

The Application of Chitosan for Environmentally Benign Process of Curcumin Dyeing of Silk Fabrics

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ABSTRACT

The present paper deals with the application of natural dyes extracted from *Curcuma longa* L. powder on silk fabrics and the assessment of chitosan coating effect on dyeability and colour characteristics. We have investigated the extraction behaviour of the curcumin as a function of ethanol concentration used as an extracting agent. It was observed that the increase in ethanol concentration increased the extractability of curcumin. In this study, curcumin was used as a source of colorant to dye silk fabrics. The dyeing process was carried out utilizing chitosan as a potential replacement for the environmentally harmful heavy metal salts in enhancing the natural dye uptake and the fastness properties. The results showed that chitosan improved the dyeing characteristics of curcumin dyed silk fabrics. Colour measurements in terms of K/S showed that the dye uptake values were found greater with higher concentration of curcumin. The binding of hydroxyl group of curcumin and polyamide of silk is suggested to take place via the formation of hydrogen bonding and ionic bonding through the reactive functional groups in chitosan. Heavy metal mordant was successfully replaced leading to environmentally friendly process of dyeing with *Curcuma longa* L. extract.

KEYWORDS: *Curcuma longa* L., natural dye, chitosan, fastness properties, dye uptake.

1. INTRODUCTION

Nowadays the interest in use of natural dyes for textile applications is growing as a result of the stringent environmental standard imposed in many countries to respond to the toxic and allergic reactions associated with synthetic dyes [1]. In spite of their inferior fastness, natural dyes exhibit better biodegradability and are generally more compatible with the environment and hence are more readily acceptable to the environmentally conscious users [2]. Other factors, such as the increasing price and non-renewable nature of raw oil from which fraction the synthetic dyes are originated, have also driven the shift toward the use of natural dyes. In the light of these considerations, natural dyes offer sustainable colorants for the future of textile industry [3].

Curcumin or bis(4-hydroxy-3-methoxyphenyl)-6-diene-3,5-dione is a C.I. Natural Yellow 3 derived from the rhizome of the plant *Curcuma longa* L., popularly called turmeric, a member of the Zingiberaceae family. Traditionally curcumin has been used as a dye for textiles. The major biologically active components of turmeric are curcuminoids, which include curcuminoids ($C_{21}H_{20}O_6$; MW 368.39), demethoxycurcuminoids ($C_{20}H_{18}O_4$; MW 338.39), and bis-demethoxycurcuminoids ($C_{19}H_{16}O_4$; MW 308.39) [4]. Curcumin is a yellow coloured phenolic pigment and is an effective antioxidant that can scavenge superoxide radicals, hydrogen peroxide and nitric oxide from activated macrophages [5]. Because of the wide spectrum of its biological activity, an extensive number of studies have been focussed on curcumin. Recently, curcumin has also been shown to display anti oxidant [5] and anti cancer [6] properties. Some studies have pointed out the possible involvement of the β -diketone moiety in the antioxidant action of curcuminoids, and the H-atom donation from the β -diketone moiety to lipid alkyl or a lipid peroxy radical as potentially more important antioxidant actions of curcumin [7].

Several investigations have been undertaken on the dyeing and functional finishing of textiles with curcuminoids or the turmeric. Shinyoung and Yiqi [8] carried out the dyeing of wool with an aqueous extract of commercially available curcumin and obtained relationship between curcumin concentration and antimicrobial activity of wool fabrics with curcumin against *E.coli* and *S.aureus*. Tsatroniet al. [9] studied the effect of chemical mordant such as aluminium sulphate, zinc chloride and potassium tartarate on wool and cotton fabrics dyed with curcumin and measured colour fastness to light, washing and rubbing. They observed improvement in fastness to light and washing of the mordant wool samples. The effect of gamma radiation on the dyeing of cotton with extract of turmeric on the colour fastness was studied by Ijazet al. [10]. The colour fastness to light, rubbing and washing properties showed that gamma irradiation has improved the dyeing characteristics from fair to good.

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The extraction of curcumin from plant materials is mainly based on the maceration method with conventional solvent extraction [11], hot and cold percolation [12], use of alkaline solution [13], an insoluble salt [14]. Moreover, the extraction of curcumin using the combination of circulation, ultrasonic, microwave, heating, pressure or enzyme treatment have also been reported [15-17]. One of the biggest challenges in using natural dyes for textile colorant is contamination of effluent streams with residual heavy metals from mordanting [18].

