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Preparation and properties of cotton fabrics dyed by Aronia (*Aronia melanocarpa*) extract and chitosan

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Abstract

Naturally occurring compounds can be utilized as coloring and finishing agents in environmentally friendly textile products. *Aronia melanocarpa* (Aronia) fruits (berries) are rich in bioactive compounds such as various form of polyphenolic compounds showing antibacterial and antioxidant, and thus numerous studies have been actively conducted on Aronia berry extract (AE) in recent years. However, most natural compounds including pigments exhibit inferior fixation and fastness when applied to cellulosic fibers. Therefore, in this study, coloring and functional compounds were extracted from Aronia berries and applied with or without chitosan to cotton fabrics to improve dyeability and functionality. The cotton fabrics were treated as follows: dyed with an AE single aqueous solution; dyed with an AE–chitosan mixture; and dyed with AE after being pretreated with a chitosan solution. Cotton fabrics dyed with AE and/or chitosan were investigated for their coloring properties and functionality, such as antibacterial properties and antioxidant capacity. It was proven that AE can be used to dye chitosan pretreated cotton fabrics, obtaining good color properties. Moreover, the cotton fabrics dyed with AE after chitosan pretreatment completely inhibited the growth of bacteria (> 99.9%) and exhibited antioxidant properties.

Keywords: Aronia, Cotton, Wool, Antioxidant capacity, Antibacterial ability

Introduction

Aronia (*Aronia melanocarpa*) belongs to the *Rosaceae* family and originates in eastern North America. Aronia berries have been used by Native Americans as both a food and traditional medicine resource (Lee et al., 2014). Aronia berries are a rich natural source of polyphenols, including hydroxycinnamic acids and flavonoids (Kulling & Rawel, 2008; Lee et al., 2014; Woo & Lee, 2021), which possess antibacterial and antioxidant properties (Moure et al., 2001). Moreover, Aronia berries have been reported to have antiproliferative and anticarcinogenic effects, and their extracts have been used for their antiarteriosclerotic, antidiabetic, anti-inflammatory, antiviral, antimutagenic, and immunomodulatory properties (Kulling & Rawel, 2008; Thi & Hwang, 2014). However, the consumption of raw Aronia berries are unpleasant owing to their harsh, sour, bitter, astringent taste, and bitter-almond smell. Moreover, Aronia is known to grow readily from seeds. Hence, the price of Aronia berries decreased significantly in South Korea (The Korea Economic Daily, 2021).

Chitosan is the second most abundant polysaccharide in nature. Chitosan has attracted significant interest because of its intrinsic therapeutic properties, including antibacterial activity, antioxidant activity, cholesterol, and triglyceride trapping effects (Maldonado-Cabrera et al., 2021; Sánchez-Machado et al., 2019). In addition, chitosan has a cationic nature owing to the protonation of amino groups on the polymer backbone, and it becomes a cationic polyelectrolyte upon dissolution in an acidic solution (Moraes et al., 2022). Thus, it was suggested that chitosan can assist cellulose in attracting the natural pigment by acting as a cationizer on the surface of cotton fibers. Cotton is a natural fiber comprised mostly of cellulose. Cotton is the preferred choice for the textile industry and consumers because of its versatility, performance, and natural comfort. Cotton is typically used in several industries and accounts for approximately half of the fibers used in apparel and various textile items (Taausif et al., 2018). However, similar to several other natural substances, cotton is known to provide the basic requirements for bacterial growth and multiplication, such as nutrients, moisture, oxygen, and temperature. This often causes an unpleasant odor, dermal infection, allergic response, and other related diseases (Singh et al., 2005; Thiry, 2001).

Therefore, this study aimed to develop an environmentally friendly textile process for coloring and functionalizing cotton fabrics using Aronia berries and chitosan. In general, the textile finishing using natural extracts show inferior fastness under the normal consuming environment such as UV radiation, wash and others. Therefore, it is expected that this study would provide an effective way to access the problem by utilizing chitosan as a sustainable cationizer in the textile process using the natural extracted functional agents. The Aronia berry extract (AE) was prepared in distilled water, and the extraction conditions used in this study were optimized in our previous study (Koh & Hong, 2022). Cotton fabrics treated with AE and AE/chitosan combinations were investigated in terms of their coloring quality and functional properties, including antibacterial ability and antioxidant capacity.

Methods

Materials and chemicals

Cotton fabric (No. 400, plain woven, 98 g/m^2) was purchased from Testfabrics, Inc. (PA, USA). Ripe *Aronia melanocarpa* berries were harvested from a local farm (Jinan, South Korea) in July 2021 and then immediately freeze-dried at JPTECH Co., Ltd. (Cheongju-si, South Korea). Freeze-dried Aronia berries were kept at −22 °C in a freezer during the experiments. Low molecular weight chitosan was purchased from Sigma–Aldrich (St. Louis, MO, USA). A 1,1-diphenyl-2-picrylhydrazyl (DPPH) free-radical compound, was purchased from Alfa Aesar (MA, USA). All other chemicals were purchased from Sigma–Aldrich (Seoul, South Korea) and used without further purification.

Preparation methods

Preparation of AE

The Aronia berries were extracted using boiling water. The freeze-dried Aronia (40 g) was immersed in 1 L of distilled water followed by boiling and extraction at 100 °C for 3 h. The extraction step was repeated once for damp Aronia berries, which were obtained after the first extraction process. Subsequently, the crude extract was filtered using filter paper (Whatman™, Tisch Scientific, OH, USA) and then concentrated by reducing it to approximately half of its original volume using a rotary evaporator. Finally, the condensed AE solution was dried to powder form using a freeze dryer (FDU-1200, Eyela, Japan). Figure 1 shows a summary of the extraction process.

