Textile & Leather Review

ISSN 2623-6281 | www.tlr-journal.com | 10.31881/TLR

Optimization Rubbing Fastness in Lyocell and Silk Fabric Dyeing with *Tinospora cordifolia* using Box-Behnken Design and *Citrus limon* Extract with Potassium Aluminium Sulfate Mordants

Ramratan Guru, Jyoti Rani

How to cite: Ramratan G, Rani J. Optimization Rubbing Fastness in Lyocell and Silk Fabric Dyeing with *Tinospora cordifolia* using Box-Behnken Design and *Citrus limon* Extract with Potassium Aluminium Sulfate Mordants. Textile & Leather Review. 2023; 6:475-497. https://doi.org/10.31881/TLR.2023.105

How to link: https://doi.org/10.31881/TLR.2023.105

Published: 12 September 2023

Optimization Rubbing Fastness in Lyocell and Silk Fabric Dyeing with *Tinospora cordifolia* using Box-Behnken Design and *Citrus limon* Extract with Potassium Aluminium Sulfate Mordants

Ramratan GURU*, **Jyoti RANI**
School of Design, Mody University of Science and Technology, Lakshmangarh, Rajasthan, India
*ramratan333@gmail.com

Article
https://doi.org/10.31881/TLR.2023.105
Received 24 July 2023; Accepted 7 September 2023; Published 12 September 2023

ABSTRACT
The demand for environmentally friendly natural dyes has risen significantly. This study currently aims to assess the rub-fastness properties of silk and lyocell fabrics that have been dyed using Tinospora cordifolia stem extracts. This dyeing process involves the utilization of both Citrus limon and potassium aluminium sulfate as mordants. To achieve this objective, we have employed the Box-Behnken experimental research design for sample preparation. Our investigation delves into the influence of various processing factors, encompassing dye concentration, time duration, and temperature, on the rubbing fastness attributes of lyocell and silk fabrics treated with Citrus limon extract and alum mordant. The dye, sourced through an aqueous extraction method from Tinospora cordifolia stems, is subsequently employed in dyeing silk and lyocell fabrics, followed by mordanting using the meta-mordanting technique. The assessment of rubbing fastness quality is conducted using the standard test method AATCC 8 for both fabric types. Our experimental findings unveil a notable similarity in rubbing properties between lyocell and silk fabrics dyed with Citrus limon and those treated with potassium aluminium sulfate mordant. Intriguingly, fabric samples treated with Citrus limon mordants exhibit marginally enhanced rubbing (2 to 3 G to 5 G) results when juxtaposed with their counterparts treated with potassium aluminium sulfate mordant. Throughout the experimental course, it becomes evident that both temperature (35 °C, 50 °C, 65 °C) and time (40, 55 and 70 min) exert a significant influence on the wet and dry rubbing fastness characteristics of both silk and lyocell fabrics. Notably, as dyeing time and temperature increase, there is a corresponding rise in fabric dye uptake.

KEYWORDS
rubbing fastness, lyocell, silk, Citrus limon, Tinospora cordifolia, mordant, Box-Behnken method

