



Full ultraviolet shielding potency of highly durable cotton via self-implantation of palladium nanoclusters

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Abstract Unique technique is currently demonstrated for preparation of ultraviolet protective cotton fabrics with full shielding effect, via self-implantation of palladium (Pd) nanoclusters. Palladium nanoclusters were in-situ immobilized within native and cationized cotton using two different concentrations of palladium precursor (20 and 60 mM) under strong acidic (pH 2) and basic (pH 11.5) media. Cationization (50 and 100%) of cotton fabrics was performed in order to increase the accessibility of fabric for controllable implantation of palladium nanoclusters. Size distribution of palladium nanoclusters in supernatant solution was estimated via Transmission electron microscopy to be 3.2 nm. The estimated data showed that the sample prepared with the highest cationization percent and highest concentration of palladium precursor in strong alkaline medium exhibited the

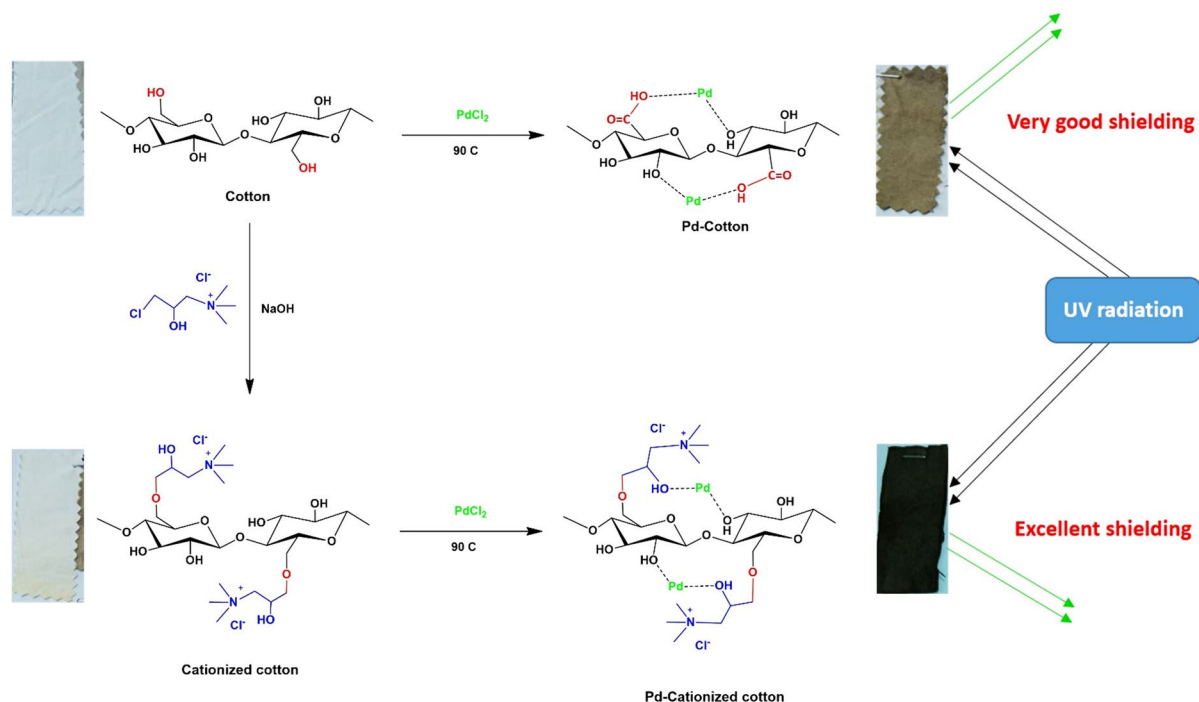
highest yellowness index, color strength and excellent ultraviolet shielding effects. The yellowness index was significantly increased from 15.67 for cationized cotton to 74.99 for the sample prepared with the highest cationization percent and highest concentration of Pd⁺² in alkaline medium (Pd-CC (100)4). Tensile strength was insignificantly decreased from 93.2 MPa for cationized cotton to 84.5 MPa for Pd-CC (100)4. Ultraviolet shielding effect was superiorly enhanced with implantation of palladium nanoclusters. The UV protection factor (UPF) was excellency increased from 1.3 (insufficient) for native cotton to 256.6 (excellent) for Pd-CC (100)4. The effect of repetitive washing cycles on the colorimetric data and the results of ultraviolet protection was also studied to affirm the effect of fabric cationization in preparation of highly durable UV-protective fabrics.

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Graphical abstract



Keywords Self-implantation · Palladium nanoclusters · Cationized cotton · Color strength · UV full shielding

Introduction

Increased concern regarding damage resulting from the exposure to microbial organisms, chemical reagents, insecticides, ultraviolet irradiation and pollutants in the last decade, has heightened the requirement for protective clothing materials. Garments today are required to be waterproofing, flame resistant, self-cleaning, pest repellent and microbicidal to protect human body from the infections, ultraviolet irradiation, chemical and biological reagents, be warmer in cold weather and comfortable in summer. Conventional methodologies of finishing application, such as pad-dry-cure or coating that are recently being applied to make the fabrics acquire the microbicidal, ultraviolet shielding, self-cleaning and fire-retardant finishing reagents, are usually combined with increase in fiber thickness, loss of smoothness and drape, lowering the washing fastness, poor of

mechanical properties and most importantly reduced the comfortability to the wearers (Durán et al. 2007; Fei et al. 2006; Mehta 2008; Rao et al. 2015; Simončič & Klemenčič, 2016; Vigneshwaran et al. 2006).

The protective clothes could be identified as textile materials that are mainly applicable to protect human body from any external threat. Moreover, there are vital safety issues correlating to their applications as well as the disposal of chemical reagents used for their contemporary finishing. Therefore, the researchers considered with the field of textile industry were continued to look for alternative finishing reagents and technologies that must be environment friendly, with high fastness, low cost and not disadvantageously affect the comfort of clothes while providing efficiency and optimum protection (Baglioni et al. 2005; Hassabo et al. 2019; Subash et al. 2012; Wong et al. 2006; Zhang et al. 2018). Cotton fabrics

were exploited in different applications and purposes owing to their outstanding properties like biodegradability, absorbability, softness, and breathability, but disadvantageous with prone to be attacked with microbes especially bacterial strains, poor ultraviolet shielding, and high flammability (Goncalves et al. 2009; Li et al. 2007a, b; Li et al. 2007a, b).

Numerous approaches were considered with investigating different techniques for acquirement different textile materials with number of additional functions, like coloration (Ahmed et al. 2018; Emam & Abdelhameed 2017), self-cleaning (Emam et al. 2018; Emam et al. 2020a, b, c; Rehan et al. 2013), optical activity (Emam et al. 2018), insect repellency (Abdel-Mohdy et al. 2008; Abdelhameed et al. 2017), microbicidal activities (Ahmed et al. 2017a, b; Emam et al. 2020; Ibrahim et al. 2021), electromagnetic interference (EMI) shielding (Gao et al. 2021; Tan et al. 2018; Wang et al. 2021), and ultraviolet shielding (Ates & Unalan 2012; Emam, et al. 2020; Khan et al. 2015; Nazari et al. 2013). In addition to, the protective masking and air-filtering textiles have been prepared for protection from the chemical warfare gases and produced via the immobilization of different functionalizing reagents, like lipophilic activated carbon with efficient adsorption action (X. Li et al. 2011).

Numerous reports were considered with the exploitation of some organic reagents for textiles functionalization, like triclosan for antibacterial potency, benzophenones for ultraviolet shielding, dimethylol dihydroxy ethylene urea for anti-crease performance, fluorocarbons for lipophilic characters, long-chain hydrocarbons and polydimethylsiloxanes for flexibility and softening (Almeida 2006; Hewson 1994). Butane tetra-carboxylic acid, citric acid and maleic acid (Welch 1988; Yang et al. 2010; Yang et al. 1998) were also exfoliated for acquiring cotton fabrics with anti-crease action.

