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Utilizing Citrus Limon in Place of Acetic Acid for an Eco-Friendly Silk Fabric Coloring Process

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ABSTRACT In this study, acetic acid and citric acid were used in place of only acetic acid to color silk fabric using Bemacid Blue N-TF dyes in 2% hues. The dyed sample was assessed using a variety of metrics and methods, including fixation efficiency (%), fastness properties, and K/S value. A sample of citric acid displayed the same color fixation and fastness characteristics. Citric acid significantly reduced the results of the tests conducted on the wastewater from both samples, both in terms of chemical and biological oxygen demand. The significant sustainable approach to acid dyeing is demonstrated by the citric acid dyed sample's comparable wash fastness of 4-5, BOD of 240 mg/L, and COD of 2375 mg/L.

INDEX TERMS acetic acid, acid dye, citric acid, silk dyeing, sustainability

I. INTRODUCTION

Citrus limon, also known as lemon in English, is a type of edible fruit from the Rutaceae family [1]. Lemon could be used in aromatherapy. Lemon juice contains citric acid (CA), which can help people relax even though it has no effect on the human body or the immune system [2], [3]. CA was primarily obtained commercially from lemons prior to the introduction of fermentation-based methods [4]. CA is colorless, weak organic acid with the chemical formula CH_2COOH-$C(OH)COOH$-CH_2COOH [5]–[8].

CA has a wide range of applications, including flavoring, acidification, and chelating [5]. We use lemon juice in certain household appliances to clean textiles and for aesthetic purposes. CA has the potential to lower water hardness and improve the efficacy of soaps and detergents [9]. CA is classified as a chemical that has no harmful effects on nature due to its easy breakdown in soil and surface water [10].

Cellulose and its derivative fabrics are the global leaders in the textile business, while animal or protein fibers continue to play a significant role

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in the higher end of the textile and fashion markets. Complex proteins comprise animal fibers. These fibers can be dyed using the same groups of dyestuffs and have typically similar dyeing characteristics. Fibers made of proteins, such as silk, are held together by sericin and fibroin filaments [11], [12].

In order to form cocoons, certain insect larvae produce fibroin, which is the primary component of the protein fiber that makes up silk. Although many insects can produce silk, only moth caterpillar silk has typically been utilized in the textile industry [13], [14]. Silk is a highly valuable natural animal protein fiber that offers several advantages such as comfortable wear, a smooth texture, excellent air permeability, and a sophisticated appearance [15]. In the textile industry, silk is frequently used to make apparel, furnishings, and other equipment. Furthermore, silk's mechanical strength and biocompatibility make it a desirable biomaterial for tissue engineering and medical applications. Scientists and researchers are interested in silk coloration because the dyeing process is inevitably linked to the production of textiles [16].

The primary dye classes used in the commercial dyeing of silk are acid dyes. Acid dyes are inexpensive, have vibrant hues, and a large color spectrum [17]–[19]. Acid dyes are applied primarily from an acidic bath, are soluble in water, and are anionic in nature. The protonated $-NH_2$ group of the silk fiber forms an ionic bond with the acidic groups of acid dyes, such as $-SO_3H$ and -COOH [17]. According to Fatma et al. [20], dyeing silk fibers in an acidic medium (pH=3) allows acid dyes to be exhausted more effectively than dyeing them at a comparatively higher pH (>3). Compared to strong sulfuric or hydrochloric acids, the use of acetic acid (AA) demonstrated superior fastness properties and compatibility with silk fibers [21].

One of the key carboxylic acids that are widely utilized in the textile industry as a chemical reagent in specific acid dyeing processes is acetic acid (CH_3COOH). It is a transparent, colorless liquid that is an organic compound and has a high blistering potential [22]. In many post-dyeing processes, acetic acid in its aqueous form can be found as a waste stream or by-product. The environment is seriously at risk from these chemicals [23]. Formic and acetic acid residues are typically found in waste streams. Acetic acids may be found in the forms of multiple derivatives such as Aluminum Acetate, Methyl Acetate, Ethyl Acetate, Acetyl Chloride, Acetamide etc. All these mentioned chemicals need to be chemically treated, recycled, or diluted before they can be broken down biologically. Human eyes, noses, and throats become irritated when acetic acid concentrations exceed 10 ppm. Serious diseases such as metabolic acidosis, disseminated intravascular coagulopathy, acute liver dysfunction, acute renal failure, and acute respiratory failure are resulted from long-term exposure to acetic acids [24]. Excessive exposure to acetic acid can cause acute kidney failure, hemolysis, and disseminated intravascular coagulation [22], [25].

