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Fashion and Textiles

RESEARCH

Sustainable functional finishing for cotton fabrics using screen-printing process and gallotannin

Kyung Hwa Hong*

*Correspondence: hkh713@kongju.ac.kr
Professor, Department of Fashion Design and Merchandising, College of Natural Science, Kongju National University, 56, Gongjudaehak-ro, Gongju-si, Chungcheongnam-do 32588, Republic of Korea

Abstract

To seek a more environmentally friendly textile finishing technique, the screen-printing method was adopted to apply functional material to cotton fabrics. In addition, gallotannin was used as a functional material because it is naturally abundant in many plant-derived substances and shows various health-promoting features such as antimicrobial, antioxidant, and other attractive properties. Therefore, a gallotannin/thickener paste was applied to the surface of cotton fabrics through the screen-printing technique, and the gallotannin-printed cotton fabrics were thoroughly investigated using scanning electron microscope (SEM), Fourier-transform infrared spectroscopy (FTIR), and other methods. The gallotannin printed area was substantially brown in appearance, and gallotannin moiety appeared to combine with cotton cellulose through heat treatment. Furthermore, functional properties of the gallotannin-printed cotton fabrics were examined in terms of antibacterial activity, deodorizing property, and ultraviolet-blocking property, of which it demonstrated excellent abilities. However, the antibacterial ability toward Gram-negative bacteria (*K. pneumoniae*) decreased as the laundry cycle increased.

Keywords: Gallotannin, Screen printing, Cotton, Functional finishing, Antibacterial ability

Introduction

Awareness of environmental concerns has been increasing globally in recent years. However, many conventional textile and apparel manufacturing processes have the potential to create a considerable environmental footprint (Koh & Hong, 2017a). Textiles are usually subjected to many processes, including scouring, bleaching, dyeing, and finishing, before being completed as final consumer items. Each process not only utilizes various toxic chemicals and a high quantity of water and energy but also creates a large volume of wastewater containing undesirable chemical compounds (Haji & Naebe, 2020). Therefore, many researchers have attempted to develop environmentally friendly textile and apparel manufacturing processes (Eid & Ibrahim, 2021; Khan et al., 2017) to preserve the natural environment. In this regard, the screen-printing process was considered a sustainable alternative to conventional textile finishing. Screen-printing has been generally used for applying coloring patterns on the surface of textile items. The technique consumes less water and energy and generates less wastewater during the process. Consequently, it imposes less burden on the environment compared to other textile dyeing or finishing processes. On the other hand, many researchers have tried to find new safe chemicals to replace conventional toxic agents and overcome the challenges of sustainability in textile wet processing (Doty et al., 2016; Fröse et al., 2019; Jose et al., 2019). We have also attempted to develop suitable natural compounds for the textile dyeing or finishing process to meet consumer demand in terms of environmental sustainability and beneficial functions of the textile items (Koh & Hong, 2014, 2017a, b, 2019; Hong, 2014). We found that gallotannin is a potential agent for environmentally friendly textile finishing as it is a natural compound with health-promoting features such as antimicrobial, antioxidant, and other attractive properties, among others. As stated by Farha et al. (2020), "Tannins show good antibacterial effects. The Web of Science Core Collection was searched for the studies reporting the antibacterial and antivirulence effects of tannins as well as potential mechanisms of action". Additionally, gallotannin shows reasonable water-solubility and heat-resistance. Since textile dyeing and finishing are mostly conducted with a water base at high-temperature, water-solubility and heat-resistance are particularly desirable for textile application.