Different types of metallic based nanomaterials were reported to efficiently applicable in various fields (Ali et al. 2021; Karatepe et al. 2021; Tümen et al. 2021), especially sewage treatment, chemical synthesis, hydrogen storage, exhaust gas treatment, and oil refinement (Adams & Chen 2011; Hughes et al. 1995; Lee et al. 2010; Mackus et al. 2015; Meyer et al. 2015; Moon et al. 2014; Zaluski et al. 1995). The priority of nanosized materials is attributed to their high surface area per volume ratio,

highly organized composition, high density of coordinative sites, high oxidation activity, and superior mechanical and thermal stabilities (Cano et al. 2017).

Numerous studies were demonstrated textiles functionalization via immobilization of various noble-metal nanostructures such as silver (Ahmed et al. 2018; Emam et al. 2016a, b; Rao et al. 2015; Simončič & Klemenčič, 2016), gold (Hanan B Ahmed et al. 2017a, b; Emam et al. 2017), titanium dioxide (Durán et al. 2007; Fei et al. 2006; Vigneshwaran et al. 2006) and zinc oxide (Baglioni et al. 2005; Hassabo et al. 2019; Zhang et al. 2018). However, palladium (Pd) nanostructures were reported to be efficiently applicable in catalysis (Abdelhameed et al. 2020; Emam & Ahmed 2019; Emam et al. 2020a, b, c; Emam et al. 2020a, b, c; Lim et al. 2010). Palladium was successfully applied as catalyst for facilitating the hydrogen absorptivity and detection owing to its high affinities in absorption of hydrogen (Adams & Chen 2011; Lee et al. 2010; Moon et al. 2014; Rikkinen et al. 2011; Zaluski et al. 1995). Moreover, palladium nanostructures were found to be more efficient in removal of dyes from the aqueous media via the heterogenous catalysis than many typically applied methodologies, like filtration, biological treatment, chemical precipitation, adsorption and techniques. According to our knowledge, no researching studies were considered with exploitation of palladium nanostructures in textile functionalization.

Herein, a novel/investigative approach for preparation of excellent ultraviolet protective cotton fabric is uniquely proposed via self-implantation of palladium nanoclusters. Whereas, for the first time, the immobilized palladium nanoclusters were functionalized in acquirement of the treated fabrics excellent ultraviolet shielding potency. The particle size of the dispersed palladium nanoclusters in supernatant solution was estimated from transmission electron microscopic images. Regulative implantation of palladium nanoclusters was proceeded via immobilization within the polymeric matrix of both native and cationized cotton fabrics. Afterward, the modified fabrics were characterized via infrared spectroscopy (FTIR), scanning electron microscopy (SEM), X-ray diffraction (XRD), colorimetric measurements (color coordinates and color strength), mechanical properties (tensile strength and elongation percentage), and UV-protective action (transmittance percent, ultraviolet protection factor UPF and ultraviolet protection rating).

Experimental work

Materials and chemicals

Palladium chloride (PdCl_2 , 99%, from Sigma-Aldrich – USA), Sodium hydroxide (99%, from Merck, Darmstadt–Germany), acetic acid and sodium carbonate were of laboratory grade chemicals. 3-Chloro-2-hydroxypropyl trimethyl ammonium chloride (69%) of technical grade chemicals (known as Quatt-188) was purchased under the commercial name CR-2000 from Aldrich. All the chemicals were used as supplied without any further purification. Mill de-sized, scoured and bleached cotton fabric, plain weave, supplied by El-Nasr Company for spinning weaving and Dyeing El-Mahallah El-Kubra, Egypt. The fabric was further purified in the laboratory by washing at 100 °C for 60 min using a solution containing 2 g/L, Na_2CO_3 and 1 g/L, non-ionic surfactant. Lastly, the fabrics were washed several times with boiling water then with cold water and finally dried at ambient conditions.

Procedure

Cationization of cotton fabrics

Chemical modification of the cotton gauze through cationization was carried out according to literature (Hashem et al. 2009, 2005). The experimental procedure adopted was as follows: 3-Chloro-2-hydroxypropyl trimethyl ammonium chloride (Quat-188) was

mixed with sodium hydroxide solution with a molar ratio of 2 (NaOH):1 (Quat-188). The cotton gauze samples were padded in this mixture in two dips and two nips, and then squeezed to a wet pick-up of about 100%. The cotton gauze was dried at 40 °C for 10 min and cured at 120 °C for 3 min. Thus, treated cotton gauze samples were washed with cold water and 1% acetic acid, followed by several washing cycles and finally dried under the normal laboratory conditions.

Self-implantation of palladium nanoclusters

The palladium nanoclusters were self-implanted in to the native and cationized cotton fabrics by dipping method. In this procedure, pieces of cotton fabrics (0.25 g) were impregnated in distilled water and stirred till temperature reached 90 °C then palladium salt solution with specific concentration (20 and 60 mM) was added with liquor ratio of 2:50 at two different pH (2 & 11.5) and left for continuous stirring at 90 °C for 30 min. Afterward, the fabric pieces were air dried before they were instrumentally analyzed. Table 1 represented the samples that were prepared in the current approach under different experimental conditions.

Temperature = 90 ± 3 °C, Time = 30 min, where, 50 and 100 referring to the experimental percent of Quaternary ammonium salt used for cationization.

Characterization and instrumental analysis

Geometrical shape and size distribution of the self-implanted palladium nanoclusters were estimated by using a high-resolution transmission electron microscope (JEOL-JEM-1200; Japan). Size distribution of palladium nanoclusters was evaluated with 4 pi analysis software (from USA) for at least 50 particles. The treated fabrics were characterized via the high-resolution scanning electron microscope (HRSEM Quanta FEG 250 with a field emission gun, FEI Company, Netherlands). Elemental analysis was also estimated using an energy dispersive X-ray analyzer (EDAX AME-TEK analyzer). The infrared spectra of the treated fabrics were obtained by using a Jasco FT/IR 6100 spectrometer. Their spectral mapping data were ranged from 4000 to 400 cm^{-1} and were determined with 4 cm^{-1} resolution and 64-time scanning with a rate of 2 mm/sec. Additionally, the prepared fabrics

Table 1 Description of the Pd-modified cotton fabrics

Samples	Fabric	PdCl_2 (mM)	pH
Pd-C1	Cotton	20	2.0
Pd-C2	Cotton	60	2.0
Pd-C3	Cotton	20	11.5
Pd-C4	Cotton	60	11.5
Pd-CC (50)1	Cationic (50) Cotton	20	2.0
Pd-CC (50)2	Cationic (50) Cotton	60	2.0
Pd-CC (50)3	Cationic (50) Cotton	20	11.5
Pd-CC (50)4	Cationic (50) Cotton	60	11.5
Pd-CC (100)1	Cationic (100) Cotton	20	2.0
Pd-CC (100)2	Cationic (100) Cotton	60	2.0
Pd-CC (100)3	Cationic (100) Cotton	20	11.5
Pd-CC (100)4	Cationic (100) Cotton	60	11.5

were characterized by powder X-ray diffraction using X'Pert MPD diffractometer system from Philips, at room temperature. Diffraction patterns were estimated in the diffraction angle (2θ) range of 3.5–50° using monochromatized (Cu $K\alpha$ X-radiation at 40 kV, 50 mA and $\lambda = 1.5406 \text{ \AA}$).

