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Effect of Chitosan and Tannin Treatment on the Functional Manifestation of *Coptidis Rhizoma* Dyed Fabrics

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Abstract

Lifestyle of Health and Sustainability (LOHAS) influences the development of hygienic and health-oriented functional textile products; consequently, there has been an increase in the prospects for environmentally friendly natural dyeing products with functional and sensitive characteristics. Therefore, the present study expresses a functional manifestation on fabrics by treatment with chitosan and tannin using natural *Coptidis Rhizoma*. Cotton and wool fabrics dyed with *Coptidis Rhizoma* after treatment with chitosan and tannin. Consequently, the antibiosis of fabric dyed with *Coptidis Rhizoma* was excellent with 99.9% bacterial reduction for *Staphylococcus aureus* and *Klebsiella pneumoniae* in both fabrics. The deodorization rate was more than 90.0% in both fabrics after 120 minutes. The UV-A protection rate was 88.2% and the UV-B protection rate was 87.7% for cotton. The UV-A protection rate was 88.2% and the UV-B protection rate was 84.3% for wool fabric. Therefore, both of the dyed fabrics showed excellent UV protection by treatment with only chitosan and tannin, without mordants. Therefore, chitosan and tannin treatments were able to manifest functionalities in the fabrics dyed with *Coptidis Rhizoma*.

Key words**:** *Coptidis Rhizoma*, Chitosan, Tannin, Functionality

I. Introduction

In recent years, the quality of human life has been considerably improved, and the LOHAS lifestyle has been spreading to pursue sustainability and global environmental preservation, as well as individual mental and physical health. This tendency has influenced the development of hygienic and health-oriented functional textile products, thereby leading to the growth of the environmentally-friendly natural dye product market with functional and emotional characteristics.

Natural dyes are eco-friendly because they consist of natural components. However, since they have a weak binding force with fiber polymers, metal mordants are used to promote the physical bonding force between fibers and natural dyes. These metal mordants are mostly heavy metals, which can harm the human body and, when excessive amounts of them are used, the defect phenomena such as scratches, fading, or spots, are occurred. In addition, wastewater discarded after dyeing can cause environmental pollution. Therefore, in the context of resource depletion and environmental pollution problems, the use of chitosan can reduce the use of mordants.

Chitosan is a useful polymer substance that can be recycled as a resource, has affinity with natural dyes, is the second richest natural polymeric material. Chitosan serves as a metal mordanting agent, as it is a polyamine that has been used as a natural chelating agent for a long time (Kim, 2000). In addition, it has a high affinity with natural dyes and facilitates the

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process of adsorption of mordant to dyes (Kang, 2002). In the case of cotton fiber, it is necessary to treat the metal mordant, because it is low reactivity with the pigment component of most natural dyes. However, compared to cotton fibers, wool fibers can be easily dyed, because wool fibers have -NH_2 group which is more reactive than -OH group. Therefore, chitosan treatment will contribute to the enhancement of dyeability between fibers and natural dyes. Because structure of chitosan is that one of the -OH groups in cellulose molecular structure are replaced with -NH_2 group, so that the -NH_2 group in chitosan molecular structure promotes effective dyeing of cotton fibers (Jeon et al., 2004).

Among natural products, phytogenic and monochromatic basic dyes, which contain berberine, an alkaloid pigment, can be expected to have a physiological activity. However, due to the high price, the *Coptidis Rhizoma* which has been more frequently used as a herb medicine, rather than as a dye. Therefore, *Coptidis Rhizoma* was replaced with *Phellodendron amurense,* because the latter contains the same berberine pigment and has a relatively easy dyeing and lower cost. *Coptidis Rhizoma* is a perennial herbaceous plant belonging to the *Ranuculaceae* family (Kim et al., 2012; Pukyong National University, 2008). Its main constituent is berberine, an isoquinoline alkaloid alkaline (Fig. 1).

It also contains components such as palmatine, coptisine, epiberberine, worenine, and lignan. Specifically, berberine has been reported to have antimicrobial function against Gram-positive and Gram negative bacillus, as well as a variety of bioactivity and antioxidative properties such as anti-inflammatory action, hemostasis, and reducing blood pressure (Kim, 2008). In addition, the antimicrobial activity of *Coptidis Rhizoma*, when it is extracted with hot water (Choi, 2010), is twice higher than that of *Phellodendron amurense* (Doh, 1999), although the two are similar in terms of their principal components.