Cellulosic fibers are widely utilized in the fashion and textile industry because of their inherent features such as hydrophilicity, comfort, durability, and easy-care. However, cellulosic fibers are natural fibers and can attract bacterial growth and multiplication for they can provide nutrients, moisture, oxygen, and optimum temperature. Thus, objectionable odor, dermal infection, allergic reactions, and other related diseases are common complaints when such materials are used for clothing and bedding purposes (Singh et al., 2005; Teker, 2020). Thus, a wide range of antimicrobial agents has been applied to natural fiber textiles (Koh & Hong, 2017b). On the other hand, even though antimicrobial agents can prevent bacterial infectious diseases and other objectionable odors, their abuse and overuse can result in potentially adverse effects including toxicity to humans and/or the environment, emergence of multidrug-resistant bacteria, and so on. Thus, natural compounds from plants are a potential source of effective antimicrobial agents because they often pose low toxicity to humans and animals (Farha et al., 2020). Gallotannins are natural polymers extracted from several plants such as gall nuts, black currant, and sumach tree leaves. They are formed by the subsequent esterification of hydroxyl groups of D-glucose and gallic acid. They possess antimicrobial, antioxidant, and anti-inflammatory abilities, among others (Engels et al., 2009; Jourdes et al., 2013). Therefore, in this research, it was attempted to develop environmentally friendly finishing process to functionalize cotton fabrics using a screen-printing technique and natural multifunctional agent 'gallotannin' and investigate the properties and functionalities of gallotannin-printed cotton fabrics.

Methods

Sample material and chemicals

Bleached and desized cotton fabric (No. 400; plain woven 98 g/m^2) was purchased from Testfabrics Inc. (West Pittston, PA). Gallotannin (ACS reagent) was purchased from Sigma-Aldrich (St. Louis, MO, USA). Thickener for screen-printing (Hanisol 130 N,

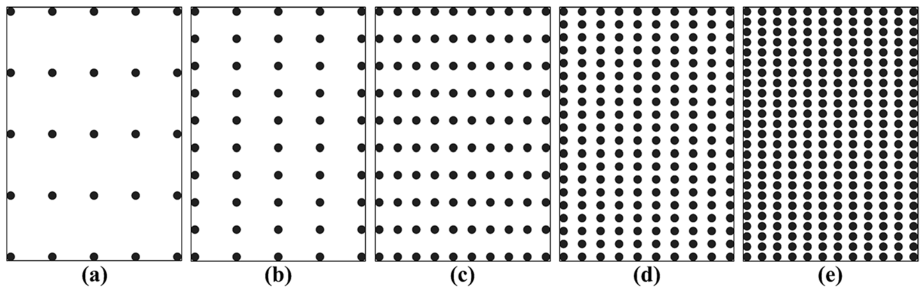

Fig. 1 Screen patterns for printing; the number of dots on 210 × 297 mm screen: **a** 25, **b** 50, **c** 100, **d** 200, **e** 300

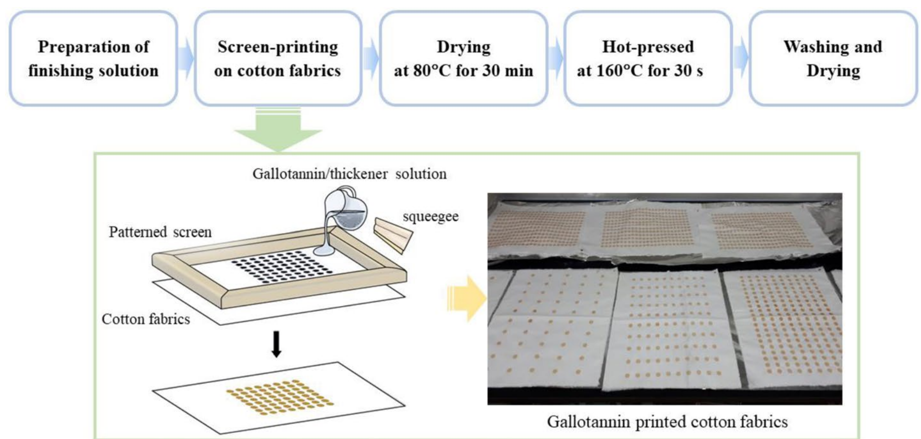

Fig. 2 Schematic diagram showing the screen-printing process

acrylic copolymer) was provided by Hansong industry Co., Ltd. (Shiheung-si, South Korea). All other reagents were used as received, without any further purification.

