD-XRF was unable to identify either Dy or Eu due to their very low concentrations. The elemental contents identified by either XRF or EDX were comparable to the molar ratios used in the preparation of RESAO and RTV-RESAO-Ex nanocomposites.

Figure 4 shows the FTIR spectral analysis applied to inspect the functional substituents on linen fabrics. The typical absorbance peaks of blank linen were observed at 3341 cm$^{-1}$ attributed to stretching hydroxyl, 2905 cm$^{-1}$ owing to stretching aliphatic $\text{C}–\text{H}$, 1025 cm$^{-1}$ assigned to stretching ether ($\text{C}–\text{O}$), and 1451 cm$^{-1}$ attributed to bending aliphatic $\text{C}–\text{H}$. It was discovered that the intensities of both the bending and stretching bands of the aliphatic $\text{C}–\text{H}$ increased after coating with the aliphatic-rich RTV. Furthermore, the hydroxyl group’s stretching vibration was shown to diminish.

Table 2. Wavelength-Dispersive X-ray Fluorescence (WD-XRF) Detected Elements of Coated Linen Fabrics

| element | RESAO-1 (wt %) | RESAO-7 (wt %) | RESAO-10 (wt %) |
|---------|---------------|---------------|-----------------|
| Si      | 87.33         | 60.64         | 47.80           |
| Sr      | 4.95          | 13.40         | 18.53           |
| Al      | 7.72          | 25.96         | 33.67           |

Stretch and bend vibrations were used to monitor the Exolit AP 422 ammonium group at 2905 and 1451 cm$^{-1}$, while the bands of P–OH, P–O, and P–O–P were monitored at 1558, 1317, and 1157 cm$^{-1}$, respectively. The bend vibration of Si–O caused an absorption peak at 564 cm$^{-1}$. There were no significant shifts in the measured bands or variations in their intensities to confirm an efficient and complete coating of textiles.

3.3. Hydrophobic Activity. The spray-coated fabrics were examined for their hydrophobic screening properties (Figure 5 and Table 3). The RTV-RESAO-Ex layer spray-coated onto the fibrous textile surface was thin. Filling in spaces and voids between fibers, the RTV-RESAO-Ex composite was monitored to generate a rougher surface. The contact angle of blank linen (uncoated linen) was not identified ($0^\circ$) owing to its high wettability. The contacting angle of phosphor-free coated linen (RESAO-0) was enhanced to 139.8$^\circ$. The contact angle of the RESAO-1-treated linen was further enhanced to 140.4$^\circ$. The contact angle increases significantly from 140.4$^\circ$ (RESAO-1) to 152.4$^\circ$ (RESAO-8) with increasing RESAO ratio. However, when the quantity of ASO was further increased, the static contact angle decreased from 152.4$^\circ$ (RESAO-8) to 151.3$^\circ$ (RESAO-10). As the amount of RESAO nanoparticles trapped on the fabric’s surface increases, roughness increases as well.55 However, the greater increase of RESAO nanoparticle concentration can significantly reduce the spaces between those RESAO particles. This might have a detrimental impact on the surface roughness, resulting in lower static contact angles.57 To provide a smoother surface, RESAO nanoscaled particles were packed into the spaces between fibers. As a result, surface roughness was reduced by increasing the total RESAO nanoparticle concentration more than RESAO-8. RESAO-8 might be considered the optimal total RESAO content in this aspect. The sliding angles of the treated and untreated textiles were compared. The linen hydrophobicity was found to increase with increasing RESAO, leading to a much higher increment in the wettability time than blank linen (5 s). The present approach can thus be described as a simple and low-cost treatment procedure that does not need the use of complicated tools or lengthy processes. Aside from that, the present straightforward technology can be used.
to industrially produce luminescent, flame-retardant, and hydrophobic linen products for a variety of uses, including textile packaging and other protecting fabrics. Silicone-coated fibers are distinguished by their hydrophobic nature and their ability to permeate oil while holding water. A hydrophobic textile with probable water–oil separation characteristics may be made using the current method.

3.4. Flame Resistance Assessment. Because it was completely burned, the blank (uncoated) linen sample did not pass the flame test, as indicated in Table 4. However, when exposed to a fire, the coated linen showed outstanding flame-retardant efficacy, with burn progress being tracked and stopping instantaneously when the flame supply was removed. The char length of the phosphor-free fabric (RESAO-0) was highly improved to 52 mm compared to the uncoated blank fabric. The char length of the sprayed textile was found to further decrease as the RESAO ratio increased. Exolit AP 422 was cross-linked with RTV to increase the fire resistance property. Increasing the RESAO ratio from 0.05 to 1.25% reduced the char length from 51 to 40 mm, respectively. However, the char length did not alter much when the RESAO ratio was increased from 1.25 to 2%. Exolit AP 422, an ecologically friendly and formaldehyde-free flame retardant, has been to replace the Pyrovatex-based flame retardants, which are expensive and harmful to the environment and human health. As a result, the present technology can lower the amount of harmful formaldehyde emitted by Pyrovatex.