II. EXPERIMENT

A. MATERIALS

The lemon (pH of 2.5) and 100% pure raw silk woven fabric (GSM 180 before degumming) were bought from the Dhaka, Bangladesh (BD) local market. From RH corporation in Dhaka, BD, acid dyes were obtained: Bemacid Blue N-TF (Acid Blue). Additional relevant chemicals and auxiliary materials, such as soda ash (98.2%), acetic acid (97%), wetting agent (95%), sequestering agent (95%), leveling agent (100%), and detergent (92%), were obtained from Southeast University's laboratory complex in Dhaka, Bangladesh.

B. DEGUMMING OF SILK FABRIC

The process of degumming involves taking out the sericin, a sticky material made by the silkworm that keeps the silk strands together. Sericin undergoes hydrolytic cleavage when dissolved in hot water, which may vary according to the time variation [26]. Another name for it is silk scouring. Due to the minimal effect of soap on the fiber degradation, soap-degummed silk exhibits good lightness strength, and softness [27], [28].

TABLE I
DEGUMMING RECIPE OF SILK

Reagent/ Conditions	Amount
Soda ash	2 gm/L
Detergent	2 gm/L
Fabric weight	5 gm
Temperature	90°
Time	40 min
M:L	1:30

$$\text{Chemical/ Auxiliaries required} = \frac{\text{total liquor} \times \text{gm/L}}{1000} \quad (1)$$

The degumming procedure was executed by utilizing the recipe given in Table 1, which was sourced from Gulrajani M.L. [26]. The required amount of chemicals and auxiliaries was calculated using the equation 1.

To perform degumming, all materials and textiles were combined with the necessary volume of water in a sealed pot. The procedure was carried out using a sample dyeing machine from Rapid Eco Dyer (Xiamen, China). The degummed sample was collected after finishing the process, and was properly washed and dried for further experiments.

C. DYEING OF SILK FABRIC

The dyeing process was carried out in exhaust method using 2% shade of Acid Dyes (Blue). This process was done at 95-100° for 40 min. The amount of dye required was calculated using equation 2, and all other chemicals were calculated using equation 1. For proper comparison between Acetic acid and Citric acid samples, all chemicals and auxiliaries were used in the same amounts. The dyeing recipe is provided in Table II.

$$\text{Dyes required} = \frac{\text{Sample wt.} \times \text{Shade\%}}{100} \quad (2)$$

TABLE II
DYEING RECIPE OF SILK

Reagent	Amount
Acid dye	2%(OWF)
Acetic acid/ Citric acid	1 gm/L
Wetting Agent	1 gm/L
Sequestering Agent	1 gm/L
Levelling Agent	1 gm/L
Fabric wt.	4 gm
Temperature	95-100^{0}C
Time	45 min.
pH	4.5-5.5
M:L	1:40

The silk fabrics were dyed using the same ecodyer machine. Using acetic acid and citric acid, a shade of 2% blue color was produced. After gathering, the dyed samples were properly cleaned, dried, and stored for use in the upcoming tests.

D. COLOR STRENGTH(K/S) & REFLECTANCE% TEST OF SILK FABRIC

All dyed samples were tested for reflectance, and color strength was measured using the data Color 650TM (data Color, USA). Three different light sources; D65-10, A-10, and F02-10, were used while wavelength ranged between 400 to 750 nm, and the opening of the aperture plate was 17 mm. The relationship between color strength and the reflectance % are as follows:

$$K/S = \frac{(1-r)^2}{2r} \quad (3)$$

E. FASTNESS TESTS OF SILK FABRIC

1) COLOR FASTNESS TO RUBBING

The Crock Meter apparatus was utilized to measure Colorfastness (CF) to Rubbing, which refers to the degree of color transfer that occurs when a colored fabric is rubbed both wet and dry on a specific test cloth. The ISO 105×12 method was used for this process. The test specimen was rubbed back and forth in a straight line for ten cycles with a downward force using a white, conditioned rubbing cloth.

2) COLOR FASTNESS TO WASH

CF to wash was done in the WASHTEC-P machines by following the ISO-105 C06 test method to check the ability of our dyed samples to maintain any of its color characteristics after being washed with regular household detergents. Here, a test specimen measuring 100 x 40 mm was sewn to a multi-fiber of same size and fastened together by sewing along one of the shorter edges.

Using a solution of 4 gm/L ECE detergent and 1 gm/L sodium perborate, the prepared sample was washed for 40 minutes at 40^0C while being agitated with 25 pieces of still balls. After that, the sample underwent hot rinse and was correctly dried in a drier set at 60^0C.

