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Dyeing of curcumin and durable press finishing with citric acid on cotton fabric using one step process

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Abstract

Cotton is an affordable and durable option for clothing. However, cotton garments tend to wrinkle easily. Therefore, cotton fabrics were studied to improve their wrinkle resistance properties. In this work, curcumin was mixed in an aqueous solution of citric acid and its catalyst and applied to cotton fabric using pad-dry-cure process for both the dyeing and finishing properties. Aloe vera extract and chitosan were used to compare for the reaction of their functional groups with carboxyl groups of citric acid on cotton fabrics. The mixture of curcumin and citric acid solution showed anti-wrinkle properties on the treated cotton. There were no functional groups of curcumin detected on the FTIR spectrum of the fabric. Furthermore, acetyl group of chitosan and methyl in acetyl group of aloe vera extract were found on the spectra of the fabrics treated with each compound. It was also found that the treated fabric showed good UV protection properties and good antibacterial gram-positive properties. This fabric has physical potential to be used for healthcare, hygiene and medical textile products.

1. Introduction

Nowadays, cotton is still one of the most favorable fibers for apparel products. This is due to its good properties such as strength, water absorption and breathability [1,2]. These properties prominently make cotton fabric comfortable to wear. Nevertheless, cotton has poor wrinkle recovery property [3]. This drawback can be overcome by a durable press finishing process using certain cross-linking agents. The most commonly used agent at the present is N, N'-Dimethyl-4,5-dihydroxyethylene urea (DMeDHEU) which is assigned in the category of a non-formaldehyde-containing type [4]. Polycarboxylic acids are also other types of cross-linking agents recently stated as an alternative for wrinkle resistant finishes [5]. They function as anti-wrinkling agents via an esterification reaction with hydroxyl groups of cotton fibers. As comprising of more than one carboxyl group, polycarboxylic acids can simultaneously undergo chemical reactions with other organic compounds containing either hydroxyl groups or amino groups. Citric acid is one of the polycarboxylic acids which is a non-formaldehyde based, environmentally acceptable, wide availability, and cost-effective agent [6,7]. In addition, the successful bonding of aloe vera extract and chitosan on cotton fabric in the presence of citric acid has been previously researched [8].

Curcumin, a natural colorant, consists of 3 hydroxyl groups in their structure and is challenging to investigate its possibility to simultaneously link with cotton fibers in the presence of citric acid. Curcumin is of interest since it provides not only dyeing properties, but also various biological activities. These include antioxidant, anti-inflammatory, anti-microbial, and anti-carcinogenic effects [9]. Therefore, multifunctional properties are expected to be achieved and this work may initiate the trial of combining dyeing and finishing processes for cotton and for future work on other fibers.

2. Experimental

2.1 Materials

The fabric used in this research was bleached 2/1 twill cotton with a weight of 173.9 $g \cdot m^{-2}$ and a fabric thickness of 0.34 mm. The fabric structure of 40/1z × 40/1z with 112 yarns per inch in both warp and weft directions. Citric acid (Ajax Finechem, Australia) was used as a crosslinking agent. Sodium hypophosphite monohydrate (KemAus, Australia) was used as catalyst. Curcumin (Aldrich, USA), chitosan (Aldrich, USA), and aloe vera extract (Nardev Chemie, Thailand) were used as coloring/finishing compounds. Coconut oil (Aldrich, USA) was used as an emulsifier.

2.2 Preparation of treated cotton fabric

All experiments in this work were divided into 2 steps as follows:

Step I: Durable press finish with citric acid

Dry cotton fabric was immersed into the finishing solution containing 6%w/v to 8%w/v citric acid and 3%w/v to 7%w/v sodium hypophosphite before being padded within the range of 70% to 75% pick up. The fabric was then dried and cured at 100°C for 1.50 min

and at 150°C for 2 min, respectively using PI-I/M6 mini-stenter machine (Copower Company, Thailand). The finished fabric was washed one time according to AATCC 124-2018. The washing mode was set at temperature of 41 ± 3°C and tumble dried after washing.