Preparation methods

Preparation of finishing paste and screen

The finishing paste was prepared as follows; 5.3 g of acrylic thickener was added to 30 wt.% gallotannin aqueous solution dropwise whilst stirring. Then, the viscous solution was placed under ambient conditions (21 ± 3 °C, 68 ± 3% R.H.) for 24 h to degas the solution. Patterns of the screen for screen-printing were generated using the Adobe Illustrator program consisting of black and white areas (as dots patterns) to generate paste flow-through and non-flow-through regions on the screen, respectively. The screens were made from 800 mesh polyester fabric on a wooden frame. Five series of screens were prepared according to flow-throw regions (radius of the dot = 4.5 mm; number of dots on a screen (210 × 297 mm): 25, 50, 100, 200, 300), as shown in Fig. 1.

Screen-printing process

The fabrication process of the developed method is displayed in Fig. 2. The finishing paste was poured on each screen on cotton fabric and spread with squeegee by traveling one cycle of back and forth. Then, the printed fabrics were dried in a convection oven at 80 °C for 30 min and hot-pressed at 160 °C for 30 s. Finally, the fabrics were thoroughly washed with distilled water and dried completely.

Measurement and analysis methods

Add-on (%) of the gallotannin printed on the cotton fabrics was measured using the weighing method based on the weight changes of the fabric before and after printing.

Surface morphologies of the gallotannin-printed cotton fabrics were observed using a high-resolution field emission scanning electron microscope (SEM, Tescan, Brno, Czech Republic).

Changes in the molecular structures on the gallotannin-printed cotton fabric were analyzed using an infrared spectrometer (Fourier-transform infrared spectroscopy, FTIR). The FTIR spectrum analysis device (100 FTIR spectrum, Perkin-Elmer, MA, US) was used with a resolution of 4 cm^{-1}, and attenuated total reflection (ATR) was used to obtain the results.

Color changes in the gallotannin-printed cotton fabrics were investigated using a Datacolor spectrophotometer (Technical Color Solution, Karachi, Pakistan), and the values of changes in colors (ΔE) were compared using L*, a*, and b* values.

The ability of the gallotannin-printed cotton fabrics to prevent microbial growth and retention was tested using *Staphylococcus aureus* (*S. aureus*) (ATCC 6538; a Gram-positive bacterium) and *Klebsiella pneumoniae* (*K. pneumoniae*) (ATCC 4352; a Gram-negative bacterium) cultures according to an established protocol (KS K 0693).

$$\text{Bacterial reduction (\%)} = \frac{(B - A)}{B} \times 100 \tag{1}$$

In formula (1), A and B represent the surviving bacterial cells (colony-forming units in mL^{-1}) on the plates inoculated with a bacterial solution derived from the printed fabric and a control solution derived from untreated fabric, respectively. To identify the antimicrobial ability of the gallotannin-printed cotton fabrics against laundering, washing cycles were conducted based on KS K ISO 105 C06:2010, A2S (washing temperature: 40 ± 2 °C, washing time: 30 min, 0.4% ECE standard solution + 0.1% natrium used, 10 still balls).

Deodorant properties of the gallotannin-printed cotton fabrics were determined against ammonia and acetic acid using a detector tube method. Fabric samples (10×10 cm) were each put in a Tedlar bag (5 L) which was air-tightly sealed. Subsequently, target gases (100 ppm of ammonia or 50 ppm of acetic acid) were each injected into the bag with 3 L of air. The bags were stably placed under ambient conditions (21 ± 5 °C) for 2 h, and then the gas concentration in the bag was determined using a detector tube.

$$\text{Deodorization rate (\%)} = \frac{(C - A)}{C} \times 100 \tag{2}$$

In formula (2), A and C represent the gas concentration in the bag containing fabric samples and the gas concentration in the bag without fabric samples, respectively.

The ultraviolet (UV)-blocking ability of the gallotannin-printed cotton fabrics was determined based on the 'test method for ultraviolet protection rate and ultraviolet protection factor of textiles (KS K 0850:2019)' proposed by the Korean Agency for Technology and Standards.

