ACTIVITATEA ANTIBACTERICIDĂ ȘI UV PROTECTOARE A ȚESĂTURII DE MĂTASE VOPSITĂ CU PERICARPUL DE *GARCINIA MANGOSTANA*
ANTIBACTERIAL ACTIVITY AND UV BLOCKING OF SILK FABRICS DYED WITH PERICARP OF *GARCINIA MANGOSTANA*

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The extract from mangosteen pericarp (MP) was applied to a silk fabric. Its dye ability of silk fabric has been studied together with its colorfastness, antibacterial activity and UV protection properties. The mordant are aluminum potassium sulfate, ferrous chloride and sodium hydroxide were used to dye fabric. The color fastness to washing, water, perspiration, light and crocking of the dyed samples was determined according to AATCC test methods. In this study the UV protection properties were investigated on silk fabrics. The chemical functional groups of the dyes that were characterized by using fourier transform infrared spectroscopy (FTIR). Our results revealed that MP dyed silk fabric exhibited fair to good and good to very good fastness to washing and crocking, fair to very good light fastness, except for fabric mordanted with sodium hydroxide, whose rating were very poor to poor light fastness. The water and perspiration fastness ratings were good to very good, except for fabric mordanted with aluminum potassium sulfate. Silk fabrics mordanted with ferrous chloride and dyed with MP usually showed good UV protection levels even if undyed. Antibacterial activity of this dyed silk was confirmed by exposing the fabric to Staphylococcus aureus. The fabric dyed with MP extract reduced the number of viable organisms by 98% mordanted with ferrous chloride. These extracts gave polyphenols, gallic acid and carboxylic acid contents. Therefore, it was suggested that MP has the potential in producing functional dyes that could be imparted into the silk dyeing natural colorant system and incorporated in several kinds of cloths.

Keywords: *antibacterial, natural dyes, color fastness, UV protection*

1. Introduction

Nowadays, the interest towards natural dye is growing as our lives are affected by pollution. Costumer concerns more about the quality of goods to avoid toxic or allergic reactions. The production process together with the use of material and ethical problems may affect consumer's decision. The growth of natural dye for textile development is steady [1]. Natural dyes can be obtained from plants, animals and minerals [2]. These natural dyes have been successfully applied to natural fiber fabrics such as cotton [3], wool [4-6], silk [7-8] and flax [9].

The mangosteen-fruit is dark purple or reddish pericarp, with white, soft and juicy edible pulp with a slightly acid and sweet flavor and a pleasant aroma [10]. Mangosteen is known as ''the queen of fruits" because it is one of the best tasty tropical fruits. The pericarp of mangosteen-fruit has been used as a medicinal agent by Southeast Asians for centuries in the treatment of skin infections and wounds [11-12], amoebic dysentery [13-14], etc. In Ayurvedic medicine the pericarp of mangosteen-fruit has widely used against inflammation and diarrhea [15], and cholera and dysentery [16]. Mangosteen has been shown to contain a variety of secondary metabolites such as prenylated and oxygenated xanthones [17-18].

Xanthones have been isolated from pericarp, whole fruit, bark, and leaves of mangosteen. Several studies have shown that xanthones obtained from mangosteen-fruit have remarkable biological activities [19]. α-, β- and γ-mangostins, garcinone E, 8- deoxygartanin and gartanin are the most studied xanthones shown in Figs. 1. In addition, synthetic xanthones have been used in several studies. Antioxidant, antitumoral, anti-inflammatory, antiallergy, antibacterial, antifungal and antiviral are some of the reported activities of xanthones isolated from mangosteen pericarp which are benefits in the medicinal properties.

Overexposure to solar UV radiation has been identified as causing an increased incidence in skin problems such as sunburn, premature aging, allergies and skin cancers [20]. In order to avoid or limit these health risks, it is important to reduce the UV ray exposure with clothing, accessories and shade structures made of protective materials. Textiles have been shown to provide UV blocking properties but these characteristics depend on fiber type, fabric construction and nature of finishing chemicals.