3) COLOR FASTNESS TO PERSPIRATION

As previously mentioned, the multifiber cloth and the dyed sample were ready for the CF to perspire. The ISO 105 E04 method was followed in the testing procedure, which was conducted using the Perspiration Tester M/C.

For the alkali and acid media, two separate samples with pH values of 8 and 5.5, respectively, were prepared. To ensure adequate soaking, we put one sample in each solution and left it for 30 minutes.

After the solution had been drained, the composite sample was encased between two 7.5 x 6.5 cm glass plates and pressed down firmly, using about 4.5 kg of force. Later it was warmed up at 37 ± 2^{0}C for four hours. Once completed, the sample was cleaned with water and placed in an oven dryer set to 60°C to ensure proper drying.

4) BOD AND COD TEST

Our colored wastewater sample was incubated at 20°C for five days to determine BOD (Biological Oxygen Demand), which was then determined by comparing the oxygen level before and after incubation.

COD testing was performed while wastewater samples were placed in vials with potassium dichromate and sulfuric acid and heated to 150^{o}C for two hours. The findings were then obtained by reading the vials in a spectrophotometer.

III. RESULTS AND DISCUSSION

A. DEGUMMING LOSS (%) OF SILK FABRIC

The fractionation of sericin into various components is carried out by dissolving it in hot water for different time periods, during which the sericin undergoes hydrolytic cleavage. The degumming loss (%) was calculated using equation 4. The weight loss was found to be about 20% which implies the effective sericin removal of the silk fabric; i.e., appropriate degumming releases the amino acid-based peptide bonds of sericin [26], [29].

$$\text{Degumming} = \frac{Weight\ before\ degumming - Weight\ after\ degumming}{Weight\ before\ degumming} \times 100\% \qquad (4)$$

The water droplet was rapidly absorbed in a circular form, demonstrating the increase in absorbency. The sample fabric was observed to have a better hand feel and greater luster.

Additionally, the spectrophotometer was utilized to measure the reflectance value of pre- and post-degum silk fabric samples to validate the sample. According to the data in Table 3, our post-degummed silk fabric has a higher light reflectance than the pre-degummed fabric, indicating that we have achieved a well-degummed fabric [30].

TABLE III
REFLECTANCE VALUE OF PRE AND POST DEGUM SILK FABRIC

Sample	400 (nm)	500 (nm)	600 (nm)	700 (nm)	750 (nm)
Pre-Degum	69.637	82.001	85.425	86.589	86.809
Post-Degum	76.772	87.971	89.455	90.076	90.066
Differences	-7.135	-5.97	-4.03	-3.487	-3.257

B. DYEING OF SILK FABRIC

The dyed samples were observed visually. All samples were found evenly dyed, and visually very less difference could be seen between the same color and same shade (%) of AA and CA dyed samples. The sample images are attached in Figure 1.

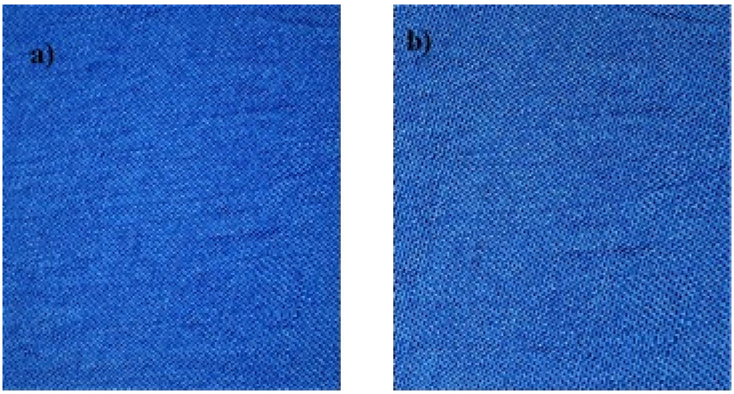

Figure 1. Dyed Silk Fabic; a) AA Sample, b) CA Sample

C. COLOR STRENGTH(K/S) & REFLECTANCE% OF SILK FABRIC

The K/S color strength values of our dyed sample were used to determine the depth of color, where K represents the absorption coefficient and S represents the scattering coefficient. The color strength and reflectance % can be related using equation 3.

The K/S data obtained from the spectrophotometer is shown in Table 4. In this instance, it is evident that the samples dyed with AA had superior color strength (K/S) to those dyed with CA. Despite this, the difference is minimal and insignificant. From the equation 3 we can also identify that for the increasing of K/S the reflectance would be higher [31], [32].

