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Study of Fastness, UV Protection, Deodorization and Antimicrobial Properties of Silk Fabrics Dyed with the Liquids Extracted from the Gallnuts, Areca Nuts, and Pomegranate Peels

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Abstract. The purpose of this research is to study the fastness, UV-protection, deodorization, and antimicrobial properties of silk fabrics dyed with liquids extracted from the gallnuts, areca nuts, and pomegranate peels.

1 Introduction

In textile industry, to synthetic dyestuffs and pigments are widely used because of their various range of colours, better colour fastness properties and low prices [1]. However, synthetic dyestuffs and pigments are ruled out by many producers because of their toxicity and carcinogenic effect, being not bio-degradable as well ecological [2]. Recently, the textile finishing industry tends to restrict the use of such synthetic dyestuffs and pigments in order for human health and environmental purposes. As a result, the use of natural dye has begun to increase for their better properties as being bio-degradable, non-toxic, origination no problem to human health and waste water contaminant [3-5]. Natural dyes are environmental friendly, low toxic and less allergenic. Due to these advantages, over the last decade the use of natural dyes has gained momentum in food, pharmaceutical, cosmetic and textile dyeing industry [6]. For many years, scientists have investigated the deodorizing/aroma [7], insect-repellent [8], flame retardant [9], protection against to UV rays [10] of plants dyeing and usability in the textile industry. Unlike the synthetic dyes, colorants derived from the nature are thought to be safe because of their non-toxic, non-carcinogenic and biodegradable nature [11]. Natural dyes mainly consist of phenolic compounds which play an important role in plant growth and reproducibility. Many of them have antioxidant activity and are also considered as antibacterial and anti-inflammatory compounds. They have been widely used as herbal medicines as well as natural dyeing agents. Phenolic compounds based on their different chemical structure, are divided to groups corresponding to flavonoids, quinones, curcuminoids and tannins [12]. Tannin is an astrigent vegetable product found in awide variety of plant such as bark, wood, fruit pods, leaves, roots and plant galls. Tannins are defined as naturally occurring water soluble polyphenolic compounds of high molecular weight (about 500-3000) containing phenolic hydroxyl groups to enable them to form effective crosslinks between proteins and other macromolecules [13].

The purpose of this research is to study the fastness, UV-protection, deodorization, and antimicrobial properties of silk fabrics dyed with the liquids extracted from the gallnuts, areca nuts, and pomegranate peels contained tannins. The light, dry cleaning, rub, and perspiration fastness of the dyed silk fabrics was evaluated. The UV protection factor of the dyed silks with SPF calculated in wavelength range of 290-400 nm range. The deodorization activity was made from concentration of residual ammonia gas in a container. The antimicrobial activity of the dyed silks was measured against *Staphylococcus aureus* and *Klebsiella pneumoniae*.

2 Theoretical background

2.1 Gallnut

Gallnuts are outgrowths of plant tissues produced when irritants are released by the larvae of gall insects such as those of the Cynipidae family, the gall wasps. This extract contains the highest naturally occurring levels of tannin (gallotannin, 50-75%), as well as smaller molecules such as gallic acid and ellagic acid. Additionally, this extract is known to possess pharmaceutical properties, including anti-inflammatory, antibacterial, and detoxifying properties [14]. Figure 1 showed the chemical structure of tannin(Gallnut tannin) contained in gallnut and image of gallnut dried.

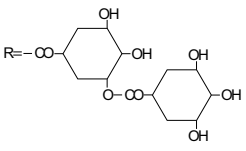

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Figure 1. Chemical structure of tannin(Gallnut tannin) contained in gallnuts and image of gallnuts dried.

2.2 Areca nut

Areca nut (*Areca catechu* L.), belonging to the family Palmae(or Arecaceae), native to Malaysia, widely cultivated in Indonesia, Sri Lanka, Hainan province, Guangdong province, Yunnan province and other places in Southeast Asia, is one of th most widely used South-China medicine resources [15]. Areca nut is popular chewable items used in traditional herbal medicine [16-18]. Areca nut exhibits multiple therapeutic properties like, aphrodisiac [19], antihypertensive [20, 21], wound healing [22], hypoglycemic [23, 24] and antidepressant [25]. It is one of the most commonly used drugs in the world, containing alkaloids, tannins, polyphenols, sugars, and lipids that have anthelmintic, antifungal, antibacterial, anti-inflammatory, and antioxidant activities [26]. Tannins are another characteristic component of Areca nut, and the main types are condensed tannins (also called proanthocyanidins). The main classes of tannins in A. catechu are the catechuins (Figure 2) [27-30].