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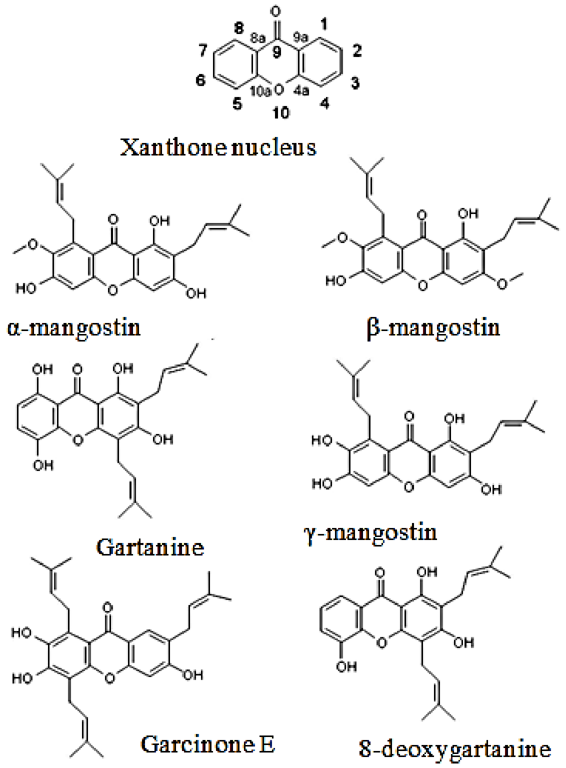


Fig.1- Chemical structure of the most studied xanthones.

Dyed fabrics are more protective than undyed ones and the protection level rises with the increase in dye concentration [21]. In general, light colours reflect solar radiation more efficiently than dark ones [22], but part of the radiation penetrates more easily through the fabric thanks to multiple scattering. Moreover, most of the studies on this topic concern synthetic dyes. The high compatibility with the environment of naturally dyed textiles and their lower toxicity and allergic reaction have been arousing growing interest in the last 15 years and, for this reason, many studies have focused on the multifunctional properties of dyeing plants extracts, as shown by Islam et al.[23]. Nonetheless, as regards the UVprotection properties of natural dyes, few researches have been performed on natural fabrics [24-26] and most of these concern animal fibers, as reported by Grifoni et al. [27]. Very few studies exist on the UV protection properties of natural dyes in combination with fabrics made of vegetable fiber [23-24,27] maybe because very few natural dyes provide vegetable fibers with strong colours without the aid of mordants. An ecofriendly natural dyeing can however be achieved by replacing metal mordant with natural mordant, like tannic acid or other vegetable tannins [28], even if metal mordants such as potassium alum and aluminium sulphate can also be used in ecofriendly natural dyeing as their environmental toxicity is almost nil [29]. Tannins are water soluble phenolic compounds that have been used on textiles for several hundred years both as a pre-treatment and post-treatment factor to increase wash fastness [30] and light fastness [31], e.g. in cotton fabrics. The evaluation of the level UV protection properties of natural colours needs to be supported by knowledge of the dyes chemical structure, absorption characteristics in the UV region, interaction and complexation with the premordanted substrate, as well as the ability to block or absorb the hazardous UV rays [27].

The objectives of this study were to explore the potential of mangosteen pericarp has investigated the dyeing, color fastness, ultraviolet protection properties and antibacterial activity of silk fabric using an aqueous extract of mangosteen pericarp as a natural dye. Different factors affecting dyeing ability were examined.

2. Experimental Details

2.1. Materials, extraction and dyeing

Mangosteen pericarp was collected in July at Songkla province, Thailand. A commercially scoured and bleached silk fabric (81.4 gm^{-2}, plain weave) was used, the method of pre-mordant dyeing was considered to be the most suitable for this study because it has the ability to improve the colorfastness of the dyed samples [32]. Amount of silk, dyeing, mordant and water were used with the weight ratio of 0.50:1.00:0.04:40.00. Three different mordants were used, aluminum potassium sulfate ($AlK(SO_4)_2{\cdot}12H_2O$), Iron (III) chloride hexahydrate ($FeCl_3{\cdot}6H_2O$) and sodium hydroxide (NaOH). The UV-vis spectra value of dye in the solution form was measured by UV-Vis Spectrometer with integrating sphere attachment (Genesys 10S UV-vis spectrophotometer, Thermo Scientific). The FTIR transmittance spectra of the samples were also analyzed in order to confirm chemical functional groups of the dyed.

