



E-ISSN: 2278-4136

P-ISSN: 2349-8234

<https://www.phytojournal.com>

JPP 2023; 12(5): 135-146

Received: 20-07-2023

Accepted: 25-08-2023

Author details are given below
the references.

***Impregnation of textile cotton material with Cymbopogon citratus*-mediated silver nanoparticles and investigations by light, electron and hyperspectral microscopies**

Jean Yves Sikapi Fouda, Agnes Antoinette Ntomba, Philippe Belle Ebanda Kedi, Thi Hai Yen Beglau, Marcus Fetzer, Till Strothmann, Tchangou Armel Florian, Sone Enone Bertin, Vandi Deli, Emmanuel Jean Teinkela Mbosso, Gustave Leopold Lehman, Emmanuel Albert Mpondo Mpondo, Gisele Etame Loe, Francois Eya'ane Meva and Christoph Janiak

DOI: <https://doi.org/10.22271/phyto.2023.v12.i5b.14722>

Abstract

The present study aims to design a cheap and environmentally friendly cotton textile impregnated with green synthesized silver nanoparticles from *Cymbopogon citratus* leaves. The silver nanoparticles were characterized by ultraviolet spectroscopy and powder X-ray diffraction. Analytical differences between impregnated and non-impregnated cotton textiles were assessed by light, and transmission electron microscopy (TEM). Hyperspectral microscopy reveals the presence of silver nanoparticles inside *in situ* impregnated cotton material and was coupled to emission spectroscopy to differentiate from cotton fabric giving insight for material quality control. TEM presents high-density dispersions of silver nanoparticles inside the cotton materials and lowest sizes for the *in situ* impregnated cotton material. The *in situ* nanomaterial shows differences in maximum emission compared to the cotton fabric, and the particles could be visualized by dark field microscopy. Water and nanoparticle solution retention capacities of the cotton fabrics were determined. The experiment suggests that the *in situ* impregnated cotton fabric had outstanding washing resistance.

Keywords: Cotton textile, Silver nanoparticles, *Cymbopogon citratus*, *Impregnation*, hyperspectral microscopy, washing resistance, transmission electron microscopy

Introduction

Nanotechnology is one of the most promising technologies of the 21st century. It is the theory of ability to convert the nanoscience to useful applications by observing, measuring, manipulating, assembling, controlling and manufacturing matter at the nanometre scale ^[1]. Today, nanotechnology plays a key role in numerous areas such as the agri-food industry, water and sanitation, electronics, energy production, or healthcare ^[2]. Nanoparticles are found in products used daily by consumers, such as electronics, batteries, sporting equipment, food packaging, dietary supplements, cosmetics, personal care products, drugs, medical devices, and clothing ^[3]. Clothing provides different types of comfort: protection against mosquito bites, sun protection but also psychological, tactile, and thermal ^[4, 5, 6]. Psychological comfort relates to fashion and acceptance in society; tactile comfort refers to the material of the fabric and its feel, and thermal comfort refers to the clothing's ability to retain body heat ^[6]. Nanotextiles are conventional textiles including nanomaterials, adding functionalities such as flame retardancy, self-cleaning, dirt and water repellency, ultraviolet radiation protection, or antibacterial property ^[7]. Other recent developments include the integration of advanced nanogenerator technology, self-charging ability, textile triboelectric nanogenerator, or silver nanowires in smart textile technology ^[8-11].

A textile is any fabric or material made of threads obtained by assembling fibres or filaments ^[12]. A magnification of 10× is necessary to observe the fabric structure while the observation of textile fibres requires a total magnification of 400 ^[13]. There are four categories of nanotextiles: nanocomposite textiles (nanotechnological properties integrated into the fibrous components, before the fabric is manufactured); nanofibrous textiles (made of fibres obtained at the nanoscale); unfractionated nanotechnological textiles (the properties have been improved in order to increase, for example, their adhesiveness, which is important in the case

Corresponding Author:

Francois Eya'ane Meva

¹ Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, PO Box 2701 Douala, Cameroon

² Institute for Inorganic Chemistry and Structural Chemistry, Heinrich-Heine University Düsseldorf, 40204 Düsseldorf, Germany

of coating their surface); nano-finished textiles (a nanotechnological property is added after the basic fabric has been manufactured) [14].

The integration of nanomaterials in textiles can be done by *impregnation*. For example, the incorporation of bioactive nanoparticles such as Ag/Zn and Ag/TiO₂ based nanocomposite fillers is used to functionalize synthetic fibres in the rotating fibre mass [15]. Multifunctional cotton and polyester knitted fabrics were produced by exhaustion in an ultrasonic bath containing TiO₂ nanoparticles, citric acid, and sodium hypophosphite as catalyst, followed by drying and curing [16]. In another study, Montazer and coworkers used silver nanoparticles (AgNPs) with 1,2,3,4-butantetracarboxylic acid (BTCA) as a cross-linking agent and sodium hypophosphite as a catalyst by the padding technique to impart antibacterial properties to knitted and nylon fabric without yellowing [17]. A sulfated cross-linked cotton fabric of β -cyclodextrin was synthesized using EDTA, with increased antibacterial activity via filling with ZnO, TiO₂ and colloidal solution of AgNPs separately, followed by curing at 120 °C for 3 min [18]. Enzymatic pre-treatment of wool and polyester blends with protease and lipase hydrolyses the wool and polyester surfaces, respectively. This was done by dipping them in an ultrasonic bath containing TiO₂ nanoparticles (TiO₂NPs) with BTCA followed by curing at 180 °C for 15 min in order to achieve remarkable multifunctional properties (self-cleaning, antibacterial and UV protection) [19].

In situ generation of nanoparticles and their deposition on the surface of tissues is a one-step process. Copper and silver nanoparticles have been successfully deposited on cotton fabrics by sonochemical irradiation or by means of chemical reduction with sodium citrate as reducing agent in presence of chitosan as chelating agent [20, 21]. Some recent studies conferring multifunctional properties alone or in combination with the staining properties of cellulose-containing fabrics in a one-step process have been done, for example: The development of multifunctional cellulosic pigment imprints using AgNPs, ZnONPs, and TiO₂NPs individually and in the presence of different binders [22]; the enhancement of antibacterial functionality of cotton, linen and cellulosic viscose fabric using AgNPs/PVP hybrid [23]; or the enhancement of antibacterial, UV protection and self-cleaning properties of cotton fabric by treatment with silver, silica by dip coating method [24]. The nanoparticles incorporated into the central part of the filaments have no positive impact on antibacterial performance [25].

In order to produce inexpensive, environmentally friendly nano finished textile fabrics with potential use in healthcare or malaria vector control, we have impregnated locally obtained fabrics with nano silver. Metallic silver is the most used nanoparticle in the textile industry for its antimicrobial and anti-odour properties [26]. This study presents the characterization by light microscopy, scanning electron microscopy and hyperspectral microscopy coupled with emission spectra of cotton fabrics impregnated with silver nanoparticles obtained from *Cymbopogon citratus* leaves aqueous extract. The retention properties of the textiles were determined.

Lemongrass (*Cymbopogon citratus*) is a perennial herb belonging to the Poaceae family. It grows well in tropical and semi-tropical regions [27]. The grass grows in dense clumps that can grow up to 1.8 m in height and approximately 1.2 m in width with a short rhizome [27]. In traditional medicine, the leaf decoction or essential oil is used against gastrointestinal

pain, coughs, herpes, fever, headache, cardiac disorders, sickle cell anemia, flatulence, vomiting, dyspepsia, jaundice, insomnia, snake bite, whooping cough, elephantiasis and depression. It is used alone or in combination for malaria control [27]. The most important molecules responsible for its therapeutic properties are: citral, myrcene, limonene, tannins, nerol acetate, neryl acetate, linalool, geranyl acetate, terpinene-4-ol, β -caryophyllene, geraniol, saponosides, phenolic compounds, and flavonoids [27].