Preparation of dyeing and treatment solutions

Dyeing and treatment solutions were prepared according to the following three processes: (i) AE dyeing (one-step process), (ii) AE–chitosan mixture dyeing (one-step process), and (iii) AE dyeing after chitosan pretreatment (two-step process). For (i), 10% (w/v) AE solution was prepared by dissolving AE powder in distilled water. For (ii), an AE–chitosan (10%/1% (w/v)) mixture was prepared by mixing the same amount of each AE aqueous solution (20% (w/v)) and chitosan aqueous solution (2% (w/v) containing 1% (v/v) acetic acid), and shaking the mixture in a water bath for 24 h (30 °C, 130 rpm).

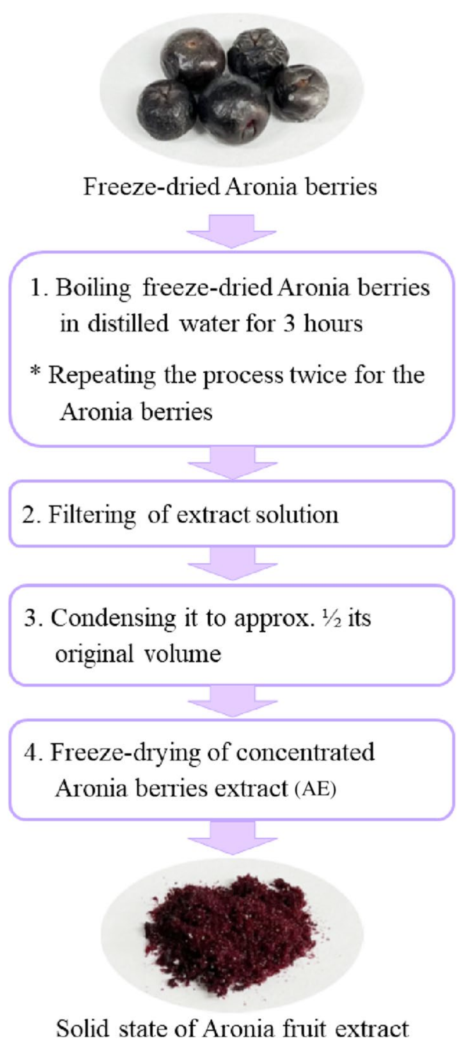

Fig. 1 Schematic process of Aronia berries extract powder preparation

Finally, for (iii), chitosan (1% (w/v) containing 0.5% (v/v) acetic acid) and AE aqueous solutions (10% (w/v)) were prepared.

Fabric dyeing

The cotton fabric was cut into $20 \times 20\ cm^2$ samples, and each sample was immersed in a container containing each dyeing or treating solution. An infrared (IR) dyeing machine (Lab IR dyeing machine, Daelim Starlet Co., Ltd., Gyeonggi-do, Korea) was used for the dyeing and treatment processes. All the steps were performed at 100 °C for 60 min (heating rate: 2.5 °C/min). The ratio of fabric weight to solution was 1:18, and the rotational speed of the bath was 45 rpm. The samples were then washed thoroughly with deionized water and dried at 65 °C for 2 h in a convection oven. For the two-step process (iii), the sample was repeatedly subjected to an identical process (from the IR dyeing machine to the drying oven) in the treating and dyeing bath solutions.

Measurement and analysis methods

Analysis of AE and dyeing solution

The total anthocyanin content of the AE was determined using the pH differential method described by Lee et al. (2005). The diluted AE (25 ppm) was mixed with buffer solutions of pH 1.0 or 4.5 (ratio of sample to buffer solution = 1:4), and the absorbance was separately measured at 520 and 700 nm using an ultraviolet–visible spectrophotometer (UV–vis, Biochrom Libra S22, Santa Barbara, CA, USA). The anthocyanin content was expressed as mg cyanidin-3-glucoside equivalents (C3G) per L of extract.

The total phenol content of AE was determined using the method described by Singleton and Rossi (1965) and Velioglu et al. (1998). 100 μL diluted AE (1 ppm) was mixed with 750 μL Folin–Ciocalteu reagent (ten-times diluted), followed by the addition of 750 μL sodium carbonate (7.5%). After the mixture was allowed to react for 60 min in the dark, the absorbance was measured at 725 nm. A calibration curve was obtained by plotting the absorbance values *versus* chlorogenic acid concentrations (25–200 mg/L), and the total phenol content was expressed as mg chlorogenic acid equivalents (CAE) per L of the extract.

The rheological properties of the treatment and dyeing solutions were measured in the steady rate sweep mode using an advanced rheometric expansion system (ARES, Rheometric Scientific, UK) at 25 °C.

Characteristics of dyed fabrics

The color change of the dyed cotton fabrics was investigated using a spectrophotometer (CM-2500d, Konica Minolta, Inc., Osaka, Japan) in terms of the L*, a*, and b* color parameter values and total color difference (ΔE*ab). Color strength (K/S) was analyzed using the Kubelka–Munk equation:

$$\mathrm{K/S} = \frac{(1-\mathrm{R})^2}{\mathrm{R}} \tag{1}$$

where *R* denotes the decimal fraction of the reflectance of the dyed fabric.