INTRODUCTION

The geographical diversity of India, along with the diversity of climatic zones and altitudes, has resulted in rich biodiversity, which has given rise to a variety of dye-producing shrubs and perennials from its gifted flora and fauna. Ancient people, in particular, used readily available vegetable minerals and animal-origin materials. In India, it was widely used for dyeing clothes and other materials. The new crop pigments are being sought to meet the growing demand for natural dyes. Natural dyes are substances made from natural resources. Although plants are the primary source of natural dyes, nowadays dyes are usually classified as being made from minerals, plants, microbes, or animals according to their source. Natural dyes are sustainable because they are renewable and biodegradable [1]. Natural dyes used in industrial products have medicinal properties and improve the aesthetic value of the textile. Natural dyes are distinctive in that they are non-carcinogenic and eco-friendly. There is an increasing demand for environmentally friendly, non-toxic dyes, especially for health-sensitive applications such as dyeing fabrics and food colouring [2]. Recently, for these applications, dyes derived from natural resources have emerged as an important alternative to potentially harmful synthetic dyes and pose the potential for effective extraction methods. The most important step in fabric manufacturing is dyeing [3,4]. The primary factor that has a significant impact on consumer purchasing decisions is colour. The garment will attract the customer because of its colour. Most natural dyes can be dissolved in water, and an aqueous method is used to dye textiles [5]. The dye is typically applied in an aqueous solution, and a mordant may be required to improve the stability of the dye on the fibre [6,7]. The colour uniformity, firmness properties, and poor adherence to natural dyes are all improved by the use of mordants in the natural dyeing process. Copper sulfate, chromium, ferrous sulfate, and stannous chloride are the most commonly used mordants. Mordants based on metal salts form a strong bond between the dye and the fabric, improving fastness. The word "mord" is a French word from which the word "mordant" was derived [8]. To enhance the colour-fastness properties of the textile, both natural and chemical mordants are being used in the textile sector. Mordants help in the absorption and fixation of dye in the fibre and also prevent bleeding and fading of dyes. When the fabric is dyed with natural and chemical mordants, different colours are obtained in the same colour. As a result, the final colour of the textile is determined not only by the dye but also by the dye concentration and mordant [9,10]. *Tinospora cordifolia* is an important medicinal plant and is widely used in the medical field. The scientific name of Giloy is *Tinospora cordifolia*. *Tinospora cordifolia* is commonly known as Gurchin in Sanskrit, Gurcha, and *Tinospora cordifolia*. *Tinospora cordifolia* is a climber shrub that grows on various trees. *Tinospora cordifolia*, which grows on the neem tree, is known as neem Giloy [11]. Neem Giloy is regarded as the best Ayurvedic practitioner. The mythological word "Guduchi" is referred to as the heavenly nectar that saved the celestial beings from old age and kept them young forever. It belongs to the family Menispermaceae. The native habitat of *Tinospora cordifolia* is mainly in China, India, and the tropical regions of Australia and Africa. Guduchi (*Tinospora cordifolia*) is highly valued in terms of medicinal use and global trade [12,13]. *Tinospora cordifolia* is a climbing shrub with heart-shaped, greenish-yellow leaves, found at higher elevations. *Tinospora cordifolia*, given its many health benefits, is often referred to as Amrita, which means "the root of immortality." *Tinospora cordifolia* stems are rather succulent, with long, fibrous, fleshy aerial roots extending from the branches [14]. The *Tinospora cordifolia* bark of the plant is watery and greyish-brown in colour. The maximum height of the *Tinospora cordifolia* plant is 300 meters [15]. The various active components derived from the plant are diterpenoids, steroids, alkaloids, glycosides, lactones, and aliphatic compounds. These compounds are isolated from different parts of the whole plant [16].

Mordants play a significant role in the natural dyeing process. To improve the colour fastness property of textiles, chemical and natural mordants are being utilised. Therefore, salts of transition metals are used as a mordant agent in natural dyeing, which is also true of chemical mordants like Fe, Cu, Mu, etc. Mordants contain heavy metal ions such as chromium, iron, cobalt, nickel, copper, or aluminium, the residue of which in dyeing wastewater poses a significant waste disposal problem [17,18]. For this reason, there is a need to replace chemical mordants with natural mordants. Natural mordant contains abundant tannin acids, which are responsible for improving the fastness property of textiles. Alum (potassium aluminium sulfate) and lemon (*Citrus limon*) were used in this study to improve the natural dye fastness of silk and lyocell fabrics. Alum is a chemical compound that is commonly used in the textile industry to improve fastness. *Citrus limon* (L.) Burm. F. It is a Rutaceae family tree with evergreen leaves and yellow edible fruits [19]. Lemon extracts have been widely studied due to their medicinal properties, which include their main constituents such as citric acid, tannin, ascorbic acid, minerals, and flavonoids, which have antioxidant, anti-inflammatory, anti-allergic, antiviral, antiproliferative, antimutagenic, and anticarcinogenic activities [20-22].

Several previous studies have highlighted the environmental drawbacks associated with the use of alum mordants in textile industries. Alum, a commonly used mordant, has been linked to harmful ecological consequences due to its potential to release toxic substances into water systems during dyeing processes. These findings have prompted a growing concern for the environmental impact of textile dyeing and finishing procedures. In response to these concerns, researchers have begun to explore natural alternatives to alum mordants. Numerous studies have investigated the efficacy of various plant-based compounds, such as tannins, myrobalan, and pomegranate peel, as potential mordants. These natural substances have shown promise in enhancing colour fastness while posing significantly fewer environmental risks compared to alum. Furthermore, investigations into the colour fastness enhancement of textiles, particularly silk and lyocell fabrics, have gained attention. Previous research has demonstrated the significance of mordanting processes in achieving improved colour retention and durability of dyed textiles. This has spurred interest in identifying effective and sustainable methods to enhance colour fastness without compromising the quality and aesthetics of the fabrics [23,24].