Results and discussion

Add-on (%)

Figure 3 shows the add-on (%) of gallotannin-printed cotton fabrics as a function of the dot numbers (printed area) on 210 × 297 mm cotton fabrics. The printed surface areas (%) on each fabric are as follows: 2.55% for the fabric containing 25 ea dots, 5.10% for the fabric containing 50 ea dots, 10.19% for the fabric containing 100 ea dots, 20.40% for the fabric containing 200 ea dots, and 30.58% for the fabric containing 300 ea dots. The amount of gallotannin applied on cotton fabrics through the screen-printing process increased with increasing the number of dots on fabrics and then reached a somewhat equilibrium at approximately 200 ea of dots on the fabric. Furthermore, it was found that the cotton fabrics just printed with gallotannin/thickener paste and dried at 80 °C for 30 min (Fig. 3a) have lost some weight after the sequent process; curing, washing and drying (Fig. 3b). This is because un-bonded gallotannin moiety and thickener were washed off through the rinsing process even though the printed fabrics were cured at a high temperature (160 °C/30 s). However, the cotton fabrics printed with above 200 ea of gallotannin dots remained with approximately 2.8% add-on after completing the entire fabrication process.

Surface morphology and characteristics

Figure 4 shows the surface morphologies of pristine cotton fabric, gallotannin-printed cotton fabric, and the gallotannin-printed cotton fabric after five cycles of laundering. The gallotannin/thickener coating was distinctly observed on the surface of the printed cotton fabric (Fig. 4b). However, the coating substrate was significantly reduced by five cycles of laundering, even though some trace of the coating was still observed on the cotton fabric (Fig. 4c).

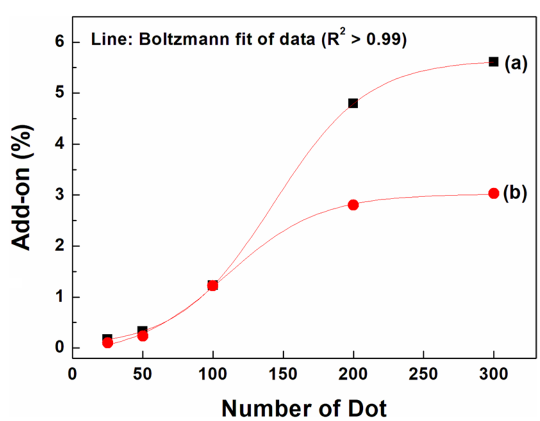

Fig. 3 Add-on (%) of gallotannin-printed cotton fabrics: **a** after gallotannin printing and drying, **b** after completing the whole printing process including printing, drying, curing, washing, and drying

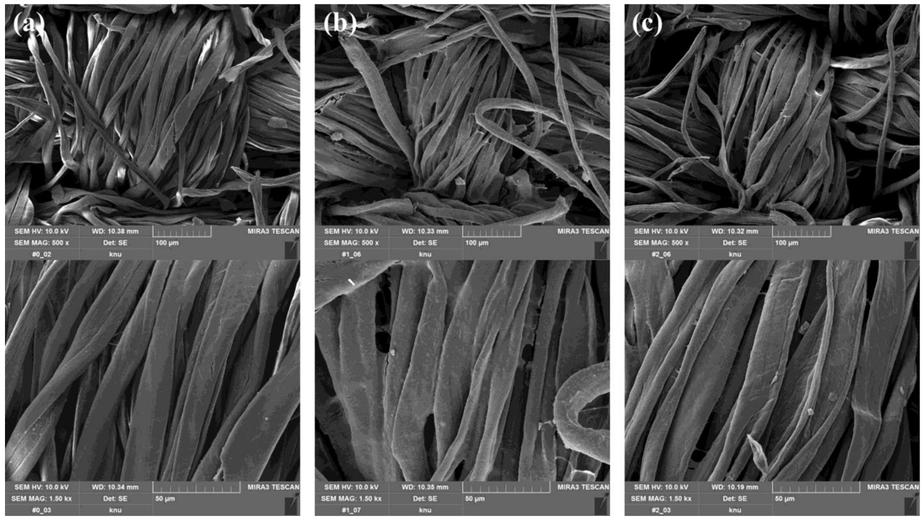

Fig. 4 SEM images of cotton fabrics: **a** pristine, **b** gallotannin printed, and **c** gallotannin printed after five cycles of laundering (magnification: ×500 in top row, ×1500 bottom row)