As shown in <Fig. 2>, tannin is a water soluble polyphenol compound widely distributed in leaves, shells, grains, and fruits (Wu et al., 2004). It has a hydrolyzable tannin, which is easily degraded by acids or enz-

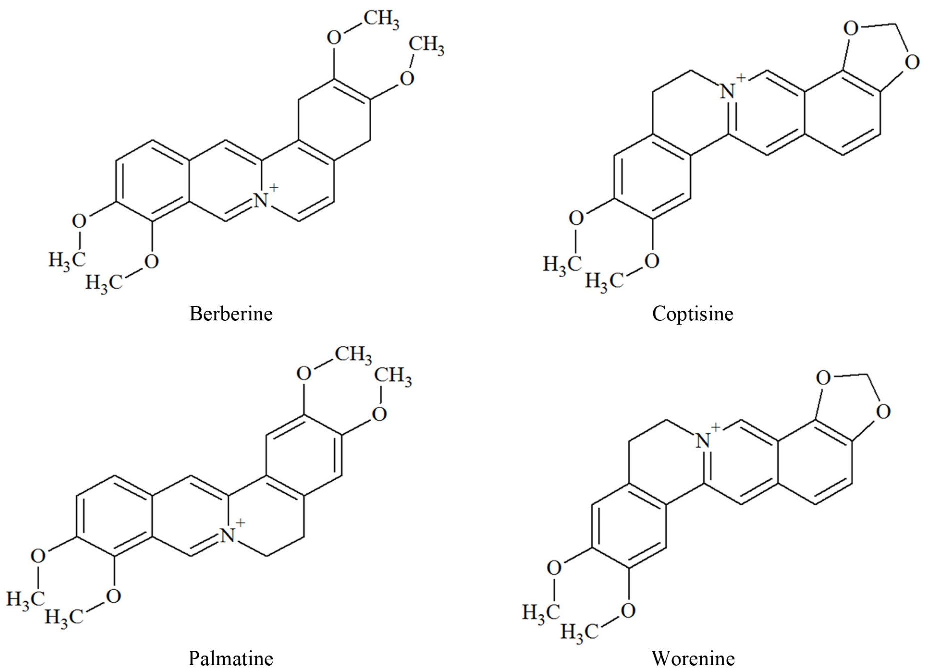

Fig. 1. The main constituents of *Coptidis Rhizoma*.

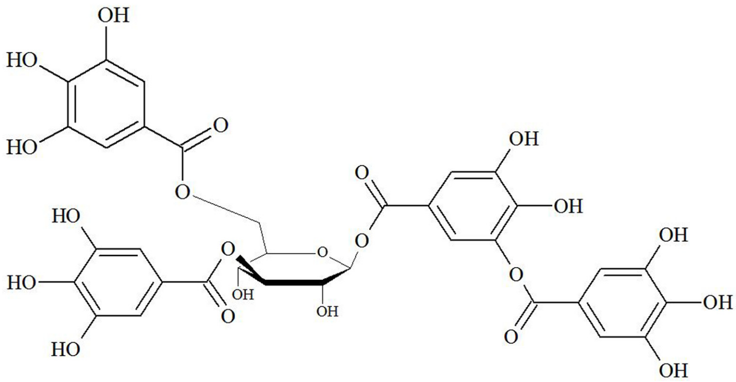

Fig. 2. Chemical structure of tannin.

ymes, and a condensed tannin, which is stable and less reactive (Salunkhe et al., 1990). The hydrolyzable type is pyrogallol tannin based on gallic acid and containing -OH group, -COOH group and their esters. Condensed type is based on flavone, and only -OH group is reacted with catechol-based tannin (Sul et al., 1995). However, tannin treatment of fibers has been used for adhesion of basic dyes to cotton fibers and for imparting excellent dyeing affinity and resisting the printing effect to wool fibers. In the case of *Coptidis Rhizoma*, berberine, which is the main component, it is difficult to adhere to vegetable fibers because of its basicity; however, it can enhance the effect of dyeing through tannin treatment. In the case of tannin having a high molecular weight, it also has a good affinity with wool fabric (Kwon et al., 2006). Animal fiber is dyed bright yellow, but has a disadvantage of poor light fastness. Since the ionic bond formed between the cationic group berberine dye and the anionic group of wool fiber is easily broken by the sunlight, through tannin treatment, light fading suppression can be expected (Lee et al., 2000).