Conventionally, heavy metal ions such as copper, cadmium, iron, aluminium, and tin were used in mordanting fabrics to enhance the natural dye uptake and fastness properties [19]. In that case, criticism that the dyeing processes using natural dyes is as polluting as that of pre-metalized synthetic dyes found its strongest ground. Not only they can cause environmental problems, but these heavy metals have also raised serious health concerns regarding their long-term effects to skin. Research has been carried out with a view to minimize the amounts of mordant chemicals [18]. What is needed, however, is a novel approach in fixing natural dyes on textile fibres without the use of heavy metals.

Chitosan has got a wide application in textile dyeing and finishing as a substitute for the various chemicals used in textile processing [20]. It has been used as a pre-treatment agent in dyeing of cotton in textile printing, wool dyeing and shrink proofing and in durable press finish. Marzyeh et al [21] studied to investigate the grafting of chitosan on wool fabric using anhydrides and its properties such as antimicrobial and anti-felting. The microbial reduction against both *E. coli* and *S. aureus* was measured for chitosan grafted-acylated wool fabric sample. This study shows that the microbial reduction was better against *E. coli* bacteria. The anti-felting behaviour was improved by a chitosan grafting. It can be concluded that the grafting of chitosan increased the number of hydrophilic groups such as NH_2 and $-\text{OH}$ and increased the formation of hydrogen bonds by water molecules and decreased hydrophilicity of fabric.

Treatment with carboxyl methylchitosan makes it possible to dye cotton in bright shades with cationic dyes having high wash fastness. Treated samples showed good antimicrobial activity against *E. coli* and *S. aureus* at 0.1 % concentration as well as improved wrinkle recovery. The effect was found to be durable for five laundering cycles [22]. Saidehet al. [23] studied the effects of pH, chitosan concentration and reaction time on chitosan grafting of acylated silk on the basis of weight gain of acylated-grafted silk fibres. The weight gain of 12% after 12 hours was achieved at 20 mg/l initial concentration chitosan with pH 7. The grafting of acylated silk with chitosan provided a significant enhancement of dye uptake compared with results in the absence of grafting.

In this study, chitosan was used as environmentally-friendly substitutes for the conventional heavy metal mordants in the dyeing of silk fabrics with *Curcuma longa* L. extract.

2. MATERIALS AND METHODS

2.1 Materials and Preparation

Silk fabrics were obtained from Rumah Sutra Alam, Ciapus, Bogor, West Java. Chitosan (DD 90%) was purchased from the Laboratory of Badan Tenaga Atom Nasional, Jakarta, Indonesia. Fresh *Curcuma longa* L. was purchased from Rawamangun market in East Jakarta. 2 kg of *Curcuma longa* L. was chopped into small pieces and dried in oven at 70°C for 48 h. The dried *Curcuma longa* L. was ground to fine powder, passed through a 100 mesh sieve and kept in an airtight container at 4°C until further use.

All chemicals used were of analytical reagent grade. Deionised distilled water used throughout the experiment was produced by Milli-Q water purification system. 4-aminoantipyrine ($\text{CH}_3\text{-C}_9\text{H}_7\text{N}_3\text{O-CH}_3$), and potassium hexacyanoferrate(III) $\text{K}_3\text{Fe}(\text{CN})_6$ were purchased from Merck. Hydrochloric acid, acetic acid, citric acid and sodium borate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) were purchased from Aldrich. Curcumin standard compound was purchased from Sigma. HPLC grade solvents were purchased from J.T. Baker. Food grade 95% ethanol was used in all extraction experiments. Other analytical or biochemical grade organic solvents and chemical reagents were purchased from local suppliers.

A stock solution ($1000 \mu\text{g.l}^{-1}$) of standard curcumin was prepared daily by dissolving 25 mg of the curcumin in ethanol and completing the volume up to 25 ml with ethanol. Working standard solutions of curcumin (5, 10, 20, 30 and $50 \mu\text{g.l}^{-1}$ curcumin) were prepared by appropriate serial dilution of the curcumin stock solution in ethanol.

The 4-AP solution 0.1 M was prepared by dissolving 10.1625 g of 4-AP in 500 ml water and prepared daily. The potassium hexacyanoferrate(III) solution 0.001 M in alkaline media was prepared by dissolving 0.1646 g of potassium hexacyanoferrate(III) in 0.5 % Na_2CO_3 in 500 ml water and prepared daily. The borate buffer solution pH 9.0 was prepared by dissolving the calculated amount of 0.025 M sodium borate in deionised water and 0.1 M HCl solution is used to adjust the pH to 9.0.