While previous studies have contributed valuable insights into the challenges posed by alum mordants and the potential of natural alternatives, there remains a need for comprehensive research that systematically evaluates the performance of these alternatives on specific fabric types. This current study aims to build upon these foundational findings by conducting a thorough exploration of natural mordant alternatives for silk and lyocell fabrics, with a focus on both environmental impact and colour fastness enhancement.

In this research, textiles were dyed using herbal medicinal plants, and the stability property of the fabric was improved by using natural and chemical mordants on that fabric. Nowadays, there is an increasing need for natural products due to sustainability. Natural dyes have low fastness properties, so it is very important to mordant the dyed fabric to improve the dyeing fastness. Mordanting is done with both chemical and natural mordants. Because chemical mordants harm the environment. It is for this reason that in this experiment, a comparative study of both chemical and natural mordants was conducted to find the best mordant that can achieve the highest fastness properties while maintaining sustainability. The meta-mordanting has been done on protein (silk) and semi-synthetic (lyocell) fabrics.

The objective of this study is to address the environmental challenges stemming from the detrimental effects of alum mordants. The research was prompted by the growing concerns about the ecological consequences of alum-based processes in textile industries. In response, this investigation aims to explore and evaluate natural mordant alternatives. By focusing on silk and lyocell fabrics, the study seeks to enhance the colour fastness of these textiles while minimizing the ecological footprint. The ultimate goal is to provide sustainable solutions that maintain or improve fabric quality while reducing the environmental impact, contributing to a more environmentally friendly and responsible approach to textile dyeing and finishing processes.

EXPERIMENTAL

Materials and methods

Ready-to-dye lyocell and 100% silk fabrics were procured for that test from Shahi Exports Pvt. Ltd. The stems of the *Tinospora cordifolia* plant were collected at the Mody University of Science and Technology campus in Laxmangarh, Rajasthan. The stems of *Tinospora cordifolia* were cleaned with water to get rid of any impurities, dried in the shade at room temperature, and then powdered in an electric grinder.

The textile was mordanted using both natural and synthetic mordants; potassium aluminium sulfate (chemical) and *Citrus limon* extract (natural) were utilized. Potassium aluminium sulphate and *Citrus limon* were purchased from the local market of Laxmangarh. In that experiment, *citrus lemon* juice and powdered form potassium aluminium sulphate were employed to enhance the fastness property of the dyed textile.

Aqueous extraction

The dried stem powder of *Tinospora cordifolia* was processed using an aqueous extraction method to obtain the dye.

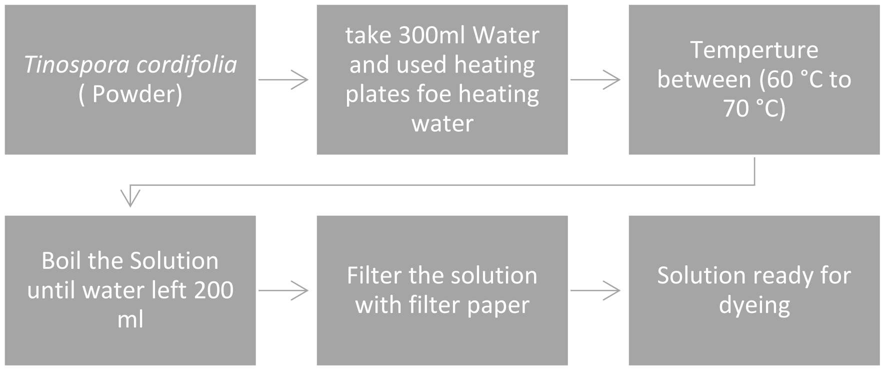


Figure 1. Dye extraction method

This method is also known as the boiling process. For the extraction of the dye from *Tinospora cordifolia* powder, 300 ml of water was poured into a beaker, and the water was heated on a heating plate to a temperature ranging from 60 °C to 70 °C. *Tinospora Cordifolia* powder was added to the water when the temperature reached 60 °C, and then the dye solution was boiled until only 200 ml of the dye solution remained in the beaker. Following this, the boiling solution was filtered twice using filter paper. At this point, the dye solution was fully prepared for dyeing the textile using this method [25].