Therefore, in the present study, we aimed to investigate the functionalization by the physiological activity of natural materials by dyeing natural fibers. To improve the efficiency of dyeing of natural dyes, chitosan, a natural polymer compound with a good affinity to both fibers and dyes, was used. Tannin was treated to improve fastness, and mordants were employed to fix the dye and change color. By examining the physiological activity of the *Coptidis Rhizoma* and by evaluating the functionalities such as antimicrobial activity, deodorization and ultraviolet light blocking rate, we also intended to provide basic data for the development of high value added eco-friendly materials.

II. Experimental

1. Materials and Reagents

1) Materials

Cotton fabrics (Standard Adjacent Fabrics for Staining of Fastness Test: KS K 0905) (Korean Agency for Technology and Standards [KATS], 2015) and wool fabrics (Standard Adjacent Fabrics for Staining of Fastness Test: KS K ISO 105-F01) (KATS, 2014a) were used (Table 1).

Table 1. Characteristics of fabrics

Fiber	Weave	Thickness (mm)	Fabric count (Threads/cm)		Weight (g/m^2)
			Warp	Weft	
Cotton 100%	Plain	0.22	41	32	120
Wool 100%	Plain	0.35	22	19	128

2) Dyes and Extraction

Coptidis Rhizoma, dyes, were supplied by natural-dyeing inc. Dyeing solutions were obtained by extraction from *Coptidis Rhizoma* powder using water as extraction solvent at 90°C for 2 hours. Next, impurities were filtered with a polyester filter fabric, and distilled water was added there to prepare a dyeing solution of a predetermined concentration.

3) Reagents

Aluminium potassium sulfate dodecahydrate ($AlK(SO_4)_2 \cdot 12H_2O$), Stannic (II) chloride anhydrous ($SnCl_2$), Iron (II) sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$), Copper (II) acetate monohydrate ($(CH_3COO)_2Cu \cdot H_2O$)) were used as mordants; acetic acid (CH_3COOH) was also used; all reagents were grade 1.

2. Experimental Method

1) UV Spectrum of Dye Component

To investigate the absorbance of the dyeing solution at the maximum absorption wavelength, the absorbance of the solution with 5% (o.w.b.) concentration diluted to 0.5% (v/v) was measured using a UV-VIS spectrophotometer (T-60, UK) at 300-700nm.

2) FT-IR Spectrum of Dye Component

To confirm the structure of powdered *Coptidis Rhizoma*, FTIR spectrophotometer (4200 type A) was used to measure the infrared spectra in the wavenumber of 500-4000cm^{-1}.

3) Chitosan Treatment

The chitosan used in present study was supplied from Chembio Co., Ltd. and its characteristics are shown in <Table 2>. The chitosan solution was prepared by dissolving chitosan in a 1% (v/v) acetic acid aqueous solution at the concentration of 1% (w/v). The fabrics were immersed in the prepared chitosan solution for 30 minutes and then made pass through a mangle to be wet pick up of the chitosan solution to 100±2%. The fabric treated with chitosan was thoroughly washed with flowing water, pre-dried, and then heat-treated at 120°C for 3 minutes.

Table 2. Characteristics of chitosan

Particle condition	powder
Degree of deacetylation	95.0%
Particle size	80 mesh
Average molecular weight	3.0×10^5
Viscosity (in 1% acetic acid)	36 cps
Moisture content	6.0%
Residue on ignition	0.13%

4) Tannin Treatment

The tannin treatment used a hydrolysable tannin ($C_{76}H_{52}O_{46}$, MW: 1701.23), and were supplied by Samchun Chemicals Co., Ltd. The fabrics treated with chitosan were added to the tannin solution at the concentration of 5% (o.w.f.), treated at the temperature of 70°C for 1 hour at the bath ratio of 50:1, sufficiently washed with water, and then naturally dried at room temperature.

