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Simultaneous Thermochromic Pigment Printing and Se-NP Multifunctional Finishing of Cotton Fabrics for Smart Childrenswear

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Abstract

Thermochromic (TC) pigments offer significant potential for functional and aesthetic design of smart textile materials. In this study, TC (blue and red) pigments were applied to cotton fabrics and printed on especially designed childrenswear by flat screen printing technique. The antibacterial and ultraviolet protection functionalities have been implemented into the fabrics under study by using selenium nanoparticles. The factors affecting the printing process were studied and the optimum formula was screen printed to produce the pattern's designs of childrenswear. After conducting several tests, the results showed a significant color-changing effect depending on temperature, the color fastness properties to light, wash, and rubbing were excellent. Antibacterial activity of printed fabrics was very good against *Bacillus cereus* and *Escherichia coli* bacteria and the anti-ultraviolet protection was found to be very good. The printed fabrics can be as protective childrenswear as shown in this work.

Keywords

thermochromic pigments, selenium nanoparticles, multifunctional finishing, antibacterial activity, ultraviolet protection, smart kid's wear

Thermochromic (TC) dyes and pigments change shade reversibly with a relatively small change in temperature. TC colorants have achieved particular importance in nontextile applications, such as plastic strip thermometers, medical thermography, food packaging, and nondestructive testing of engineered articles and electronic circuitry, but their use in textile systems appears to be far less widespread (Chowdhury et al., 2013). In recent years, there has been growing interest in the use of

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leuco dye–based TC pigments as smart materials in textile and fashion design. Fabric displays have been created for new forms of communication and expression (Jacobs & Worbin, 2005; Kooroshnia, 2013; Peiris et al., 2011). Of those, leuco dye–based TC printed fabrics were combined with skin technology to create interactive textile displays, enabling us to reconsider relationships between human behaviors and the surrounding environments (Kooroshnia, 2013). In combination with a suitable binder system, TC colorants can be applied to textile substrates by means of continuous (pad-dry-cure) or screen-print application (Christie et al., 2007; Dawson, 2010; Lloyd, 2015; Mather, 2001; Tang & Stylios, 2006). Furthermore, TC printed fabrics have been combined with skin technology to explore ways to control and develop dynamic textile expressions. However, most of the previous researchers have focused on combining TC printed fabric with computational technology and exploring ways of heating or cooling the TC printed fabric.

Selenium (Se) is one of the necessary trace elements in the human body. Approximately 40–300 μg of Se are needed daily in the diet of a normal adult (Deepa & Ganesan, 2014; Tran & Webster, 2013). High concentrations of selenium (such as 3,200 μg or above/day) are toxic for the human body and other living organisms (Navarro-Alarcon & Cabrera, 2008; Tran & Webster, 2013). Researchers have found that organic selenium inhibits bacterial growth (Ratushnaya et al., 2002); additionally, selenium nanoparticles (Se-NPs) have high biological activity and low toxicity, plus help to soothe redness (Ingole et al., 2010; Srivastava & Mukhopadhyay, 2015; H. Wang et al., 2007; Xia, 2007). Selenium has excellent antioxidant properties (Barnaby et al., 2011; Torres et al., 2012) and antifungal properties (Shahverdi et al., 2010). Various strategies so far employed for the synthesis of nano-selenium include pulsed laser ablation (Quintana et al., 2002), a solution phase approach (Gates et al., 2002), electrokinetic techniques (M. Wang et al., 2010), a vapor deposition route (Filippo et al., 2010), and a wet chemical method (Ingole et al., 2010). These methods are characterized by elevated temperature and high pressure and are hazardous to the environment (Zhang et al., 2011). The main synthetic approach for preparing Se-NPs is chemical reduction, which requires reducing and stabilizing agents. The use of stabilizer may hinder the technological application, specifically in the biological origin, because the chemical structure of stabilizers has a toxic effect (Ingole et al., 2010). To overcome these hurdles, microorganisms and plant extracts were exploited as a possible green alternative to physical and chemical methods (Hariharan et al., 2012). In the worlds of innovative textile products, hygiene, and active lifestyle, selenium has received great attention recently; this has created a rapidly increasing market for a wide range of uses, including antibacterial, UV protection, flame retardancy, softness, easy care, water and oil repellency, and enhancing the antimicrobial activity of pigment printing (Abou Elmaaty, Abdelaziz, et al., 2018; Abou Elmaaty, Elnagar, et al., 2018; Hebeish et al., 2015; Ibrahim et al., 2015; Ibrahim, Abou Elmaaty, et al., 2013; Ibrahim, Eid, et al., 2013; Ibrahim, Eid, & Abdel-Aziz, 2017; Ibrahim, Eid, El-Aziz, et al., 2017; Ibrahim, Eid, Elmaaty, et al., 2013). In addition, researchers (Elmaaty et al., 2018) have presented papers on how to strengthen and functionalize TC pigments printed with antibacterial materials. In this work, we aim to apply recent advancements in the field of antibacterial, UV-blocking, TC-colored textiles to textiles that are functionalized with Se-NPs' surface-coating printing technology. Consequently, we researched the printing and finishing parameters of TC, mainly concentration, curing time, temperature, and toxicity and then printed the TC pigments at their optimum conditions for designed childrenswear.