The color level was determined using the method described by Chong et al. (1992). The color level of the dyed cotton fabrics was calculated by measuring the reflectance values over the visible spectrum ($\lambda = 390$–700 nm) at 10 nm intervals. Each fabric sample was measured eight times by randomly varying the measurement spot. The relative unlevelness index (RUI) was calculated using the following equation:

$$RUI = \sum_{\lambda=390}^{700} C_\lambda V_\lambda \tag{2}$$

where C_λ and V_λ denote the coefficient of variation of the reflectance value measured at each wavelength and the photopic relative luminous efficiency function, respectively. The degree of color levelness is described according to the RUI value (Table 1).

Color fastness for the dyed cotton fabrics was investigated to washing (KS K ISO 105 C06:2010, A2S, washing temperature: 40 ± 2 °C, washing time: 30 min, 0.4% ECE standard solution + 0.1% sodium, 10 still balls) and to light (KS K ISO 105-B02:2014, xenon arc lamp, blue scale).

The surface morphology of the dyed cotton fabrics was observed using high-resolution field-emission scanning electron microscopy (SEM, Tescan, Brno, Czech Republic).

The molecular changes in the dyed cotton fabrics were analyzed using Fourier-transform infrared spectroscopy (FT-IR) and X-ray photoelectron spectroscopy (XPS). FT-IR analysis was performed with a resolution of 4 cm^{-1} using an FT-IR spectrometer (100 FT-IR spectrum, Perkin–Elmer, MA, US) in attenuated total reflection (ATR) mode. XPS measurements were performed using a spectrometer (MultiLab. ESCA 2000,Thermo VG Scientific, Loughborough, UK). The spectrometer was equipped with an Al–Kα X-ray source set at 15 kV and 0.72 eV. To compensate for the surface-charging effect, all the binding energies were referenced to a C_{1s} neutral carbon peak at 284.48 eV. The surface elemental composition was determined using peak area ratios.

The antibacterial ability of the dyed cotton fabrics was determined 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). The bacterial reduction was determined using the following equation:

$$\text{Bacterial reduction}(\%) = \frac{(\mathrm{B} - \mathrm{A})}{\mathrm{B}} \times 100 \tag{3}$$

Table 1 Suggested interpretation of RUI values

Relative unlevelness index (RUI)	Visual appearance of levelness
< 0.2	Excellent levelness (unlevelness not detectable)
0.2–0.49	Good levelness (noticeable unlevelness under close examination)
0.5–1.0	Poor levelness (apparent unlevelness)
> 1.0	Bad levelness (conspicuous unlevelness)

where *A* and *B* denote the surviving bacterial cells (colony-forming units per mL) on plates inoculated with a bacterial solution derived from the cotton fabric and a control solution, respectively.

The antioxidant capacity of the dyed cotton fabrics was measured using the DPPH assay (Alger, 1997). The fabric sample (500 mg) was immersed in 30 mL 0.15 mM DPPH/methanol solution and allowed to react for 1 h in the dark. The absorbance of the solution was measured at 517 nm using a UV–vis spectrophotometer (S-3100; Scinco, South Korea). The DPPH scavenging ability was calculated using the following equation:

$$\text{DPPH scavenging activity}(\%) = \frac{\mathrm{C} - \mathrm{S}}{\mathrm{C}} \times 100 \tag{4}$$

where *C* and *S* denote the absorbance of the DPPH/methanol solutions from the containers without and with the fabric sample, respectively.

Results and discussion

Analysis of AE

Anthocyanins are water-soluble pigments that display colors ranging from pink to scarlet, purple, and blue, and are found in plant matter (Sudhakar et al., 2016). Anthocyanins are hydrogen donors and possess unpaired electrons, and are thus able to effectively eliminate a variety of reactive oxygen radicals, consequently revealing antioxidant capacity (Wang et al., 2022). Moreover, several studies have shown that anthocyanins have a wide range of pharmaceutical effects, including retinoid regeneration, immunity improvement, antiaging, anti-inflammatory, and antitumor effects (Grobelna et al., 2019; He & Giusti, 2010; Kalisz et al., 2020). The total anthocyanins in the AE prepared in this study was 10.83 ± 0.52 mg (C3G)/g.

In addition, phenolic compounds are important plant constituents with redox properties responsible for antioxidant activity (Aryal et al., 2019; Siddhuraju & Becker, 2003). The hydroxyl groups in plant extracts are responsible for scavenging free radicals and inducing antioxidant activity. The phenolic content was measured using the Folin–Ciocalteu reagent, and the results were calculated from a calibration curve ($y = 0.0063x - 0.0209$, $R^2 = 0.9991$) of chlorogenic acid concentrations (25–200 mg/L). Consequently, the content of total phenolic compounds in the AE was 10.83 ± 0.52 mg (CAE)/g. Velioglu et al. (1998) reported that the antioxidant activity of plant materials correlated with the phenolic compounds.

Therefore, the cotton fabrics dyed with AE were expected to exhibit antioxidant activity. However, the powder form of AE was difficult to handle because of its deliquescence under atmospheric conditions. Thus, it was suggested that an AE stock solution should be prepared for further experiments.