The flame-retardant linen sample (RESAO-8) was washed to examine the effects of washing on its durability. RESAO nanoparticles and Exolit AP 422 were attached to the fabric surface utilizing RTV as a trapping bulk. As shown in Figure 6, the char length was reported after each laundry cycle. For 35 washes, the char length was observed to gradually widen. However, after 35 wash cycles, the sample was found to completely burn.

3.5. Mechanical and Colorimetric Screening. The primary goal of using the spray-coating procedure is to create a water-repellent textile surface while yet allowing the cloth to move and breathe freely. It was found that applying RTV-RESAO-Ex nanocomposites using the spray-coating procedure had a significant influence on the fabric’s physical properties. The char length was reported after each laundry cycle. For 35 washes, the char length was observed to gradually widen. However, after 35 wash cycles, the sample was found to completely burn.

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Table 3. Hydrophobic Data Screening of Blank (Uncoated) and Treated Linen

| sample     | CA (deg) | SA (deg) | WT (min) |
|------------|----------|----------|----------|
| blank      | 0        | 0        | 0        |
| RESAO-0    | 139.8    | 15       | 30       |
| RESAO-1    | 140.4    | 15       | 35       |
| RESAO-2    | 141.0    | 15       | 45       |
| RESAO-3    | 142.5    | 15       | 45       |
| RESAO-4    | 144.0    | 14       | 55       |
| RESAO-5    | 145.3    | 13       | >60      |
| RESAO-6    | 148.0    | 11       | >60      |
| RESAO-7    | 151.6    | 10       | >60      |
| RESAO-8    | 152.4    | 9        | >60      |
| RESAO-9    | 151.8    | 8        | >60      |
| RESAO-10   | 151.3    | 8        | >60      |

CA represents contact angle, SI represents sliding angle, and WT represents wettability time.

Table 4. Impacts of RESAO Content on Linen Flammability

| sample | DCL (mm) | MCW (mm) |
|--------|----------|----------|
| Blank  | Complete Burning        |
| RESAO-0| 52       | 17       |
| RESAO-1| 51       | 18       |
| RESAO-2| 51       | 17       |
| RESAO-3| 50       | 18       |
| RESAO-4| 47       | 17       |
| RESAO-5| 45       | 18       |
| RESAO-6| 42       | 17       |
| RESAO-7| 40       | 18       |
| RESAO-8| 40       | 17       |
| RESAO-9| 39       | 18       |
| RESAO-10| 40      | 18       |

DCL represents the damaged char length, and MCW represents the maximum char widths.
addition of RESAO. There were minor differences in a* and b* after increasing the RESAO quantity, indicating the creation of a transparent layer on the textile surface. The inherent characteristics of the sprayed fabrics were not significantly impacted by the photoluminescent, fire-resistant, and superhydrophobic coated film.

3.6. Photoluminescence Spectral Analysis. The spray-coating method was used to embellish the fibrous linen surface with the produced RESAO nanoscaled particles. Photographs of RESAO-7 were taken under visible daylight and UV irradiation (365 nm), as shown in Figure 7. Those dissimilar conditions led to a wide range of colorimetric changes, including white in daylight and green under UV rays. To keep RESAO attached to the fabric, RTV was used as a kind of organic matrix. Because the phosphorescent layer formed by trapping RESAO and Exolit AP 422 in the RTV film has the same photoluminescence wavelength as the RESAO powder, no significant faults in the nanocomposite film’s phosphorescence characteristics were identified. Using linen that is superhydrophobic, flame-retardant, and photoluminescent, this work presents the first example of multifunctional linen fibers. Illustration of both excitation and fluorescence spectra for the coated linen fabrics is shown in Figures 8 and 9. The

Table 5. Comfort Screening of Blank and Coated Fabrics

| sample     | BL (cm) | weft | warp | AP (cm²·cm⁻²·s⁻¹) |
|------------|---------|------|------|------------------|
| blank      | 3.97    | 4.42 | 44.29|
| RESAO-0    | 4.10    | 4.66 | 43.83|
| RESAO-1    | 4.93    | 5.36 | 42.75|
| RESAO-2    | 5.07    | 5.53 | 42.46|
| RESAO-3    | 5.23    | 5.96 | 42.11|
| RESAO-4    | 5.50    | 6.17 | 41.89|
| RESAO-5    | 5.73    | 6.46 | 41.30|
| RESAO-6    | 5.80    | 6.17 | 40.78|
| RESAO-7    | 6.04    | 6.35 | 40.42|
| RESAO-8    | 6.15    | 6.52 | 40.06|
| RESAO-9    | 6.29    | 6.73 | 39.94|
| RESAO-10   | 6.48    | 6.90 | 39.47|

“BL represents the bend length, and AP represents the air permeability.