2.2 Extraction of *Curcuma longa* L.

For macerating extractions as a control for traditional extraction methods, 5 g of *Curcuma longa* L. powder was extracted with a 10-fold excess volume of solvents (w/v) using 100W ultrasonic treatment for 1 h.,

unless otherwise stated. The defatted rhizome was further extracted by benzene for separating the curcumin. The curcumin extract was filtered through double layers of filter papers or centrifuged at 5000 g for 10 min. and used for the chemical determination of components or filtered through a 0.45 μm membrane. The supernatant was evaporated to dry at 60°C by means of rotary evaporator. The curcumin content were weighed in petri dishes and dried in circulating air oven (40°C) to constant weight.

2.3 Curcumin content

A suitable aliquot of the curcumin solution was prepared by dissolving 50 ml of curcumin in 25 ml ethanol 80%. A 10 ml aliquot was added to a mixture containing the 2 ml borate buffer pH 9.0 solution, 5 ml potassium hexacyanoferrate 0.005 M, three drops of 4-AP reagent, and the resulting solution was shaken for 2 min until the solution became red in colour. The absorbance of the curcumin was determined by UV-Vis spectrophotometer (Shimadzu 160 A) at 456 nm using a standard curve from analytical grade curcumin ranging from 1.0 to 10.0 mg/ml. Each sample was solubilized in ethanol and properly diluted. Experiments were performed in triplicate. The values of curcumin were expressed in mg curcumin/g dry weight of *Curcumin longa* L.

2.4 Degumming of silk fabrics

Silk fabrics were degummed in the degumming bath containing citric acid solutions at the concentration of 30%, and Noigen HC nonionic detergent (0.2%) at 98°C for 30 min. The material-to-liquor ratio was 1:20. After degumming, samples were washed with cold and warm water and finally dried immediately at 80°C for 1 h, and then kept at room temperature for 48 h. Degumming ratio, which correspond to the amount of sericin removed by different degumming treatment, was calculated from the weight loss of silk fabrics before and after degumming treatment using the equation (1),

$$\%D = \frac{W_a - W_b}{W_a} \times 100\% \quad (1)$$

where W_a and W_b are the weights of the dried fabrics before and after degumming, respectively.

2.5 Pre-treatment of silk fabrics with chitosan

A 2% (w/v) stock solution of chitosan was prepared by dissolving the required amount of chitosan in a 4% (v/v) aqueous acetic acid solution. Degummed silk fabrics samples (50 g) was then immersed directly in 2% (v/v) aqueous solutions of chitosan (2 l) at 50°C for 20 h. Afterwards, the silk fabrics was removed and dried at 100°C for 30 min, and then cured at 115°C for 5 min, rinsed with water at 50°C and allowed to dry at room temperature.

2.6 Dyeing using Curcumin extract

Curcumin powder was dissolved by boiling it in ten-fold water for one hour. The untreated and pre-treated silk fabrics were dyed at a liquor ratio of 1:10. Dyeing was conducted in a laboratory scale exhaust-dyeing machine at 90°C for 60 min. At the end of dyeing process, the dyed fabrics were rinsed in a detergent solution for 10 min at 60-70°C, followed by running hot water and cold water successively until no colorants come out. The samples were subsequently air dried under the shade and stored in an envelope.

2.7 Characterization of the silk fabrics

Fourier transform infrared (FTIR) spectra (transmission) were measured on a Perkin-Elmer FTIR spectrophotometer 2000 in the range of 4000-400 cm^{-1} at a resolution of 4 cm^{-1} . The sample and analytical grade potassium bromide (KBr) were dried at 100°C overnight.

Scanning electron microscopy (SEM) was used to examine the surface of the pre-treated silk fabrics. Pieces were cut from films and were mounted directly on stubs, freeze-dried and coated with gold. Samples were examined and photographed under a scanning electron JSM 6400, JEOL, Tokyo, Japan with the built-in films Image Analysing System. The printed silk fabrics were crosscut in the warp and weft directions to investigate the depth of the ink penetration.

2.8 Dye exhaustion (E%) and colour measurement (K/S)

The dyeability of *Curcuma longa* L. extract was measured and evaluated based on the spectral reflectance of dyed samples using VIS-spectrophotometer (Minolta CM3600d). The percentage of dyebath exhaustion was measured on UV-Vis Shimadzu spectrophotometer at the maximum λ of the dye used. The percentage of dyebath exhaustion (%E) was calculated according to the equation (2),

$$\%E = \frac{c_o - c_e}{c_o} \times 100\% \quad (2)$$

where C_o and C_e are the concentration of dyebath before and after dyeing, respectively.