Rheological property of solution

The rheological properties of the treatment and dyeing solutions used in this study were measured (Fig. 2). The chitosan polymers played a significant role in increasing the viscosity and stress of the solutions. Moreover, even though AE alone did not affect the rheological properties in water (Fig. 2a), the AE constituents led to a slight increase in the viscosity and stress of the dyeing solution containing chitosan (Fig. 2c). This suggests

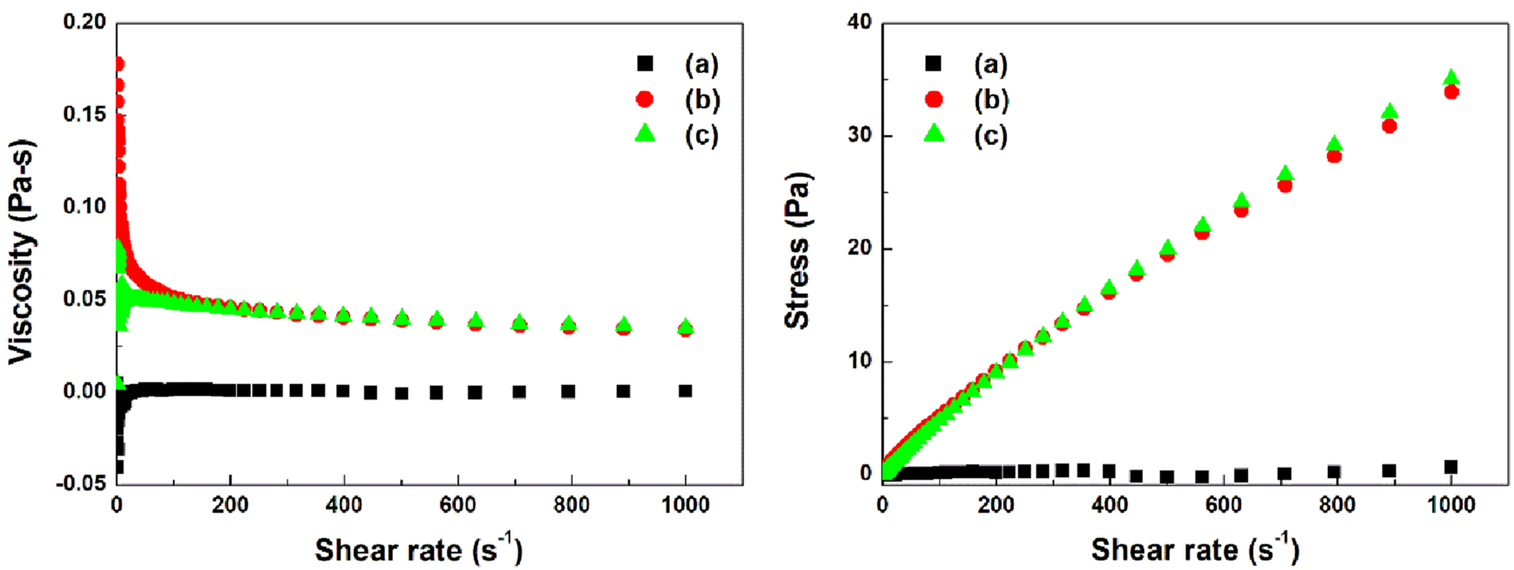

Fig. 2 Rheological properties of treating and dyeing solutions: **a** Aronia berries extract aqueous solution (10% (w/v)), **b** chitosan solution (1% (w/v)), and **c** Aronia berries extract–chitosan (10%/1% (w/v)) mixture

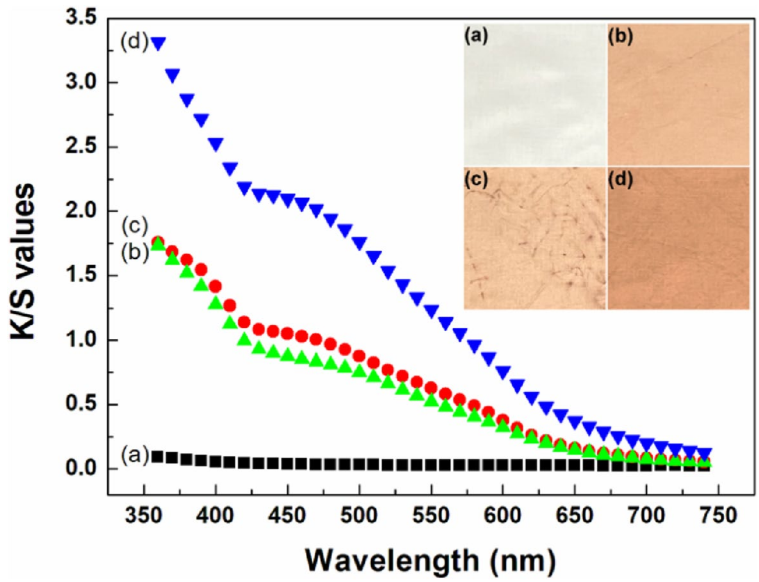

Fig. 3 K/S values and color appearance of cotton fabrics: **a** untreated, **b** dyed with Aronia berries extract, **c** dyed with Aronia berries extract–chitosan mixture, and **d** dyed with Aronia berries extract after chitosan pretreatment

Table 2 Colorimetric data of cotton fabrics

Fabric sample	L*	a*	b*	DE*ab	RUI
Untreated	91.12	− 0.05	1.94	–	–
Dyed with AE alone	66.46	10.49	14.22	29.50	0.09
Dyed with AE–chitosan mixture	68.54	9.61	13.56	27.17	0.58
Dyed with AE after chitosan pretreatment	57.03	11.73	15.60	38.57	0.13

Total color difference (DE*ab) = $[(DL)^2 + (Da)^2 + (Db)^2]^{1/2}$

AE Aronia berries extract, *RUI* Relative unlevelness index

that the addition of AE to the chitosan solution formed a more rigid structure because the abundant tannins in the AE acted as crosslinkers between chitosan polymers.