Table 1. Fabric specification's

Fabric composition	Fabric type	Fabric structure	Fabric GSM, g/m^2	EPI, ends/cm	PPI, picks/cm
Silk	Woven	Plain weave	85	47	28
Lyocell	Woven	Plain weave	95	45	25

Table 2. Factors and their corresponding level of variations

Factor (variables)	Corresponding variables levels		
	Low (-1)	Middle (0)	High (1)
Dye concentration (g/ml)	3	5	7
Time (min)	40	55	70
Temperature ±1 (°C)	35	50	65

Mordanting and dyeing process

In this research, the samples were meta-mordanting together with natural and chemical mordant. 200 ml of dye solution, 1.5 g of potassium aluminium sulfate, and 16 ml of citrus juice were used in this experiment. Fabric samples made from 100% silk and lyocell (sample weights: 3.5 g, respectively) were used for dyeing. A beaker dyeing machine was used to dye silk and lyocell fabric. The temperature increased by 2 °C each minute as the dyeing began at 35 °C, 50 °C, 65 °C (± 1 °C). After dyeing, the samples were washed with soap, followed by hot and cold water, and then were allowed to air dry at room temperature in the shade [25,26].

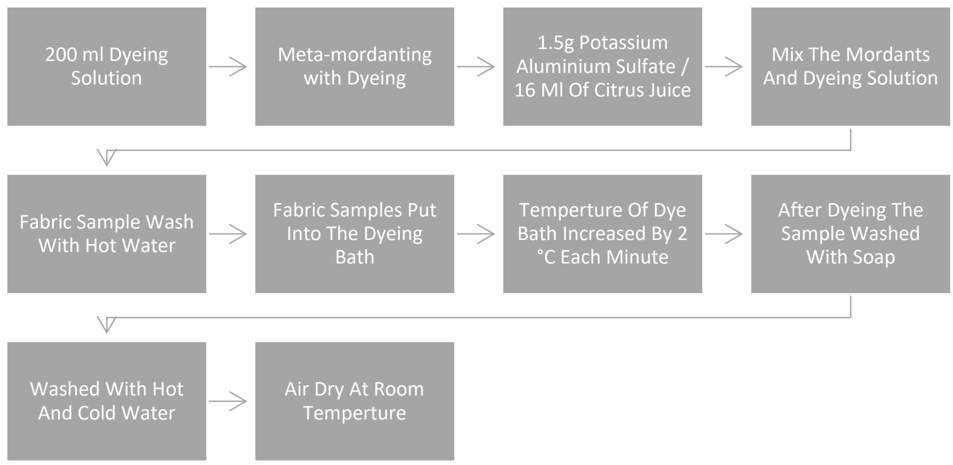


Figure 2. Process of mordanting and dyeing the samples

Methodology

The experimental design employed in this study followed a Box-Behnken framework, aiming to optimize the colour strength of natural dye extracts from *Tinospora cordifolia* for textile dyeing. The coded values of process parameters of dyeing and standard runs of the experimental design were as per Table 3. Three independent variables were considered, each with three levels, and three centre points were incorporated into the design. The rubbing fastness of the dyed textiles was meticulously evaluated following AATCC 8 standards. The assessment utilized a crock meter to measure both dry and wet rub resistance. The ISO 105-A02 grayscale was employed to quantify colour transfer, and a manual crock meter facilitated the assessment process, ensuring precise and consistent results.

Adherence to established textile standards is pivotal within the industry. The AATCC 8 standard test method, sanctioned by the American Association of Textile Chemists and Colorists, assigned grades from 1 to 5 to indicate fabric rubbing fastness. The study aimed for compliance with acceptable quality, necessitating a minimum grade of 4 for dry rubbing and 3 for wet rubbing. To enhance rubbing fastness, innovative techniques involving *Citrus limon* and Potassium aluminium sulfate were applied to silk and lyocell fabrics. The outcomes of these interventions were systematically recorded and are presented in Table 4.

The Dye Uptake % test holds fundamental significance across diverse sectors, including textiles, pharmaceuticals, and materials science. This method serves as a cornerstone for assessing substrate absorbency and material characteristics. In practice, a specimen was immersed in a dye solution to enable diffusion and absorption over a specified timeframe. The quantification of absorbed dye as a percentage followed the Equation 1:

$$\%\,dye\ uptake = \frac{\text{absorbance before dyeing} - \text{absorbance after dyeing}}{\text{absorbance before dyeing}} \times 100 \qquad (1)$$

A colourimeter quantified pre- and post-dye absorption in silk and lyocell fabrics, shedding light on the dye solution's incorporation during the textile dyeing process. This comprehensive approach enriched our understanding of intricate material properties and their behaviour under specific conditions.