2.9 Colour fastness test

The dyed silk fabrics were tested according to ISO standard methods. The specific tests were for colour fastness to washing (ISO: 3361 / 1979) and rubbing (ISO: 766 / 1988). The fastness ratings were evaluated by comparing with Grey Scale standard ISO-1976. The changes in shades were related to the standard Grey Scale rating (rating is 1-5: 1 is poor and 5 is excellent). Assessments were conducted for changes of colour shade and staining to adjacent layer(s) of silk and/or cotton upon the test procedures.

The wash fastness of the dyed silk fabrics samples were measured in Launder-o-meter as per the ISO 105-C06:1994 (2010) specifications. A layer of parallel lengths of dyed silk were sewed between the two pieces of adjacent fabrics (silk and cotton). The samples were washed with 5 g/l non-ionic detergent at 50°C for 45 min in Launder- o-meter under condition IIIA of the AATCC Test Method 124-2001 to determinethe colour change of fabricsand staining assessment after laundering.

Dry and wet rub fastness of the dyed silk fabric samples were tested using a Crock-meter as per Indian standard IS 766:1988 (Reaffirmed 2004) based on ISO 105-X12:2001 by mounting the fabric on panel and giving ten strokes for both dry and wet rub fastness tests.

2.10 Physical properties of treated silk fabric samples

The treated silk fabrics were characterized in terms of bending length, wrinkle recovery angle (WRA) in dry state and whiteness index (WI) to see if the treatment had any detrimental effect on the physical properties. The bending length was measured using a bending length tester (Paramount Stiffness Tester) based on the cantilever principle.

The wrinkle recovery property of the fabrics was measured as per the standard method: AATCC TestMethod 66-1975. The whiteness of treated fabrics was measured by CIEwhiteness index using Gretag Macbeth Colour eye 7000A.

3. RESULTS AND DISCUSSION

3.1 The effect of ethanol concentration on extracted curcumin content

Using traditional maceration method, various concentrations of a food grade ethanol and extraction conditions were tested to optimize the extraction of curcumin from *Curcuma longan*L. As shown in Fig. 1, the extraction efficiency of curcumin showed a rapid increase with the increasing ethanol content from 0.5 to 1.5 mg/g, and then followed by a slow decrease. The extraction efficiency decreased when the ethanol concentration was higher than 80%. The best solvent for curcumin was determined to be 80% ethanol using the ultrasonic method.

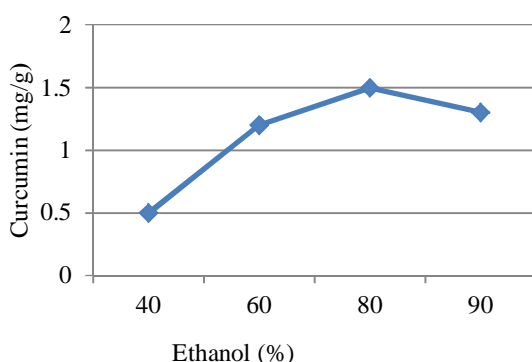


Fig. 1. Curcumin content with various concentrations of ethanol

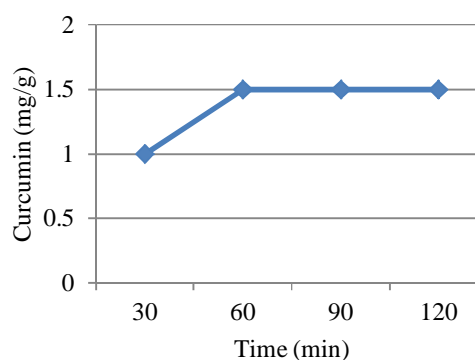


Fig. 2. Curcumin content extracted at various extraction times

3.2 The effect of extraction time on extracted curcumin content

The extraction of curcumin from *Curcuma longan*L. powder was carried out in various durations. The extracted curcumin content (mg/g) at various extraction times is shown in Fig. 2. It was observed that the curcumin content increased from 1 to 1.5 mg/g after one hour. Thus, one hour was chosen as the extraction time of curcumin.