Rivero et al. (2010) reported that the mechanical properties of tannic acid–chitosan blends were superior to those of neat chitosan owing to the reinforcing effect of the crosslinker throughout the chitosan polymer matrix. They also determined that the addition of tannic acids significantly increased the tensile strength of tannic acid–chitosan composite films to approximately 29%; however, it did not affect the flexibility of the film.

Characteristics of cotton fabrics dyed with AE and AE/chitosan combinations

Color appearance

Figure 3 and Table 2 show the colorimetric data of the cotton fabrics dyed with AE, with the AE–chitosan mixture, and with AE after chitosan pretreatment. It was observed that the a* and b* colorimetric values were the highest on the cotton fabrics pretreated with chitosan and then dyed with AE. Subsequently, both the a* and b* values decreased for the fabric dyed with AE and the fabric dyed with the AE–chitosan mixture. The L* value gradually decreased as the a* and b* values increased. Therefore, the cotton fabric dyed with AE after chitosan pretreatment exhibited a significantly increased reddish and yellowish color and a darker shade compared to the other dyed cotton fabrics. The results indicated that chitosan cationized the cotton surface during pretreatment, and consequently assisted cotton cellulose in forming more hydrogen bonds with the AE constituents (Oliveria et al., 2017).

A schematic representation is shown in Fig. 4 (Agarwal et al., 2018). However, when the cotton fabric was dyed with the AE–chitosan mixture, chitosan hindered the AE pigment access to cotton cellulose and consequently induced uneven AE dyeing on the cotton surface (RUI = 0.58 'Poor levelness'). The other two dyed cotton fabrics exhibit excellent levelness (Table 2). The unlevelness of dyeing were also visually observed from the photos in Fig. 3. This is because the chitosan polymer chains bonded prior to AE in the mixture, and thus prevented AE pigments from evenly accessing the cotton surface. This assumption is supported by the rheological observations of the AE–chitosan and chitosan solutions (Fig. 2).

In addition, the color fastness to washing and light of the dyed cotton fabrics was investigated. No significant differences were observed among the dyed cotton fabrics: color fastness to washing: 3 − 3.5; color fastness to light: 2 − 2.5.

Surface morphology and molecular change

Figure 5 shows the surface morphologies of the cotton fabrics dyed with AE, with the AE–chitosan mixture, and with AE after chitosan pretreatment. Some traces of the treatment were observed on the surface of the cotton fabrics dyed with the AE–chitosan mixture (Fig. 5c) and dyed with AE after chitosan pretreatment (Fig. 5d) owing to the addition of the chitosan polymer. However, it was determined that the amount of coating on the surfaces of both fabrics was not significant, as it affected the touch

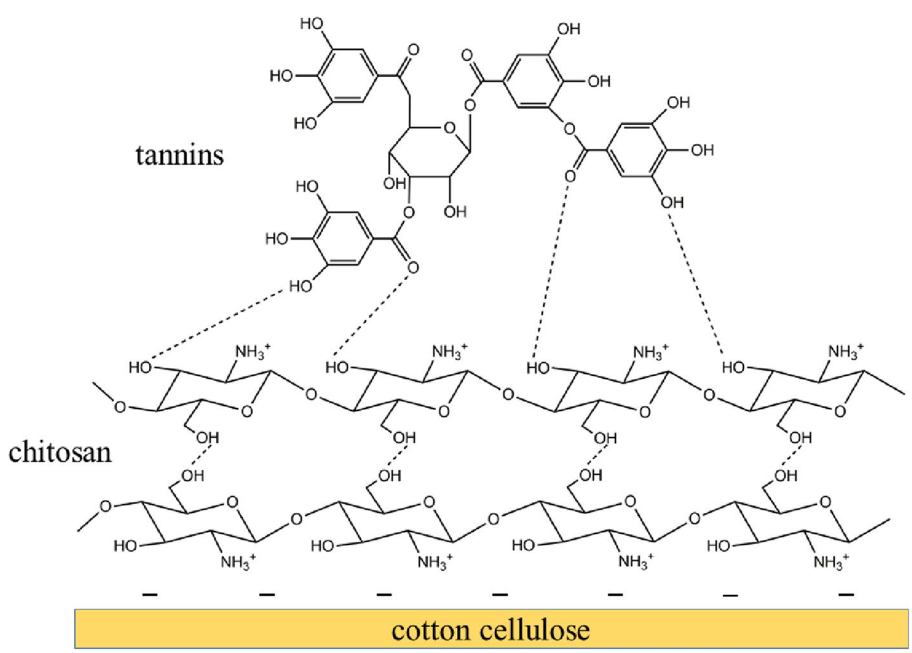

Fig. 4 Schematic representation of cotton fabric dyed with Aronia berries extract after chitosan pretreatment