The colorimetric data (L, a^* , b^* , absorbance, color strength [K/S], whiteness index E313 [D65/10] and yellowness index E313 [D65/10] of the treated fabrics were estimated using a spectrophotometer attached with a pulsed xenon lamp (UltraScan Pro, Hunter Lab, USA). The color coordinate parameters of L^* , a^* , and b^* corresponded to the lightness (black/white, 0/100), red/green ratio (+/–), and yellow/blue ratio (+/–), respectively (Hanan B Ahmed et al. 2017a, b; Ahmed et al. 2018). The measurement was performed three times for estimation of the average values. The mechanical properties of the treated fabrics were investigated, while, tensile strength (MPa) and elongation at break (%) for the fabrics were estimated related to ASTM method D2256–66 T by using the strip test methodology on the tester instrument (Asano machine MFG. Co. Ltd., Japan). Transmission spectral results (T%) for ultraviolet irradiation

(UVR) over pristine and cationized cotton before and after the successive implantation of palladium nanoclusters were estimated using JASCO V-750 spectrophotometer (Japan) in the range of 280–400 nm with two nanometers interval. Additionally, ultraviolet protection factor (UPF), resistance in UV-A (315–400) region (UVA) and in UV-B (280–315) region (UVB) were predicted using the AATCC test method 183–2010.44. For each sample, estimation was estimation for two different times, and the average was calculated.

Results and Discussion

Synthesis of palladium nanoclusters

In spite of immobilization of various metallic nanoobjects for textile finishing was extensively studied (Baban et al. 2010; Gorenšek & Recelj 2009; Gulrajani et al. 2008; Ko et al. 2007; Maneerung et al. 2008; Perelshtein et al. 2008; Saengkiettiyut et al. 2008; Wu et al. 2010), but there were no researching approaches that were considered with exploitation of

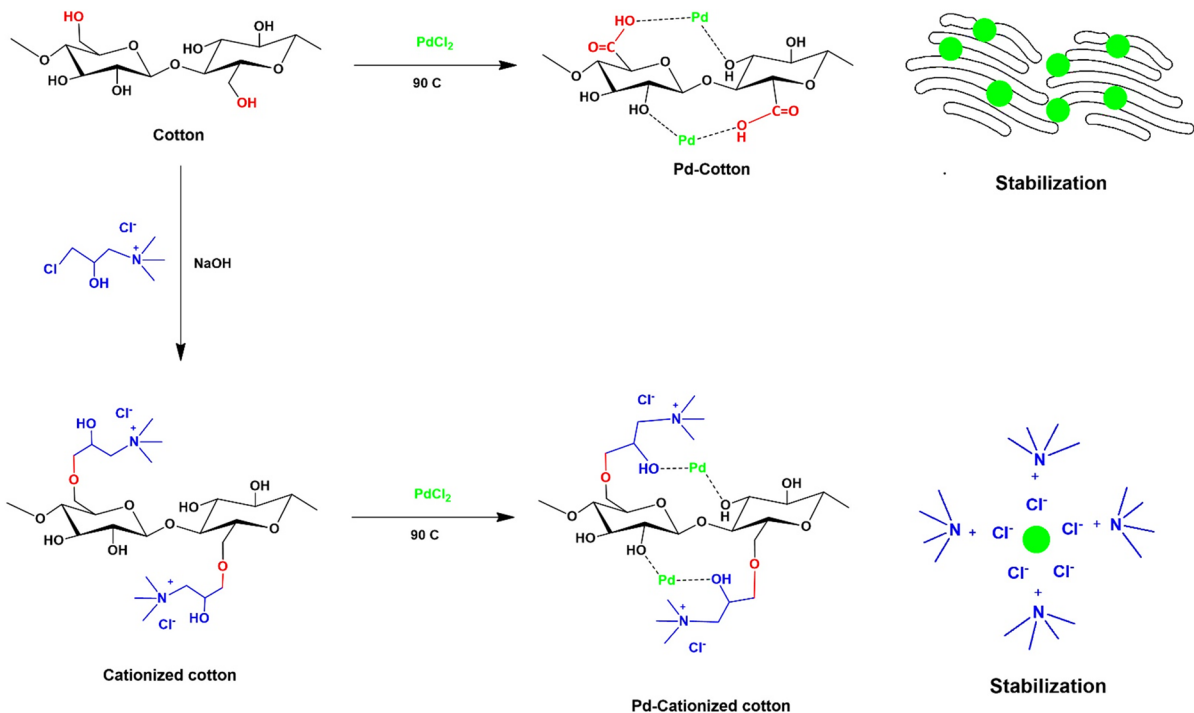


Fig. 1 Schematic for formation of Pd-modified cotton fabrics

Fig. 2 TEM images for the supernatant solution of Pd after modification of cotton fabrics; **a** Pd-C3, **b** Pd-CC (50)3 and **c** Pd-CC (100)3

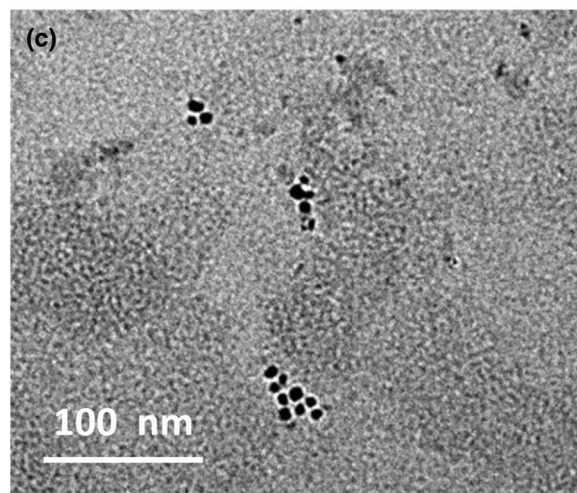
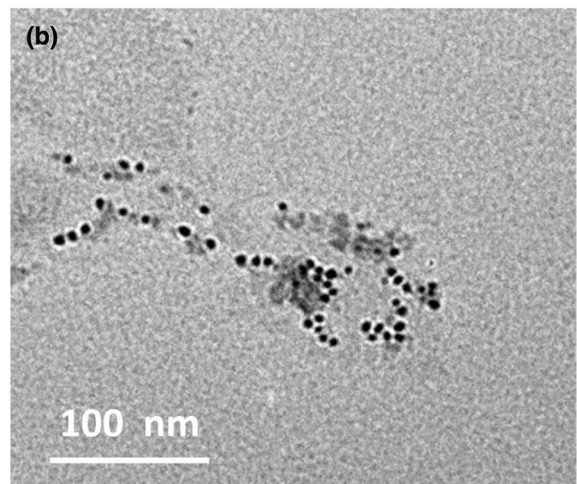
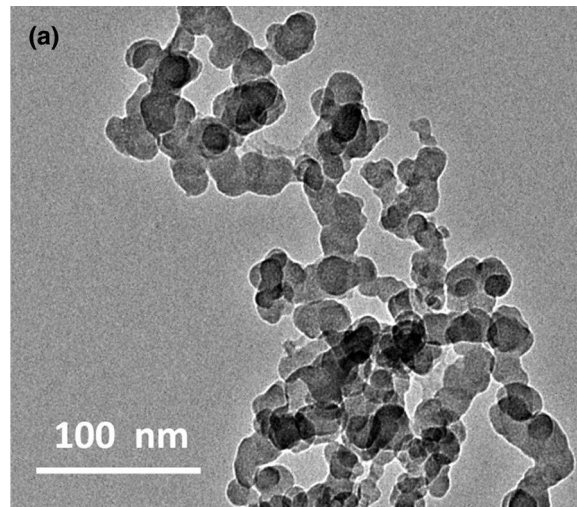
palladium nano-objects for textile finishing. Additionally, no researching reports were studied the direct implantation of palladium nanoclusters within textile matrix. The main challenge of the current approach is to study investigate the superior role of palladium nanoclusters in textile functionalization. Herein, the leverage effect for the direct implantation of palladium nanoclusters on coloration, mechanical properties and UV-protection efficiency of cotton fabrics was systematically studied.

In accordance to literature (H. B. Ahmed et al. 2017a, b; Ahmed et al. 2018; Emam et al. 2016a, b), self-implantation of palladium nanoclusters within cotton fabrics could be hypothesized as the cellulosic building blocks of cotton fabrics with the terminal/ alcoholic groups might assist in reduction of palladium ions for ingrainings of palladium nanoclusters that could be highly stabilized within the intermolecular spaces of cotton polymeric matrix. Moreover, fabric cationization was performed for cotton before self-implantation of palladium nanoclusters in order to achieve the fulfill goal. Fabric cationization is supposed to enhance the implantation and stabilization of palladium nanoclusters within fabric matrix, while, such chemical modification acquired the fabric more accessible reducing groups to act more actively for ingrowth and stabilization of the requisite nanostructure (Fig. 1).