3.3. The effect of degumming time on degumming ratio

Fig. 3 shows the results of citric acid degumming tests on silk fabrics and their comparison with blank samples. The degumming ratio (%D) of silk fibres treated with 30% citric acid solution for 10 min, 30 min, 45

min, and 60 min were 6.5%, 25.3%, 22.0%, and 25.4%, respectively. The effect of various degumming times on degumming ratio of silk fabrics treated with citric acid was evaluated. Fig. 3 shows degumming ratio and amount of residual sericin for the silk fibres treated with citric acid at 98°C for various degumming time starting from 30 min. Initially, %D of silk fabrics increased gradually with time and then attained the saturation level at about 25%. The amount of residual sericin, which remains on the raw silk fabrics, decreased with increasing time. The size of raw silk decreased gradually with the increasing time. Becker *et al.* (1995) confirmed that sericin content in *B. mori* cocoons varies from 19% to 28% and reported that it gradually declined during cocoon spinning. From the obtained results it can be inferred that almost all of the sericin was removed after degumming with 25% citric acid.

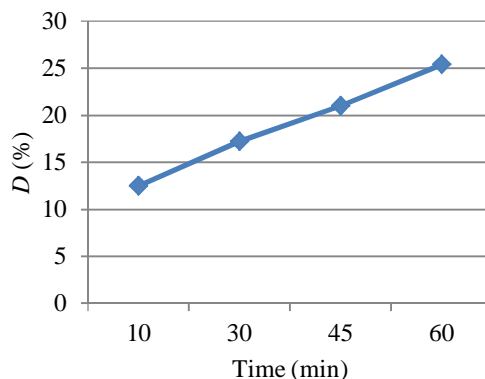


Fig. 3. Degumming ratio of silk fibres with various degumming times

3.4 Dye exhaustion and colour measurement

Both untreated and pre-treated silk fabrics showed yellow colour after dyeing. The high affinity of silk for the natural dyes in general is most probably due to the secondary amine groups ($>\text{NH}_2$) in the polyamide structure of silk, which become cationic ($>\text{NH}_2^+$) in water and available for ionic interaction with the anionic phenolic compounds ($-\text{O}-$) of natural dyes. Except for hydroxyl groups that are available for hydrogen bonding, such an active anchor sites do not exist in silk cellulose. This observation provides a clue as to the approach in finding substitutes for heavy metal mordants.

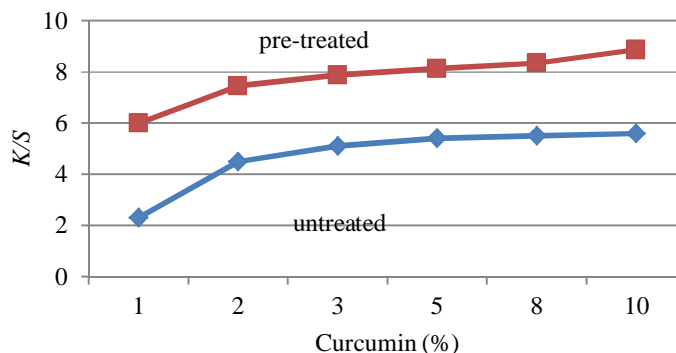


Fig. 4. K/S values of curcuminoids dyed silk fabrics at various concentrations of curcumin

The pre-treated fabrics, as expected and shown by the higher K/S values in Fig. 4, have darker colours than the untreated fabrics, which demonstrates that pre-treatment chitosan is effective in increasing the uptake of curcuminoids colorant just as the conventional heavy metal mordants are. In both cases, the cationic polymers imparted positive charges on the fibre surface, providing active sites to anchor anionic moieties such as phenolic compounds in the curcuminoids extracts on the textile fibres. Accordingly, as seen in Fig. 4, the colour uptake increased with the increase in curcuminoids concentration.

Further observation showed that chitosan, in this study, is a more efficient fixing agent, which can be observed by the fact that it gave higher K/S . Since chitosan was first dissolved in acetic aqueous solution and then applied to silk, the primary amines ($-\text{NH}_2$) in chitosan would exist in $-\text{NH}_3^+$ cationic form. As mentioned before, these $-\text{NH}_3^+$ cations would attract the anionic phenolic compounds in the curcuminoids.

The percentage exhaustion of dyes of silk fabrics samples for various concentration of curcumin dyes is recorded in Fig 5. Curcumin dyes used in this study showed higher dye exhaustion on chitosan treated silk than untreated silk sample. This could be explained based on the forces of repulsion and attraction expected to occur during dyeing process. These forces arose due to the presence of free hydroxyl groups in silk, cationic and anionic groups present in dyes, and amino (NH_3^+) ions in chitosan, besides other factors.