2.2. Determination of color fastness

The color fastness to washing, light, crocking, perspiration, and water of the dyed samples were determined according to AATCC Test Method 61-2010, AATCC Test Method 16-2004 Option 3, AATCC Test Method 18-2007, AATCC Test Method 15-2009, and AATCC Test Method 107-2009, respectively.

2.3. UV measurements

The UV protection factor (UPF) was determined on three fabric samples ($3x1cm^2$). Each sample was taken from the center of the fabric, fixed on a common slide frame and placed in a Genesys 10S UV-vis spectrophotometer with thin film holder equipped with an integrating sphere to measure both direct and diffuse transmitted light. Each sample was positioned at right angles to the light beams. Transmission measurements were made

Table 1

UPF categories with relative transmittance and protection level

UPF range	Protection category	$UVBE_{eryt}$ transmittance (%)
<15	Insufficient protection	>6.7
15-24	Good protection	6.7-4.2
25-39	Very good protection	4.1-2.6
40-50, 50+	Excellent protection	≤2.5

in the 280-400 nm range with a 1 nm step (AATCC Test Method 183-2004). UPF was calculated according to Equation (1):

$$UPF = \frac{\sum_{280}^{400} E_\lambda S_\lambda \Delta_\lambda}{\sum_{280}^{400} E_\lambda S_\lambda T_\lambda \Delta_\lambda} \quad (1)$$

where: E_λ is the relative erythemal spectral effectiveness, S_λ is the solar spectral irradiance, T_λ is the average spectral transmittance of the specimen (measured) and Δ_λ is the measured wavelength interval (nm). UPF values higher than 40 were reported as 40 corresponding to the highest UV-protection category (excellent protection, Table 1).

2.4. Antibacterial activity against *Staphylococcus aureus* (*S. aureus*)

Antibacterial activity was characterized in terms of percentage reduction in visible growth of bacteria. For a qualitative, relatively and easily executed method to determine residual antibacterial activity of textile materials, refer to AATCC Test Method 100-2004.

Typical gram positive and gram negative micro-organisms *Staphylococcus aureus* (ATCC 6538). The initial bacterial concentration was kept about 10^5 CFU/ml used for all experiments. The percentage reduction in the number of colony forming units between the untreated and treated fabrics after incubation for 24 h at 37 ± 1 °C was calculated using the following equation:

$$100(B - A)/B = R \quad (2)$$

Where R is the percentage reduction in bacteria, and B and A are the numbers of CFU/ml of bacteria measured for the untreated and the treated silk fabrics respectively.

3. Results and discussion

3.1. The coloration and fastness of dyed silk fabrics

Figure 2 shows the silk fabrics color with MP dyed with various mordants. The obtained colors (Fig. 2) show that silk fabrics dyed without mordant had grey color, while those mordanted with ferrous chloride and aluminum potassium sulfate produced of black and dark purple color shades, respectively. With sodium hydroxide, the color reddish brown shade. This may be associated with the change of ferrous chloride into a ferric form by reacted with oxygen in the air. Ferrous and ferric forms coexist on the fiber and their spectra overlap, which results in a shift of max and thus consequently a color change to a darker shade [33]. Additionally, the tannins in the MP extract combine with sodium hydroxide to form complexes, which also result in brown shade of silk fabric.

The intensity of the extracted liquor samples was measured in terms the level of absorption on a UV-visible spectrophotometer. From Figure 3 it was found that the wavelength of maximum absorption for the extracted liquor was at 278 nm. This was the wavelength selected for UV-visible spectrophotometric measurements.