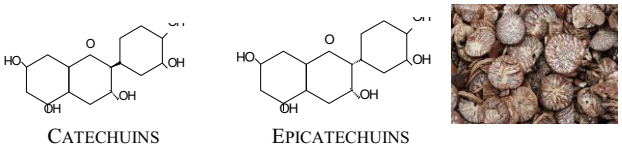

Figure 2. Chemical sructure of tannin(Catechuins and Epicatechuins) contained in areca nuts and image of areca nuts dried.

2.3 Pomegranate

Pomegranate (*Punica granatum* L.) belongs to the Punicacea family [31-33]. The cultivation of pomegranate is native to the Middle East and was later known in the Mediterranean. Pomegranate peels are rich in tannins [34-36]. They have been used traditionally for their medicinal properties as anticancer, anti-inflammatory, antioxidant and antithelminthic [37, 38] and for other purposes such as tanning, dyeing [39, 40] and heavy metal removal [41]. Pomegranate peels are characterized by an interior network of membranes comprising almost 26-30% of total fruit weight and are characterized by substantial amounts of phenolic compounds, including flavonoids (anthocyanins, catechins and other complex flavonoids) and hydrolysable tannins (punicalin, pedunculagin, punicalagin, gallic and ellagic acid) [42-44]. Gallic acid, ellagic acid and punicalagin, in addition to their free radical-scavenging properties, also possess antibacterial activites against intestinal flora, particularly enteric pathogens, i.e., Escherichia coli, Salmonella spp. Shigella spp., as well as Vibrio cholera [45-48]. Figure 3 showed the chemical structure of pomegranate tannin(Ellagic tannin) contained in pomegranate peels and image of pomegranate peels dried.

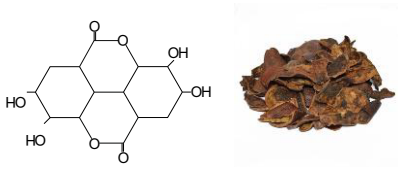

Figure 3. Chemical structure of pomegranate tannin(Ellagic tannin) contained in pomegranate peels and image of pomegranate peels dried.

3 Experimental materials and methods

3.1 Experimental materials

Silk: Silk used in this study was purchased from Testfabrics Inc. (West Pittston, PA)., and the characteristics are as shown on the Table 1. The silk was purchased from Testfabrics Inc. (West Pittston, PA).

Table 1. The Characteristics of silk

Fiber	Weave	Yarn Number		Fabric Counts (Threads/in.)		Weight (g/㎡)
		Warp	Weft	Warp	Weft	
Silk	Plain	21D	21D/2	56	39	26

Gallnut, Areca Nut, Pomegranate: Gallnuts and areca nuts were acquired from online from Cheongmyeong herbs(http://www.good1075.com), and pomegranates were purchased from a local market in Korea.

3.2 Experimental methods

UV-Vis/NIR Spectra: 1g dried gallnuts, areca nuts and pomegranate peels was added to 100 ㎖ ethanol respectively, and they were extracted at room temperature for 24 hours, and filtered. The filtered extracts respectively were used as samples for UV-Vis analysis. The measurement of the UV-absorption characteristics was conducted in the range of 190-800 nm by using an ultraviolet-Visible/Near Infrared spectrophotometer (Varian Cary 5000).

FT-IR Spectra: The dried and grinded powers of gallnuts, areca nuts and pomegranate peels were analyzed with Fourier Transform Infrared Spectrometer (Bruker TENSOR27). Each samples were scanned registering the spectrum with 32 scans with a resolution 0f 4 cm^{-1} in the wave number range between 4000 and 600 cm^{-1}.

The extraction treatment of gallnut, areca nut and pomegranate peel: Gallnuts, areca nuts and pomegranate peels were extracted in liquor ratio of 1:20 at the boiling temperature for 20minutes. Each solutions were filtered with filter paper. The process was repeated 2 times. The liquid extraction combined first and second extract liquid was used as solution for dyeing.