Materials and methods

Textile and chemicals

Cotton textiles were obtained from the Cotton Industry of Cameroon (CICAM). They were cut into 10 × 10 cm size and kept in a dust-free environment. Silver nitrate (99.99%) was purchased from MERCK (Germany) and prepared as a 1 mM solution. The distilled water used throughout the experiments was obtained from a 2001/2 Water Still (GFL, Germany).

Plant collection

Fresh leaves of *Cymbopogon citratus* (Figure 1) were collected in Douala at Massounbou Pk 21, Littoral region, Cameroon, in January 2019, dried and authenticated at the National Herbarium of Cameroon by comparison with a voucher specimen previously deposited under the reference number N° 18628/SFR/Cam.



Fig 1: The picture of *Cymbopogon citratus* plant

Plant extract preparation

Ten grams of fresh leaves of *Cymbopogon citratus* were finely cut, washed with running tap water and distilled water. *Cymbopogon citratus* leaves were heated for 5 minutes at 80°C in a beaker containing 100 mL of distilled water. After cooling, the mixture was filtered using Whatman filter paper n°1 and the extract obtained was stored at 4 °C for further use.

Silver nanoparticles synthesis

Silver nanoparticles mediated by *Cymbopogon citratus* (AgNPs-CC) were obtained from previously reported protocols with some modifications [28]. The *Cymbopogon citratus* aqueous extract was mixed with 1 mM aqueous silver nitrate solution in a 1:5 ratio (giving 60 mL solution), under aseptic conditions and incubated at room temperature in the presence of light for 5 hours. A colour change of the solution was observed. The obtained solution was centrifuged for 1 hour at 6000 rpm and the supernatant was then removed. The isolated nanoparticles were rinsed twice with distilled water and dried in an oven for 24 hours at 104 °C. The obtained pellets were weighed to determine their mass.

Ultraviolet visible spectroscopy

Ultraviolet-Visible spectroscopy was performed using an aliquot of 2 mL of AgNPs-CC suspension on a UV Line 9100-9400 Spectrophotometer from SI Analytics (Germany). The spectra were taken in the wavelength range of 320 to 1100 nm at different interval times (10 minutes, 20 minutes, 40 minutes, 50 minutes and 120 minutes).

Powder X-ray diffraction (PXRD)

The PXRD measurements of purified silver nanoparticles were carried out using a Panalytical Empyrean Serie 2 X-ray diffractometer (Cu-K α 1 1.54060 Å, Cu-K α 2 1.54443 Å, Cu-K α 1.39225 Å) by preparing a thin film on silicon substrate.

Textile Impregnation

In a typical experiment, 10 cm x 10 cm (100 cm²) cotton fabric material was subjected to simple dipping into distilled water or AgNPs-CC with magnetic stirring and *in situ* synthesis of AgNPs-CC according to Vinatoru and coworkers with some modifications [29]. The pieces were immersed in the nanoparticle-containing solution under magnetic stirring. After the fabric was removed and dried, it was subjected to a washout test to check the effectiveness of the *impregnation*.

Impregnation by soaking of nanoparticles

Single dip *impregnation* was done by soaking a batch of 10 samples of 10 cm x 10 cm each in a 120 mL of water and nanoparticles solution under magnetic stirring at 3000 rpm for 1 hour.

Impregnation by an *in situ* method was carried out according to Vinatoru and coworkers, with slight modifications [30]. Briefly, the textile was soaked in a beaker containing 100 mL of 1 mM silver nitrate solution. A constant stirring of 3000 rounds/min or rpm was applied with a magnetic stirrer for 5 minutes. 20 mL of the plant extract was added and the system was stirred for 2 hours, after which the textile was removed and dried at 25 °C.

Determination of the retention capacity

Ten samples of 10 cm x 10 cm were soaked in a small plastic container containing 300 mL of distilled water or nanoparticle solution for 10 minutes, then allowed to drain over the container for an additional 10 minutes. The mean weight of the solution left and retained was determined.

Washout test evaluation of the washing resistance

After the fabrics were impregnated and dried, their resistance to washing was evaluated by dipping two samples for 15 minutes under magnetic stirring at 3000 rpm at temperatures of 25 °C and 60 °C. Five washing cycles were made. Washout resistance was assessed by measuring absorbance at the fixed characteristic plasmon resonance wavelength of 450 nm using the spectrophotometer.

Thermogravimetric analysis

Thermogravimetric analyses were performed using a TG 209 F3 Tarsus thermal gravimetric analyzer (NETZSCH-Gerätebau GmbH, Germany). The temperature range for the

analysis was 30 °C – 1000 °C at a heating rate of 10 °C/min under a nitrogen atmosphere (20 mL/min).

Transmission electron microscopy

Cotton samples were incubated for 2h with 50% Epon, 50% Aceton for 2h, followed by two times 2h infiltration in pure Epon resin. Samples were polymerized at 60°C for three days. Ultrathin sections were stained for 5 min with uranyl acetate in ethanol, washed, stained 4 min with lead citrate and again washed. Electron micrographs were recorded at a JEOL JEM-2100 Transmission Electron Microscope at 200 kV with a TVIPS F416 camera system. The images were analysed using Fiji software.

Hyperspectral microscopy and emission spectroscopy

The samples were analysed by a CytoViva (Auburn, AL) hyperspectral microscope, and the spectral differences between the samples were determined by analysis of emission spectra. The CytoViva hyperspectral microscope is specifically designed for optical imaging of microscopic sample elements and the creation of a spectral image where each pixel of the image contains the full visible near-infrared (Vis-NIR) reflectance or emission spectrum from 400 nm-1000 nm at high spectral resolution (1.5 nm). The CytoViva system used Olympus microscope components (Tokyo, Japan) and was equipped with CytoViva's patented enhanced darkfield illumination condenser optics (US7, 564, 623, US7, 542, 203). An aluminium halogen light source (Dolan Jenner, Boxborough, MA) was used with the darkfield optics because it produces spectrally smooth illumination for hyperspectral imaging. These enhanced darkfield optics are designed to create high signal-to-noise darkfield optical images of samples. In these images, the sample elements appear bright (high signal) against a dark (low noise) background. The microscope was equipped with a 10 nm step translational stage (Prior Scientific, Cambridge, UK) which serves as a "push broom" mechanism for the line scan hyperspectral image capture. A transmission diffraction grating hyperspectral imager, which acts as a prism (Specim Spectral Imaging, Oulu, Finland) was used for spectral delineation. A CCD was integrated into the spectrograph, which is used to capture the spectral data from the spectrograph. The system includes customized CytoViva ENVI 4.8 hyperspectral image capture and analysis software, which integrates all microscopy and hyperspectral image capture components. In addition to managing the image capture process, this software includes custom hyperspectral image analysis capabilities for pixel level spectral characterization, mean spectral comparison and spectral mapping within a sample image and between sample images.

Results and discussion

Visual observation

Visual observation within the first hour of the reaction between silver nitrate solution and *Cymbopogon citratus* fresh leaf extract shows a change of colour from pale yellow to dark brown due to the increase in the density of nanoparticles in the solution (Figure 2).