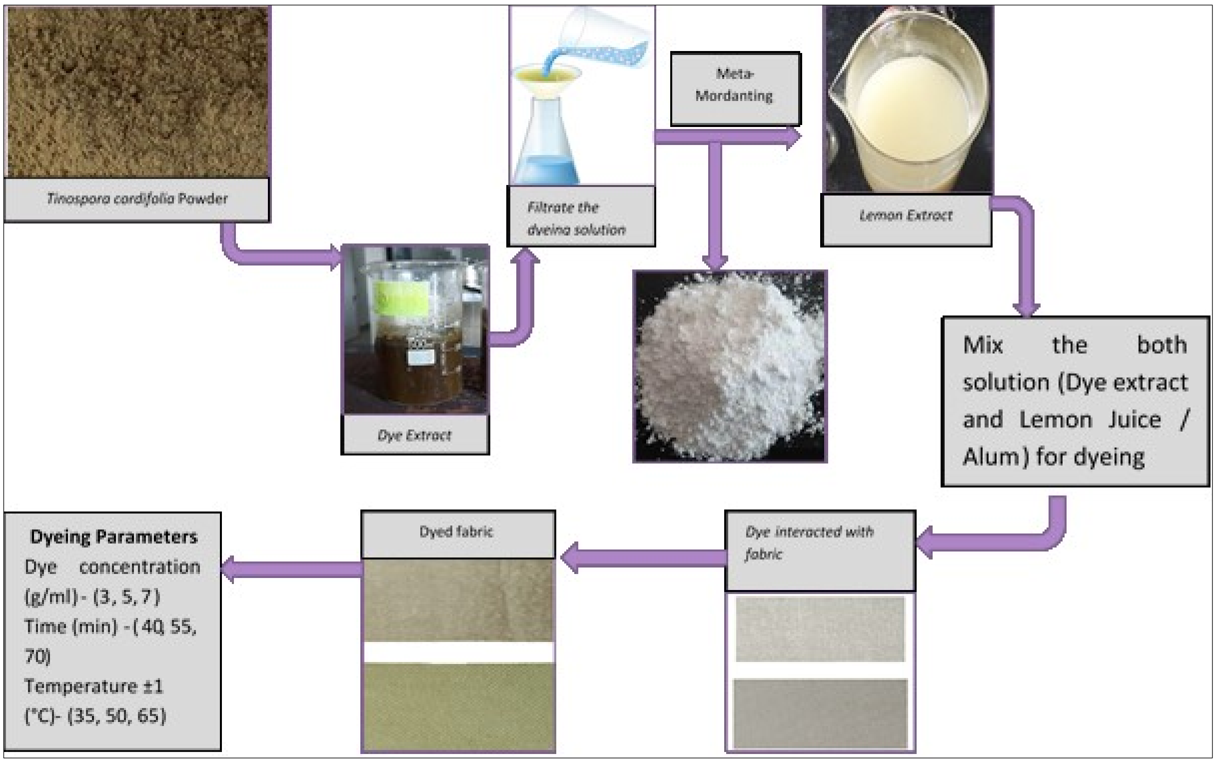


Figure 3. Mechanism between natural dyes and fabric

Table 3. Box-Behnken design for three variables (coded value)

Standards run	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Dye concentration (g/ml)	-1	1	-1	1	-1	1	-1	1	0	0	0	0	0	0	0
Time (min)	-1	-1	1	1	0	0	0	0	-1	1	-1	1	0	0	0
Temperature ±1 (°C)	0	0	0	0	-1	-1	1	1	-1	-1	1	1	0	0	0

RESULTS AND DISCUSSION

Dyeing under optimized conditions

Tinospora cordifolia extract was used to dye the lyocell and silk fabrics according to Table 3. To increase the dye fastness property of silk and lyocell fabric, natural (*Citrus limon*) and chemical (potassium aluminium sulfate) mordants were used.