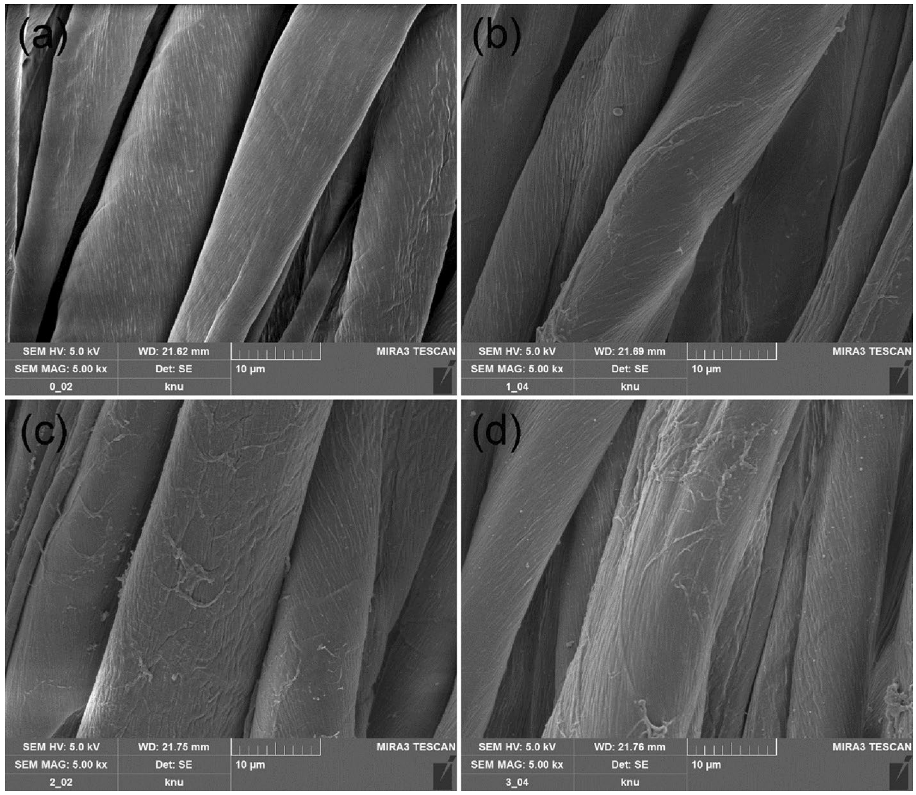

Fig. 5 SEM images (× 5000) of cotton fabrics: **a** untreated, **b** dyed with Aronia berries extract, **c** dyed with the Aronia berries extract–chitosan mixture, and **d** dyed with Aronia berries extract after chitosan pretreatment

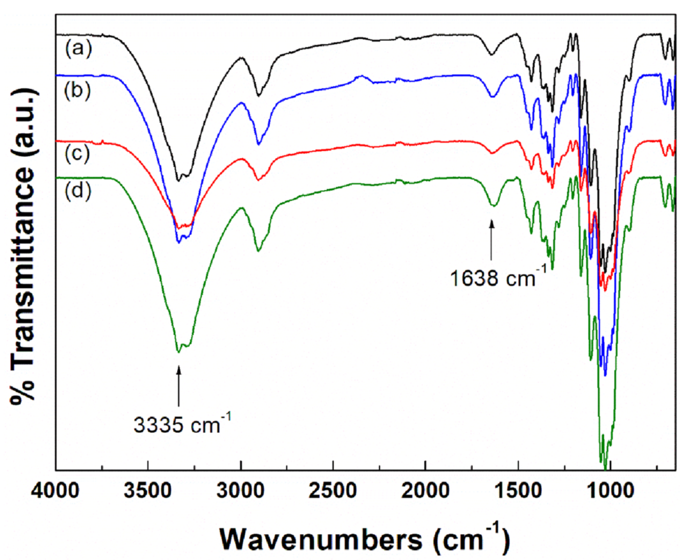

Fig. 6 FTIR-ATR spectra of cotton fabrics: **a** untreated, **b** dyed with Aronia berries extract, **c** dyed with Aronia berries extract–chitosan mixture, and **d** dyed with Aronia berries extract after chitosan pretreatment

of the fabrics or blocked the interstices between the fibers. In addition, small and bright beads (sugar crystals) were observed on the surface of the cotton fabric dyed with AE after chitosan pretreatment (Fig. 5d), indicating that the AE components were more attached on the surface of the coating.

The molecular changes on the surface of the dyed cotton fabrics were investigated by FT-IR spectroscopy (Fig. 6). All spectra exhibit typical bands assigned to cellulose in the region of 1630–900 cm^{-1} (Hospodarova et al., 2018; Hong & Sun, 2008). In addition, the broad peak at approximately 3335 cm^{-1} is characteristic of the stretching vibration of the hydroxyl group in polysaccharides. The peak located at 1638 cm^{-1} corresponds to the vibration of water molecules absorbed by cellulose (Oh et al., 2005). The peak intensity of the hydroxyl group increased in the spectra of the cotton fabric dyed with AE (Fig. 6b), and the cotton fabric dyed with AE after chitosan pretreatment (Fig. 6d). However, the peaks decreased significantly in the spectrum of the cotton fabric dyed with the AE–chitosan mixture (Fig. 6c). This suggests that the AE, containing abundant hydroxyl groups (Fig. 4), endowed more hydroxyl groups on the surfaces of both cotton fabrics dyed with AE alone and dyed with AE after chitosan pretreatment.

Furthermore, fewer hydroxyl groups were observed on the surface of the cotton fabrics dyed with the AE–chitosan mixture because chitosan on the fabric surface has fewer hydroxyl groups in the polymer structure than cellulose. This was supported by the XPS analysis results of the cotton fabric surfaces (Fig. 7). It was observed that nitrogen had the highest presence on the surface of the cotton fabric dyed with the AE–chitosan mixture (1.84 wt.%), and decreased for the fabric dyed with AE after chitosan pretreatment (0.87 wt.%), and the fabric dyed with only AE (0.01 wt.%). This implies that the cotton fabric dyed with the AE–chitosan mixture was covered by more chitosan surface than the other dyed fabrics.