5) Dyeing and Mordanting

(1) Dyeing

Dyeing condition was the concentration of 5% (o.w.b.) *Coptidis Rhizoma* extract solution with the bath ratio 1:50 at 80°C for 90 minutes. Dyed fabrics were washed with water and naturally dried at room temperature.

(2) Mordanting

The mordants were treated with four kinds of mordants (Al, Sn, Fe, Cu) at the concentration of 5% (o.w.f.) with the bath ratio 1:50 in a shaking incubator at 50°C for 30 minutes. The mordanting method was performed using pre-mordanting (mordanting-washing-drying-dyeing-washing-drying).

6) Antibiosis

The antimicrobial activity of the dyeing fabric was evaluated by Bioassay test according to KS K 0693 (KATS, 2016). *Staphylococcus aureus* (ATCC 6538) (American Type Culture Collection [ATCC], 2018b) and *Klebsiella pneumoniae* (ATCC 4352) (ATCC, 2018a) were used. The bacterial reduction rate (%) was determined by the method of bacterial count, which is the relative decrease rate of each viable cell count after culturing for 18 hours for the control and test specimens, and the bacteriostatic reduction rate was calculated using <Eq. 1>.

$$\textit{Bacterial reduction rate}\ (\%) = \frac{M_b - M_c}{M_b} \times 100 \quad \cdots\cdots \text{Eq. 1.}$$

where, M_b: the number of microbe in blank specimen incubated for 18 hours

M_c: the number of microbe in test specimen incubated for 18 hours

7) Deodorization

Deodorization of dyed fabrics was evaluated using the gas detection method which measures the rate of disappearance of ammonia gas after injecting a 10cm ×10cm dyed fabrics and an aqueous ammonia solution (concentration 500μg/mL) in 30, 60, 90, and 120 minutes in a test environment of 20°C and 65% RH. Then, ammonia gas concentration was measured. The deodorization rate was calculated using <Eq. 2>.

$$\textit{Deodorization rate}\ (\%) = \frac{A - B}{A} \times 100 \quad \cdots\cdots \text{Eq. 2.}$$

where, A: Gas concentration in a sealed jar of the blank

B: Gas concentration in a sealed jar of the sample

8) UV Protection

The UV blocking rate of the dyeing fabrics was determined according to KS K 0850 (KATS, 2014b). Herein, the ultraviolet ray blocking rate is a percentage of the ultraviolet ray that is not transmitted when the ultraviolet ray is transmitted through the sample. The test was performed with a UV-VIS spectrophotometer (V-670, Japan) equipped with an integrating sphere and measured at the wavelength interval of 5nm in the wavelength range of 280 to 400nm with a Xenon arc light source. The transmittance of ultraviolet ray A (320-400nm) and the transmittance of ultraviolet ray B (280-320nm) were divided to obtain the percentage of ultraviolet ray transmittance. The UV transmittance rate was calculated using <Eq. 3>–<Eq. 4>, and UV protection rate was calculated using <Eq. 5>.

$$\text{UV-A transmittance rate (\%)} = \frac{T_{315} + T_{320} + \ldots + T_{395} + T_{400}}{18} \quad \cdots\cdots \text{Eq. 3.}$$

$$\text{UV-B transmittance rate (\%)} = \frac{T_{280} + T_{285} + \ldots + T_{310} + T_{315}}{8} \quad \cdots\cdots \text{Eq. 4.}$$

$$\text{UV protection rate (\%)} = 100 - \text{UV transmittance rate (\%)} \quad \cdots\cdots \text{Eq. 5.}$$

where, T: UV transmittance rate through the fabric sample at wavelength λ

III. Results and Discussion

1. UV-VIS Spectrum of Dye Component

<Fig. 3> shows the results of the UV-VIS absorption spectrometry after the dilution of the solution after filtration of the 5% concentration of the hydrothermal extract of *Coptidis Rhizoma* powder. According to the results, the maximum absorption wavelength was observed at around 345nm, the weak absorption peak appeared at around 420nm, and the absorption of visible light hardly occurred at above 500nm. These findings are consistent with the results of previous studies (Bae, 2003; Bai, 2000; Cho & Kang, 2000) that examined the absorption peak of berberine standard reagent and *Coptidis Rhizoma.* Therefore, our results confirm that the pigment is berberine. In addition, it