Table 4. Control factors and corresponding observed responses

Runs	Variables			Responses with potassium aluminium sulfate mordant				Responses with *Citrus limon* mordant			
				Dry rubbing		Wet rubbing		Dry rubbing		Wet rubbing	
	Dye concentratio	Time (min)	Temp. ±1 (°C)	Silk fabric	Lyocell Fabric	Silk fabric	Lyocell fabric	Silk fabric	Lyocell fabric	Silk fabric	Lyocell fabric
1	-1	-1	0	4 G	4.5 G	4 G	4 to 5 G	4 G	5 G	4 to 5 G	4 to 5 G
2	1	-1	0	4 G	4.5 G	4G	4 G	4 G	4 to 5 G	4 to 5 G	4 to 5 G
3	-1	1	0	4 to 5 G	4 G	4 G	5 G	5 G	5 G	4 to 5 G	5 G
4	1	1	0	4 to 5 G	4 to 5 G	4 to 5 G	4 G	4 to 5 G	4 to 5 G	4 G	4 G
5	-1	0	-1	3 G	2 to 3 G	3 G	3 G	3 to 4 G	3 G	2 to 3 G	3 G
6	1	0	-1	3 to 4 G	3 G	3 G	3 to 4 G	3 to 4 G	3 to 4 G	3 G	3 G
7	-1	0	1	4 to 5 G	5 G	4 G	4 to 5 G	5 G	5 G	4 to 5 G	4 G
8	1	0	1	4 to 5 G	4 to 5 G	4 G	4 to 5 G	4 to 5 G	4 to 5 G	5 G	4 to 5 G
9	0	-1	-1	2 to 3 G	3 G	2 to 3 G	3G	3 to 4 G	3 G	3 G	2 to 3 G
10	0	1	-1	3 to 4 G	4 G	3 to 4 G	4 G	4G	4 G	3 to 4 G	3 G
11	0	-1	1	4 G	4 G	4 to 5 G	4 G	4 to 5 G	4 to 5 G	4 to 5 G	4 G
12	0	1	1	4 to 5 G	5 G	5 G	4 to 5 G	5 G	5 G	4 to 5 G	5 G
13	0	0	0	4 to 5 G	5 G	4 to 5 G	4 G	5 G	4 G	4 G	4 to 5 G
14	0	0	0	4 G	5 G	4 to 5 G	4 G	5 G	4 to 5 G	4 to 5 G	4 to 5 G
15	0	0	0	4 to 5 G	4 to 5 G	5 G	3 to 4 G	4to 5 G	5 G	4 G	4 G

**where G denotes grade*

Dry rubbing fastness property of silk and lyocell fabric mordanting with potassium aluminium sulfate mordant

It was found that factors such as dye concentration, time, and temperature have a significant impact on the rubbing fastness of silk and lyocell fabrics. Different levels of time and temperature affect the rubbing fastness properties of samples of silk and lyocell fabric meta-mordanting with potassium aluminium sulphate mordant and dyed with *Tinospora cordifolia*, according to Box-Behnken's experimental design. Figure 4 shows that the highest grade of silk fabric is 4 to 5 Grade, and the lowest grade is 2 to 3. According to the 5, 6, 9, and 10 runs, they get the lowest grade of dry rubbing fastness properties at the temperature of 35 °C in these cases, but every run's dyeing time was different at 45, 55, and 65 min. When the dyeing time was high 65 min, the grade value was 3 to 4 grade to obtain the fastness property. The dry rubbing fastness property increases with temperature. Whereas silk fabric was meta-mordanting with dye at 55 °C temperature, good rubbing fastness was achieved. The 55 °C temperature on runs 1, 2, 3, 4, 13, 14 and 15 (13, 14 and 15 runs are middle value). The excellent dry rub fastness properties of silk fabrics were obtained when the temperature was 65 °C. The highest dyeing time value of the rubbing fastness according to runs 7, 8, 11 and 12 is 5 grade is the highest value and the lowest value is 4 grade.

According to Figure 4, lyocell fabric has also achieved good rubbing fastness properties over the highest time and temperature. In the case of runs no. 5, 6, 9 and 10, they all of them have a temperature of 35 °C. The lowest grade value of rubbing fastness of lyocell fabric obtained 2 to 3 grade at a temperature of 35 °C at a peak time of 65 min was 4 grade. In terms of runs 1, 2, 3, 4 and middle values (13, 14 and 15), they all had a temperature of 55 °C. Good rubbing fastness property of lyocell fabric is achieved when the temperature is 55 °C. In the shortest time, a grade value of 35 min is 4 grade and at the highest time, a grade value of 65 min is obtained 5 grade. Runs 7, 8, 11 and 12 have 65 °C temperature. The excellent rubbing properties of Lyocell fabric were achieved when the temperature was 65 °C. Grade value 4 grade is achieved in the shortest time in 35 min and the grade value 5 grade is achieved in 65 min at the highest time.