Materials and Methods

Materials

Cotton fabric (poplin, 158 g/m^2) was purchased from the Misr Helwan Company (Helwan, Cairo) for spinning and weaving. The fabric was cleaned by scouring for 2 hr at a boil using an aqueous

Table 1. Recipe of the Pigment Printing Paste.

Constituents	Paste (kg^{-1})
TC pigment	X g
Thickener	20 g
Binder	100 g
Fixer	20 g
Selenium nanoparticles	Y ml
Dextrosil	10 ml
Water	820 ml

solution containing 1% sodium hydroxide. It was then thoroughly washed and air-dried at room temperature, to be ready for further processing. Printofix® Binder N 86 (acrylate-based copolymer, anionic, SPI, Egypt), Fast print® thickener 600 (polyacryate inverse emulsion, anionic, SPI, Delta for Chemical Industry Co., Egypt), and blue TC pigments with activation temperature 38 °C were purchased from HALI INDUSTRIAL Co., Ltd. (China), along with the fixing agent, Dextrosil. All chemicals were used as received.

Methods

Synthesis of Se-NPs. The Se-NPs in this study were prepared according to an economically and environmentally friendly method described in the literature (Srivastava & Mukhopadhyay, 2015). The cytotoxicity of Se-NPs was analyzed on human cells (HFB_4) by MTT assay. This assay assessed the mitochondrial activity of the viable cells by measuring its ability to reduce Yellow Tetrazole (MTT) into purple formazan crystals (Sabela et al., 2018).

Printing. Printing pastes were prepared using the recipe shown in Table 1. The paste was applied to substrates using the flat-screen technique. Printed samples were then dried at 80 °C for 3 min and cured at 150 °C for 3 min by means of a high-temperature steamer from R. B. Electronic & Engineering Pvt. Ltd. A print paste was executed with varying TC and Se-NP concentrations, along with varying curing times and temperatures, to achieve the optimum printing recipe.

Characterization. The color uptake, expressed as the color strength (K/S) value of the obtained pigment print, was performed using a spectrophotometer (model: CM3600A; manufacturer: Konica Minolta, Japan). K/S values are calculated at the wavelength of maximum absorption of the color's (λ_{max}) reflectance curve at K/S value 370. The fastness properties of the printed fabric enabled us to find the fixation of the printed pigment from the fabric by using a Launder O meter machine, as stated in AATCC T M 61-1996 (American Association of Textile Chemists and Colorists, 1996b); a CGOLDENWALL dry wet rubbing fastness tester, as stated in AATCC T M 8-1996 (American Association of Textile Chemists and Colorists, 1996a); and a light-fastness tester a carbon arc lamp, as stated in AATCC T M 16A-1972 (American Association of Textile Chemists and Colorists, 1972). The tensile strength test of unprinted and printed cotton samples was performed with a Universal testing machine (Tinius Olsen EN ISO 13934-1; 1999—model H25KT; International Organization for Standardization, 1999). The surface morphology of selected fabric samples was observed with a scanning electron microscope (SEM) model Joel JSM-6510, Japan, with energy dispersive X-ray (EDX) spectroscopy for composition analysis. The antibacterial activity was assessed quantitatively against gram-positive bacterium (*Bacillus cereus*) and gram-negative bacterium (*Escherichia coli*) on the blank, printed cotton, and cotton/polyester blended fabrics with Se-NPs, according to AATCC T M 100-2004 (American Association of Textile Chemists and Colorists, 2004); the result was expressed as the percentage of bacteria reduction. UV-protection functionality, expressed as UV-protection factor (UPF), was assessed according to the AS/NZS 4399:1996. Durability to washing was evaluated according to AATCC T M 61(2A)-1996 (American Association of Textile Chemists and Colorists, 1996c) after five washing cycles. The color-changing ability of different printed fabrics was tested by exposing the fabrics near a heat gun with a temperature of 38 °C.