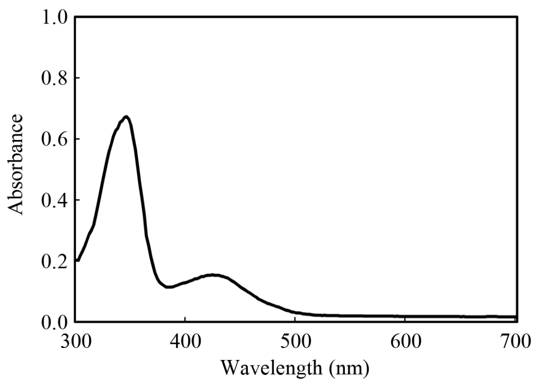

Fig. 3. UV-VIS spectrum of *Coptidis Rhizoma* dye component.

can be seen that the extract of *Coptidis Rhizoma* is yellow, because it hardly absorbs the light at green and red wavelengths.

2. FT-IR Spectrum of Dye Component

The FT-IR spectrum of *Coptidis Rhizoma* powder for the analysis of the components of *Coptidis Rhizoma* is shown in <Fig. 4>. According to the results, a broad absorption peak due to the hydroxy group present in the dye is shown at about 3421cm^{-1}. C=C stretching vibration shows two peaks at 1635cm^{-1} and 1508cm^{-1}, so it has characteristic peaks of aromatic compounds, which are distinguished from alkene compounds having one peak at around 1500cm^{-1}. The C-O stretching vibration peak of the aromatic ether compound was observed at around 1234cm^{-1}, and the N-H stretching vibration peak appeared at 3500 to 3600cm^{-1}. However, the stretching vibration peak of the tertiary amine without the N-H bond were observed near 1273cm^{-1} and 1031cm^{-1}, respectively. Therefore, the characteristic peaks of the FT-IR spectrum are consistent with the chemical structure of *Coptidis Rhizoma* with various aromatic rings containing berberine, coptisine, and palmatine.

3. Antibiosis of *Coptidis Rhizoma* Dyed Fabrics

Human skin and textile products have bacteria or microorganisms that cause illness or odor. In order to prevent these consequences, an antimicrobial effect is needed. Wool, which is a protein fiber, is expected to exhibit a lower bacteriostatic effect than cotton fabrics, because wool fabric does not inhibit the growth of microorganisms (Han & Choi, 2000).

<Fig. 5> shows the results of evaluating the antibiosis of dyed fabrics with chitosan and tannin treatment by using determination of viable count using *Staphylococcus aureus* and *Klebsiella pneumoniae*. First, 80.0% of cotton fabric untreated with chitosan and tannin, 0.0% of untreated wool fabric, and 99.9% of chitosan and tannin- treated fabrics processed with *Staphylococcus aureus* showed the reduction of bacteriostasis. On the other hand (Park & Oh, 2003), pneumococcus enters the human body through bedding, clothes, air, mouth, nose, ear, and skin. In the case of chitosan and tannin untreated dyed fabrics, the rate of bacteriostatic reduction was as low as 20.3% for cot-

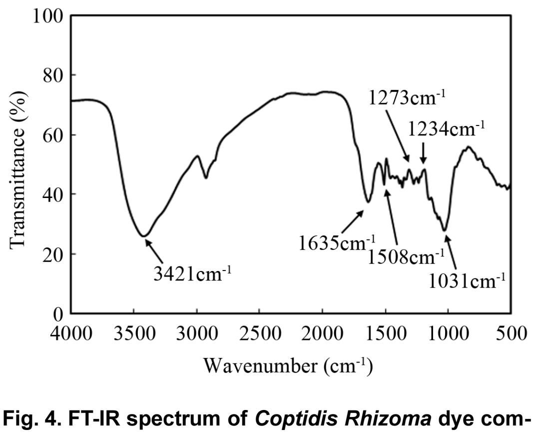

Fig. 4. FT-IR spectrum of *Coptidis Rhizoma* dye component.