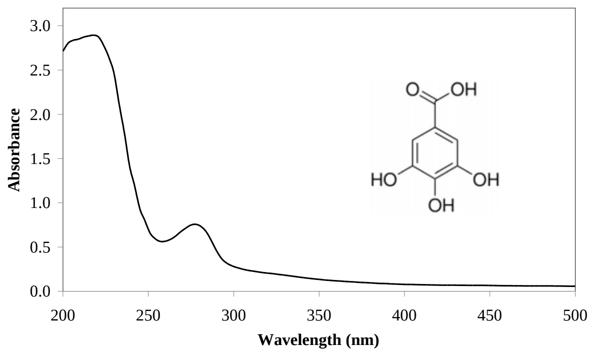


Fig. 3 - UV-Visible absorption spectrum of extracted MP solution (insert picture shows chemical formula of gallic acid).

Figure 3 shows that the maximum absorption values of the extracted MP dyed were found in the 250-350 nm wavelength range, which is similar to the absorption range for gallic acid (Fig. 3). Gallic acid (Fig. 3), a polyphenol monomer, esterifies and binds with the hydroxyl group of a polyol carbohydrate such as glucose. These structure which are known to have antibiotic, antioxidant and anti-inflammatory activity [32]. When the glucose core is esterified with five

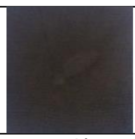
Color obtained				
Type of mordants	Without mordanting	$FeCl_3$	$AlK(SO_4)_2$	NaOH

Fig. 2 - Silk fabrics color with MP dyed with various mordants.

Table 2

Colorfastness to washing at 40°C (AATCC Test Method 61-2010)

Fastness	Type of mordants			
	Without mordant	NaOH	$FeCl_3$	$AlK(SO_4)_2$
Color change	3.0	4.0	3.0	3.5
Color staining				
ACETATE	4.5	4.5	4.5	4.5
COTTON	3.5	3.5	3.5	3.5
NYLON	4.5	4.5	4.5	4.5
SILK	4.5	4.5	4.5	4.5
VIACOSE RAYON	4.0	4.0	4.0	4.0
WOOL	4.5	4.5	4.5	4.0

Table 3

Colorfastness to water (AATCC Test Method 107-2009)

Fastness	Type of mordants			
	Without mordant	NaOH	$FeCl_3$	$AlK(SO_4)_2$
Color change	4.5	4.5	4.5	4.5
Color staining				
ACETATE	4.5	4.5	4.5	4.0
COTTON	4.0	4.5	4.0	3.5
NYLON	4.0	4.5	4.0	2.5
SILK	4.0	4.5	4.0	3.5
VIACOSE RAYON	4.5	4.5	4.5	4.0
WOOL	4.5	4.5	4.5	3.0

Table 4

Colorfastness to perspiration (AATCC Test Method 15-2009)

Fastness	Type of mordants			
	Without mordant	NaOH	$FeCl_3$	$AlK(SO_4)_2$
Color change	4.0	4.0	4.0	4.0
Color staining				
ACETATE	4.5	4.5	4.5	3.5
COTTON	4.5	4.5	4.5	3.5
NYLON	4.5	4.5	4.5	3.5
SILK	4.5	4.5	4.5	3.0
VIACOSE RAYON	4.5	4.5	4.5	3.5
WOOL	4.5	4.5	4.5	3.0

Table 5

Colorfastness to light (AATCC Test Method 16-2004 Option 3)

Type of mordants	Color change	
	20 hr.	40 hr.
Without mordant	3.5	3.0
NaOH	2.5	1.5
$FeCl_3$	3.5	3.0
$AlK(SO_4)_2$	4.5	4.0

Table 6

Colorfastness to crocking (AATCC Test Method 18-2007)

Type of mordants	Color staining	
	Dry	Wet
Without mordant	4.0	3.0
NaOH	4.5	4.0
$FeCl_3$	4.0	3.0
$AlK(SO_4)_2$	3.5	3.0

or fewer galloyl groups, the resulting compounds are defined as gallotannin precursors.