Design process. The design process of childrenswear was initiated by testing how dynamic prints behaved with TC/Se-NP pigments in relation to children's bodies. These garments were screen printed with different designs using an overall optimum condition in order to monitor the increase in children's body temperatures by changing the design color with any uptick in body temperature. Two TC pigments (blue and red) were used in the implementation of pattern design ideas for childrenswear.

Results and Discussion

Statistical Analysis

All tests have been performed by taking the average of three (sample) readings. The standard error of the mean was calculated according to the equation given below and found to be $+ (-)$ 0.1,

$$SE_x = \frac{S}{\sqrt{n}},$$

where S = sample standard deviation and n = the number of observations of the sample.

Cytotoxicity Test of Synthesized Se-NPs

Table 2 and Figure 1 illustrate the cytotoxicity of the prepared Se-NPs against HFB_4 cell lines. The results indicated that no significant cytotoxicity was observed at a concentration less than 0.195 μM/ml. However, Se-NPs' cytotoxicity increased gradually from 1.17% to 94.13% as concentrations of Se-NPs were increased from 0.195 to 25 μM/ml as shown in Table 2 and Figure 1. The IC_{50} of prepared Se-NPs was 1.014 μM/ml (80.07 μg/ml). Other researchers have evaluated the cytotoxicity of biologically synthesized Se-NPs as anticancer agents on the Michigan Cancer Foundation-7 (MCF-7) cell line and reported that the IC_{50} value of Se-NPs was 41.5 μg/ml (Forootanfar et al., 2013). Se-NPs had a lower cytotoxicity on normal human cells in comparison with several nanoparticles such Ag-NPs (Akter et al., 2018).

Effect of TC Pigment and Se-NPs' Concentration on Color Strength (K/S)

It is obvious from Figure 2A and B that TC pigment concentration was directly proportional with color uptake expressed as color strength (K/S values). Moreover, increasing the pigment concentration from 0.15 up to 0.25 g/kg in the printing paste provided nearly constant values in the depth of shade. This may be attributed to form and size of pigment particles; chemical class and functionality of colorants; compatibility with other ingredients; extent of location, distribution, and agglomeration; and the extent of pigment molecules' accommodation, incorporation, and fixation onto the binder/fabric matrix. As for the effect of the Se-NPs' concentration on the K/S values, Figure 3A and B shows that increasing Se-NP concentration from 0 to 4 ml in the printing paste results in a remarkable improvement in the depth of the obtained prints (from K/S = 3.5–6.05). A further increase in the concentration of Se-NPs had no effect on the color strength due to the saturation of the fabric by nanoparticles.