Step II: Process of mixing curcumin/ aloe vera extract/ chitosan into the finishing solution

A 0.1 g of curcumin/ aloe vera extract/ chitosan was individually prepared with a coconut oil as an emulsifier. This was then mixed well in the solution of citric acid and sodium hypophosphite. Firstly, the fabrics were immersed into the mixture and then followed by the pad-dry-cure method using the same conditions as step I.

2.3 Physical test

After preparation of the test specimen, all fabrics were tested in the standard testing atmosphere with a relative humidity of 65 ± 2% and a temperature of 21 ± 2°C for 24 h prior to test. The physical tests were also conducted. They were as follows:

(1) Smoothness appearance of fabrics after repeated home laundering according to AATCC test method 124-2018,

(2) Wrinkle recovery of woven fabrics: Recovery angle according to AATCC test method 66-2017,

(3) Standard test method for tearing strength of fabrics by falling-pendulum type (elmendorf) apparatus according to ASTM D 1424-21,

(4) Breaking force and elongation of textile fabrics (strip method) by ASTM D 5035-11 method.

2.4 FTIR-ATR technique evaluation

The treated and untreated cotton fabrics were investigated and compared for their functional groups using a Nicolet 6700 Fourier Transform Infrared Spectroscopy (FTIR) (Thermo Scientific, USA). The transmittance of each sample was recorded between 4,000 cm^{-1} and 650 cm^{-1}.

2.5 Color measurement

The K/S and CIE L*a*b* values of untreated and treated cotton fabrics were determined by Datacolor Check II spectrophotometer (Datacolor, USA). The L* value represents the lightness with values from 0 (black) to 100 (white). The a* and b* values defined red-green axis and yellow-blue axis from positive to negative, respectively. The K/S value was calculated using the Kubelka-Munk equation.

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

where R is the reflectance of fabrics at maximum wavelength

2.6 Water absorbency of fabrics

The untreated and treated cotton fabrics were evaluated according to AATCC 79-2014 standard. This method is based on observation-based technique. In brief, one drop (0.5 mL) of water was delivered from a fixed height onto the fabric at an ambient temperature of 21 ± 2°C, relative humidity levels of 65 ± 2%. Then, the water drop was observed and measured for how long it took the water drop to disappear.

2.7 UV protection properties

The UV protection properties of fabric were evaluated in accordance with standard AS-NZS 4399-2017. The UV protection properties in terms of UV protection factor (UPF) and the UV radiation blocked (UVA and UVB) were measured using a UV-VIS spectrophotometer. In brief, the fabric specimens were cut and mounted without tension on a slide frame ready for measurement. The spectrophotometer was performed in the wavelength range of 290 nm to 400 nm with a wavelength resolution of 5 nm. For each fabric sample, four specimens were measured and the mean UPF was calculated according to Equation 2. In addition, the UPF of the fabrics was classified into specific UPF ratings as shown in Table 1.

$$UPF = \frac{\sum_{290}^{400} E_\lambda \cdot S_\lambda \cdot \Delta_\lambda}{\sum_{290}^{400} E_\lambda \cdot S_\lambda \cdot T_\lambda \cdot \Delta_\lambda} \quad (2)$$

where S_λ = the solar spectral irradiance (in $Wm^{-2}Nm^{-1}$)
E_λ = the erythemal spectral effectiveness from CIE 1987
T_λ = the spectral transmission through the textile
Δ_λ = the bandwidth (in nm), and λ is the wavelength (in nm)

2.8 Antibacterial properties

The treated and untreated fabrics were tested according to the AATCC test method 100-2012 in their effectiveness against Gram-positive (*S. aureus*) and Gram-negative (*E. coli*) bacteria. The fabrics were briefly introduced into a 100 mL nutrient broth inoculated with the microbes. The cultures were incubated for 24 h at 37°C. Microbial inhibition was determined by the reduction in the number of bacterial colonies using the following Equation:

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

where B = the number of survival bacteria before contact with fabric
A = the number of survival bacteria after contact with fabric

Table 1. AS/NZS 4399-2017 UPF classification system.