Table 2 shows that the wash fastness ratings of both with-out mordanted and mordanted dyed samples were fair to good (3–4), whose rating was good to very good (4–5). This drastic color change may be attributed to (a) the ionization of the hydroxyl groups in the dye molecules under the alkaline condition of the standard detergent solution [34-35] or (b) the decomposition of the dye itself, resulting in a colorless or a differentially colored compound [35]. The ratings obtained for color fastness to water in terms of the degree of color change and color staining were good to very good (4 to 5), except for the fabric mordanted with aluminum potassium sulfate, whose rating were poor to very good (2.5–4) as shown in Table 3. The color fastness to perspiration fabrics dyed with and without mordants ranged from 4 to 4–5 (good to very good), except for the fabric mordanted with aluminum potassium sulfate, whose rating were fair to good (3–4) as seen in Table 4. The light fastness was fair to very good (3–4.5), except for the fabric mordanted with sodium hydroxide, whose rating were very poor to poor (1-2.5) depend on the exposure to light time irradiation as shown in Table 5. Color fastness to crocking is found to be in the range of 3–4 to 4-5 (fair to good and good to very good), when subjected to wet and dry rubbing respectively, as seen in Table 6.

3.2. FTIR analysis

The band frequencies of the spectra obtained with their assignment are given in Figure 4. The FTIR spectra of the MP dye consisted of six main groups of absorption bands in the wavelength range of 400–4000 cm^{-1} (Fig. 4). The intense band detected in the 3300-3500 cm^{-1} region and 1300 cm^{-1} originated from compounds with –OH groups such as water and ethanol, which are major compounds in these samples.

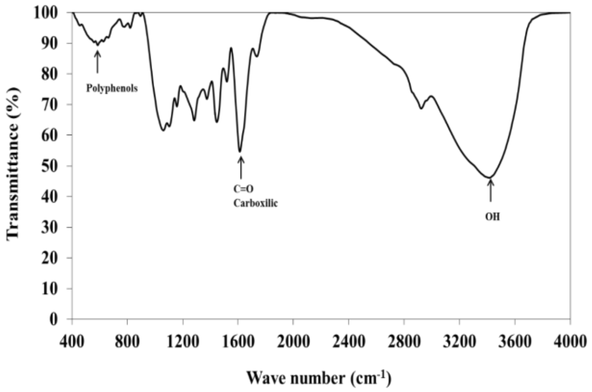


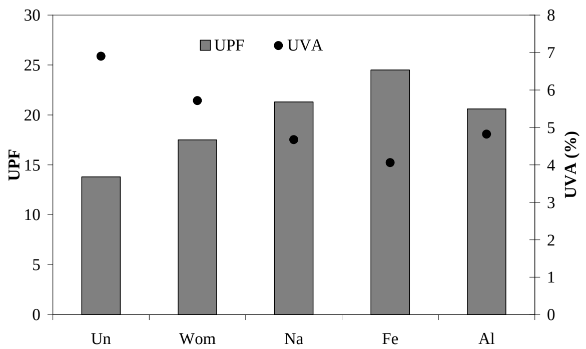
Fig. 4 - FTIR spectra of mangosteen pericarp dye.

The bands in the region of 2900-3000 cm^{-1} can be assigned to the C-H (chlorophyll) [36-38]. The bands in the region of 400-800 cm^{-1} can be assigned to the polyphenols [39-40]. These wavelengths are part of the fingerprint region and include infrared typical absorption of phenolic molecules such as the stretching band of carboxylic (C=O) groups (1600-1630 cm^{-1}). The peaks at 2973, 2931 cm^{-1} are attributed mainly due to $-CH_3$, $-CH_2-$ stretching vibration [36,39, 41]. The peaks in the range of 1160-1340 cm^{-1} and1250-1310 cm^{-1} are attributed mainly aromatic compound or phenols groups [42].