In more details, according to literature (Fürstner 2008; Kiwi & Graetzel 1979) that were considered with stabilization of metal nanoparticles by ionic liquids, it could be depicted that, cationization of fabrics with quaternary ammonium salts acted in higher efficiency for stabilization of nanoclusters via the electrochemical and steric conceptions, as halogen ions were supposed to be very strongly adsorb on the surface of palladium nanoclusters, while, the surrounding quaternary alkylammonium cations were suggested to constrain close contact between the nanoclusters.

TEM and size distribution

The geometrical features and topography of the implanted palladium nanoclusters that were stably dispersed within the supernatant solution containing



the treated fabrics were shown in transmission electron microscopic images (Fig. 2) from which the size distribution (Supplementary file, Fig. S1) was estimated. The microscopic images displayed the successful implantation of palladium nanoclusters with size distribution of 41.3 ± 10.1 nm in alkaline medium (pH of 11.5) in the supernatant solution of native cotton treated with 20 mM of palladium chloride. Quite smaller sized palladium nanoclusters (3.2 ± 0.6 nm) were ingrained and well dispersed within the supernatant solution of cationized fabric treated with palladium precursor under the same experimental conditions, reflecting the pre-eminent effect of cationization in enhancement the accessibility of fabric/cellulosic backbone in the ingrowth of palladium nanoclusters. While, duplication of the percentage of quaternary ammonium salt exploited for cationization from 50 to 100% is insignificantly affected on particle size (4.6 ± 1.7 nm).

SEM

The topography of the surface of the pristine and cationized fabrics before and after implantation of palladium nanoclusters. In order to show the effects of palladium precursor concentration and cationization on the successive implantation of palladium nanoclusters, scanning electron microscopic images (SEM images), energy dispersive x-ray (EDX signals), and the elemental analysis (supplementary file, Fig. S2) of cationized cotton, Pd-C4, Pd-CC(50)4, Pd-CC(100)3 and Pd-CC(100)4 were plotted in Fig. 3. Before successive implantation of palladium nanoclusters, the surface of the cationized fabric seemed to be smoothy, and the specialized signals of carbon, oxygen, nitrogen and chlorine were obviously detected (Fig. 3a). After implantation of palladium nanoclusters, the nanoclusters were clearly dispersed on the surface of the modified fabrics. Whereas, from EDX data, the characteristic peaks of carbon, oxygen, nitrogen, chlorine, and palladium were significantly shown, that affirmed the successive implantation of nanoclusters within the fabrics (Fig. 3b). Moreover, cationization were shown to extensively enhance the implantation of palladium nanoclusters to be regulatory uploaded on the fabric surface as shown in Fig. 3b&c, while, duplication of the cationization percent from 50% (Fig. 3c) up to 100% (Fig. 3e) resulted in more accessibility for implanting of more

condensed nanoclusters, which could confirm the postulated mechanism of palladium nanoclusters implantation. By comparing between the microscopic images of the modified fabrics after implantation of palladium nanoclusters (Fig. 3d & e), it is obviously shown that, increment of the concentration of palladium precursor resulted in more dense masses of nanoclusters on the surface of the fabric.

FTIR

The chemical compositions of quaternary ammonium salt, pristine cotton, cationized cotton and fabrics immobilized with palladium nanoclusters were investigated via FTIR (Fig. 4). From the spectral data it could be depicted that the quaternary ammonium salt, pristine and cationized cotton were characterized with absorption signals of O–H ($3330\text{--}3289$ cm^{-1}) and aliphatic C–H (2888 cm^{-1}) and weak bands of C=O (1620 cm^{-1}), asymmetric C–O–C (1472 cm^{-1}), and C–C (1072 cm^{-1}) (Emam & Abdelhameed 2017; Emam & Bechtold 2015). After implantation of palladium nanoclusters, all of the absorbance bands were retained and new band of O–Pd (833 cm^{-1}) was obviously detected, that affirmed the cooperation of the fabric functional groups in stabilization of palladium nanoclusters. Additionally, the absorption bands of C=O (1620 cm^{-1}), H–C–H, and asymmetric C–O–C became more intense, that also affirmed the role of the fabric accessible groups in reduction and stabilization of palladium ions for successive implantation of palladium nanoclusters.

XRD patterns

The aforementioned illustration affirmed the successful implantation of palladium nanoclusters within the fabric matrix through chemical bonding. Figure 5 is plotted for XRD data of cotton and cationized cotton before and after implantation of palladium nanoclusters, and from the represented data it could be declared that, cotton was detected with three characteristic bands at $2\theta = 14.9^\circ$, 16.7° and 21.5° corresponding to the crystalline structure of cellulose. While, after immobilization of palladium nanoclusters, two characteristic peaks for palladium Pd (111) and Pd (200) were detected at $2\theta = 31.8^\circ$ and 45.4° , respectively, for face centered crystalline Pd (JCPDS data number 89–4897 card) (Emam & Ahmed 2019;

Fig. 3 SEM photos for the Pd-modified cotton fabrics; **a** cationized cotton, **b** Pd-C4, **c** Pd-CC (50)4, **d** Pd-CC (100)3 and **e** Pd-CC (100)4

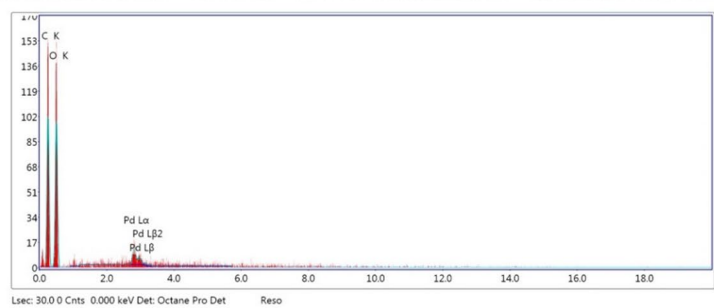
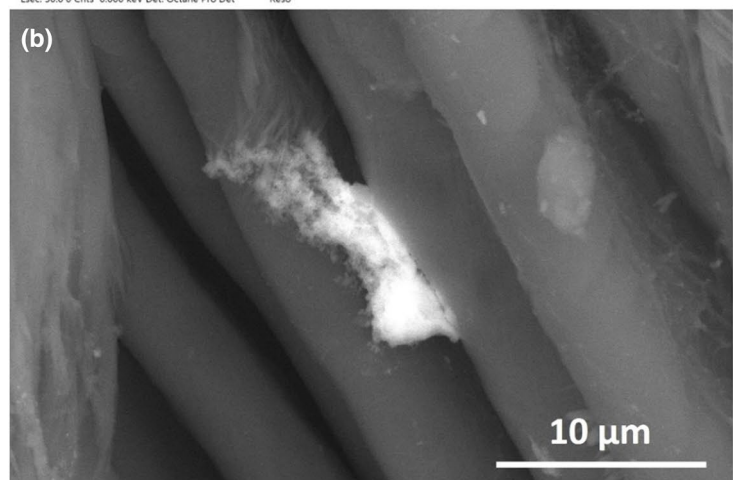
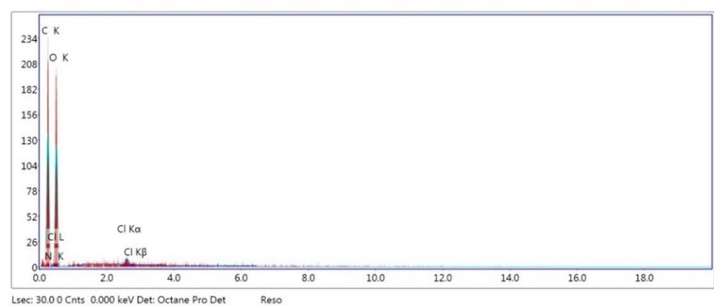
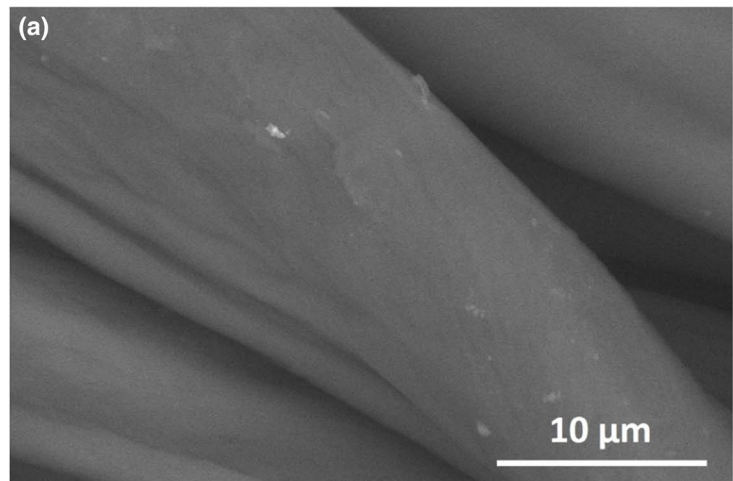