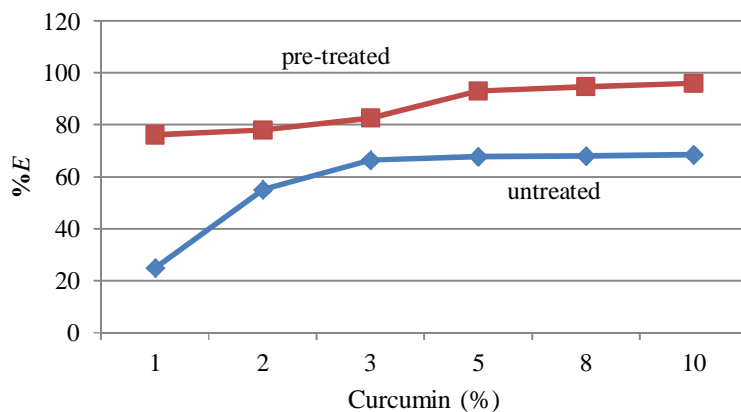


Fig. 5. %E value of curcumin dyed silk fabrics at various concentrations of curcumin

3.5. FTIR spectroscopy

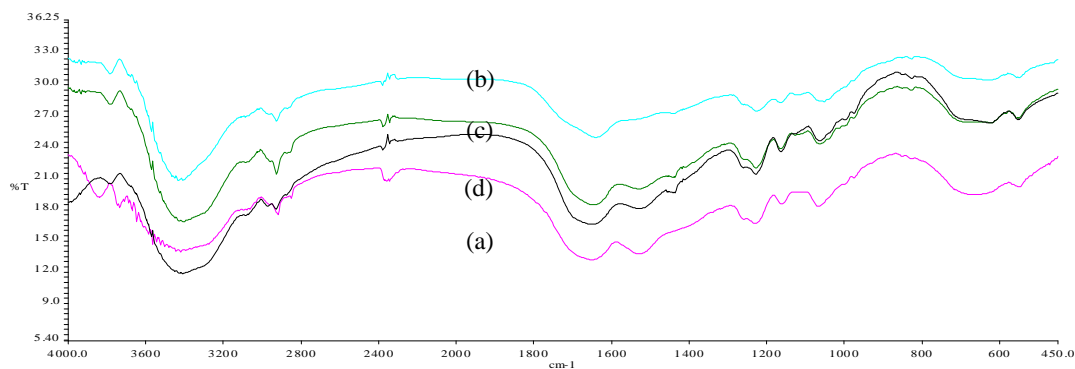


Fig. 6. FTIR spectra of: untreated silk (a), degummed silk (b), chitosan-treated degummed silk (c), and chitosan-grafted degummed silk after curcumin dyeing (d).

FTIR spectra were investigated to manifest the existence and formation of the newly formed bonds over the degummed silk due to the treatment of the samples with citric acid and chitosan. FTIR spectrum of raw silk is characterized by strong bands at above 3000 cm^{-1} (OH and NH stretching vibrations) and various amide bands in the range of $1700\text{--}600\text{ cm}^{-1}$, which are typical of polypeptides and proteins and allow their conformational characterization [20]. FTIR spectrum of degummed silk fabric (Fig. 6(b)) showed reduction in the intensity of the absorption and partially disappearance of the peaks at 2250 cm^{-1} and 1516 cm^{-1} that might be attributed to large amount removal of cyanide and amide groups respectively in sericin from the surface of silk samples by anhydride molecules. Fig. 6(a) represents the FTIR spectrum of chitosan grafted on degummed silk samples. It is seen that the omitted peaks 2870 cm^{-1} (symmetric CH_3 stretching) and 1550 cm^{-1} (NH stretching of chitosan) reappeared with high intensity absorption. Chitosan interaction with curcuminoid molecules was reflected by the lowering absorption intensity of symmetric CH_3 stretching (2870 cm^{-1}) in Fig. 6(c).

These changes might be a proof of the presence of amine groups of the grafted chitosan on degummed silk samples. Comparison of the FTIR spectra of degummed silk and chitosan-grafted samples (Fig. 6(b) and (a)) clearly indicated that the grafting treatment did not produce any considerable structural changes. These findings supported the results of the SEM images (Fig. 7) that it was more probably the external fibre layers that were engaged in the reaction of silk, citric acid, and chitosan. Based on the finding of this study, the grafting mechanism of chitosan using degummed silk can be proposed.