TABLE IV

K/S VALUES OF BLUE COLOR DYED SAMPLES

Name	360	400	500	600	700	750
Standard Sample*	1.343	0.724	0.907	6.41	0.166	0.013
Sample 1	1.208	0.659	0.828	5.768	0.158	0.01
Sample 2	1.21	0.649	0.81	5.783	0.154	0.01
Sample 3	1.26	0.667	0.833	5.872	0.159	0.01

Note. * Dyed with AA

D. FASTNESS TESTS OF SILK FABRIC

1) COLOR FASTNESS TO RUBBING

Table 5 displays the dry and wet rub fastness characteristics of our dyed samples. Similar results were obtained from the AA and CA samples, suggesting that CA had no negative effects on the fabric samples [33].

TABLE V

RUBBING FASTNESS COMPARISON

Condition	Sample Dyed with AA	Sample Dyed with CA
Dry	4-5	4-5
Wet	4-5	4-5

2) COLOR FASTNESS TO WASH

Table 6 demonstrates the wash fastness results of AA and CA samples. Similar results for both samples are displayed here, confirming how well CA works with silk textiles.

TABLE VI

WASH FASTNESS COMPARISON

Multi Fiber Strip	Sample Dyed with AA	Sample Dyed with CA
Acetate	4-5	4-5
Cotton	4	4
Nylon	2	2
Polyester	4-5	4-5
Acrylic	4-5	4-5
Wool	2	2

3) COLOR FASTNESS TO PERSPIRATION

Our dyed samples, which are shown in Table 7, were treated in acid and alkali solutions to examine their properties related to perspiration fastness. The AA sample fastness result on a nylon strip was found to be 3 for both acid and alkali conditions, which is better than the CA sample fastness result of 2-3. Again, the CA sample fastness result on a polyester strip was found to be 4-5 for both acid and alkali conditions, which is better than the AA sample fastness result of 4. The researchers Scheurell et al. [34] claimed that a higher degree of color fading corresponds to a lower fastness result. Here, there is more fading in the nylon portion, but there is less fading in the multifiber fiber strips overall.

TABLE VII

PERSPIRATION FASTNESS COMPARISON

Condition	Multifiber strip	Sample Dyed with AA	Sample Dyed with CA
Acid	Acetate	4- 5	4-5
	Cotton	4	4
	Nylon	3	2-3
	Polyester	4	4-5
	Acrylic	4-5	4-5
	Wool	4-5	4-5
Alkali	Acetate	4-5	4-5
	Cotton	4	4
	Nylon	3	2-3
	Polyester	4	4-5
	Acrylic	4-5	4-5
	Wool	4-5	4-5

4) BOD AND COD TEST

To check the sustainability approach, the BOD and COD test was done, and the data are illustrated in table 3. The CA wastewater sample showed better sustainability compared to AA wastewater sample. The 240 mg/L BOD level indicates that the lower amount of oxygen would require for the microorganism to treat the wastewater. Again, the complexity of acid dyes and other necessary chemicals or auxiliaries may explain the need for increased oxygen in wastewater treatment for chemical processes, yet notable lower COD values of CA compared to AA wastewater were still observed.

TABLE VIII

BOD AND COD COMPARISON BETWEEN SODA ASH AND SODA ASH SUBSTITUTE SAMPLE

Sample	BOD (mg/L)	COD (mg/L)
AA waste waster	2050	24175
CA waste water	240	2375

E. CONCLUSION

The purpose of this study was to investigate the effects of using citrus limon juice instead of organic acetic acid when dyeing silk fabrics. Greater color yield was observed for both AA and CA at the same dose of 2% in the deep shade scenario. Similar effects to those of an AA sample can be obtained by a CA in terms of color uniformity and fastness. The process could be environmentally friendly because of its low BOD and COD values. Finally, while accounting for environmental and chemical costs, synthetic CA can be used commercially to dye textiles with acid without replacing the hazardous AA.

Author Contribution

Mainul Morshed: conceptualization, supervision, project administration. **Zahanara Akter Mim:** investigation, methodology, data curation, validation. **Sabbir Ahmed Akash:** investigation, methodology, data curation, validation. **Nubaia Akter Sanjida:** investigation, methodology, data curation, validation. **Sabbir Ahmed Sad:** writing – review & editing. **Sanjana Afrin:** writing – review & editing. **Lucky Akter:** writing – review & editing.

Conflict of Interest

The authors of the manuscript have no financial or non-financial conflict of interest in the subject matter or materials discussed in this manuscript.

Data Availability Statement

Data supporting the findings of this study will be made available by the corresponding author upon request.

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