3.3. Transmittance and UPF measurements

The results obtained by previous author [27, 43] in similar experiments in which the mordants used during the dyeing process did not show any UV-absorbing capacity; in the present experiment the first mordanting markedly affected the UV-protection properties of all silk fabrics. The UPF value remains lower than 15 (the minimum protection level as defined in Table 1). The protection category of all other silk fabrics moved from no protection to good protection for $FeCl_3$, NaOH and $AlK(SO_4)_2$ were used as mordant. The different effect of mordanting in this experiment in comparison with those previously mentioned was ascribable to the use of natural mordant containing tannins. Indeed, the main constituents of the MP are gallic acid (Fig.3), tannic acid, ellagic acid and xanthones contents [19, 44]. Tannins absorb UV radiation with efficiency similar to carotenes and anthocyanins [19, 44], and provide the same protection from UV damage that accessory pigments do. Similar behavior was also shown in Figure 5, however the UPF value of MP with $FeCl_3$, NaOH and $AlK(SO_4)_2$ dyed silk fabric slightly exceeded the threshold of 15, corresponding to a good UV protection, even if the UPF variability was high.

Generally, in all the cases in which UPF reached at least the good protection level UVA transmittance was also in range 4.2-6.7% (Fig. 5),



Note: Un= Undyed, Wom= With-out mordanting, Na=NaOH, Fe = $FeCl_3$ and Al = $AlK(SO_4)_2$.

Fig.5 - UPF (bars) and UVA transmittance (black dots) for dyed silk fabrics mordanted. Bars and dots are means of three measurements.

which the American Association of Textile Chemists and Colorists (AATCC). The threshold above which photosensitive skin disorders, like chronic actinic dermatitis and solar urticarial can be aggravated [45]. Nevertheless high UPF do not necessarily imply low transmission in UVA wavelengths, as already found by previous researcher [22, 27, 46]. In this study, for example, $FeCl_3$, NaOH and $AlK(SO_4)_2$ with MP dyed that had UPF corresponding to good protection, showed UVA transmittance values slightly in range 4.2-6.7%.

3.4. Antibacterial activity of silk fabrics samples dyed with mangosteen pericarp

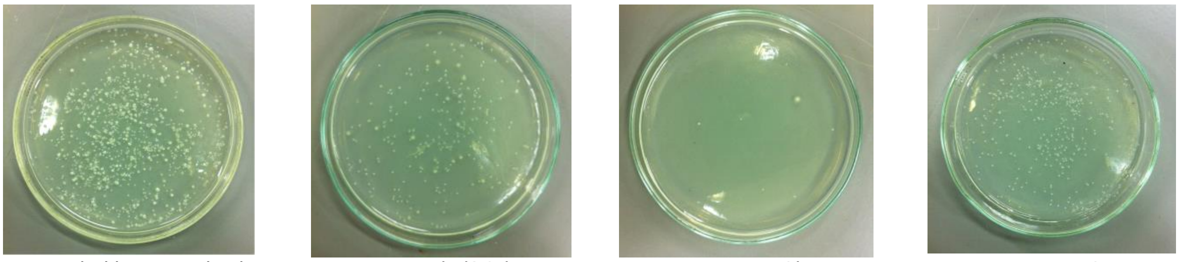
The aim of this study was to evaluate the antimicrobial activity of MP with various mordants. The silk fabrics dyed with MP were tested for against *S. aureus* and the results were compared with control. Each test was performed in triplicates. Textiles have long been recognized as a media to support the growth of bacteria as they provide excellent environment for bacterial growth and prolife ration. The antibacterial textiles can protect not only the textile itself from damage but also the textile user against pathogenic or odor causing the bacteria. So it is highly desirable that the growth of bacteria on textiles is minimized in their use and storage [2, 32]. It is fortunate to discover the antibacterial activity of mangosteen pericarp.