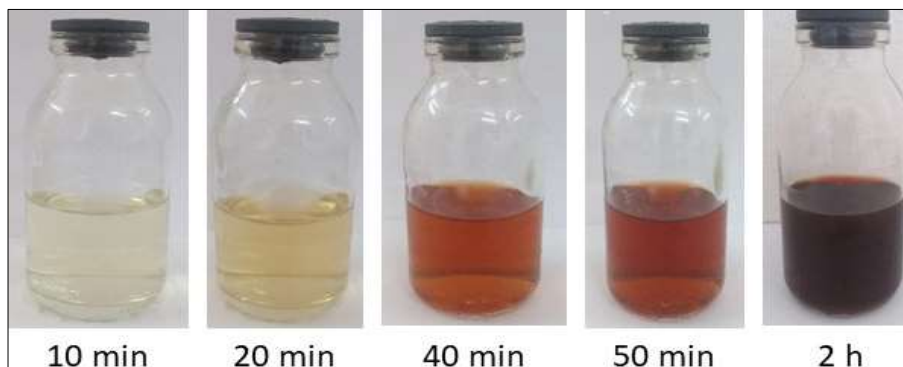


Fig 2: Colour change during silver nanoparticles synthesis from silver nitrate and *Cymbopogon citratus* extracts

UV-Visible spectroscopy results

UV Visible spectroscopy was used to confirm the visual observations made. It was noticed that the intensity of the UV-Vis absorption bands increases with time (Figure 3) [28]. A characteristic plasmon resonance peak centred at 450 nm and belonging to the range of surface plasmon resonance band

of AgNPs (400-600nm) was obtained. After two hours of reaction, the regular shape of the UV-Visible spectrum shows that the nanoparticles were stable with no sign of aggregation [31]. The reaction was carried out in the presence of the photocatalytic action of light.

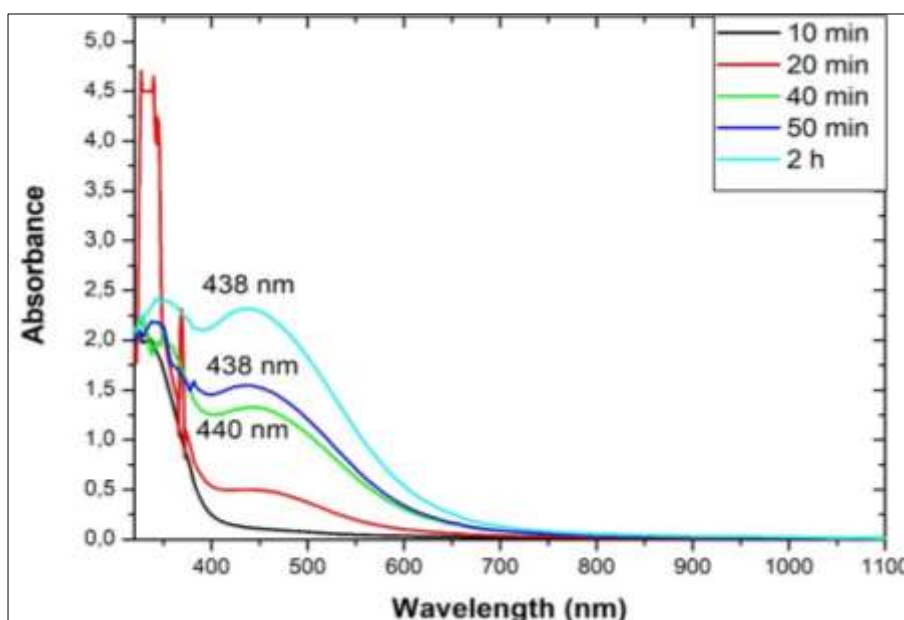


Fig 3: Ultraviolet visible spectrum of AgNPs-CC formation during two hours; a characteristic plasmon resonance band appears between 430 and 440 nm.

Powder X-ray diffraction result

Figure 4 shows the powder X-ray diffraction (PXRD) pattern of AgNPs mediated by *Cymbopogon citratus*. The nanoparticles generated by *Cymbopogon citratus* are

crystalline and composed of AgCl (in the majority 82%) and Ag (18%) phases (Figure 4; Table 1). A similar situation has been observed for silver nanoparticles obtained from *Selaginella myosurus* or *Psidium guajava* [31, 32].

Table 1: Characteristics of powder X-ray diffraction signals

No.	Pos. [°2Th.]	d-spacing [Å]	Height [cts]	FWHM Left [°2Th.]	Size nm
1	27.76	3.210	439.8	0.156	54.81■
2	32.18	2.779	946.0	0.156	55.38■
3	37.95	2.369	397.7	0.125	70.34□
4	38.09	2.361	502.3	0.125	70.37□
5	44.23	2.046	82.64	0.749	11.97■
6	46.17	1.965	428.8	0.1872	48.20■
7	54.75	1.675	107.8	0.312	29.96■
8	57.40	1.604	89.64	0.374	25.28□
9	64.40	1.445	78.23	0.374	26.20■
10	67.40	1.388	29.68	0.374	26.65■
11	76.68	1.242	70.05	0.499	21.20■
12	77.3355	1.233	81.88	0.2496	42.60□

AgCl crystallite size: 34 nm; Ag crystallite: 52 nm. Determined by the Scherer equation.

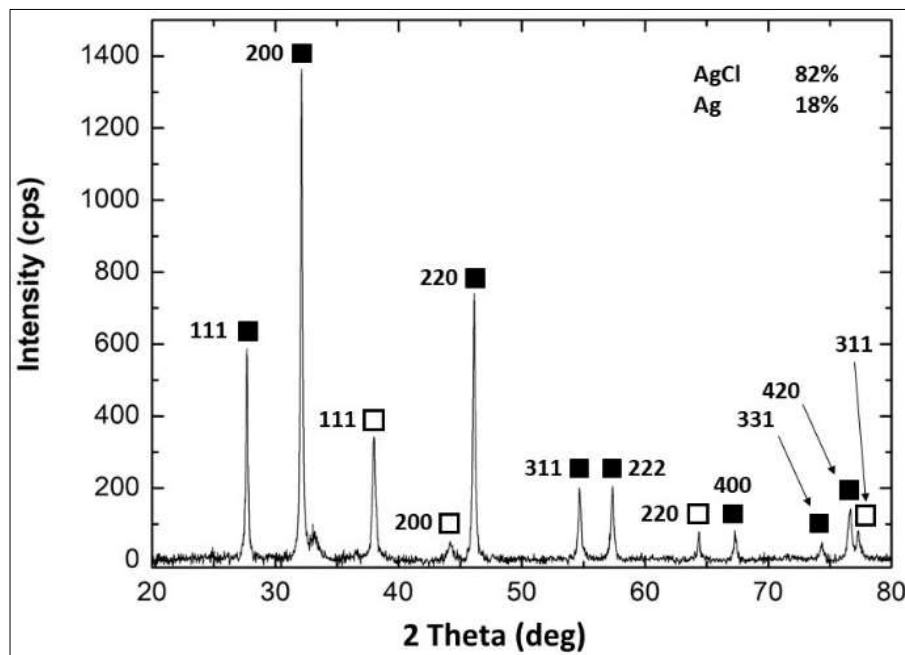


Fig 4: Powder X-ray diffraction pattern of the synthesised AgNPs-CC. Dark square and white square correspond to AgCl pattern (JCPDS file: 31-1238) and Ag pattern (JCPDS file: 65-2871), respectively.

Fabric retention capacities

The studied cotton fabric (Table 2) has the capacity to absorb more water and matter than its own weight (Figure 5). This retention is greater than that of 100% cotton (Navy-Blue with stripes), which has a retention of 125%^[33]. Both cotton fabrics release 65% water based on the weight of the material plus that of the liquid. The amount of water released is higher than the amount of nanoparticles solution released. This can be due to the disruption of Van der Waals forces between water molecules in the nanoparticle solution. Cotton terry

towel fabric produced with zero twist yarns for pile loops exhibited higher water absorption (889%) and a high absorption rate^[34]. Measurement of the capacity of hydrophilic fibre cotton fabric to retain water and AgNPs-CC solution showed that the cotton absorption capacity is lower than Kerlon and higher than Tergal^[35]. Buch and Leary show that 100% cotton (Navy-Blue with stripes) absorbs more water than rayon and polyester^[33]. This high absorption and expelling of water makes cotton an interesting material in the field of sports because it does not block perspiration.