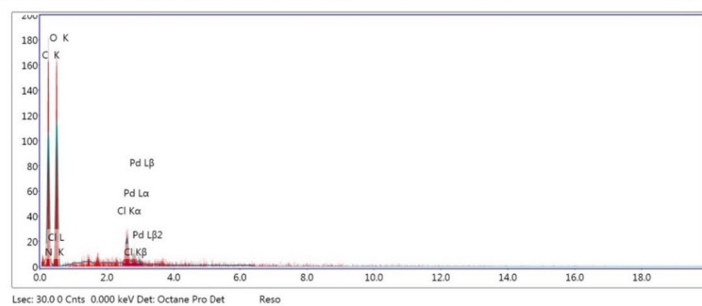
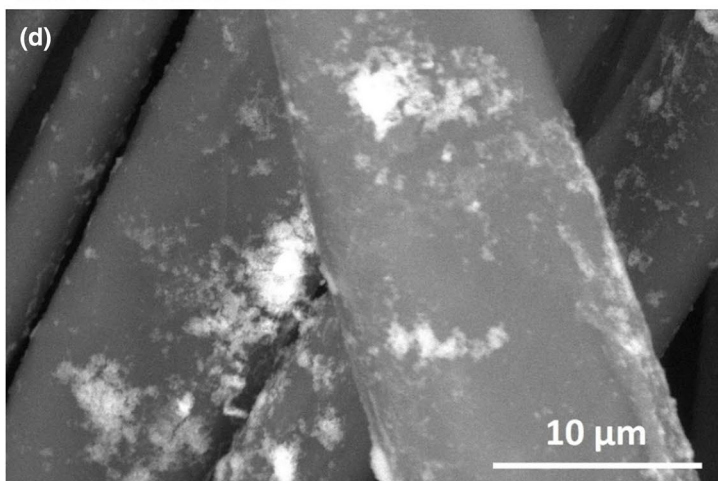
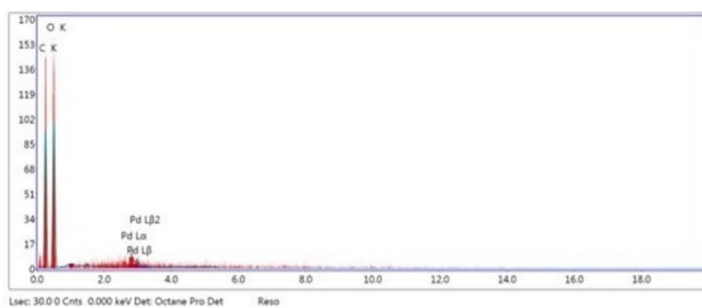
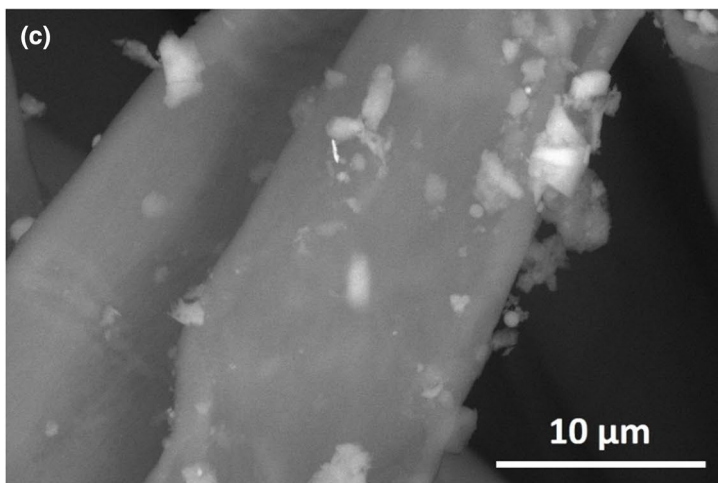
Fig. 3 (continued)