3.6 Morphology of silk surface

For the control samples, which were not treated with citric acid 30% as shown in Fig. 7(a), the surface characteristic of the silk fibre is fairly rough. This rough surface was clearly an evident to the large amount of sericin coating. The sericin appears as some partially non-uniform coating on the surface of the fibroins and various granules and impurity deposits are visible in the vacant spaces in-between the fibroins. Different surface morphologies and fibre damages of the raw silk fibres and degummed silks in various conditions are observed according to the SEM micrographs. The micrographs of samples degummed for 98 min show good degumming result and no sign of destruction or damage on the silk fibres surfaces (Figs. 7(b-d)). The fibres surfaces are mostly smoother due to sericin complete removal, which show that only very shallow longitudinal striations were attributable to the fibrillar structure for the truly degummed silk fibres (Fig. 7(b)). Chitosan-grafted sample showed a rougher texture by the presence of chitosan particles, which also gave additional weight (Fig. 7(c)). Sample subjected to curcumin dyeing was even rougher and heavier with additional curcumin dye particles (Fig. 7(d)).

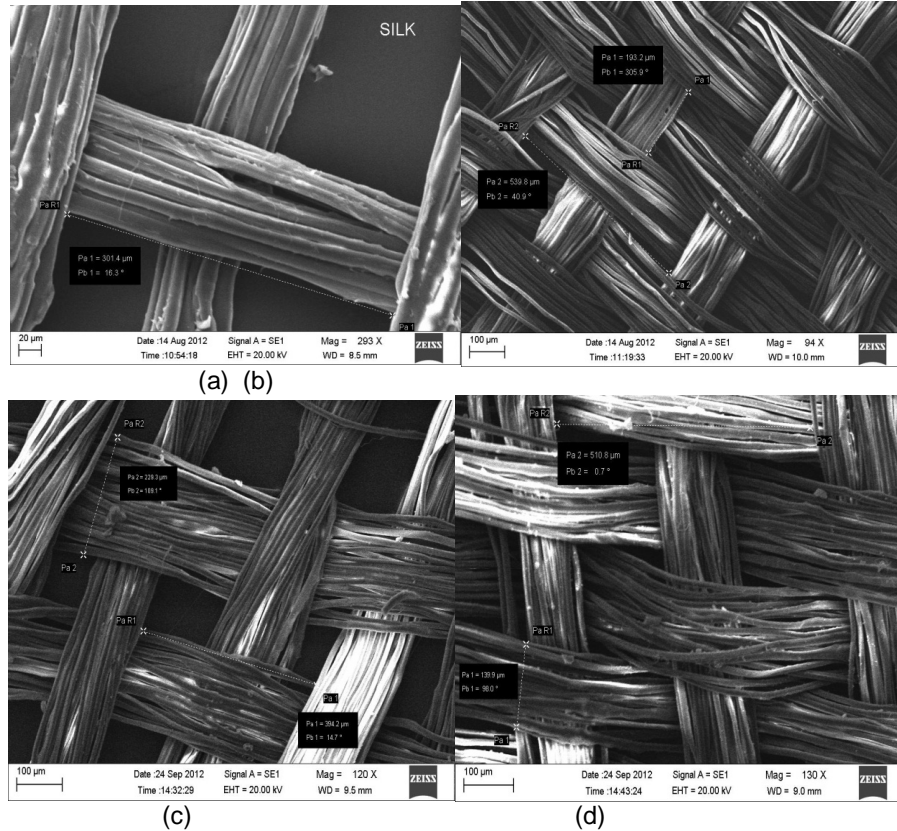


Fig. 7. SEM images of silk samples: (a) untreated silk, (b) degummed silk, (c) chitosan-grafted degummed silk, and (d) chitosan-grafted degummed silk after curcumin dyeing.

3.7. Differential scanning calorimetry (DSC)

Thermal measurement is a powerful tool for detecting fine structural changes of fibres. Fig. 8 shows the DSC curves of chitosan-grafted silk fabrics. The endotherm peak at 218.42°C for the degummed silk samples, which can be attributed to the helix melting, was shifted to 225.95°C for chitosan-grafted fabrics due to the formation of strong bonds such as amide.