UPF range	UV protection category	% of UV radiation blocked
15-29	Minimum protection	93.3
30-49	Good protection	96.7
50, 50+	Excellent protection	98

3. Results and discussion

3.1 Physical and mechanical properties of untreated and citric acid treated fabrics

The physical properties and the FTIR spectra for untreated fabric and treated fabric with a solution composed of 6%w/v to 8%w/v citric acid and 3%w/v to 7%w/v sodium hypophosphite were reported in Table 2-3 and Figure 1, respectively.

As seen in Table 2, the evaluation of the physical properties of the finished fabric showed that citric acid could enhance the smoothness appearance of cotton from SA-1 to a higher level, SA-2 to SA-3. Also, the total crease recovery angle increased from 150.2º to 206.3º. Both the citric acid and sodium hypophosphite concentration has been correlated with smoothness appearance and total crease recovery angle for the finished fabrics. The explanation of these crease recovery results is due to citric acid consisting of three carbonyl groups with sodium hypophosphite catalyst at a high temperature forming a cyclic anhydride intermediate. This intermediate reacts immediately with the hydroxyl groups of cellulose producing ester linkage [10-11].

According to the spectra results shown in Figure 1, a new sharp peak emerged at 1723 cm^{-1} of the citric acid treated cotton spectrum and is attributed to carbonyl (C=O) stretching of the ester bond. It is an indication of esterification reaction between carboxylic groups of citric acid and hydroxyl groups of cellulose.

Compared to untreated cotton fabric, the tensile and tearing strength retention of the treated fabric were reduced in both warp and weft directions (Table 3). A higher concentration of citric acid solution tended to decrease the tensile and tearing strength retention of the fabrics. Moreover, the tensile and tearing strength retention of the fabrics decreased with the increase in sodium hypophosphite concentration. Similar results were seen in the studies of Dhiman and Chakraborty [12] and Ahmed *et al*. [13]. An optimum condition of the finished cotton fabric used in this research was 6%w/v citric acid and 4%w/v sodium hypophosphite for acceptable tensile and tear strength retention (loss less than 40%), good smoothness appearance, and high total crease recovery angle.

3.2 Color measurement of untreated and citric acid mixed curcumin treated cotton fabrics

After the fabric was finished and dyed with a mixture of citric acid and curcumin, the cotton fabric was initially evaluated for its color and handle properties. The results are shown in Table 4 and Figure 2.

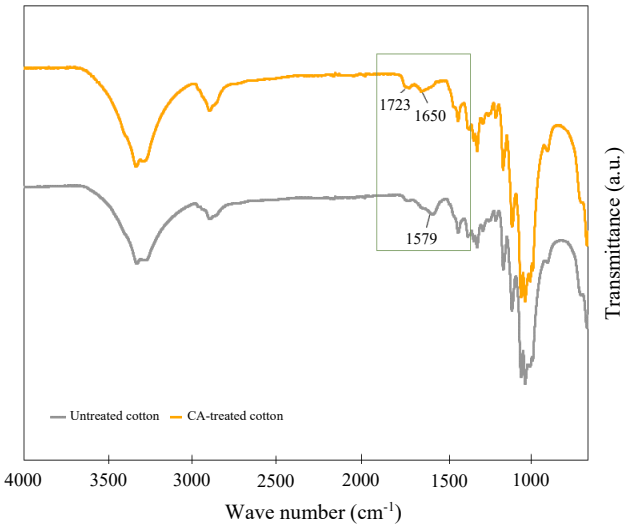

Figure 1. FTIR spectra of untreated (untreated cotton) and citric acid treated cotton fabrics (CA-treated cotton).

Table 2. Physical properties of cotton fabrics before and after finishing with citric acid.