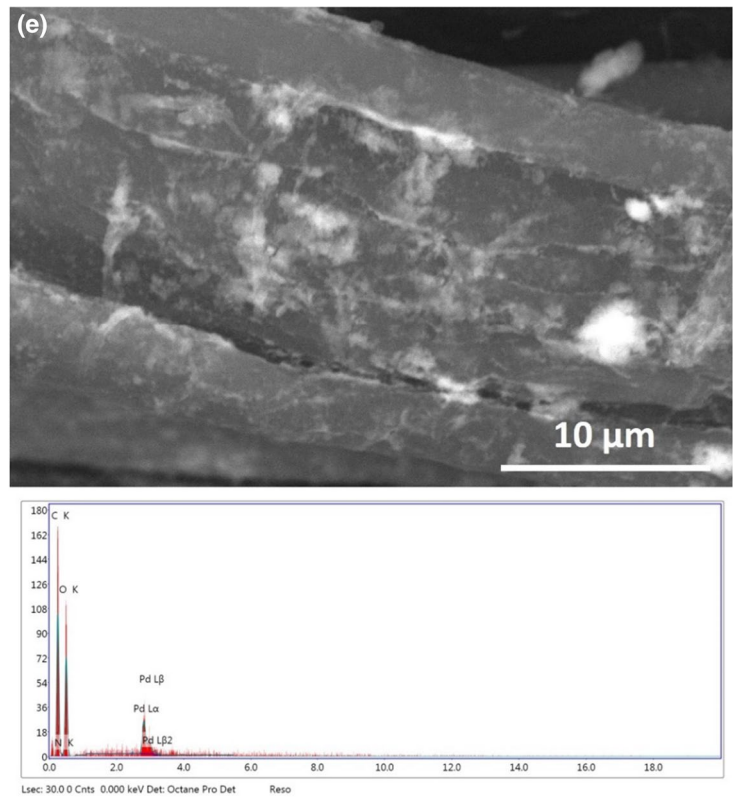
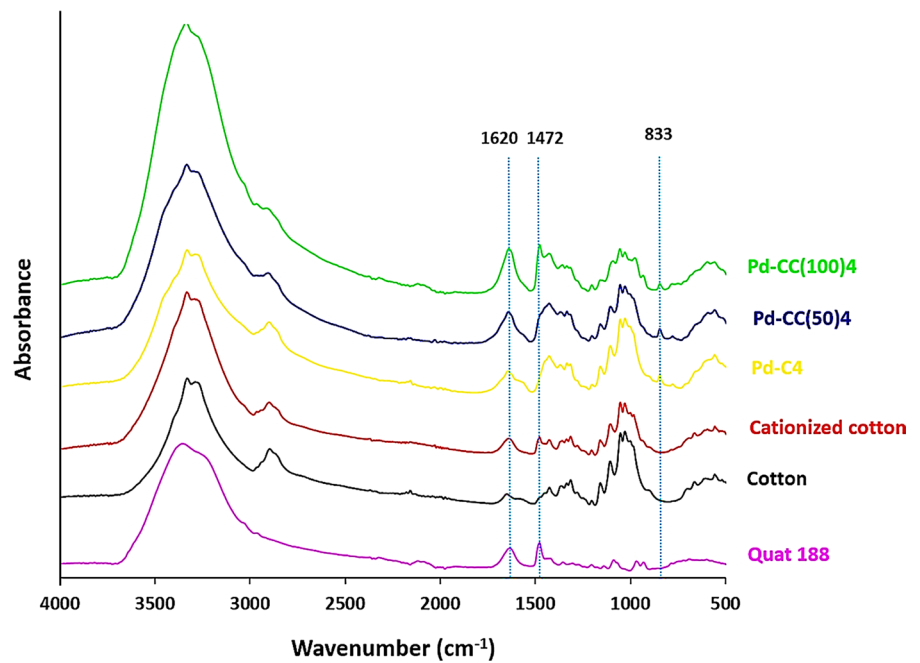
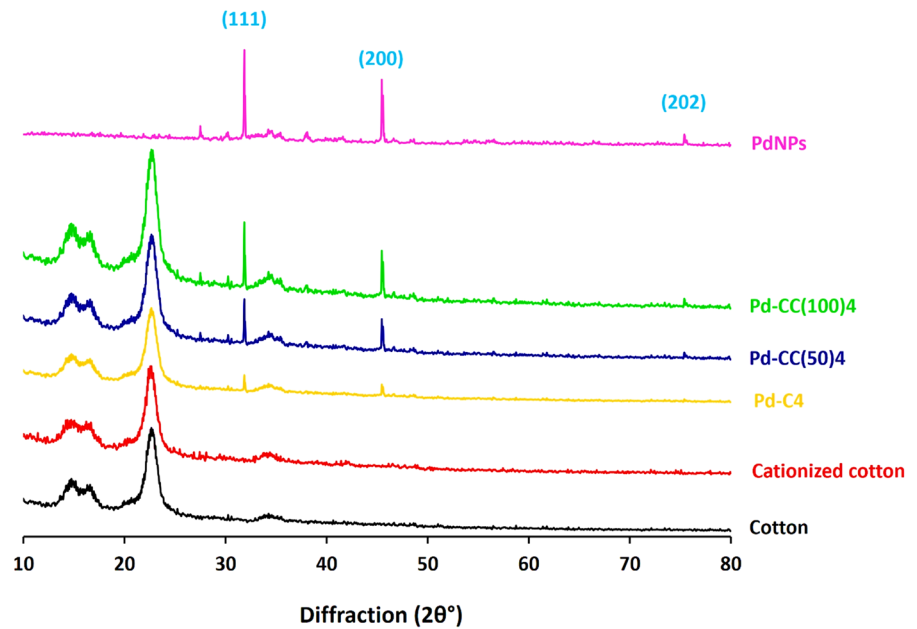
Fig. 3 (continued)**Fig. 4** FTIR for the Pd-modified cotton fabrics

Fig. 5 XRD patterns for the Pd-modified cotton fabrics



Emam, et al. 2020a, 2020b, 2020c; P. Liu et al. 2016a, 2016b; Liu et al. 2016a, 2016b). Moreover, for cationized cotton modified with palladium nanoclusters, an additional peak for Pd (202) at $2\theta = 75.4^\circ$ was appeared, to affirm the effect of cationization in increment the accessibility of fabric for regulative implantation for more of palladium nanoclusters.

Colorimetric data and mechanical properties

From the visual observation of the samples imaged in Fig. 6 it could be noted that, treatment of fabrics with the demonstrated technique resulted in fabric coloration, while, the color was changed from white for native cotton to creamy white with cationization, and under different experimental conditions, the fabric color was developed to yellowish, brownish and lastly to dark black color. The color data of the pristine cotton and cationized cotton after implantation of palladium nanoclusters are presented in Table 2 and supplementary file (Fig. S3). From the plotted data in Table 2, Yellowness index (YI) of the cationized cotton was extremely higher than that of pristine cotton fabrics. For fabrics prepared under alkaline condition, with increment of the concentration of palladium precursor and with duplication of the cationization percentage up to 100%, the lightness (L^*) and whiteness

index (WI) were significantly decreased, while, color strength (K/S), darkness (b^*) and yellowness index were significantly increased.

From the plotted results in Fig. 6, it could be observably shown that, cationized fabrics were exhibited with higher absorbance and color strength values. Additionally, absorbance and color strength were extensively higher for fabrics prepared under alkaline conditions rather than that prepared under acidic pH with increment of palladium salt concentration and duplication of cationization percentage from 50% up to 100%. So, it could be summarized that, yellowness degree, absorbance and color strength could be ordered as follows: Pd-CC (100)4 > Pd-CC (100)3 > Pd-CC (50)4 > Pd-CC (50)3 ≥ Pd-C4 > Pd-C3 > cationized cotton > cotton.

The effects of palladium nanoclusters implantation within the polymeric matrix for both of pristine and cationized cotton on the mechanical properties were monitored via estimation of tensile strength and elongation percentage and the results were tabulated in Table 3. Cationization of fabrics and immobilization of palladium nanoclusters resulted in decrement in the tensile strength and elongation percentage. Retention of Elongation percentage and slight diminishing in tensile strength were observed with duplication of cationization percentage. Cationized fabrics immobilized with palladium nanoclusters under alkaline

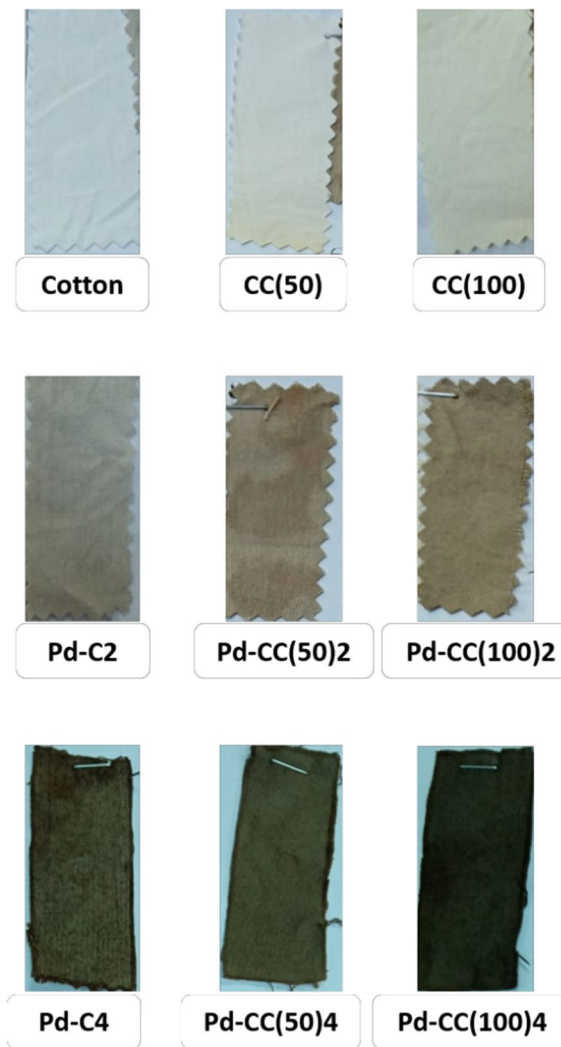


Fig. 6 Photographs for the Pd-modified cotton fabrics

conditions were characterized with lower tensile strength and elongation percentage than that prepared under acidic conditions. Increment of palladium concentration from 20 up to 60 mM resulted in nonsignificant decrement in tensile strength and retention of elongation. This could be logically attributed to that, the chemical medication and immobilization of palladium nanoclusters within the intermolecular spaces of fabric polymeric matrix, resulting in bond breaking within the polymeric chains. However, the decrement in tensile strength for the fabrics after implantation of palladium nanoclusters could be acceptable for application as clothing materials.