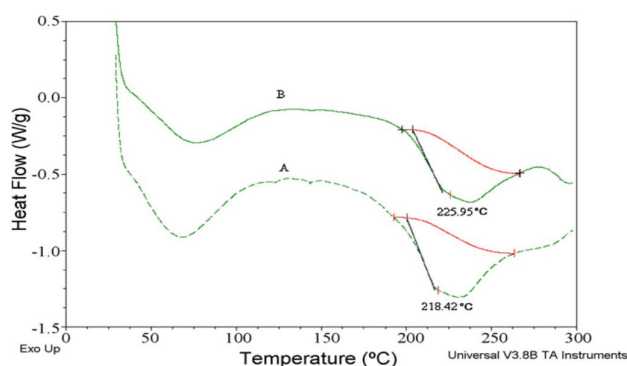


Fig. 8. DSC curves for silk samples: (a) degummed silk, and (b) chitosan-grafted silk

3.8 Determination of fastness properties

Table 1 shows the fastness properties of the silk samples dyed with the extracted dye. There was no change in fastness properties of dyes and the results showed that the dyed samples have high rating of good to very good fastness (4-5). This is because the non-saturation properties of functional groups such as C=C (ethylene) and C=O (carbonyl) in the natural dye enabled their binding and adsorption by the fiber and hence increasing colour fastness to washing and rubbing.

3.8.1. Washing fastness

The wash fastness of fabric dyed with and without degumming and with chitosan premordanting for curcumin natural dyes (ethanolic extract) are indicated in Table 1. These results were assessed in the usual way in terms of the grey scale values for the staining of adjacent wool material and alteration in shade. It is indicated that sample with natural dye extract did not alter in colour shade (good to very good rating of 4-5). With regard to staining to cotton, the samples showed various results from rather poor to good rating (2-4).

3.8.2 Rub fastness

Silk fabric samples dyed with curcumin using 1% chitosan as fixation agent (premordant) have shown a better rub fastness in comparison to corresponding samples without fixation. Dry rub fastness of non-fixation curcumin dyed samples were found to have fairly good to good rating of 3-4 on the grey scale whereas fixation curcumin dyed samples showed good rub fastness rating of 4. Wet rub fastness values of all curcumin dyed samples were found to have fairly good to good rating of 3-4.

Table 1. Fastness properties of silk fabric dyed with curcumin extracts with and without mordanting

Dyeing	Washing Fastness		Rubbing fastness		
	Shade change	Staining to cotton	Dry staining	Wet staining	Shade change
Dyeing without degumming	4-5	Silk: 3 Cotton: 3-4	4	3-4	4-5
Dyeing with degumming	4-5	Silk: 2-3 Cotton: 3-4	3	3-4	4
Dyeing with degumming and premordanting	4-5	Silk: 3-4 Cotton: 4	4	3-4	4-5

3.9 Physical properties of chitosan-treated silk fabric samples

Since chitosan is a high molecular weight polymer, its application to silk can affect the silk texture and other physical properties. To ensure that the chitosan treatment had no undesirable effects, treated silk was characterized in terms of several parameters related to its appearance and feel. The physical properties of fabric samples were compared in terms of wrinkle recovery angle (WRA), Whiteness index (WI), and bending length (BL).

All treated samples showed much higher wrinkle resistance compared to control samples (Table 2). Wrinkle recovery angle was slightly lower for chitosan-treated samples as compared to the untreated ones. The difference in wrinkle recovery angle between chitosan-treated and untreated fabric samples could be due to the low solubility of chitosan in water. The solution in this case was much less viscous. Thus, the film deposited on silk in case of the former would be finer and more flexible than the latter.

All treated fabrics showed slightly lower whiteness.

Another physical property tested was bending length, which is a measure of stiffness of fabric samples. All treated samples showed slightly higher bending length/stiffness than the control sample, indicating that the silk feel was not affected adversely by the treatment.

Table 2. Physical properties of silk fabric samples

Fabric and treatment	WRA	WI	BL	
			Warp	Weft
Silk fabric	117.24	85.74	2.2	1.9
Silk fabric - degummed	147.54	80.60	3.1	2.5
Silk fabric - degummed and chitosan- treated	148.12	79.20	2.6	2.1
Silk fabric - dyed	148.12	77.25	2.48	2.0

4. Conclusion

Silk fabrics were successfully dyed by curcuminoids using chitosan as fixation agent. Pre-treatment by chitosan increased the dye uptake. The depth and direction of colour changed with the increase of curcuminoids concentration. Most importantly, heavy metal fixation was successfully replaced, leading to a safer and more sustainable, environmentally friendly process of dyeing textile with natural dyes.

5. Acknowledgement

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