Citric acid (%w/v)	Sodium hypophosphite (%w/v)	Smoothness appearance	Total crease recovery angle, degrees (º)
0	0	SA-1	150.2 ± 0.8
6	3	SA-2	158.6 ± 1.7
6	4	SA-3	206.3 ± 1.3
6	5	SA-3	192.0 ± 0.9
8	5	SA-2	165.3 ± 2.4
8	6	SA-3	194.2 ± 2.1
8	7	SA-3	180.4 ± 1.7

Table 3. Tensile and tearing strength retention of untreated and treated fabrics.

Citric acid	Sodium hypophosphite	Tensile strength retention		Tearing strength retention	
(%w/v)	(% w/v)	warp	weft	warp	weft
0	0	100	100	100	100
6	3	89.2	85.6	78.3	83.2
6	4	86.1	75.5	68.8	76.9
6	5	81.6	71.1	62.5	71.6
8	5	77.8	72.6	67.0	62.2
8	6	77.5	72.7	68.8	61.5
8	7	65.4	73.0	66.1	57.3

Table 4 Color values of untreated and citric acid mixed curcumin treated cotton fabric.

Citric acid (%w/v)	Sodium hypophosphite (% w/v)	K/S at 440 nm	CIE L*a*b*		
			L*	a*	b*
0	0	0.02	98.76	-0.03	4.23
6	3	3.29	90.08	-3.16	51.03
6	4	3.72	90.56	-3.11	55.83
6	5	3.64	89.11	-2.15	55.85
8	5	3.76	90.64	-3.19	56.40
8	6	3.57	90.18	-2.78	54.76
8	7	3.78	90.51	-2.93	53.82

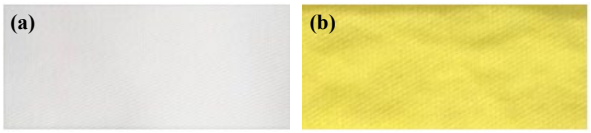

Figure 2. Photographs of cotton fabric (a) untreated fabric (b) cotton fabric treated with 6% w/v citric acid and 4% w/v sodium hypophosphite.

It was found that the handling properties of fabric treated with the mixture of citric acid and curcumin was similar to that of the citric acid treated cotton fabric. In the case of the citric acid treated fabric dyed with curcumin, the dyed fabric exhibited a yellow color which became slightly darker (L* value decreased), yellower (b* value increased) and less red (a* value decreased) compared to the untreated cotton fabric.

3.3 FITR analysis

Due to curcumin having three hydroxyl groups, other natural substances containing more hydroxyl groups or other functional groups reacted more easily to the carboxylic groups that were selected and compared. Thus, aloe vera extract and chitosan were used in this experiment and compared to curcumin. The FTIR spectrum for untreated and treated cotton fabrics with curcumin, chitosan, and aloe vera are displayed in Figure 3.

When compared to the spectrum of the untreated cotton fabric, new peaks were observed at the wave number of 1725 cm^{-1}, 1719 cm^{-1}, 1653 cm^{-1}, 1359 cm^{-1}, 1247 cm^{-1}, and 1000 cm^{-1}. Typically, peaks at 1725 cm^{-1}, 1247 cm^{-1}, and 1000 cm^{-1} were attributed to the vibration stretching of carbonyl carbon (C=O), asymmetric C-O bond attached to the carbonyl carbon with the involvement of the alpha carbon-carbonyl carbon C-C bond, and the second asymmetric C-O moiety in the ester, respectively. These three peaks were found in the spectrum of the citric acid treated cotton dyed with curcumin.

Compared to the spectrum for citric acid treated cotton dyed with curcumin, only two peaks at 1247 cm^{-1} and 1000 cm^{-1} were noticed from the spectra for the citric acid treated cotton finished with aloe vera extract and the citric acid treated cotton finished with chitosan. The peak position at 1725 cm^{-1} seemed to have shifted to 1719 cm^{-1} and was also related to the stretching vibration of the carbonyl group (> C = O) for the ester bond. The reason could be due to the competition in esterification of the carboxylic groups of citric acid. They could react not only with the hydroxyl groups of cotton, but with hydroxyl groups of aloe vera extract or amino groups of chitosan as well. A decrease in peak intensity was also noticed.