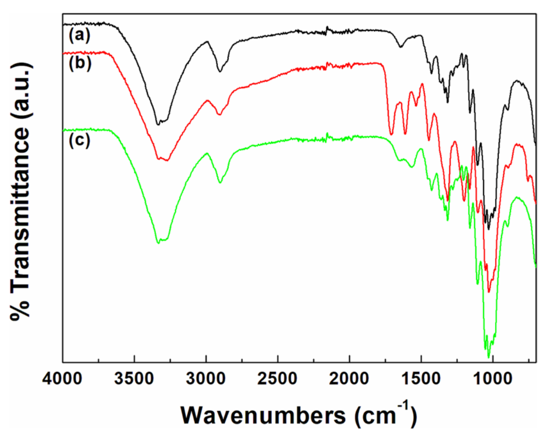

Fig. 5 FTIR-ATR spectra of gallotannin-printed cotton fabrics: **a** pristine, **b** gallotannin printed, **c** gallotannin printed after five cycles of laundry

Figure 5 displays the FTIR spectra of pristine cotton fabric, gallotannin-printed cotton fabric, and gallotannin-printed cotton fabric after five cycles of laundering. All fabrics showed cotton characteristic absorption bands such as O–H stretching vibrations in the range of 3500–3200 cm^{-1}, C–H stretching absorption at approximately 2907 cm^{-1}, and C–O asymmetric bridge stretching and C–O–C pyranose ring skeletal vibrations at approximately 1052 cm^{-1}, β-glycosidic linkages at approximately 895 cm^{-1} (Shukla & Athalye, 1994). However, new peaks were observed in the cotton fabrics after gallotannin printing, as shown in Fig. 5b. A strong peak at approximately 1707 cm^{-1} is possibly attributed to C=O stretching from a residual acrylic compound (Magalhães et al., 2012) in the thickener, and a peak at approximately 1610 cm^{-1} is attributed to C=C stretching from aromatic rings in the gallotannin structure (Jahanshahi et al., 2016). This result is easily assumed due to the notable observation of the gallotannin/thickener coating

Table 1 Color revelation characteristics of gallotannin printed cotton fabrics

Cotton fabrics	Lightness (L*)	Red/Green Value (a*)	Blue/Yellow Value (b*)	ΔE
Pristine	90.66	− 0.07	0.84	–
Gallotannin printed	72.71	6.82	21.68	28.35
Gallotannin printed after five cycles of laundering	81.39	1.15	19.01	19.64

Table 2 Antibacterial ability of gallotannin printed cotton fabrics as a function of the number of gallotannin printed dots on 210 × 297 mm cotton fabrics

Number of printed dot (Printed area on fabrics (%))	*S. aureus*	*K. pneumoniae*
25 (2.55)	98.8	86.1
50 (5.10)	99.6	98.6
100 (10.19)	99.9	99.1
200 (20.40)	99.9	99.6
300 (30.58)	99.9	99.8

on the printed cotton fabrics, as shown in Fig. 4b. A significant amount of coating was washed off through five cycles of laundering, as shown in Fig. 4c. However, it was found that the gallotannin trace remained after five cycles of laundering because peaks at 1663 cm^{-1} (vibration of the carbonyl C=O) and 1563 cm^{-1} (aromatic C=C bond stretching) originating from gallic acid were observed, as shown in Fig. 5c (Koh & Hong, 2014). Therefore, it was presumed that the chemical interactions took place between the carboxyl group of gallic acid originating from gallotannin (one of the hydrolysable tannins) and hydroxyl group in cotton cellulose, which reinforces the interaction between gallotannin functional moiety and cotton fiber (Gao et al., 2019).

Color revelation characteristics

Table 1 presents the color revelation characteristics of gallotannin-printed cotton fabrics and the gallotannin-printed cotton fabric after five cycles of laundering. It was observed that a*, b* values increased; however, L* values decreased on the surface of the gallotannin-printed cotton fabrics. This indicates that the cotton fabric became darker, and the color changed to more reddish and yellowish through the gallotannin printing process. However, the color of the gallotannin-printed cotton returned to the initial pristine cotton color to some extent after five laundering cycles. Since thickener was used during the screen-printing process to apply the functional material 'gallotannin' to the surface of cotton fabric, the gallotannin moiety un-bonded to cotton cellulose was thought to be easily removed with the thickener, as shown in Fig. 4c.