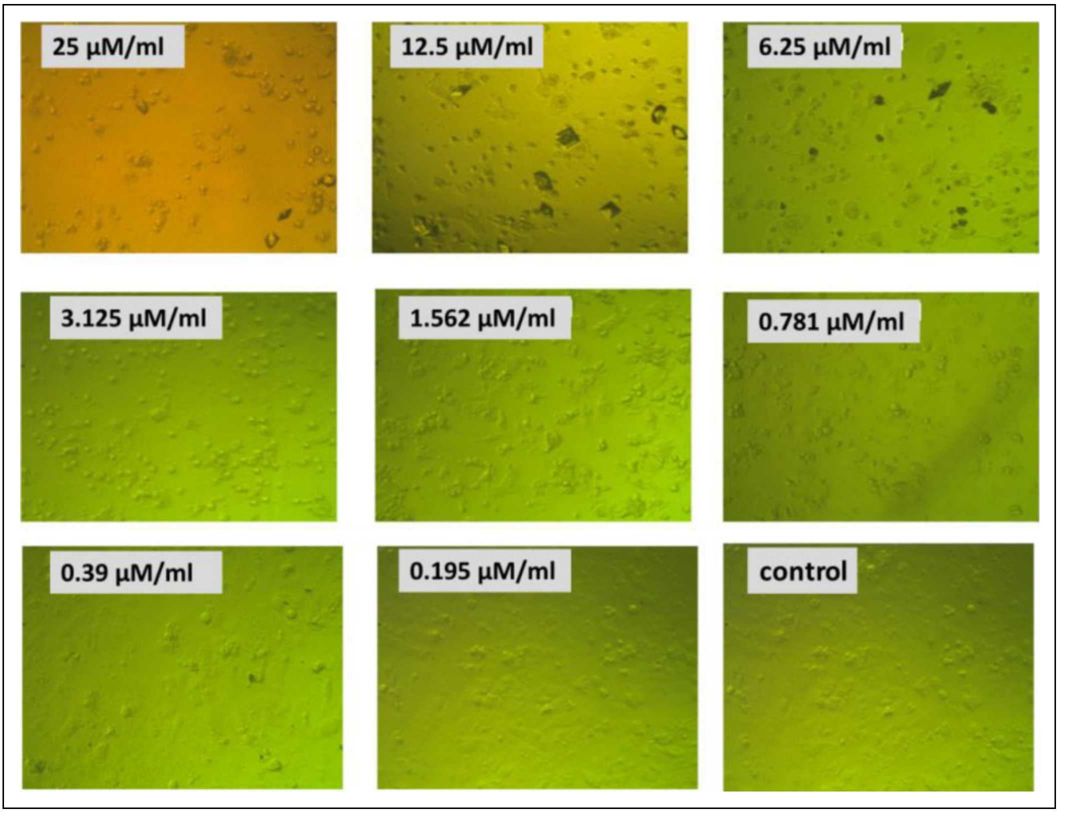

Figure 1. Effect of Se-NPs at different concentrations on HFB_4 cells' viability.

Table 2. Cytotoxicity and IC_{50} of Synthesized Selenium Nanoparticles (Se-NPs).

Se-NPs Concentration (μM/ml)	Toxicity (%)	IC_{50} (μM/ml)
0.195	1.17	1.014
0.39	9.39	
0.781	52.58	
1.562	74.53	
3.125	86.85	
6.25	89.79	
12.5	92.61	
25	94.13	

Effect of Curing Temperature and Time on Color Strength (K/S)

To investigate the influence of curing conditions on color strength (K/S values), different curing temperatures (100, 130, 150, and 170 °C) and durations (3, 6, and 10 min) were applied after printing fabrics with the TC/Se-NP printing paste; the results are shown in Figures 4A and B and 5A and B. It can be concluded from Figure 4 that increasing the curing temperature from 150 °C to 170 °C increases the color strength from 6.05 to 6.5; however, it also caused the fabric to yellow. Thus, 150 °C was selected as the optimum curing temperature. On the other hand, the maximum K/S value, 6.05, was recorded after 3 min as shown in Figure 5. Increasing curing time above 3 min caused a