Table 2: Chemical and physical characteristics of cotton fabric

Type of fabric (Code)	Type of fibre (%)	Metric number		Thread count (number strands/cm)		g/m ²	Weave type
		Warp	Weft	Warp	Weft		
Loincloth (P30A)	Cellulose (100%)	51.38	35.01	25	15.0	116.81	Canvas

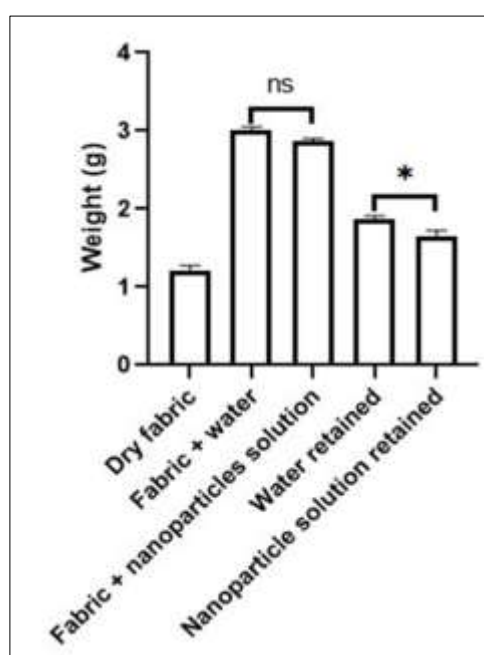


Fig 5: Weight of fabrics (dry and wet) and soaking solutions. ns = no statistical difference between fabric + water and fabric + nanoparticle solution ($p > 0.05$); * $P = 0.0239$, The difference between retained water and retained nanoparticle solution retained was statistically significant. Each bar is expressed as mean \pm ESM.

Retention capacity, visual inspection, and light microscopic observation of impregnated cotton fabric

The ability of nanoparticles to impregnate fabrics depends on the method used. Several methods exist for the *impregnation* of silver nanoparticles in textiles [25], *single dip impregnation* and *in situ impregnation* are used in this work for the *impregnation* cotton by biogenerated silver nanoparticles. Modern techniques such as plasma technology, sol gel, sonochemistry or ultra-signification are used for *in situ impregnation* [25]. These techniques are not very accessible in countries with limited resources. In the present study, these modern methods were replaced by a simpler one at the laboratory scale. The measurement of the cotton fabric capacity to retain AgNPs-CC in diluted solutions (31.25 ppm to 250 ppm), is independent of the concentration in the range studied (Figures 6 and 7).

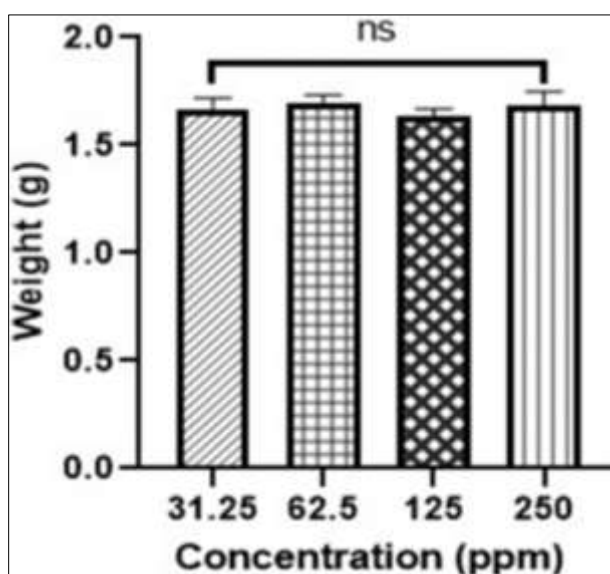


Fig 6: The nanoparticle solution was retained as a function of the concentrations used to impregnate cotton fabrics. Each bar is expressed as mean \pm ESM. ns = not statistically significant ($P > 0.05$).

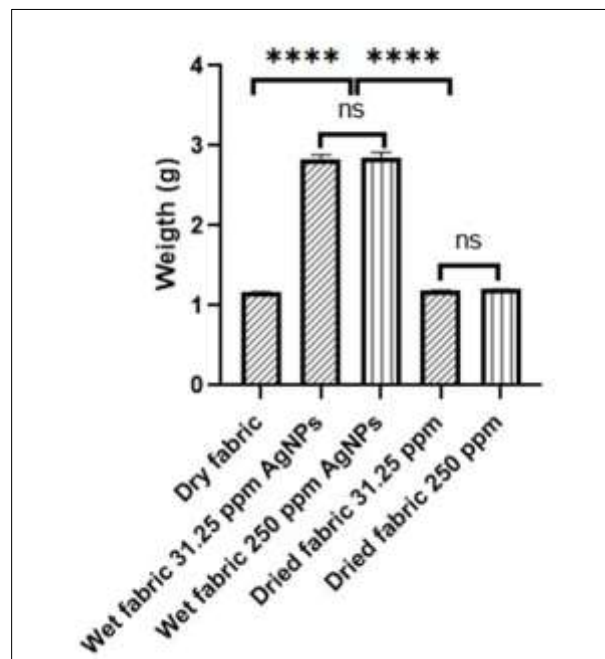


Fig 7: Retention capacity of impregnated cotton fabric. Dry and dried fabric weight was not different statistically ($p > 0.05$) in the range of concentrations used. Each bar is expressed as mean \pm ESM. ns = not statistically significant ($p > 0.05$).

Gedanken and collaborators method of metal oxide *impregnation* suffers from low penetration of nanoparticles into the bulk material [36]. Vinatoru and colleagues reported a two-step method with immersion in the salt followed by *in situ* conversion to oxide nanoparticles [29]. The dry dip-impregnated and *in situ*-impregnated cotton fabrics were subjected to optical microscopy. The difference between both types of fabric obtained was not clear. As with the synthesis of silver nanoparticles by reaction of plant extract with silver salt, the first observation of the *impregnation* of silver nanoparticles was inspected by a change of colour of the textiles. White fabric samples turned brown, a characteristic colour of silver nanoparticles (Figure 8) [37].

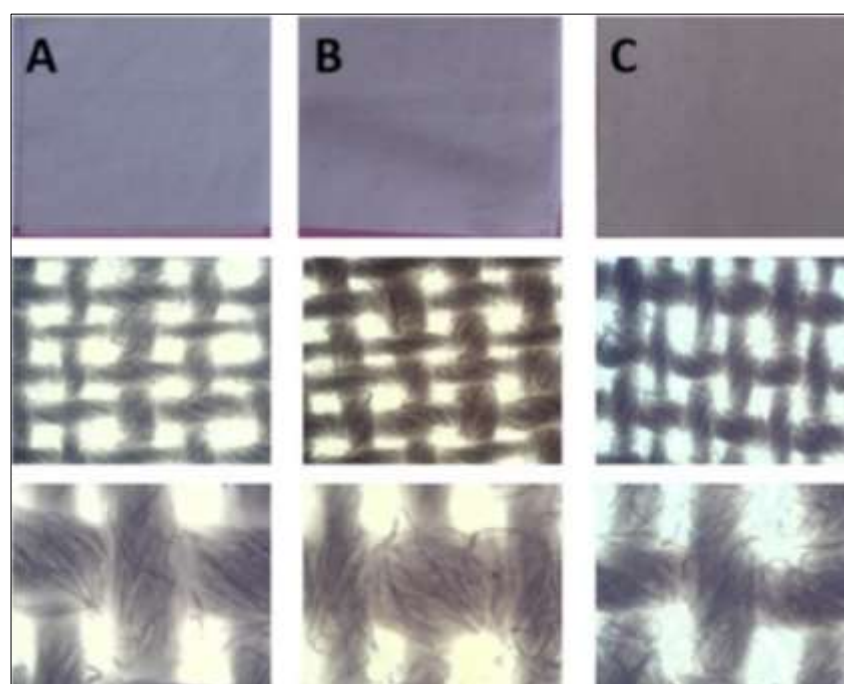


Fig 8: Macroscopic view and optical images of cotton fabric (10 x and 50 x). A: non-impregnated, B: simple impregnated and C: *in situ* impregnated

Effect of temperature and washing cycles

The *in situ* impregnated fabric with the nanoparticle solution shows a better resistance to cold and even warm washing (Figure 9). Cumulative absorbance presents a linear behavior that can allow prediction of washing cycles related to solution absorbance measurement. For example, a cumulative absorbance of 0.15 will be achieved for 87 cycles using the linear equation for simple soaking at 60 °C. The power to retain the nanoparticles is maximal for fabric generated *in situ* at 25 °C. Thus, it was selected for further analysis.