UV protection

Cotton based textile materials were widely applicable in various purposes owing to their outstanding characters like biodegradability, softness, and breathability, but they are disadvantageous with prone to microbial attacking, low capability in protection from ultraviolet irradiation, and high flammability (Goncalves et al. 2009; Q. Li et al. 2007a, 2007b; Y. Li et al. 2007a, 2007b). It was recently reported that special metallic oxides like, titanium dioxide, silicon dioxide, zirconium dioxide, magnesium dioxide and zinc oxide, in addition to some of polymeric materials were successfully applicable for coating of cotton fabrics to acquire the treated fabrics characteristic functional properties (Charpentier et al. 2012; Gandhi et al. 2010; Hojjati et al. 2007).

Ultraviolet protection factor (UPF, Table 4) and transmission percent (T%, supplementary file, Fig. S4) could be ascribed as key factors for evaluation of the ultraviolet protection action for all the prepared samples. Ultraviolet shielding property was analyzed within wavelength range of 280–400 nm, as shown in supplementary file (Fig. S4). Transmittance percent of cotton fabric is ca. 66% and 83.3% for both UV-A and UV-B, respectively. The blocking percent of the treated fabrics indicated with minor transmittance of UV radiation compared to untreated ones (UVA blocking percent 34.0% & UVB blocking 16.7%), i.e., T% of all the prepared samples was observably diminished after implantation of palladium nanoclusters compared to that of the untreated fabric (UVA T% 66.0 & UVB T% 83.3). The blocking percentage of treated cotton fabrics showed that the percent of blocking for UV-B radiation was higher than that of UV-A for all samples prepared from native cotton, in contrast to that prepared from cationized cotton (Table 4). The increment of concentration of palladium precursor (UVB T% for Pd-C3 was 1.6%, while, for Pd-C4 was 1.0%) and processing the palladium implantation under alkaline conditions resulted in lowering the detected transmission percent. Therefore, Pd-C4 sample (UVA T% 0.9 & UVB T% 1) was exhibited by the highest UVA & UVB blocking percent of 99.1% and 99.0%, respectively, compared to the other three samples pristine cotton.

Figure S4 (supplementary file) demonstrated that, the decrement of transmission was significantly higher for the cationized cotton samples compared to

Table 2 Colorimetric data for the Pd-modified cotton fabrics

Fabric samples	L*	a*	b*	WI E313 [D65/10]	YI E313 [D65/10]	K/S (370 nm)
Cotton	86.49±0.98	1.02±0.12	- 2.65±0.21	81.28±2.23	- 4.81±0.43	0.58±0.22
Pd-C1	75.78±0.74	1.18±0.23	6.08±0.78	16.31±2.01	14.76±1.73	1.06±0.42
Pd-C2	71.39±0.96	1.72±0.42	7.76±1.10	- 1.75±0.49	19.83±1.88	1.65±0.65
Pd-C3	67.46±1.45	3.16±0.76	11.41±1.15	-27.86±2.17	31.35±2.16	8.70±1.23
Pd-C4	63.54±1.24	4.59±0.54	15.06±1.92	- 53.97±4.48	42.88±2.60	9.39±1.38
Cationized (50) Cotton	84.05±0.30	0.43±0.08	2.59±1.01	52.39±4.08	5.83±1.51	0.72±0.35
Pd-CC (50)1	60.30±0.66	5.12±0.53	15.59±1.82	- 72.05±5.58	45.06±2.52	3.1±0.54
Pd-CC (50)2	57.41±0.45	6.20±0.86	19.54±1.56	- 89.11±5.14	56.93±3.35	4.5±0.68
Pd-CC (50)3	45.18±0.72	7.05±1.02	20.39±2.21	- 101.00±6.28	63.76±3.15	9.64±0.88
Pd-CC (50)4	41.99±0.83	7.76±1.00	21.61±2.04	- 109.01±6.84	65.04±3.06	11.82±1.5
Cationized (100) Cotton	83.13±0.40	0.10±0.10	7.53±1.21	24.14±1.82	15.67±1.52	0.72±0.38
Pd-CC (100)1	54.96±0.83	5.22±0.59	17.21±1.44	- 91.36±3.86	51.47±2.26	3.24±0.62
Pd-CC (100)2	45.19±0.37	6.44±0.88	18.97±1.72	- 101.00±4.25	59.75±2.77	5.2±0.83
Pd-CC (100)3	41.99±0.44	7.58±0.40	21.05±1.92	- 119.79±5.66	70.04±3.31	11.17±1.53
Pd-CC (100)4	39.70±0.52	8.22±0.72	24.03±2.20	- 124.25±4.83	74.99±2.80	15.25±1.88

Table 3 Mechanical properties of the Pd-modified cotton fabrics

Samples	Tensile strength (MPa)	Elongation (%)
Cotton	107.9	17
Pd-C1	104.1	16
Pd-C2	109.2	16
Pd-C3	106.5	15
Pd-C4	103.7	13
Cationized (50) Cotton	99.1	15
Pd-CC (50)1	97.7	15
Pd-CC (50)2	95.4	16
Pd-CC (50)3	96.0	14
Pd-CC (50)4	93.6	14
Cationized (100) Cotton	93.2	13
Pd-CC (100)1	94.4	14
Pd-CC (100)2	90.3	13
Pd-CC (100)3	87.0	12
Pd-CC (100)4	84.5	12

that prepared from native cotton. Duplication of cationization percent and alkalinity were notably affected on diminishing of transmission percent, whereas, increment of concentration for palladium salt under alkaline conditions was shown to insignificantly affect the transmission. The estimated results showed that UVA T% & UVB T% were sharply decreased

from 34.6 to 41.4% for cationized (50) cotton (UVA blocking 53.4% & UVB blocking 41.4%) to 2.7% & 1.8% for Pd-CC (50)2 (UVA blocking 97.3% & UVB blocking 98.2%) and superiorly to 0.7% and 0.5% for Pd-CC (50)3 (UVA & UVB blocking 99.3%) and Pd-CC (50)4 (UVA & UVB blocking 99.5%), respectively. Whereas, the lowest transmission percent of 0.4% for all the prepared samples was exhibited by Pd-CC (100)4 (the highest UVA & UVB blocking percent of 99.6%). These could affirm the effects of cationization, alkalinity and concentration of palladium precursor in implantation of greater amounts from palladium nanoclusters

UPF & UV protection rating were also evaluated in accordance to the estimated values of transmission percent (Table 4). The UPFs of native cotton, cationized (50) cotton and cationized (100) cotton fabrics were 1.3, 5.6 and 8.1, respectively. They were estimated to increase up to 103.4, 198.9 and 256.6 for Pd-C4, Pd-CC (50)4, and Pd-CC (100)4, respectively. The highest UPF was found in Pd-CC (100)4 to affirm the superior cationization effect on the efficient implantation of higher amounts of palladium nanoclusters within the fabric matrix to reveal higher blocking action for ultraviolet radiation, while, palladium nanoclusters were superiorly acted as excellent UV absorbers for preparation of UV protective textiles. Moreover, the demonstrated data for ultraviolet protection rating it could be declared that, all the

Table 4 Ultraviolet protection results for the Pd-modified cotton fabrics

Samples	UVA (T%)	UVB (T%)	UVA Blocking	UVB Blocking	UPF
Cotton	66.0	83.3	34.0	16.7	1.3
Pd-C1	13.4	13.5	86.6	86.5	7.4
Pd-C2	4.0	4.2	96.0	95.8	24
Pd-C3	1.6	1.6	98.4	98.4	76.2
Pd-C4	0.9	1.0	99.1	99.0	103.4
Cationized (50) Cotton	34.6	41.4	53.4	46.6	5.6
Pd-CC (50)1	2.9	2.3	97.1	97.7	39.8
Pd-CC (50)2	2.7	1.8	97.3	98.2	50.1
Pd-CC (50)3	0.7	0.7	99.3	99.3	147.8
Pd-CC (50)4	0.5	0.5	99.5	99.5	198.9
Cationized (100) Cotton	15.3	11.6	84.7	88.4	8.1
Pd-CC (100)1	2.1	1.2	97.9	98.8	76.2
Pd-CC (100)2	1.9	1.2	98.1	98.8	76.2
Pd-CC (100)3	0.8	0.7	99.2	99.3	137.9
Pd-CC (100)4	0.4	0.4	99.6	99.6	256.6