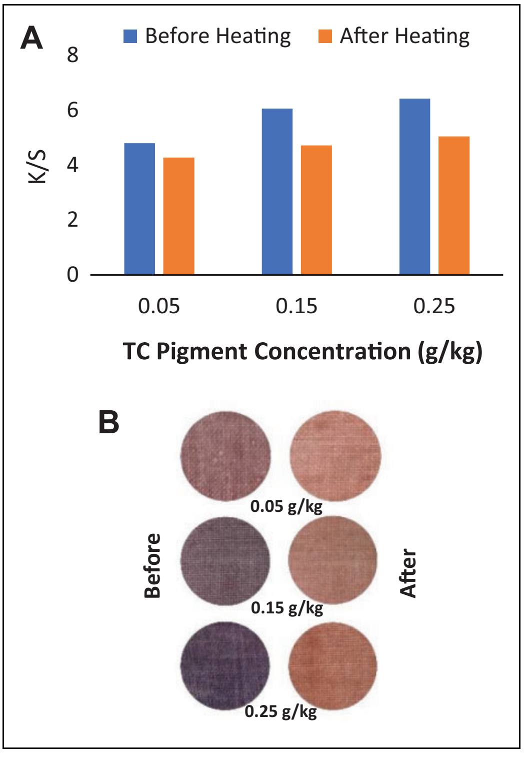

Figure 2. Effect of thermochromic (TC) pigment concentration on K/S (A) and six shades for each TC pigment concentration (before and after heating) (B).

decrease in the color strength. This may be attributed to the fact that the binder, which enclosed the pigment molecules, formed a film under curing conditions.

Physical Properties

The durability of printing on cotton fabrics with TC/Se-NPs (using overall optimum conditions) was evaluated in terms of color fastness mainly washing, rubbing, and light in addition to tensile strength (elongation and maximum force). Table 3 shows that the observed changes in the mechanical behavior of the fabrics were in a range acceptable for standard cotton fabrics. According to this result, we concluded that printing fabric with TC/Se-NPs did not cause a significant damage to the structure of the yarn. This fact was supported by conducting a Wilcoxon signed ranks test, the results of which suggest that there is no significant change between unprinted and printed samples. As for the wash and rubbing fastness, it gave excellent to very good results, respectively, even after five washing cycles. Light fastness provided moderate values, which may be attributed to the crystal violet lactone analogues (diarylphthalide compounds) and fluoran dyes that are the typical compounds used as color formers in the leuco dye–type TC pigments (Elmaaty et al., 2018)

UV Protection of Printed Cotton Fabric Using Se-NPs

UPF is actually the measure of UV radiation (Ultra violet A and Ultra violet B) blocked by the fabric. Higher UPF values mean more blocked UV radiation (Akgun et al., 2010). The UPF values

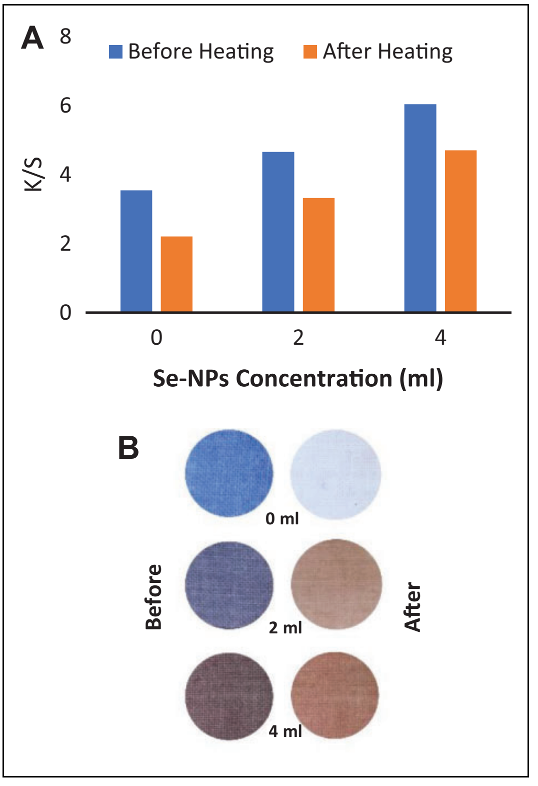

Figure 3. Effect of Se-Np concentration on K/S (A) and six shades for each Se-NP's concentration (before and after heating) (B).

are shown in Table 4; the printed cotton fabric with Se-NPs showed a very good UV-blocking ability (Alebeid & Zhao, 2017) of the printed sample, with a UPF value of 44.03.

Morphology of TC/Se-NPs Printed Sample

The TC/Se-NPs were monitored by SEM. The surface of treated samples showed some round-shaped features a few mm in diameter as shown in Figure 6C and D). These were TC/Se-NPs. No boundary between the particles and binder could be seen, which indicates that all particles were reasonably well covered by the binder.