Mordanting: Silk fabrics were mordanted by post-mordanting method using ferric mordant (0.2%), and liquor ratio for mordanting was kept at 1:30. Before the application of mordants, silk fabrics were soaked in distilled water. Water soaked silk fabrics were immersed

in mordants solutions, and mordanted at 40 ℃ for 30minutes with constant stirring. Mordanted silk fabrics were rinsed with distilled water to remove superfluous mordants.

Dyeing: Before the application of dyeing, silk fabrics were soaked in distilled water. Firstly silk fabrics were dyed in liquor ratio of 1:30 at the boiling temperature for 20 minutes with constant stirring. Secondly, the samples were left in the fluid for one night. Thirdly, the samples were washed with 1500 ㎖ of cold distilled water (Three repetitions) then squeezed and dried at room temperature.

Color Fastness Tests: The light fastness of silk fabrics dyed was conducted on Fade-O-Meter(25-FR, Atlas Electric Devices Co. U.S.A) having water cooled Xenon Arc, as per test method KS K ISO B02:2005. The dry cleaning fastness of silk fabrics dyed was measured in Launder-O-Meter(Model LP2, Co. Atlas) as per the KS K ISO 105D01:2010, specification. The dry and wet rub fastness of silk fabric dyed s was tested using Rubbing-Crock Meter(CM-5, Atlas Electric Devices Co. U.S.A) as per the KS K ISO 0650:2011. The perspiration fastness of silk fabrics dyed was measured AATCC Perspiration Tester (PR-1, Atlas Electric Devices Co. U.S.A) as per test method KS K ISO 105E04:2010.

UV protection factor: UV-protection factor was tested using UV-Vis spectrophotometer(Varian Cary 5000) as per the KS K 0850:2014, o/d. Transmission measurements were made in 290-400 nm range with a 1 nm step. SPF was calculated according to:

$$SPF = \frac{\sum_{290}^{400} E_\lambda S_\lambda \Delta_\lambda}{\sum_{290}^{400} E_\lambda S_\lambda T_\lambda \Delta_\lambda}$$

where S_λ is the solar spectral irradiance at noon for a typical summer's day in central Italy, E_λ is the CIE erythermal spectral effectiveness, T_λ is the spectral transmittance of each fabric sample and T_λ is the wavelength step.

Deodorization activity: Deodorizationrate was calculated according to:

$$\text{Deodorizationrate}(\%) = \frac{Cb - Cs}{Cb} \times 100$$

where C_b is residual gas concentration of control after 2hours, Cs is residual gas concentration of specimen after 2hours.

Antimicrobial Activity: The antimicrobial ability of the dyed samples to impede microbial growth and retention was tested using S*taphylococcus aureus* and *Klebsiella pneumoniae* cultures, according to an established protocol to test the antibacterial of textiles (KS K 0693). Antimicrobial activity was calculated according to:

$$\text{Reduction bacteria}(\%) = \frac{B - A}{A} \times 100$$

where B is the number of bacteria recovered from the inoculated control specimen incubated for 18hours, A is the number of bacteria recovered from the inoculated treated test specimen incubated for 18hours.

4 Results

4.1 Spectroscopic analysis by UV‐Vis/NIR spectra

Figure 4 shows the UV-Vis/NIR spectra of the ethanolic extraction solution of gallnuts, areca nuts and pomegranate peels in the range of 190-800 nm. As shown by Figure 4 and Table 2, two absorption bands are easily seen in the ranges from 190 to 250 nm, and from 250 to 300 nm, and another broad absorption band appears around 300-400 nm. Gallnuts presented two characteristic absorption maximum, λmax_1 around 217 nm and λmax_2 at 279 nm. Spectra of areca nuts classified as condensed tannin, presented two characteristic absorption maximum, λmax1 around 224 nm and λmax2 at 280 nm. Pomegranate peels absorbed with two λmax at 250 and 368 nm.