Ultrasonication used to impregnate has conferred a washout resistance to materials [38]. The batch process for *in situ* synthesis by Vinatoru and collaborators [29], was modified to achieve significantly increased loading of Ag⁺ ions onto the fabric. A dipping immersion in a solution of Ag⁺ ions under agitation causes an increased loading of ions and replaces ultrasonication. These ions are converted into nanoparticles by adding *Cymbopogon citratus* extract solution due to the absorption capacity of cotton at 25 °C and 60 °C.

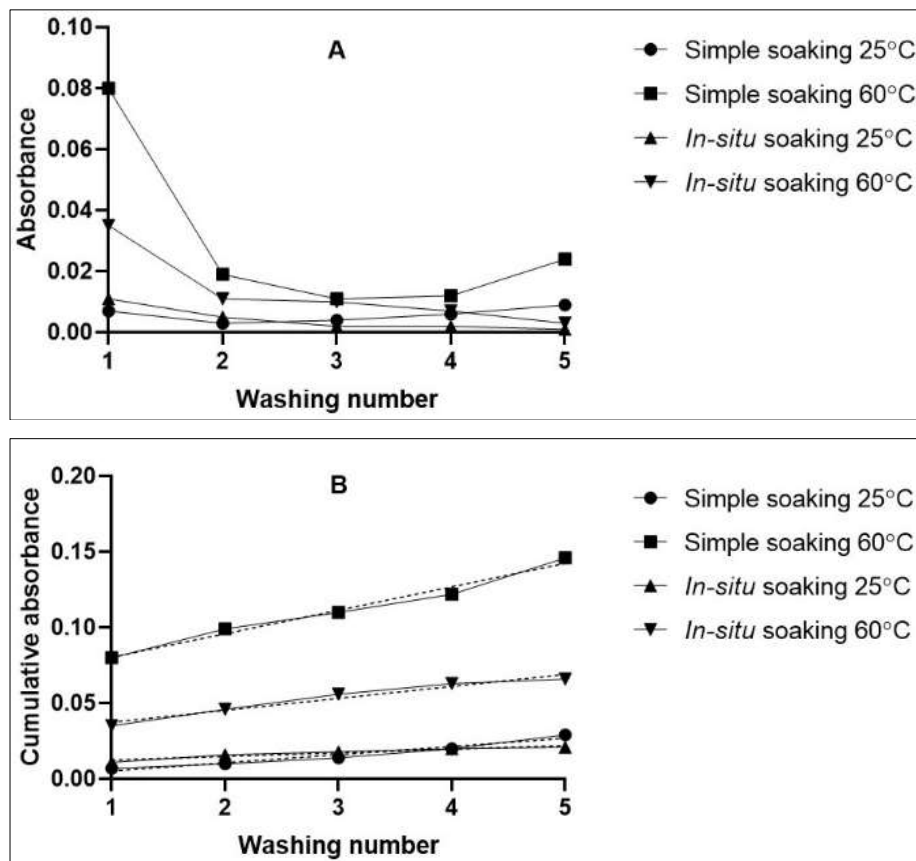


Fig 9: Absorbance (A) and cumulative absorbance (B) recorded after 5 washing of cleaning solutions at 25 °C and 60 °C of simple and *in situ* impregnated cotton fabric. The cumulative absorbance presents a linear correlation of the data (dotted lines) with equations $Y = 0.005400 \cdot X - 0.0002000$, $R^2 = 0.9529$ for simple soaking at 25 °C, $Y = 0.01550 \cdot X + 0.06490$, $R^2 = 0.9801$ for simple soaking at 60 °C, $Y = 0.002400 \cdot X + 0.01000$, $R^2 = 0.9172$ for *in situ* soaking at 25 °C and $Y = 0.007900 \cdot X + 0.02950$, $R^2 = 0.9590$ for *in situ* soaking at 60 °C. In both experiment the cleaning water is analysed.

Thermogravimetric analyses (TGA)

The thermal properties of the tested samples were analyzed by thermal gravimetric technique. Figure 10 shows that the mean weight loss of non-impregnated, simple soaking and *in situ* impregnated cotton materials was -2% in the first stage of thermal degradation (between 1 and 100 °C), indicating the loss of water molecules. The thermal decomposition of non-impregnated cotton exhibited a mass change of -69%, in the range 250-400 °C, while -78% and -76% mass change was observed for simple soaking and *in situ* impregnated samples in the same range, respectively. TGA studies have shown different steps in the thermal degradation of cellulose with weight loss due to absorbed water before 200 °C. Above that temperature, thermal decomposition and depolymerisation occur. Between 250 and 290 °C, primary volatile decomposition releases CO₂, CO, and H₂O. Volatile products

are recorded at temperatures between 290 and 350 °C, including anhydroglucoses (1, 6-anhydro-β-D-glucopyranose, 1, 6-anhydro-β-D-glucofuranose and 1, 4:3, 6-dianhydro-α-D-glucopyranose) or dehydration products of anhydroglucoses (5-hydroxymethyl-2-furfural, 2-furyl hydroxymethyl ketone and levoglucosenone) [39]. The difference in decomposition in impregnated and non-impregnated cellulose materials in this work may be attributed to the presence of secondary metabolites with the non-oxidative degradation before 200 °C and the oxidative degradation after 220 °C being in the range 350-600 °C for various plants extracts [40]. Furthermore, the residual weight of the sample treated with simple soak (4%) was 2-fold higher than the residual weight of the non-impregnated sample (2%). These effects could be attributed to the adhesion of silver nanoparticles to cotton fiber, making it not easy to decompose at high temperature [41].

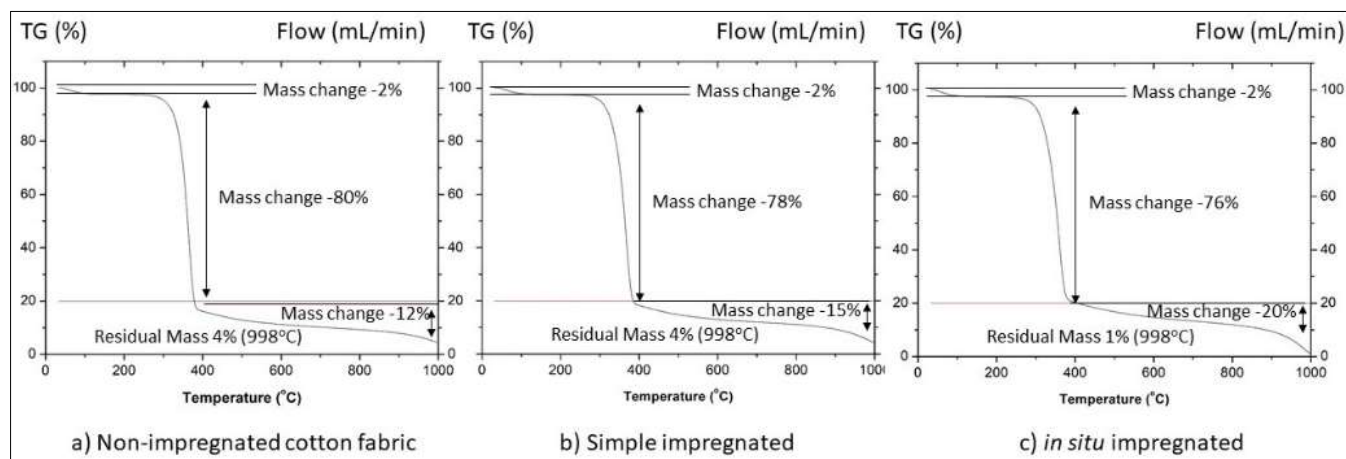


Fig 10: Thermogravimetric diagrams of non-impregnated (a), simple soaking impregnated (b), and *in situ* impregnated (c) cotton material with *Cymbopogon citratus*-mediated silver nanoparticles.