Antimicrobial ability

Table 2 presents the antimicrobial ability of gallotannin-printed cotton fabrics as a function of the dot number on the 210 × 297 mm cotton fabric. It was observed that the gallotannin-printed cotton fabrics containing more than 100 printed dots reduce > 99% of bacteria against *S. aureus* and *K. pneumoniae*. In particular, the printed fabrics seem more effective on *S. aureus* than on *K. pneumoniae*. Tannins are well known to inhibit the growth of various microbes, such as bacteria and fungi. However, the antimicrobial effect of gallotannin-printed cotton fabrics against *K. pneumoniae* was significantly reduced after laundering, as shown in Fig. 6. According to Farha's study (2020), "The minimum inhibitory concentrations (MIC) of various types of tannins are found in the range of 61.5–3200 μg/mL. Compared to Gram-negative bacteria, the MIC values of tannins are much lower for Gram-positive bacteria because of structural differences of the bacterial cell envelope". Therefore, it was thought that the gallotannin moiety was washed off with laundering, and the remaining amount of gallotannin on the fabrics after five laundering cycles placed in the range of MIC values, which are effective to *S. aureus,* a Gram-positive bacterium, but not *K. pneumoniae,* a Gram-negative bacterium.

Deodorant properties

Unpleasant odors in daily human life predominantly come from various sources including the human body and unsanitary living conditions. In particular, the growth of microorganisms on textiles may generate unpleasant body odors. It is already recognized that sweat is initially odorless; however, it becomes odorous through degradation by certain species of bacteria on the surface of clothing materials and/or human skin. Thus, antibacterial finishing applied to textiles could inhibit the growth of odor-causing bacteria and eventually prevent odor buildup in textile products. In addition, researchers found some functionalization of textiles are effective to reduce the unpleasant odor, and they believed the deodorizing mechanisms are caused by adsorption, chemical decomposition, biological decomposition, and the masking effect which occurs on the surface of textiles. Fibrous materials are innately favorable to adsorb odor molecules due to their high surface area and the active groups on the surface. Thus, both physical and chemical interactions between fibers and odor molecules are presumed to take place simultaneously during the odor adsorption process (Wang et al., 2020). Therefore, gallotannin-printed cotton fabrics were thought to be capable of reducing the generation of unpleasant odor because they have been verified to show antibacterial ability and have many active groups on their surface. The deodorant capacity of gallotannin-printed cotton fabrics was examined against ammonia and acetic acid, as shown in Fig. 7. Both

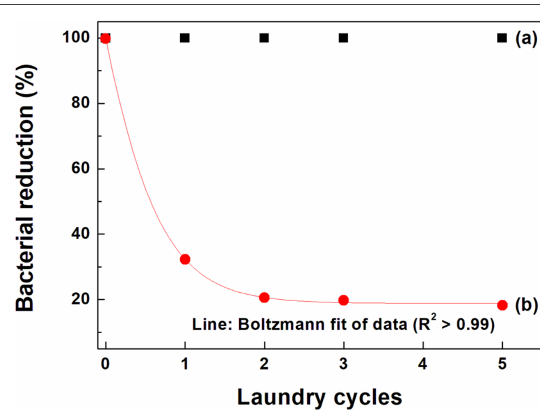

Fig. 6 Antimicrobial capacity of gallotannin-printed cotton fabrics with increasing laundering cycles: **a** against *S. aureus*, **b** against *K. pneumoniae*

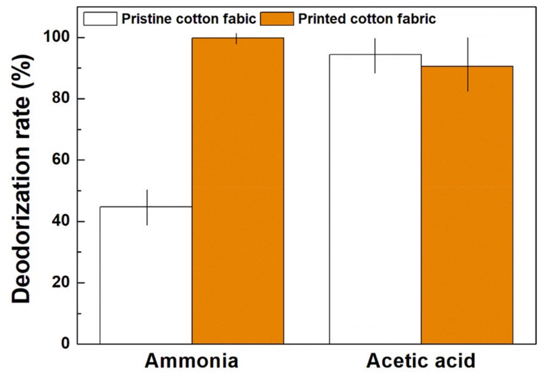

Fig. 7 Deodorization rate of pristine and gallotannin-printed cotton fabrics

Table 3 Ultraviolet (UV)-blocking rate (%) of gallotannin printed cotton fabrics

Wavelength range (nm)	Number of gallotannin printed dots on cotton fabrics sized in 210 mm × 297 mm	
	100	300
UV-R: (190–400)	48.3	74.2
UV-A: (315–400)	45.1	72.5
UV-B: (290–315)	52.4	79.7