The silk fabric achieved excellent dry rubbing fastness properties with potassium aluminium sulfate mordant as well as lyocell fabric. Time and temperature were highly influenced variables of the dry rubbing fastness property of silk and lyocell fabric. As time and temperature increase, the property of dry rubbing fastness also increases accordingly. Dye concentration has a very slight effect on the rubbing fastness property. The dye concentration has no significant effect on the rubbing fastness property of silk and lyocell fabric.

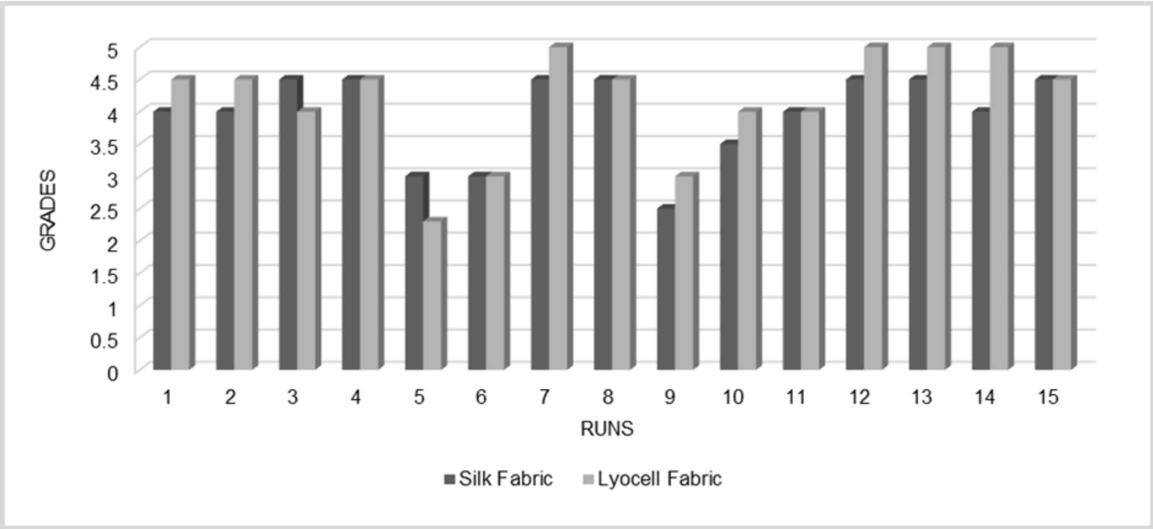


Figure 4. Dry rubbing fastness property of silk and lyocell fabric mordanting with potassium aluminium sulfate mordant

Wet rubbing fastness property of silk and lyocell fabric mordanting with potassium aluminium sulfate mordant

It has been observed that the variables of research dye concentration, time and temperature greatly affected the wet rubbing fastness property of lyocell and silk fabric. According to the experimental design Box-Behnken, various levels of temperature and time influence the wet rubbing fastness property of silk and lyocell fabric was treated with potassium aluminium sulfate mordant with meta-mordanting process and dyeing with *Tinospora cordifolia* stem extract. Figure 5 shows that the highest grade of silk fabric is 5 grade, and the lowest grade is 2 to 3 grade. Runs 5, 6, 9 and 10 have the lowest value of temperature is 35 °C. At the highest time 65 min obtained the 3 to 4 grade G and at the lowest time got the 2 to 3 grade of wet rubbing fastness property of silk fabric. Whereas increasing the time and temperature the wet rubbing fastness is also increased. The 55 °C temperature on runs 1, 2, 3, 4, 13, 14 and 15 (13, 14 and 15 runs are middle value). At the temperature of 55 °C the good wet rubbing fastness property of lyocell and silk fabric. The highest treated time 65 min achieved a 5 grade and at the lowest time 35 min had obtained the 4 grade. The brilliant dry rub fastness properties of silk fabrics were obtained when the temperature was 65 °C. The highest dyeing time value of the rubbing fastness according to runs 7, 8, 11 and 12 is 5 grade and the lowest value is 4 grade.