The Se-NPs formation was analyzed by EDX measurement shown in Figure 7. The peak was present at 1.5 keV with 16.93% weight and 4.61% atomic absorption of the analyzed spot in the printed cotton fiber surface—characteristics of Se. The presence of this peak confirms that the Se-NPs were entirely composed of Se. Some indications of Ca and Si were observed, which is associated with grid impurity used for EDS analysis. The C and O signals originated from cellulose polymer.

Antibacterial Activity of TC/Se-NPs Printed Fabric

A modified AATCC T M 100-2004 method (American Association of Textile Chemists and Colorists, 2004) was adopted for measuring the antibacterial activity of fabrics. Two strains of bacteria, *E. coli* and *Staphylococcus aureus*, were used. One piece of fiber string (20 cm; 0.31 mg) was placed in

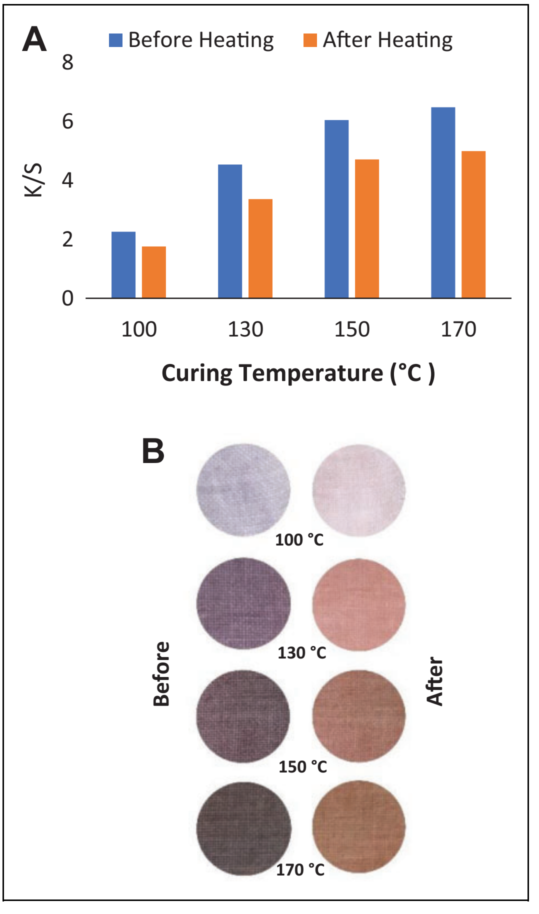

Figure 4. Effect of curing temperature on K/S (A) and eight shades for each curing temperature (before and after heating) (B).

a 1.5 ml microcentrifuge tube; then 200 µl of saline containing 107 CFU/ml (log 7) of the organism were added to the tube, completely covering the fiber. This was incubated for 24 hr at the appropriate temperature for the inoculated culture. After 24 hr, agar plates were spread with the supernatant using a spiral platter (Model 4000, Spiral Biotech). After bacterial incubation at 37 °C, colonies were counted. Plate counts were determined by an automatic spiral plate reader (Q count, Spiral Biotech). The reduction in bacteria growth count was calculated and compared to cotton control (without any additive) as follows:

$$\text{Reduction of bacteria}\,(\%) = ((A - B)/A) \times 100,$$

where A is the initial number of bacterial colonies and B is the final number of bacterial colonies after treatment with TC/Se-NPs printed fabric.

As shown in Table 5, the cotton fabric printed with TC/Se-NPs showed a high percentage of bacterial reduction against two pathogenic bacterial strains. The printed fabric with TC/Se-NPs showed an average reduction of 89.9% against *B. cereus* and 99.9% of against *E. coli.* The high antimicrobial activity of Se-NPs may be attributed to Se-NPs' large surface area, which provides better contact with microorganisms.