ammonia and acetic acid are the main components of sweat and odorous body chemicals. Gallotannin-printed cotton fabrics revealed a much higher deodorization rate of ammonia gas than pristine cotton fabrics. However, there is no significant difference between pristine and gallotannin-printed cotton fabrics in the deodorization rate of acetic acid. The acetic acid molecule appears to be already effectively adsorbed on the surface of cotton fabrics regardless of if the fabrics were treated due to the fibrous structure. Therefore, it was found that the gallotannin-printed cotton fabrics showed excellent deodorant ability (> 99.8% deodorization rate of ammonia and 90.5% of deodorization rate of acetic acid). This result is similar to previous research including Rathinamoorthy and Thilagavathi (2014) who utilized herb *Terminalia chebula* extract as a functional agent for textile finishing. *Terminalia chebula* was reported to contain many phenolic compounds including gallic acid, ellagic acid, and chebulinic acid. Through a subjectively human sensory test, they found the textile finished with *T. chebula* extract showed great performance with up to 47% odor reduction.

UV-blocking ability

The UV-blocking rate of gallotannin-printed cotton fabric was examined according to a 190 to 400 nm wavelength range of UV radiation. The values displayed in Table 3 are relative UV-blocking rates of the printed fabrics compared to those of pristine cotton fabric. It was observed that the gallotannin-printed cotton fabrics showed a UV-blocking rate under the whole range of UV radiation, and the blocking capability was more effective under a shorter wavelength range of UV radiation. This is because phenolic compounds in gallotannin effectively absorb UV radiation, particularly in the wavelength range of 260–280 nm (Choi & Joen, 2009; Koh & Hong, 2019). However, the gallotannin printed area on cotton fabrics seemed to affect the UV-blocking rate. Thus, the UV-blocking rate would be improved by increasing the gallotannin printed area on the surface of cotton fabrics. Furthermore, it was thought that the gallotannin printing on clothing fabrics for outdoor activities could be beneficial to prevent the UV radiation we are exposed to daily and protect our skin.

Conclusions

Screen-printing is a simple technique used to apply coloring patterns on the surface of fabrics. It is well-known to consume less water and energy and produce less wastewater during the process. Thus, it was thought that the technique could be an alternative textile finishing method to conventional methods generating environmentally harmful impacts. Additionally, gallotannin has been identified as a potential material to impart many beneficial functionalities on fabric products in previous studies. Therefore, functionalized cotton fabrics were manufactured using a screen-printing process and gallotannin based agent in this study. Overall, it was found that the gallotannin-printed cotton fabrics revealed antibacterial ability against *K. pneumonia,* a gram-negative bacterium, as well as *S. aureus,* a gram-positive bacterium. The antibacterial ability against *S. aureus* was maintained up to five cycles of laundering; however, that against *K. pneumonia* was impaired. Additionally, the gallotannin-printed cotton fabrics showed a deodorant property toward body odorous compounds including ammonia and acetic acid. Finally, it was observed that the gallotannin-printed cotton fabrics showed UV-blocking ability, which was dependent on the gallotannin printed area on cotton fabrics. Consequently, gallotannin printing on cotton fabrics can be beneficial to contain the growth of some bacteria causing unpleasant odor or diseases on clothing materials. Furthermore, the gallotannin printed clothing materials could protect our skin from irritation by UV radiation in daily life.

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Authors' contributions
KHH conducted all the experimental work, analyzed data, and wrote the manuscript. The author read and approved the final manuscript.

Authors' information
Kyung Hwa Hong is a professor at the Kongju National University.

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Availability of data and materials
The datasets generated and analyzed during the current study. And, they are available from the corresponding author on reasonable request.

Declarations

Competing interests
I declare that the author has no competing interests.

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