Transmission electron microscopy

Transmission electron microscopy analysis was carried out to further prove the formation of silver nanoparticles in the cotton material, after simple soaking and *in situ impregnation* with *Cymbopogon citratus* leaves-mediated nanosilver. It is apparent from Figure 11 that polycrystalline nanograins of mostly spherical shape are distributed within the cotton material. The presence of a few agglomerates appears in the *in situ* impregnated material. The size distribution was determined by analysis of a section of 100 particles. A mean size of the silver particles inside the simple dipped cotton impregnated material of 7.2 ± 1.4 nm was found significantly high compared to the mean size inside the *in situ* impregnated

cotton material 5.9 ± 0.7 nm (see Figure 12). The size distribution of both materials was found to be smaller than in the case of silver nanoparticle-loaded poly (acrylamide-co-itaconic acid)-grafted cotton fabric (12.5 nm) for both studied *impregnations* [42]. An almost similar size distribution 0-35 nm was obtained for silver mediated *Cymbopogon citratus* compared to silver nanoparticle-loaded poly (acrylamide-co-itaconic acid) inside cotton fabric 0-30nm [42]. The distance distribution was determined by analysing 50 couples of neighbour silver nanoparticles. Distances between silver center was found smaller for the *in situ* impregnated material due to the spreadability and dispersibility of the cotton fibre network during material fabrication.



Fig 11: TEM images of non-impregnated (a), simple soaking impregnated (b) and *in situ* impregnated (c) cotton material with *Cymbopogon citratus*-mediated silver nanoparticles.

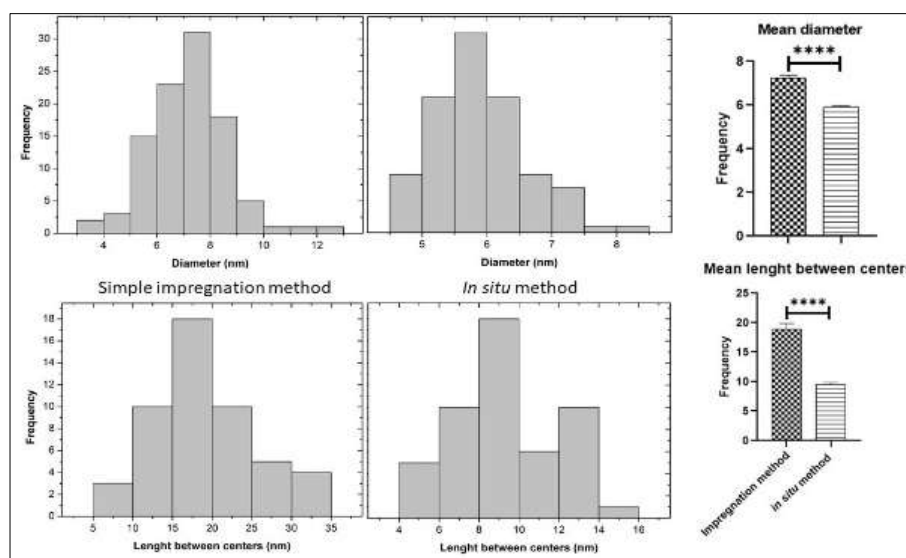


Fig 12: Fiji analysis of 100 centres and 50 couples of nanoparticles neighbours for simple impregnated and *in situ* impregnated materials. Significantly different mean diameters of silver nanoparticles of 7.2 ± 1.4 and 5.9 ± 0.6 nm are obtained for simple dipping and *in situ* impregnated materials, respectively. The mean distance between centre of 19.0 ± 6.6 and 9.5 ± 2.7 nm is obtained for simple dipping and *in situ* impregnated materials respectively. $P < 0.0001$

Hyperspectral microscopy coupled emission spectroscopy

The presence of nanoparticles was studied by hyperspectral microscopy of the Cyto Viva technology of the cotton fabric (white) and the *in situ* impregnated with AgNPs-CC. The first microscopy experiment did not reveal a significant difference between the impregnated and unimpregnated samples (Figure 13). The UV-Vis spectrum, on the other hand, shows a bathochromic shift of the emission wavelength from 625 nm (unimpregnated) to 675 nm (impregnated) allowing quality control on different textiles. A region of interest (ROI) selected from the microscope provides the average spectral response of the fabric area. Darkfield microscopy in a second experiment (Figure 14) allows subtraction of background and

reveals the green coloured luminous nanoparticles which is one of the characteristic of silver nanoparticles by the CytoViva technology microscopy analysis and the appearance of red pixels. Silver nanoparticles of about 20 nm can be observed. The maximum plasmonic resonance values of the CytoViva technology do not correspond to the one obtained in visible ultraviolet experiments of nanoparticle preparations from plant extract (400 to 600 nm) in aqueous medium. When the distance between two AgNPs is comparable to the size, the Coulombian interaction between the charge density oscillations (from the surface plasmons) leads to hybridization of the individual plasmons of AgNPs, resulting in new renormalized plasmon energies [38].

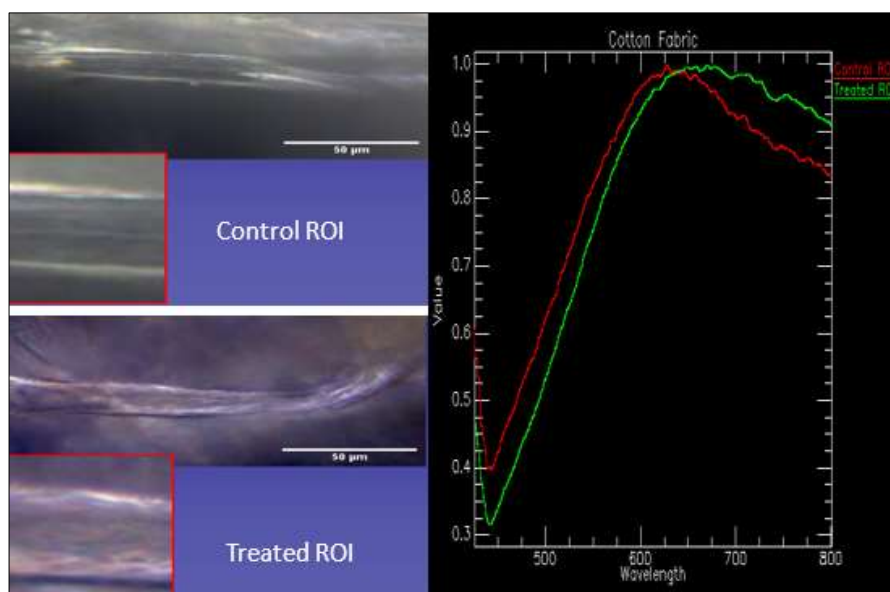


Fig 13: Image of unimpregnated non-impregnated and *in situ* impregnated cotton fabric with AgNPs-CC by Cytoviva hyperspectral microscopy technology coupled to visible ultraviolet