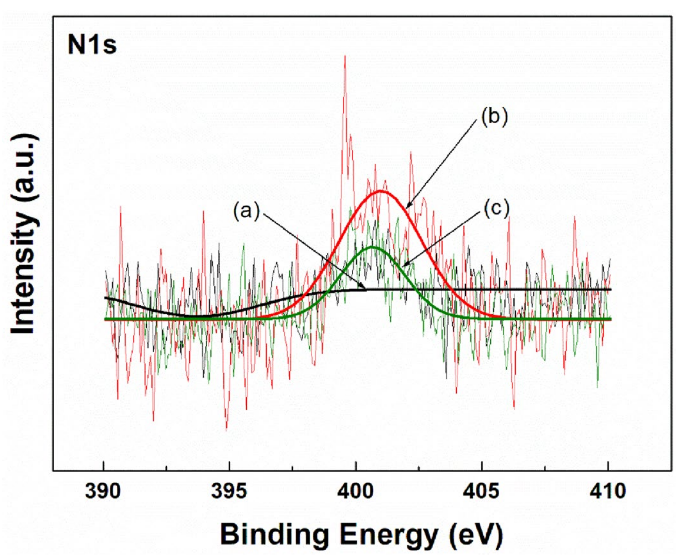

Fig. 7 XPS narrow scans of cotton fabrics: **a** dyed with Aronia berries extract, **b** dyed with Aronia berries extract–chitosan mixture, and **c** dyed with Aronia berries extract after chitosan pretreatment

Table 3 Antibacterial activity of cotton fabrics

Fabric sample	*S. aureus* (%)	*K. pneumoniae* (%)
Untreated	12.6	10.6
Treated with chitosan	16.8	21.4
Dyed with AE	99.5	87.8
Dyed with AE–chitosan mixture	> 99.9	98.2
Dyed with AE after chitosan pretreatment	> 99.9	> 99.9

AE Aronia berries extract

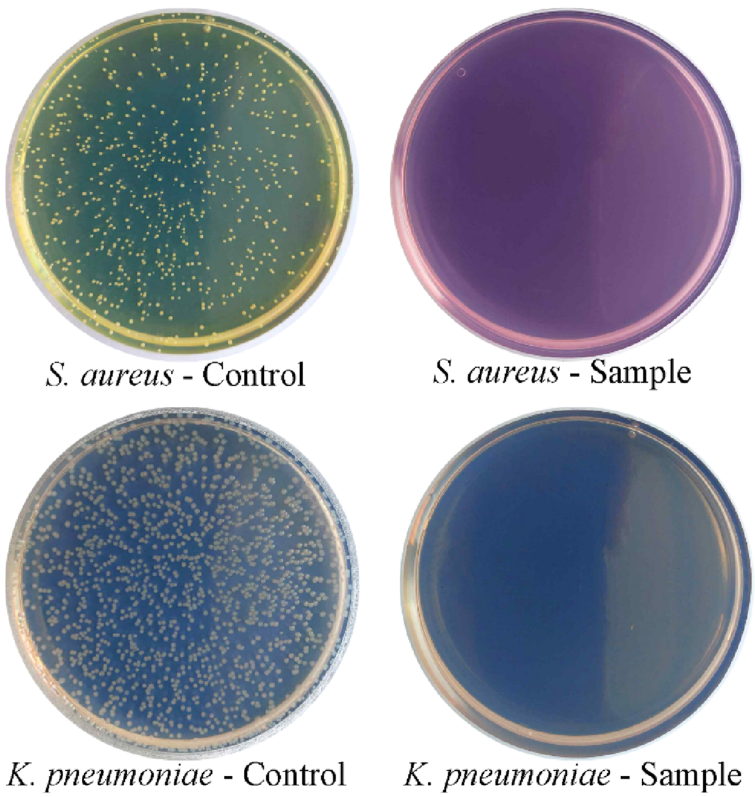

Fig. 8 Antibacterial test results of cotton fabrics dyed with Aronia berries extract after chitosan pretreatment

Antibacterial ability

Table 3 summarizes the antibacterial ability of cotton fabrics dyed with AE, with the AE–chitosan mixture, and with AE after chitosan pretreatment. The cotton fabric dyed with AE after chitosan pretreatment reduced the colony numbers of *S. aureus* and *K. pneumoniae* (> 99.9%) the most effectively (Fig. 8). In addition, the cotton fabrics dyed with the AE–chitosan mixture and only AE exhibit less antibacterial ability (Table 3). In particular, the antibacterial ability significantly decreased against the gram-negative bacterium *K. pneumoniae*. This is typical for multiple plant-born antibacterial compounds, which exhibited excellent antibacterial abilities against gram-positive bacteria but poor antibacterial abilities against gram-negative bacteria (Cisowska et al., 2011; Hong, 2021).

However, Aronia berries are a rich natural source of polyphenols. Polyphenols in food sources have been actively studied during the last decades due to their wide range of biological activities including anticarcinogenic, antiatherosclerotic, antimicrobial, and antioxidant. The therapeutic properties of polyphenols have been thought to be attributed to their antioxidative activity by scavenging free radicals. However, it is recently suggested that their capability to modulate some cellular enzyme activities be involved in contributing the biological activities (Caturla et al., 2003). Efenberger–Szmechtyk et al. (2021) reported that polyphenols can alter bacterial cell morphology by damaging the cell membrane, resulting in the leakage of intracellular material. They can increase reactive oxygen species generation and consequently induce oxidative stress in bacterial cells. They can also affect protein biosynthesis, resulting in changes in the metabolic processes.