Table 5 Colorimetric data for the Pd-modified cotton fabrics after washing

	Samples	L*	a*	b*	WI E313 [D65/10]	YI E313 [D65/10]	K/S (370 nm)
5 Washings	Pd-C3	76.63 ± 1.22	2.13 ± 0.44	4.45 ± 0.82	25.92 ± 3.23	13.43 ± 1.21	4.99 ± 0.63
	Pd-C4	74.67 ± 1.04	2.85 ± 0.18	6.28 ± 0.90	12.87 ± 1.68	19.19 ± 1.83	5.11 ± 0.81
	Pd-CC (50)3	58.29 ± 0.93	4.39 ± 0.64	14.08 ± 1.22	- 46.94 ± 2.27	37.80 ± 2.35	7.02 ± 0.82
	Pd-CC (50)4	54.73 ± 0.88	5.46 ± 0.72	16.51 ± 1.18	- 55.69 ± 3.55	46.20 ± 2.44	10.10 ± 1.12
	Pd-CC (100)3	49.70 ± 1.01	0.29 ± 0.22	5.42 ± 0.91	- 51.38 ± 2.52	44.26 ± 3.16	8.86 ± 1.27
	Pd-CC (100)4	48.56 ± 0.91	0.19 ± 0.25	0.94 ± 0.24	- 68.43 ± 3.43	53.55 ± 3.02	13.04 ± 1.76
10 Washings	Pd-C3	81.91 ± 1.42	1.53 ± 0.43	0.83 ± 0.12	54.39 ± 2.18	4.16 ± 1.01	2.14 ± 0.31
	Pd-C4	80.23 ± 1.05	1.97 ± 0.37	1.89 ± 0.32	46.28 ± 2.37	7.34 ± 1.23	2.37 ± 0.41
	Pd-CC (50)3	67.46 ± 0.72	3.26 ± 0.51	9.26 ± 1.02	- 12.73 ± 1.11	27.61 ± 1.94	5.95 ± 0.74
	Pd-CC (50)4	64.70 ± 0.48	4.15 ± 0.48	11.39 ± 1.14	- 27.78 ± 2.26	33.69 ± 2.22	7.80 ± 0.95
	Pd-CC (100)3	59.31 ± 0.85	4.85 ± 0.72	12.75 ± 1.46	- 28.00 ± 2.05	38.73 ± 1.92	7.40 ± 1.04
	Pd-CC (100)4	57.18 ± 0.67	5.53 ± 0.54	15.15 ± 1.77	- 36.47 ± 3.12	43.09 ± 2.09	10.12 ± 1.32

samples that were prepared under alkaline conditions showed very good to excellent rating, for substantiation the excellency of palladium nanoclusters as UV blocking structures.

Compared with other reported approaches in literature the UV protection properties of cotton fabrics that were currently prepared via the self-implantation of palladium nanoclusters are extensively higher than for fabrics prepared in literature via the immobilization of metallic based ultraviolet absorbers like silver, gold, zinc, copper, or titanium (Ahmed et al. 2017a, b; Emam & Bechtold 2015; Emam, et al. 2016a, b; El-Hady et al. 2021), or via the impregnation of

MOFs within the polymeric matrix of fabric (Emam & Abdelhameed 2017; Emam, et al. 2020). Therefore, the currently represented results suggested that palladium nanoclusters-coated fabrics could be expressed as potential candidates for ultraviolet shielding in textile functionalization, packaging applications and optoelectronics.

Durability

Table 5 represents the effect of repetitive washing cycles on the colorimetric data for the fabrics after

Table 6 Ultraviolet protection results for the washed Pd-modified cotton fabrics

	Samples	UVA (T%)	UVB (T%)	UVA Blocking	UVB Blocking	UPF
5 Washings	Pd-C3	3.9	3.9	96.1	96.1	22.4
	Pd-C4	3.7	3.8	96.3	96.2	23.5
	Pd-CC (50)3	2.5	2.6	97.5	97.4	40.6
	Pd-CC (50)4	2.4	2.4	97.6	97.6	41.4
	Pd-CC (100)3	1.6	1.6	98.4	98.4	72.4
	Pd-CC (100)4	1.2	1.4	98.8	98.6	75.2
10 Washings	Pd-C3	5.8	5.7	94.2	94.3	15.8
	Pd-C4	5.6	5.5	94.4	94.5	16.7
	Pd-CC (50)3	3.6	3.7	96.4	96.3	24.4
	Pd-CC (50)4	3.5	3.5	96.5	96.5	25.3
	Pd-CC (100)3	2.9	2.8	97.1	97.2	36.8
	Pd-CC (100)4	2.6	2.7	97.4	97.3	39.6

successive immobilization of palladium nanoclusters. From the tabulated data it could be depicted that, cationization acted in preparation of higher durable fabrics, as even after 10 washing cycles, yellowness index of the cationized cotton (Pd-CC (100)4, 43.09 ± 2.09) was extremely higher than that of non-cationized fabric (Pd-C4, 7.34 ± 1.23). Whereas, with duplication of the cationization percentage from 50% up to 100%, color strength (K/S) was significantly higher for fabrics prepared under alkaline condition with higher concentration of palladium precursor (Pd-C4, 2.37 ± 0.41 ; Pd-CC (50)4, 7.80 ± 0.95 ; Pd-CC (100)4, 10.12 ± 1.32). These could affirm the effect of cationization for stabilized immobilization of palladium nanoclusters within the fabric matrix. Table 6 displayed the effect of washing on the ultraviolet protection results for the fabrics modified with palladium nanoclusters. The obtained results approved the effect of cationization in successive implantation of palladium nanoclusters, stabilized within the fabric matrix with repetitive washings to prepare highly durable/functionalized fabrics. Whereas, the results showed that, even after 10 washing cycles, the ultraviolet protection factor rating was decreased from excellent (UPF, Pd-CC (100)4, 256.6) to very good (UPF, Pd-CC (100)4, 39.6) for the cationized fabrics, while, it was significantly lowered from excellent (UPF, Pd-C4, 103.4) to good (UPF, Pd-C4, 23.5).

Conclusion

In the current study, for the first time, self-implantation of palladium nanoclusters within cotton fabrics was proceeded in order to act as strong ultraviolet absorbers to acquire the treated fabrics excellent ultraviolet protection potency with full shielding effects. The self-implanted palladium nanoclusters were immobilized within the polymeric matrix of both native and cationized cotton fabrics. For all the prepared specimens, the effects of the concentration of palladium precursor, pH, and cationization percentage on the particle size of the implanted nanoclusters, in addition to, the color coordinates, yellowness index, whiteness index, color strength, tensile strength, elongation percentage were systematically studied. The effect of repetitive washing cycles on the colorimetric data and the results of ultraviolet protection were also represented. From all of the illustrated data, it could be summarized that, palladium nanoclusters acted superiorly as strong ultraviolet shielding sites within the fabric matrix. The yellowness degree and UV protection potency were shown to follow the trend of Pd-CC (100)4 > Pd-CC (50)4 > Pd-C4 > Cationized (100) Cotton > Cationized (50) Cotton > native cotton. Pd-CC (100)4 exhibited good mechanical properties and excellent ultraviolet protection potentiality, that could be attributed to the effect of cationization in the stronger implantation of palladium nanoclusters within the fabrics. It could be eventually reported that, the textile industry should address the unique presented challenge for production of excellent ultraviolet protective cotton fabrics with full

shielding effect, via self-implantation of palladium nanoclusters.

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Declarations

Conflict of interest The authors declare that they have no competing financial interest.

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