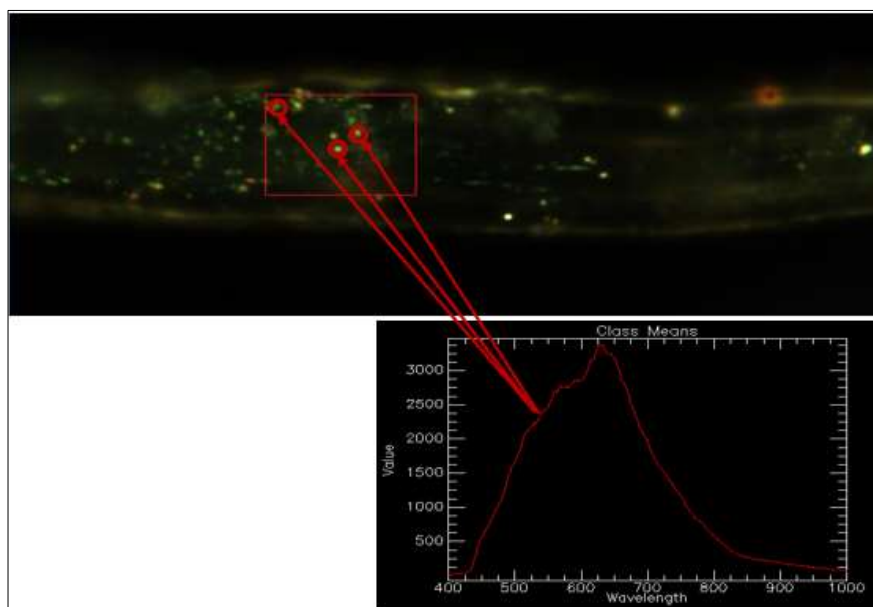


Fig 14: Image of silver nanoparticles on impregnated cotton fabric generated by *Cymbopogon citratus* by Cytoviva darkfield hyperspectral microscopy technology coupled to visible ultraviolet

Conclusion

The future of nanotechnology is promising in resource-constrained countries. In the field of textiles, there is much at stake because the development of intelligent textiles is a solution to many problems of disease prevention in the health

sector. Clothing and bedding are in most cases responsible for the transmission of many hospital-acquired infections. Nanoparticle *impregnation* allows the development of lab coats or sheets with antimicrobial properties, sutures threads or dressings with anti-inflammatory and healing properties, or

mosquito nets with intrinsic vector control properties. Silver nanoparticles were prepared by the reaction of silver nitrate and *Cymbopogon citratus* leaves. The characteristic brown colour change of silver nanoparticle is observed on the cotton material after *impregnation*. The *impregnation* of the nanoparticles was presented by soaking and *in situ* procedure, and the washing solution was studied by UV-Vis. A linear evolution of the cumulative absorbance makes it possible to predict the number of cycles to degrade the cotton fabric using different methods. If visible microscopy does not present clear differences between fabric materials, hyperspectral microscopy coupled emission spectroscopy allows one to differentiate them by a bathochromic effect of the emission wavelength. The smallest nanoparticles obtained by an *in situ* method can further be visualized using the enhanced darkfield optics technique of CytoViva technology. Transmission electron microscopy images provided evidence of mainly spherical silver nanoparticles formation inside the cotton materials. The thermal stability of the simple soaking impregnated cotton material was found to be higher than that of the untreated. The *in situ* impregnated fabric has a high washout resistance and has the potential to be used in the management of hospital structures in resource-limited countries.

Acknowledgements

The authors thank the Multidisciplinary Laboratory of the Faculty of Medicine and Pharmaceutical Sciences, Department of Pharmaceutical Sciences, for technical support. The support of Word University Service under APA 2668 for providing part equipment used is appreciated. FEM thank the Commonwealth Scholarship Commission for a generous African Academic Fellowship (grant no. CMCf-2015-3). PBEK thanks IFS for awarding a research grant (I-1-F-6137-1). Support of the Imaging Core Facility, Biocenter, University of Würzburg, Germany for the JEOL JEM-2100 Transmission Electron Microscope funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – 218894163 and support from CytoViva is acknowledged.

Data availability

Data will be made available on request.

Conflicts of interest

The authors declare that they have no conflicts of interest.

References

1. Bayda S, Adeel M, Tuccinardi T, Cordani M, Rizzolio F. The history of nanoscience and nanotechnology: From chemical-physical applications to nanomedicine. *Molecules*. 2019;25(1):112. <https://doi.org/10.3390/molecules25010112>.
2. Maksimović M. The roles of nanotechnology and internet of nano things in healthcare transformation. *TecnoLógicas*. 2017;20(40):139-153.
3. Contado C. Nanomaterials in consumer products: a challenging analytical problem. *Front Chem*. 2015;3:48. <https://doi.org/10.3389/fchem.2015.00048>
4. Londono-Renteria B, Patel JC, Vaughn M, Funkhauser S, Ponnusamy L, Grippin C, *et al*. Long-lasting permethrin-impregnated clothing protects against mosquito bites in outdoor workers. *Am J Trop Med Hyg*. 2015;93(4):869-874. <https://doi.org/10.4269/ajtmh.15-0130>.
5. Gambichler T, Altmeyer P, Hoffmann K. Role of clothes in sun protection. *Recent Results Cancer Res*. 2002;160:15-25. https://doi.org/10.1007/978-3-642-59410-6_3.
6. Pamuk O. Clothing comfort properties in textile industry. *Natural and Applied Sciences*. 2007;3(1):69-74.
7. Saleem H, Saidi SJ. Sustainable use of nanomaterials in textiles and their environmental impact. *Materials*. 2020;13:5134. 2020 <https://doi.org/10.3390/ma13225134>.
8. Dong K, Peng X, Wang ZL. Fiber/Fabric-based piezoelectric and triboelectric nanogenerators for flexible/stretchable and wearable electronics and artificial intelligence *Advanced Materials*. 2020;32(5):1902549.
9. Dong K, Wang ZL. Self-charging power textiles integrating energy harvesting triboelectric nanogenerators with energy storage batteries/supercapacitors. *Journal of Semiconductors*. 2021;42:101601.
10. Dong K, Hu Y, Yang J, Kim SW, Hu W, Wang ZL, *et al*. Smart textile triboelectric nanogenerators: Current status and perspectives. *MRS Bulletin*, 2021;46:512–521.
11. Liu X, Miao J, Fan Q, Zhang W, Zuo X, Tian M, *et al*. Smart Textile Based on 3D Stretchable Silver Nanowires/MXene Conductive Networks for Personal Healthcare and Thermal Management. *ACS Applied Materials & Interfaces*, 2021;13(47):56607-56619.
12. Haydon B. Nanomaterials and their applications in textiles – Standards – Domestic standardization for Canadian manufacturers and importers and international standardization developments. 2012 https://www.ic.gc.ca/eic/site/iccatt.nsf/eng/07154_1.html
13. Dastjerdi R, Montazer M. A review on the application of inorganic nano-structured materials in the modification of textiles: Focus on anti-microbial properties. *Colloids and Surfaces B: Biointerfaces*, 2010;79(1):5-18.
14. Smith A W. An introduction to textile materials: their structure, properties and deterioration. *Journal of the Society of Archivists*, 1999;20(1):25-39.
15. Gason F, De Wael K, Baes C. *Travaux personnels, Laboratoire Fibres et Textiles, INCC Bruxelles*. 1995-2010.
16. Hashemikia S, Montazer M. Sodium hypophosphite and nano TiO₂ inorganic catalysts along with citric acid on textile producing multi-functional properties. *Applied Catalysis A: General*, 2012;417-418:200-208.
17. Montazer M, Shamei A, Alimohammadi F. Stabilized nanosilver loaded nylon knitted fabric using BTCA without yellowing. *Progress in Organic Coatings*, 2012;74(1):270-276.
18. Selvam S, Rajiv GR, Suresh J, Gowri S, Ravikumar S, Sundrarajan M, *et al*. Antibacterial effect of novel synthesized sulfated β-cyclodextrin crosslinked cotton fabric and its improved antibacterial activities with ZnO, TiO₂ and Ag nanoparticles coating. *International Journal of Pharmaceutics*. 2012;434(1-2):366-374.
19. Montazer M, Seifollahzadeh S. Enhanced Self-cleaning, Antibacterial and UV Protection Properties of Nano TiO₂ Treated Textile through Enzymatic Pretreatment. *Photochemistry and Photobiology*. 2011;87(4):877-883.
20. Thomas V, Bajpai M, Bajpai SK. In Situ formation of silver nanoparticles within chitosan-attached cotton fabric for antibacterial property. *Journal of Industrial Textiles*. 2011;40(3):229-245.
21. Perelshtein I, Applerot G, Perkas N, Wehrschoetz-Sigl E, Hasmann A, Guebitz G, *et al*. CuO–cotton nanocomposite: Formation, morphology, and