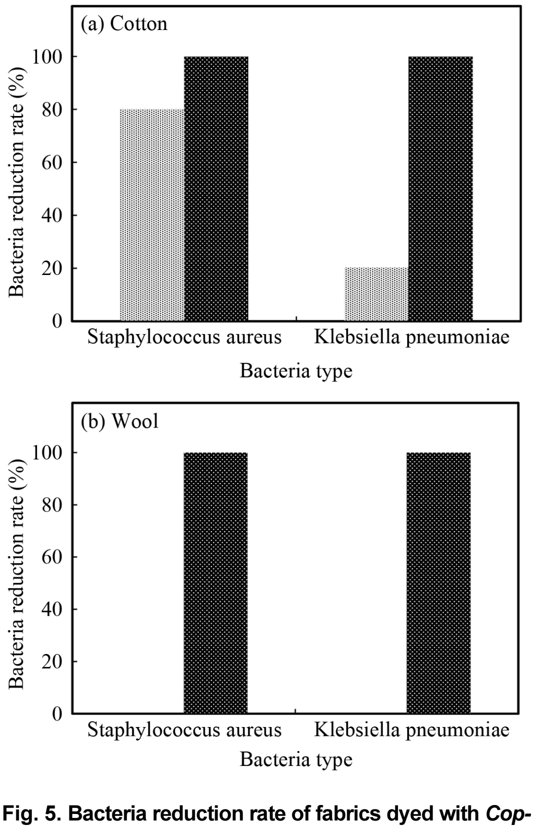

Fig. 5. Bacteria reduction rate of fabrics dyed with *Coptidis Rhizoma* extracts.

ton and 0.0% for wool fabric. In the case of dyed fabrics treated with chitosan and tannin, 99.9% bacteriostatic reduction rate was observed. Therefore, our results confirm the possibility of using dyed fabrics in the development of a functional natural material using the excellent antimicrobial activity of *Coptidis Rhizoma* dye.

4. Deodorization of *Coptidis Rhizoma* Dyed Fabrics

Today, with the growth of the desire for a comfortable space and due to the growing sensitivity to various odors in the living environment, interest in deodorants has also been increasing. Accordingly, the deodorizing effects of human-friendly natural materials are an important factor related to their functionality. The results of evaluating the deodorization of the fabric dyed twice under optimal conditions after the treatment with chitosan and tannin are shown in <Fig. 6>.

According to the results, the deodorization rate of chitosan and tannin treated dyed fabrics was improved compared with that of untreated dyed fabrics with chitosan and tannin over time, and both fabrics showed more than 90.0% deodorization rate after 120 minutes. Especially, the deodorization rate of dyed wool fabrics was significantly improved as compared with that of dyed cotton fabrics, and the degree of deodorization of wool fabrics was greater than or equal to 99.9%. We expect that the excellent deodorizing property of such dyed fabrics can be used for the development of sanitary functional materials, such as sanitary napkins, diapers, and hospital products.

5. UV Protection of *Coptidis Rhizoma* Dyed Fabrics

In recent years, the destruction of the ozone layer due to the increased air pollution leads to an increase in ultraviolet radiation dose, which causes health problems. The ultraviolet rays oxidize the melanin pigment precursor to produce melanin, which darkens the skin, UV-A (315-400nm) and accelerates aging, UV-B (280-315nm), causing t sun-burn and burn skin to deposit pigment (Kim & Choi, 2004). It is divided into UV-C (180-280nm) which has sterilizing action. The ultraviolet ray blocking performance of a functional material refers to the blocking ability of this material against UV-A and UV-B. Therefore, the ultraviolet transmittance of the fabric dyed twice after the treatment with chitosan and tannin was divided into UV-A and UV-B.