Table 6. Coloration Properties of Blank and Sprayed Substrates

| sample  | L*   | a*   | b*   |
|---------|------|------|------|
| blank   | 93.04| −3.18| 0.21 |
| RESAO-0 | 92.43| −2.92| 0.47 |
| RESAO-1 | 91.11| −2.66| 0.63 |
| RESAO-2 | 90.59| −2.30| 0.78 |
| RESAO-3 | 89.85| −1.92| 0.94 |
| RESAO-4 | 88.90| −1.73| 1.16 |
| RESAO-5 | 86.76| −1.50| 1.38 |
| RESAO-6 | 86.35| −1.56| 1.54 |
| RESAO-7 | 86.05| −1.40| 1.73 |
| RESAO-8 | 85.93| −1.14| 1.88 |
| RESAO-9 | 85.44| −0.94| 2.12 |
| RESAO-10| 84.75| −0.84| 2.38 |

3.6. Photoluminescence Spectral Analysis. The spray-coating method was used to embellish the fibrous linen surface with the produced RESAO nanoscaled particles. Photographs of RESAO-7 were taken under visible daylight and UV irradiation (365 nm), as shown in Figure 7. Those dissimilar conditions led to a wide range of colorimetric changes, including white in daylight and green under UV rays. To keep RESAO attached to the fabric, RTV was used as a kind of organic matrix. Because the phosphorescent layer formed by trapping RESAO and Exolit AP 422 in the RTV film has the same photoluminescence wavelength as the RESAO powder, no significant faults in the nanocomposite film’s phosphorescence characteristics were identified. Using linen that is superhydrophobic, flame-retardant, and photoluminescent, this work presents the first example of multifunctional linen fibers. Illustration of both excitation and fluorescence spectra for the coated linen fabrics is shown in Figures 8 and 9. The

Figure 7. Photos of RESAO-7 under daylight (a) and UV rays (b).

Figure 8. Excitation spectral analyses of various linen fabrics with different ratios of RESAO nanoparticles: RESAO-1 (a), RESAO-3 (b), RESAO-5 (c), RESAO-7 (d), and RESAO-10 (e).

Figure 9. Fluorescence spectral analyses of RESAO-7 versus ultraviolet irradiation time (10–50 s).
absorption spectra were found to increase with increasing RESAO ratio in the nanocomposite coating to indicate concentration-dependent absorption spectra (Figure 8). On the other hand, the emission spectra were also found to increase with increasing time of exposure to UV light to indicate irradiation time-dependent absorption spectra (Figure 9). Under UV light, all of the treated samples exhibited reversible emission. However, the treated linen samples with a higher RESAO content from RESAO-8 to RESAO-10 showed long-lasting phosphorescent emission because they continued to produce light even after the UV source was turned off. The samples between RESAO-1 and RESAO-7 showed fluorescence without emission bands visible after turning off the ultraviolet lamp. The linen substrates that could induce long-lasting phosphorescence were those treated with a quantity of RESAO equivalent to or greater than RESAO-8. Thus, the RESAO-7 linen sample can be reported as the optimal sample with the greener luminescent linen sample for anticounterfeiting applications. The emission wavelength was measured at 519 nm, resulting in a bright and wide emission band.

The treatment of linen with the RTV-RESAO-Ex nanocomposite indicated that as the RESAO ratio increased, so did the decay time of the coated sample. Figure 10 depicts the decay time curve of linen treated with the RTV-RESAO-Ex nanocomposite. Increases in the overall concentration of RESAO nanoparticles resulted in more intensified spectral band for the coated samples at the same wavelength. The decay time profile was nonlinear as a function of time. However, there were two distinct phases of decay, with the first stage showing a fast progression, followed by a more gradual decline. It has been common to use Dy\textsuperscript{3+} and Eu\textsuperscript{2+} as traps with the capacity to extend the phosphorescence time in long-lived phosphors. Phosphorescence emission is dependent on the density of the traps, while its persistent emission is linked to the depth of the entrapped photons. As a result, the fibers of luminous linen continue to shine at night. It has been known that the transition of Eu(II) \[4f^{7}S_{d}^{2} \leftrightarrow 4f^{7}S_{d}^{2}\] is responsible for RESAO phosphorescence. Neither Dy(III) nor Eu(III) displayed any distinctive emission bands. This shows that Eu(III) is completely exchanged to Eu(II), and the photons held by Dy\textsuperscript{3+} have been transferred to Eu\textsuperscript{2+}. The absorption spectral analysis showed a wide band (400–675 nm). This large bandwidth allows for a wide variety of electromagnetic spectrum excitation. Dy(III) causes the discharge of hole traps after the ultraviolet lamp has been removed. Eu(II) receives the released hole traps followed by the transition of Eu(II) to ground state, where it exhibits a long-lasting luminescence. As shown in Figure 11, both reversibility and photostability of cured linen were tested using repeated cycles of exposure to UV irradiation and darkness. It was initially subjected to UV radiation for 5 min and put in the dark for 10 min to discharge light and restore its original white color. After each cycle, the intensity of fluorescence was measured to indicate that the photostability of the material was high.