- antibacterial activity. *Surface and Coatings Technology*. 2009;204(1-2):54-57.
22. Ibrahim NA, Eid BM, Youssef MA, Ibrahim HM, Ameen HA, Salah AM. multifunctional finishing of cellulosic/polyester blended fabrics. *Carbohydrate Polymers*. 2013;97(2):783-793.
 23. Ibrahim NA, Eid BM, Elmaaty TMA, El-Aziz EA. A smart approach to add antibacterial functionality to cellulosic pigment prints. *Carbohydrate Polymers*. 2013;94(1):612-618.
 24. Onar N, Aksit A C, Sen Y, Mutlu M. Antimicrobial, UV-protective and self-cleaning properties of cotton fabrics coated by dip-coating and solvothermal coating methods. *Fibers and Polymers*, 2011;12(4):461-470.
 25. Ibrahim NA. Nanomaterials for Antibacterial Textiles. In: *Nanotechnology in Diagnosis, Treatment and Prophylaxis of Infectious Diseases*. Elsevier; c2015. p. 191-216.
 26. Chadeau E. Caractérisation des propriétés antibactériennes de textiles fonctionnalisés avec de l'argent ou du PolyHexaMéthylène Biguanide (PHMB). Thesis. Université Claude Bernard - Lyon I. French, 2011.
 27. Kouame NM, Kamagate M, Koffi C, Die-Kakou HM, Yao NAR, Kakou A. *Cymbopogon citratus* (DC.) Stapf : ethnopharmacologie, phytochimie, activités pharmacologiques et toxicologie. *Phytothérapie*. 2016;14(6):384-392.
 28. Eya'ane Meva F, Segnou ML, Okalla Ebongue C, Ntomba AA, Belle Ebanda Kedi P, Deli V, *et al.* Spectroscopic synthetic optimizations monitoring of silver nanoparticles formation from *Megaphrynium macrostachyum* (Benth) Milne-Redh leaf extract. *Brazilian Journal of Pharmacognosy*. 2016;26(5):640-646.
 29. Vinatoru M, James T, Anthony MB. Method for producing antimicrobial yarns and fabrics by nanoparticle *impregnation*. Patent WO2016/0878649A1, 2015.
 30. Nguepi TE. Impact of cotton textile sector trends in Cameroon, *International Journal of Research in Business & Social Science*. 2019;8(6):331-343.
 31. Belle Ebanda Kedi P, Eya'ane Meva F, Kotsedi L, Nguemfo EL, Bogning Zangué C, Ntomba AA, *et al.* "Eco-friendly synthesis, characterization, *in vitro* and *in vivo* anti-inflammatory activity of silver nanoparticles mediated Selaginella myosurus aqueous extract. *International Journal of Nanomedicine*. 2018;2018:8537-8548.
 32. Ntomba AA, Eya'ane Meva F, Eysisap Ekoko W, Kojom Foko LP, Ngo hondt E, Etame Loe G, *et al.* Biogenic synthesis of silver nanoparticles using guava (*Psidium guajava*) leaf extract and its larvicidal action against *Anopheles gambiae*. *Journal of Biomaterials and Nanobiotechnology*. 2020;11:49-66.
 33. Buch V, Leary J. Summer Ventures in Science and Math 2012, Water retention capacities of different fabrics, Appalachian State University; c2012. p. 1-7.
 34. Cruz J, Leitão A, Silveira D, Pichandia S, Pinto M, Fanguero R, *et al.* Study of moisture absorption characteristics of cotton terry towel fabrics. *Procedia Engineering*. 2017;200:389-398.
 35. Sikapi Fouda JY. Biosynthèse de nanoparticules d'argent et *impregnation* sur du tissu textile, Thesis. University of Douala, Cameroon, French; c2018.
 36. Gedanken A, Nitzan Y, Perelshtein I. Sonochemical coating of textiles with metal oxide nanoparticles for antimicrobial fabrics. Patent; c2010. WO2010/001386
 37. El-Shishtawy RM, Asiri AM, Abdelwahed NAM, Al-Otaibi MM. In situ production of silver nanoparticle on cotton fabric and its antimicrobial evaluation. *Cellulose*. 2011;18(1):75-82.
 38. Perelshtein I, Perkas N, Gedanken A. The sono chemical coating of textiles with antibacterial nanoparticles. In: *Handbook of Antimicrobial Coatings*. Elsevier; c2018. p. 235-255.
 39. Abidi N, Hequet E, Ethridge D. Thermogravimetric analysis of cotton fibers: Relationships with maturity and fineness. *Journal of Applied Polymer Science*. 2007;103:3476-3482.
 40. Malucelli LC, Massulo T, Magalhães WLE, Stofella NCF, Vasconcelos EC, Filho MASC, *et al.* Thermal and chemical characterization of *Dicksonia sellowiana* extract by means of thermal analysis. *Revista Brasileira de Farmacognosia*. 2018;28:626-630.
 41. Sheetal, Sihag S, Yadav M, Pal J. Structural and morphological characterization of nanocellulose extracted from cotton straw residue. *Journal of Water and Environmental Nanotechnology*. 2022;7:1-13.
 42. Gupta P, Bajpai M, Bajpai SK. Investigation of antibacterial properties of silver nanoparticle-loaded poly (acrylamide-co-itaconic acid)-grafted cotton fabric. *The Journal of Cotton Science*. 2008;12:280-286.

Author's details

Jean Yves Sikapi Fouda

Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon

Agnes Antoinette Ntomba

Department of Animal Biology and Physiology, Faculty of Science, University of Douala, Douala, Cameroon

Philippe Belle Ebanda Kedi

Department of Animal Biology and Physiology, Faculty of Science, University of Douala, Douala, Cameroon

Thi Hai Yen Beglau

Institute for Inorganic Chemistry and Structural Chemistry, Heinrich-Heine University Düsseldorf, Düsseldorf, Germany

Marcus Fetzer

Institute for Inorganic Chemistry and Structural Chemistry, Heinrich-Heine University Düsseldorf, Düsseldorf, Germany

Till Strothmann

Institute for Inorganic Chemistry and Structural Chemistry, Heinrich-Heine University Düsseldorf, Düsseldorf, Germany

Tchangou Armel Florian

Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon

Sone Enone Bertin

Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon

Vandi Deli

Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon

Emmanuel Jean Teinkela Mbosso

Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon

Gustave Leopold Lehman

- 1) Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon
- 2) Department of Animal Biology and Physiology, Faculty of Science, University of Douala, Douala, Cameroon

Emmanuel Albert Mpondo Mpondo

Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon

Gisele Etame Loe

Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon

Francois Eya'ane Meva

- 1) Department of Pharmaceutical Sciences, Faculty of Medicine and Pharmaceutical Sciences, University of Douala, Douala, Cameroon
- 2) Institute for Inorganic Chemistry and Structural Chemistry, Heinrich-Heine University Düsseldorf, Düsseldorf, Germany

Christoph Janiak

Institute for Inorganic Chemistry and Structural Chemistry, Heinrich-Heine University Düsseldorf, Düsseldorf, Germany