Antibacterial activity of the silk fabric samples dyed with MP under optimum conditions was measured. For *S. aureus* bacteria, the dyed silk sample caused significant reductions in the number of growing bacteria after 24 h of incubation of 37°C. The bacterial reduction (%) values of unmordanted and mordanted dyed fabrics against *S. aureus* were assessed, without mordanting (53%), aluminum potassium sulfate (65%), ferrous chloride (98%) and sodium hydroxide (74%) (Table7). Table 7 show the antibacterial results of fabrics dyed with different mordant. All samples mordant with MP dyes exerted high antibacterial activity against these bacteria. As it can be seen in Table 7 and Figure 6, the antibacterial activity of dyed samples has dramatically increased after mordanting. The samples mordanted with ferrous chloride showed excellent antibactericidal activity against *S. aureus*. The antibacterial results also revealed that the unmordanted or mordanted dyed fabrics had almost different antibacterial activity against the gram positive bacteria *S. aureus*. The bactericidal effect of dyed generally has been attributed to the decomposition of bacterial outer membranes by reactive oxygen species (ROS), primarily hydroxyl radicals (-OH) as shown in Figure 4, which leads to phospholipid peroxidation and ultimately cell death. Many metallic salts are shown to inhibit the growth of microorganisms or destroy them at very low concentrations. Metal may have toxic effects either in its free state or in metallic compounds. Mechanism of metal antibacterial action is suggested to be either through protein binding or formation of reactive oxygen species (ROS). In the first mechanism, metal ions attach covalently to the -SH groups of cellular enzymes and suppress their activity and change the bacterial metabolism. This eventually leads to the cell death. The second mechanism is based on pro-oxidant activity of aluminum. Aluminum-induced mitochondrial dysfunction, changes in reactive oxygen species and its pro-oxidant activity in plants, animals and human has been investigated both experimentally and computationally. Highly active oxygen radicals produced in this reaction attack the chemical structure of bacteria and destroy them [47,48]. We propose that as metal ions have great antibacterial properties. The phenolic components of dyes from MP are shown in Figure 4. These compounds are classified as tannins which have received great attention due to their therapeutic activities. They

Table 7

Antibacterial activities against *S.aureus* of fabrics dyed with MP extracts after dyeing.

Fabric sample	Bacterial reduction (%)
MP (Without mordant)	53
MP+$AlK(SO_4)_2$	65
MP+$FeCl_3$	98
MP+NaOH	74



MP (Without mordant) MP+$AlK(SO_4)_2$ MP+$FeCl_3$ MP+NaOH

Fig. 6 - Photo images of *S. aureus* counting test after 24 hours of incubation of 37°C on the silk fabrics dyed with MP (without mordant) and treated with various mordants samples.

are also reported to have antibacterial and anti-infective activity owing to their potential complex with proteins through hydrogen bonding, hydrophobic forces or covalent linkages [47-48]. Several studies have been found the inhibitory effect of several xanthones, isolated from mangosteen-fruit pericarp, against the growth of methicillin-resistant *S. aureus* (MRSA). α-mangostin exhibited high efficacy [44].

4. Conclusion

The purpose of this study was to investigate the colorfastness, UV protection properties and antibacterial activity of silk fabrics dyed with MP natural dyes. It was found that dyed samples present interesting color fastness, UV protection properties and antibacterial activity. Further improvement in color yield was observed with different types mordanting. Each mordant gave different color shades grey, black, dark purple and reddish brown for without mordant, ferrous chloride, aluminum potassium sulfate and sodium hydroxide respectively. MP dyed silk fabric exhibited fair to good and good to very good fastness to washing and crocking, fair to very good light fastness, except for fabric mordanted with sodium hydroxide, whose rating were very poor to poor light fastness. The water and perspiration fastness ratings were good to very good, except for fabric mordanted with aluminum potassium sulfate. Silk fabrics mordanted with ferrous chloride and dyed with MP usually showed good UV protection levels even if undyed. A very good protection level was reached by silk mordanted with ferrous chloride and dyed with MP. As well as offer effective antibacterial activity against typical gram positive (*S. aureus*).This result indicates that MP contains high polyphenolic, gallic acid, chlorophyll and carboxylic acid contents. These colorants might be alternative sources to synthetic dyes.

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