<Fig. 7> and <Table 3> show the results of evaluating the UV protection rate. In the case of untreated cotton fabrics had the UV-A protection rate of 31.3% and the UV-B protection rate of 46.7%, respectively. The cotton fabrics treated with chitosan and tannin showed 88.2% UV-A and 87.7% UV-B protection rates, respectively. Regardless of the type of mordant, the UV protection rate increased slightly. In the case

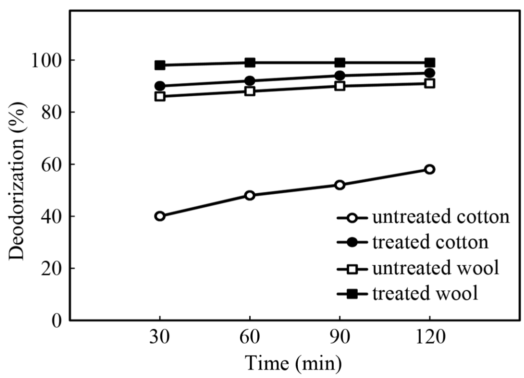

Fig. 6. Deodorization rate of fabrics dyed with *Coptidis Rhizoma* extracts.

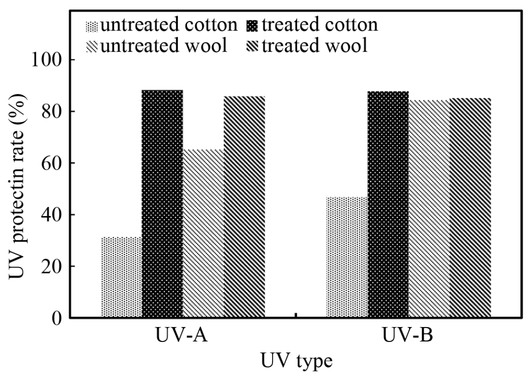

Fig. 7. UV protection rate of fabrics dyed with *Coptidis Rhizoma* extracts.

Table 3. UV protection rate of fabrics dyed with *Coptidis Rhizoma* extracts by mordants

Fabrics	Mordants	UV protection rate (%)	
		UV-A (315-400nm)	UV-B (280-315nm)
Cotton	w/o mordant	88.2	87.7
	Sn	89.2	89.2
	Fe	89.5	88.6
	Al	88.5	87.8
	Cu	89.6	88.7
Wool	w/o mordant	85.8	85.1
	Sn	89.3	88.7
	Fe	88.6	87.9
	Al	87.8	87.0
	Cu	89.7	88.8

of wool fabrics, the UV-A and UV-B protection rates of untreated fabrics were 65.2% and 85.1%, respectively. After treatment with chitosan and tannin, the UV-A and UV-B protection rates were 85.8% and 84.3%, respectively. As a result, both fabrics showed a significant increase in UV protection rate after treatment with chitosan and tannin as compared to that of untreated one, and the effect of chitosan and tannin treatment on the UV protection rate was confirmed.

IV. Conclusions

In this study, availability as a natural dye, and the functionalization of natural products by physiological activity were investigated by staining with cotton and wool fabrics using natural products. In order to increase the efficiency of natural dyeing, the components were analyzed using the UV-VIS spectrum of *Coptidis Rhizoma* extract and FT-IR spectrum of *Coptidis Rhizoma* powder. Tannin for improving fastness and chitosan were treated before dyeing. Antimicrobial activity, deodorization, and ultraviolet blocking rate were evaluated.

First, according to the results of the FT-IR spectrum analysis, *Coptidis Rhizoma* had various aromatic rings and contained berberine, coptisine, palmatine, and other components.

Second, in the case of untreated fabrics, the reduction rate of bacterium to *Staphylococcus aureus* was 80.0% in cotton and 0.0% in wool fabric. The rate of bacteriostatic reduction of cotton fabric was 20.3%, while that of wool fabric was 0.0%. In the case of dyed fabrics, which treated with chitosan and tannin, both bacteria showed a very good bacterium reduction rate of 99.9% for both strains.

Third, the deodorization rate of fabric treated with chitosan and tannin increased with times, and both fabric showed deodorization rates of over 90.0% in 120 minutes.

Fourth, UV-A and UV-B protection rates were 88.2% and 87.7% in the case of chitosan and tannin-treated cotton fabrics, respectively. However, the UV protection rate was not imfluenced by mordant types.

Finally, *Coptidis Rhizoma* has mainly been used as a medicinal product due to its high price. the results of the present study confirmed the hygienic performance of its, such as excellent antimicrobial and deodorant properties, as well as superior ultraviolet protection. Therefore, our results could be provided as the fundamental data for the development of environment-friendly materials by imparting functionality in an eco-friendly methods using natural materials.

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