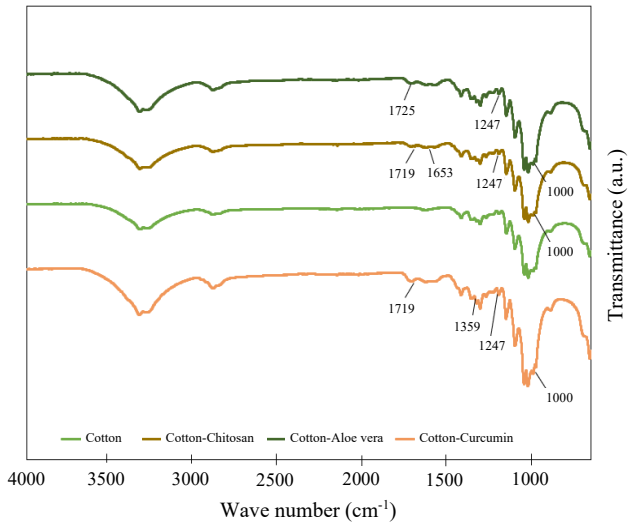

Figure 3. FTIR spectra of untreated (Cotton) and treated cotton fabrics with curcumin (Cotton-Curcumin), chitosan (Cotton-Chitosan), and aloe vera (Cotton-Aloe vera).

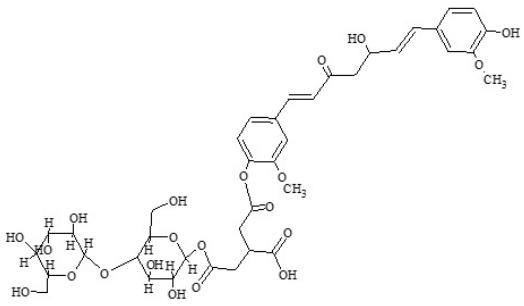

Figure 4. The possible structure of cotton fibers finished with citric acid and dyed with curcumin.

The peak positions of the spectrum for the citric acid treated cotton finished with curcumin were different from those of the citric acid treated cotton finished with aloe vera and chitosan spectra. It could therefore be suggested that no esterification reaction took place between the carboxyl groups of citric acid and hydroxyl groups of curcumin. This statement was supportive with the presence of peaks at 1653 $cm^{-1}$and 1359 cm^{-1} corresponding to the stretching vibration of acetyl group in chitosan and the C-H bending of methyl (CH_3) in the acetyl group of aloe vera extract, respectively. Whereas no peaks were indicated for the presence of curcumin on cotton. In order to ensure that the non-existence in the substitution reaction of hydroxyl hydrogens of curcumin was not due to the heterogeneous

reaction, curcumin emulsion prepared with a natural oil and polyethylene glycol fatty acid ester as an emulsifier was used in the experiment. The proposed structure for citric acid mixed with curcumin treated cotton fabric is shown in Figure 4.

3.4 Water absorption properties

Cotton, typically, loses hydroxyl groups through esterification reactions with carboxylic acids present in citric acid [5,12]. Therefore, water absorbency was tested and the results of the experiments are displayed in Table 5.

The time a water drop took to disappear from the fabric was used a measure of the wettability of the fabric. The high wettability of fabric will shorten the absorption time. Normally, a scoured and bleached cotton fabric exhibits good water absorbency, high whiteness degree, and excellent hydrophilicity.

As shown in Table 5, the time spent absorbing water in seconds of untreated and treated fabrics was found. These results indicated good wettability. There was no difference in absorption time for both the front and the back the fabric. The treated fabric still had the required level of possession and could still maintain its water absorption properties. If this treated fabric was made into clothing, it would still make the wearer feel as comfortable as the cotton fabric that has not been finished.

3.5 UPF measurement

Curcumin, a substance in turmeric, used as a natural color in this research showed a broad characteristic UV-visible absorption spectrum. Therefore, the untreated and treated cotton fabric obtained UPF and the ability to block out both UVA and UVB rays as shown in Table 6.