Moreover, the antibacterial ability of the cotton fabric treated with chitosan aqueous solution (1% (w/v)) was investigated for thoroughness. The cotton fabric treated with chitosan did not exhibit any antibacterial activity against *S. aureus* or *K. pneumoniae*. This is because the chitosan used in this study has a low molecular weight (M_w), and the M_w cannot impose an antibacterial effect on cotton fabrics. Thus, it was determined that low-M_w chitosan acts as a cationizer and/or crosslinker between cotton cellulose and AE constituents, not as a synergistic agent for AE. Consequently, the low-M_w chitosan polymers enhanced the attraction of AE functional compounds to cellulose fibers through the process, bolstering the functionalities of AE from the treated cotton fabrics. Lizardi–Mendoza et al. (2016) reported that the degree of acetylation and M_W are the main characteristics of chitosan. These have a determining effect on the functionality of chitosan, from its solubility and material-forming capacity to biodegradability and diverse bioactive attributes.

Antioxidant capacity

Figure 9 shows the antioxidant capacity of cotton fabrics dyed with AE, with the AE–chitosan mixture, and with AE after chitosan pretreatment. The cotton fabric dyed with

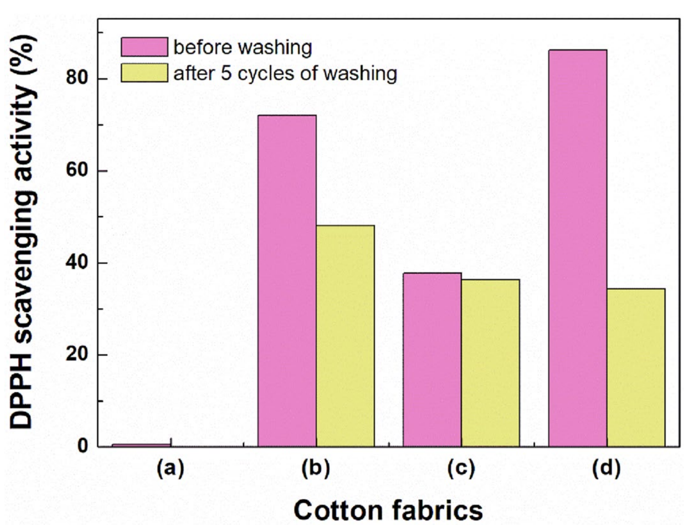

Fig. 9 Antioxidant capacity of cotton fabrics: **a** untreated, **b** dyed with Aronia berries extract, **c** dyed with the Aronia berries extract–chitosan mixture, and **d** dyed with Aronia berries extract after chitosan pretreatment

AE after chitosan pretreatment exhibited the highest antioxidant capacity (86.19%), followed by the fabrics dyed with AE (72.04%), and with the AE–chitosan mixture (37.77%). Therefore, it was determined that the AE presence on cotton fabric surfaces had a more direct effect on antioxidant capacity than on antibacterial ability.

In addition, the antioxidant capacity was determined after five washing cycles based on KS K ISO 105 C06:2010 (Hong, 2021). It was observed that the cotton fabrics dyed with AE and with AE after chitosan pretreatment exhibited a significantly deteriorated antioxidant capacity compared to the cotton fabric dyed with the AE–chitosan mixture. Hence, it was determined that chitosan functions as a crosslinker between AE and cotton cellulose, and thus prevented AE from being 'lost' during washing. However, because the absolute value of the antioxidant capacity of the cotton fabric dyed with the AE–chitosan mixture was not superior to the other two dyed fabrics even after five washing cycles, we cannot say that the cotton fabric dyed with the AE–chitosan mixture is durable to washing in terms of antioxidant capacity. According to Kim et al. (2007), DPPH radical scavenging activity is highly correlated with the amount of phenolic compounds and proanthocyanins. Therefore, it was presumed that the AE constituents, including phenolic compounds and proanthocyanins, were significantly removed from the dyed cotton fabrics following five washing cycles.

Conclusions

Aronia berries contain high levels of bioactive compounds and natural purple pigments. In addition, chitosan has a cationic nature owing to the protonation of the amino groups on the polymer backbone. Thus, chitosan treated on the cotton fabric surface was suggested to assist cotton cellulose in attracting and bonding to the Aronia berry pigments by functioning as a cationizer. Therefore, Aronia berry extract (AE) and chitosan were applied to cotton fabric in the following three ways: dyed with AE; dyed with AE–chitosan mixture; and dyed with AE after chitosan pretreatment. The dyed cotton fabrics were investigated based on coloring and functionality. The total anthocyanin and phenolic contents of the AE prepared in this study were also analyzed. Hence, it was observed that the AE contains significant amounts of total anthocyanins (10.83 ± 0.52 mg (C3G)/g) and phenolic compounds (10.83 ± 0.52 mg (CAE)/g). In addition, it was observed that the cotton fabric pretreated with chitosan and then dyed with AE exhibited the most excellent coloring and functionality (antibacterial and antioxidant). The cotton fabric dyed with the AE–chitosan mixture exhibited superior antibacterial ability, but the worst antioxidant capacity among the dyed cotton fabrics. Moreover, dyeing with the AE–chitosan mixture induced uneven coloring of the cotton fabric, likely owing to the agglomeration of AE and chitosan in the dyeing bath. Therefore, both Aronia berries and chitosan can be sustainable sources of textile coloring and functional agents that can replace toxic synthetic chemicals. The two-step process of dyeing with AE after chitosan pretreatment would be an effective method of applying AE and chitosan to cotton fabrics based on coloring and functionality.

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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.

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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.

Ethics and consent
Not applicable.

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