### 3.7. Antimicrobial and Ultraviolet Blocking Activity

The plate agar count technology was used to evaluate antibacterial effectiveness against E. coli and S. aureus bacteria and C. albicans fungus. Table 7 summarizes the antibacterial increase percentage triggered by the added quantity of RESAO. The treated linen demonstrated antibacterial activity ranging from bad, good, and very well to outstanding as the percentage of RESAO increased. The UV blocking ability of treated linen at various SAOED contents was evaluated by UPF as shown in Table 7.

![Figure 10. Decay time of various linen fabrics with different ratios of RESAO nanoparticles: RESAO-7 (a), RESAO-8 (b), RESAO-9 (c), and RESAO-10 (d).](image)

![Figure 11. Reversibility of the RESAO-7-coated linen sample at 519 nm.](image)

| RESAO (wt %) | S. aureus (Reduction %) | E. coli (Reduction %) | C. albicans (Reduction %) | UPF |
|-------------|-------------------------|-----------------------|---------------------------|-----|
| RESAO-1     | 15 ± 1.3                | 16 ± 1.0              | 0.00                      | 83  |
| RESAO-2     | 18 ± 1.1                | 20 ± 1.1              | 0.00                      | 117 |
| RESAO-3     | 22 ± 1.2                | 25 ± 1.3              | 0.00                      | 125 |
| RESAO-4     | 25 ± 1.4                | 28 ± 1.0              | 0.00                      | 164 |
| RESAO-5     | 28 ± 1.3                | 31 ± 1.0              | 0.00                      | 183 |
| RESAO-6     | 30 ± 1.0                | 35 ± 1.2              | 0.00                      | 217 |
| RESAO-7     | 32 ± 1.0                | 39 ± 1.0              | 9 ± 1.1                   | 228 |
| RESAO-8     | 33 ± 1.1                | 41 ± 1.4              | 9 ± 1.0                   | 295 |
| RESAO-9     | 33 ± 1.1                | 42 ± 1.3              | 9 ± 1.2                   | 330 |
| RESAO-10    | 34 ± 1.0                | 42 ± 1.1              | 9 ± 1.1                   | 355 |
Spray coating was employed as a simple approach to manufacture multifunctional linen fabrics. The lanthanide-doped aluminate was utilized as a photoluminescent, UV shielding, and antimicrobial agent. The ecologically friendly organic Exolit AP 422 was employed as a fire-resistant agent. The environmentally benign RTV was utilized as a superhydrophobic agent and a film matrix to hold both flame-retardant and luminous phosphor agents onto the textile surface. Immobilization of lanthanide-doped aluminate onto linen surface provides photoluminescence while maintaining the fabric’s intrinsic properties, such as air permeability and elasticity. TEM analysis was employed to explore the morphology of the prepared RESAO nanoparticles (25–55 nm). The treated linen morphology, elemental composition, emission spectra, comfortability, and mechanical qualities were all examined in depth. Different analytical methods were used to examine the treated linen surface morphology. Upon excitation of spray-coated fabrics at 366 nm, the emission wavelength was detected at 519 nm. The linen substrates that could induce long-lasting phosphorescence were those treated with a quantity of RESAO equivalent to or greater than 1.5%. Thus, the linen sample with a RESAO ratio of 1.25% can be reported as the optimal sample with the greener activity between coated and uncoated linen fabrics. The char length was used to examine the treated linen morphology and to determine the flame retardancy properties. The char length was better than that of the untreated sample. Char length was used to examine the treated linen morphology and to determine the flame retardancy properties. The char length was better than that of the untreated sample.

4. CONCLUSIONS

Table 7. The improved UV absorbance of RESAO owing to its electronic structure explains the improved UV shielding of coated fabrics with increasing RESAO ratio.

Notes
The authors declare no competing financial interest.

All data generated or analyzed during this study are included in this published article.

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