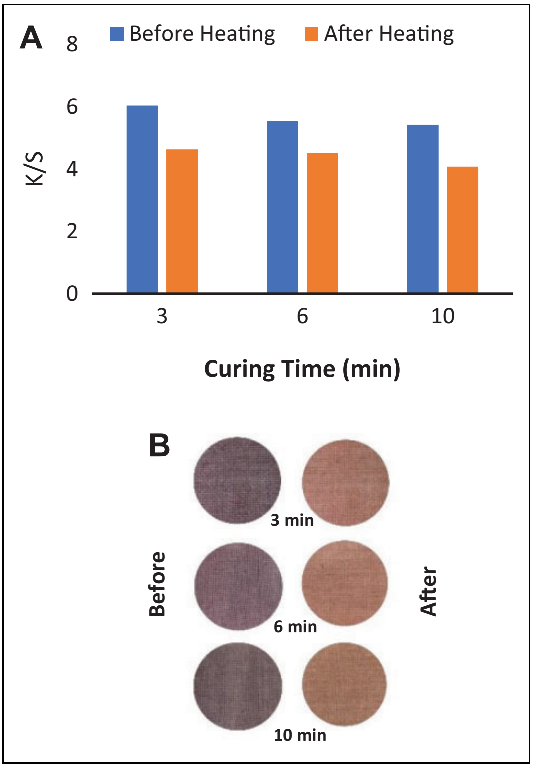

Figure 5. Effect of curing duration on K/S (A) and six shades for each curing duration (before and after heating) (B).

Table 3. Properties of Cotton and Cotton Polyester Fabrics Printed With TC/Se-NPs Using Optimum Conditions.

Sample	Wash Fastness		Rubbing Fastness		Light Fastness	Tensile Strength	
	St.	Alt.	Dry	Wet		Force,[a] *N*	Elongation,[b] %
Unprinted	—	—	—	—	—	349.4	13.80
Printed fabric	5	5	4–5	3–4	2–3	369.9	12.30
Printed fabric after five washing cycles	5	5	4–5	3–4	2.3	—	—

[a]Standard deviation for unprinted = 0.36, standard deviation for printed = 0.30, significance = .1. [b]Standard deviation for unprinted = 12.6, standard deviation for printed = 11.4, significance = .2.

Table 4. UPF Values of the Unprinted and Printed Fabrics.

Function	Before Printing	After Printing
UPF	2.46	44.03

Note. UPF = UV-protection factor.

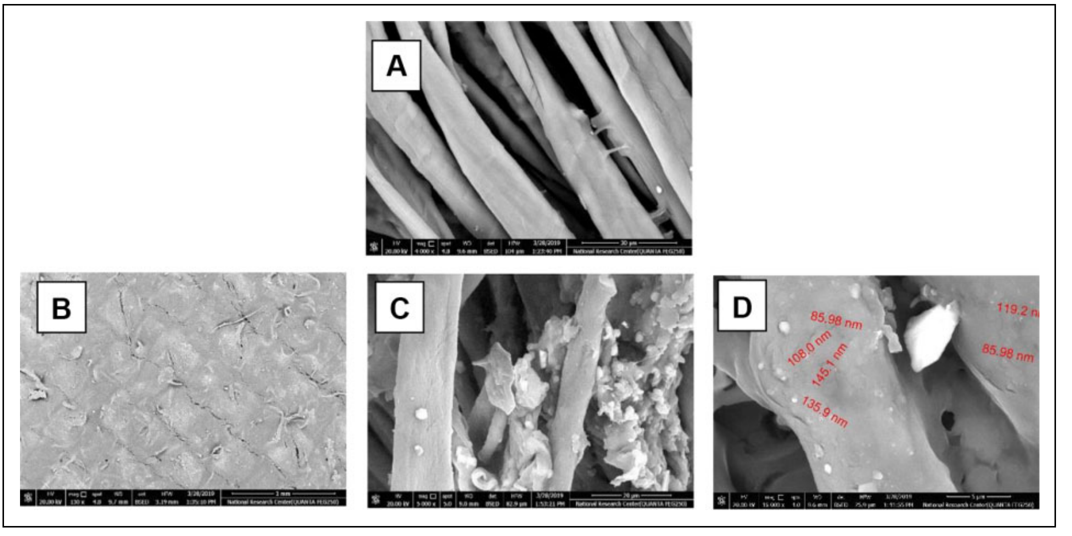

Figure 6. Scanning electron microscope of the untreated sample (A) and the blue TC/Se-NPs screen-printed sample (B, C, and D).