According to the test results in Table 6, the untreated fabric has poor UV protection properties and an extremely low UPF of 5.1, while the treated fabric displayed a minimum level of UV protection properties (UPF rating of 15) according to AS/NZS 4399:2017. This may be due to the natural assimilation properties of natural colors from turmeric with curcumin content with the highest absorption properties in both the wavelength range of 265 nm. This is the UV ray area, and at the approximate wavelength between 410 nm and 430 nm of the visible wavelength [14].

Table 5. Water absorbency time for the untreated and treated fabric.

Citric acid	**Sodium hypophosphite**	**Absorbed time (s)**	
(%w/v)	**(%w/v)**	**Front of fabric**	**Back of fabric**
0	0	1	1
6	3	1	1
6	4	1	1
6	5	1	1
8	5	1	1
8	6	1	1
8	7	1	1

Table 6. UV protection properties of untreated and treated fabric.

Citric acid	**Sodium hypophosphite**	**UPF**	**% of UVA blocked**	**% of UVB blocked**
(%w/v)	**(%w/v)**			
0	0	5.1 ± 0.4	85.1 ± 1.2	91.1 ± 0.2
6	3	14.8 ± 1.8	94.6 ± 1.1	94.2 ± 0.9
6	4	15.6 ± 1.4	95.0 ± 1.6	94.6 ± 1.3
6	5	16.1 ± 2.1	96.2 ± 3.7	93.8 ± 0.5
8	5	15.7 ± 2.3	93.9 ± 3.2	93.4 ± 1.4
8	6	15.3 ± 1.8	94.5 ± 1.9	94.7 ± 2.1
8	7	15.8 ± 2.0	94.9 ± 1.7	93.6 ± 0.8

Table 7. Efficiency of antimicrobial cotton fabric against *S. aureus* and *E. coli*.

Citric acid	**Sodium hypophosphite**	**% Reduction**	
(%w/v)	**(%w/v)**	***S. aureus***	***E. coli***
0	0	0	0
6	3	98.62	0
6	4	99.75	0
6	5	99.47	0
8	5	99.90	0
8	6	>99.94	0
8	7	>99.94	0

3.6 Antibacterial properties

Both citric acid and curcumin, a major natural color present in turmeric, exhibited antibacterial properties. The untreated and treated cotton fabrics were tested according to AATCC 100-2012 standards. The efficiency of antimicrobial cotton fabric against *S. aureus* and *E. coli* is shown in Table 7.

The fabric finished with citric acid and dyed with natural colors from turmeric at the same stage showed strong antibacterial activity against *S. aureus*. On the other hand, the growth of *E. coli* bacteria was not inhibited. *E.coli*, gram negative bacteria is more resistant to the action of the antimicrobial agent than *S. aureus*, gram positive bacteria. This is due to an additional protection layer of the outer membrane [15]. There are various research studies that used curcumin as an antibacterial agent against *S. aureus* [16-18]. *S. aureus* is a particularly pathogenic bacteria to humans that commonly causes infections in hospitals and the community resulting in food poisoning and skin and soft tissue infections [19-20]. Tyagi *et al.* [21] demonstrated that bacterial membrane damage and leakage of intracellular contents on exposure to curcumin. Abdulrahman *et al*. [22] showed that the curcumin molecule induces phototoxicity by generating reactive oxygen species (ROS) and inhibits bacterial growth. Based on this, the antibacterial action of curcumin relates to bacterial membrane disruption, inhibition of bacterial biofilm formation, induction of oxidative stress, and prevention the attachment of bacteria to the host receptors.

4. Conclusions

In this work, the one step process of dyeing with curcumin and finishing with citric acid was successfully achieved. The smoothness appearance, total crease recovery angle, tearing strength retention and tensile strength retention of fabric depends on the amount citric acid and sodium hypophosphite used. The obtained fabric shows much improved UV radiation protection and antibacterial efficacy against *S. aureus* with excellent water absorption.

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