4.2 Spectroscopic analysis by FT-IR spectra

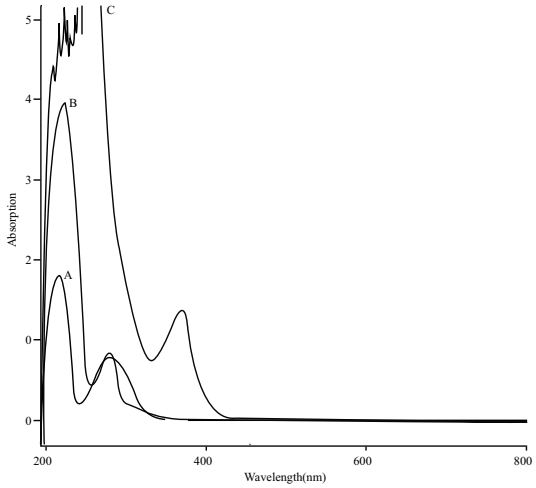

Figure 4. UV-VIS Spectra of ethanol extraction solution of gallnuts(A), areca nuts(B) and pomegranate peels(C)

Table 2. Wavelength and absorption of gallnuts, areca nuts and pomegranate peels

Samples	Wavelength(nm)	Absorption
Gallnut	217	1.813
	279	0.771
Areca nut	224	4.001
	280	0.856
Pomegranate peel	258	6.654
	368	1.366

4.3 Fastness properties

Fastness properties of silk fabrics dyed were given in Table 3. The samples showed mostly good light and dry

cleaning fastness with 4 grade. Wet rub fastness was found to be relatively better than dry rub fastness. Perspiration fastness was all excellent grades 4~5 except for the 3~4 grades from discoloration by acidity and alkalinity.

Table 3. The fastness properties of silk fabrics dyed

Dyeing Fastness			Grade
Light Fastness			4
Dry Cleaning Fastness	Discoloration		4
	Solvent Contamination		4
Rub Fastness	Dry		2
	Wet		2~3
Perspiration Fastness	Acidity	Discoloration	3~4
		Contamination(Silk)	4
		Contamination(Cotton)	4~5
	Alkalinity	Discoloration	3~4
		Contamination(Silk)	4
		Contamination(Cotton)	4~5

4.4 UV Protection Rate

UV protection rate of dyed silk fabrics was shown in Table 4. UV-A protection rate of the samples in wavelength range of 290-400 nm showed 98.3%, and UV-B protection rate of the samples in wavelength range of 290 ~ 315 nm showed 98.4%. As described above, the samples appeared very good UV protection rate.

Table 4. UV protection rate of silk fabrics dyed

	UV Protection Rate (%)	
	UV-A	UV-B
Untreated Silk	-	-
Silk Fabric Dyed	98.3	98.4

4.5 Deodorization activity of silk fabric dyed

Table 5 showed deodorization activity of dyed silk fabrics. As outlined in Table 5, the samples appeared excellent deodorization activity over 99% even after 120min. test.

Table 5. Deodorization activity of silk fabric dyed

		Deodorization Activity (%)
Untreated Silk		-
Silk Fabric Dyed	30min.	over 99%
	60min.	over 99%
	90min.	over 99%
	120min.	over 99%

4.6 Antimicrobial ativity of silk fabric dyed

The antimicrobial activity of dyed silk fabrics against *Staphylococcus aureus* and *Klebsiella pneumoniae* was assessed. Table 6 showed the antiimicrobial activity of dyed silk fabrics. The samples appeared high antiimicrobial activity activity of 99.9% against *Staphylococcus aureus* and *Klebsiella pneumoniae*.

Table 6. Antimicrobial activity of silk fabrics dyed

	Reduction of Bacteria (%)	
	Staphylococcus aureus	*Klebsiella pneumoniae*
Untreated Silk	-	-
Silk Fabric Dyed	99.9	99.9

5 Conclusion

Among dyeing fastness of dyed silk fabrics, light and dry cleaning fastness showed 4 grade. Rub fastness was 2~3 grade. Perspiration fastness was 3~5 grade. The dyed silk fabrics in wavelength range of 290-400 ㎚ appeared UV protection rate of 98.3%, and UV-B protection rate in wavelength range of 290 ～ 315 ㎚ showed 98.4%. Deodorization activity of the dyed silk fabrics appeared over 99%. The dyed silk fabrics showed high antibacterial activity of 99.9% against *Staphylococcus aureus* and *Klebsiella pneumoniae*.

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