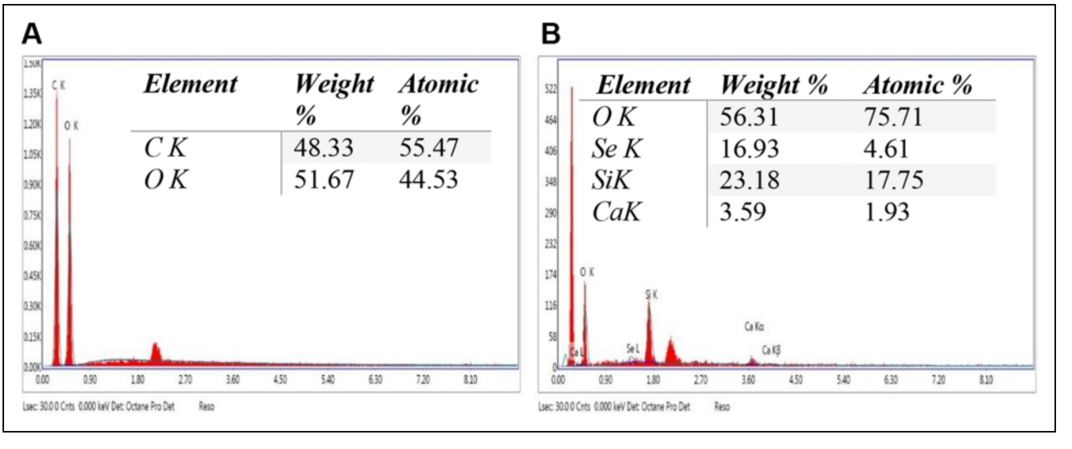

Figure 7. Energy dispersive X-ray spectroscopy spectrum of (A) unprinted cotton fabric and (B) and printed cotton fabric with TC/Se-NP pigment.

Table 5. Percentage of Bacterial Reduction on Printed Fabric with TC/Se-NPs.

Fabric Composition	*Escherichia coli* count	Reduction *Escherichia coli* (%)	*Bacillus cereus* count	Reduction *Bacillus cereus* (%)
Control	1.26×10^8	—	9.16×10^4	—
Printed cotton fabric with TC/Se-NPs	5.24×10^4	99.9	1.73×10^2	89.9

Figure 8. Cotton garments made with blue and red thermochromic pigments (left side of each pair), and light brown and beige dynamic prints (right side of each pair), showing the color-changing effect over a focused area.

Implemented Childrenswear

In this research, we explored several possibilities for developing outfits that may also function as interactive tools through using dynamic prints in the design of wear. As already known, body temperature is a major influence in changing the colors of the pattern's design from dark to light. The initial tests indicated that the interactive garments reacted to body temperature very quickly, particularly in parts of the garments that have direct contact with body parts. As shown in Figure 8, there was a shift in color from dark to light in the garment printed with two TC pigments. Children subjected to temperature rise can be identified immediately as a result of their garments' color change.

Conclusion

In this study, TC pigments were applied to cotton fabrics and tailored into childrenswear by a flat screen-printing technique. The produced fabrics have the ability to change color in response to temperature. Antibacterial and UV-protection functionality have been implemented into the fabrics under study by using Se-NPs in the same printing paste formulation in combination with TC pigment. After reaching the optimal conditions of pigment (0.15 g), Se-NPs (4 ml) with the same concentrations of other components mentioned earlier were dried at 80 °C for 3 min and cured at 150 °C for 3 min; the design process was initiated by testing how dynamic prints produced with TC/Se-NPs' pigment behaved and were activated by heat and the human body. The obtained results demonstrated that incorporation of the nominated Se-NPs into TC pigment printing paste is accompanied by an enhancement in color strength, fastness, and UPF values, along with a noticeable improvement in antibacterial functionality. Finally, we suggest that the TC pigments could be alternatively used with conventional pigments in childrenswear finishing applications according to modern fashion trends. The pigments could also provide different patterns of printed cotton products in addition to technical fabrics in the field of medical and protective wear applications.

Declaration of Conflicting Interests

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