According to Figure 5, the lyocell fabric also achieved good wet rubbing fastness properties over the peak time and temperature. In the case of Runs no. 5, 6, 9 and 10, they all of them have a temperature of 35 °C. At the peak time of 65 min and the lowest time of 35 min, at a temperature of 35 °C, 4 grade was the highest grade and 3 grade was the lowest grade value of the wet rubbing fastness of lyocell fabric. In terms of runs 1, 2, 3, 4 and middle values (13, 14 and 15), they all had a temperature of 55 °C. Good wet rubbing fastness property of lyocell fabric is achieved when the temperature is 55 °C.

At the least time value, 35 min had archive 4 grade and the peak time a grade value of 65 min is obtained 5 grade. Runs 7, 8, 11 and 12 have 65 °C temperature. The outstanding wet rubbing properties of Lyocell fabric were achieved when the temperature was 65 °C. Grade value 4 grade is achieved in the shortest time in 35 min and the grade value 4 to 5 grade is achieved in 65 min at the highest time.

Lyocell fabric with potassium aluminium sulphate mordant, the silk fabric has also achieved good dry rubbing fastness properties. Time and temperature have a significant effect on the wet rubbing fastness of silk and lyocell fabrics. The characteristic of dry rubbing fastness increases with the passage of time and increasing temperature. The wet rubbing fastness feature is unaffected by the concentration of the dye. The intensity of the colour of the fabric increases with the concentration of the dye. The property of rubbing fastness of silk and lyocell fabrics is not significantly affected by the concentration of dye.

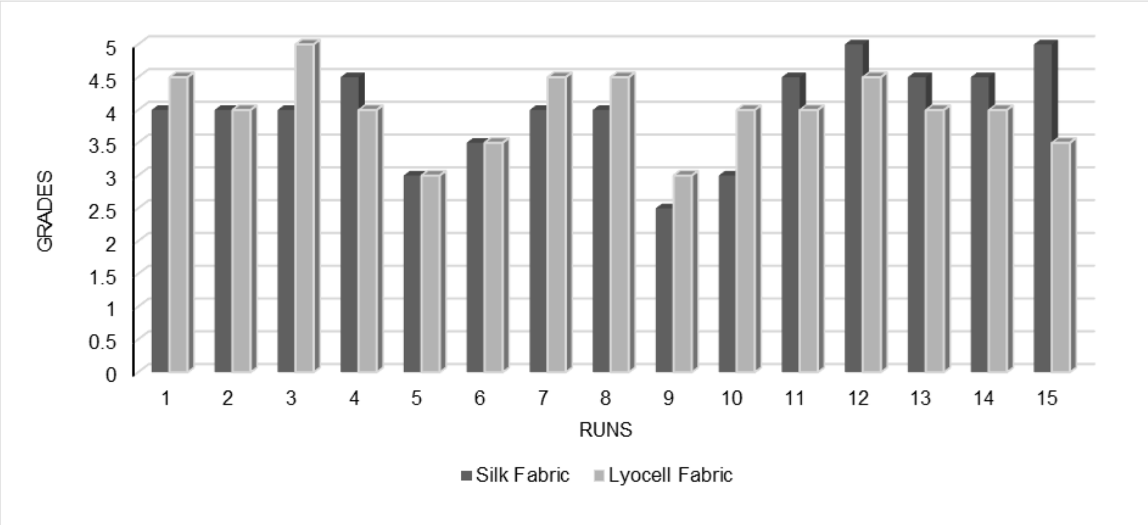


Figure 5. Wet rubbing fastness property of silk and lyocell fabric mordanting with potassium aluminium sulfate mordant

Dry rubbing fastness property of silk and lyocell fabric mordanting with Citrus limon mordant

It turned out that various variables of research dye concentration, time and temperature highly affected the dry rubbing fastness of silk and Lyocell fabrics, both fabrics treated with *Citrus limon* mordant. The Box-Behnken experimental design has been used for the sample design. The different levels of time and temperature greatly affect the dry rubbing fastness of silk and lyocell fabrics. The silk and lyocell fabric were meta-mordanting with the *Citrus limon* mordant and dyed with *Tinospora cordifolia* stem extraction. Figure 6 shows the results of dry rub fastness of silk and Lyocell fabrics according to grayscale. In the case of silk fabric. The minimum temperature in these runs 5, 6, 9 and 10 is 35 °C. In maximum time 4 grade is obtained in 65 min and in fleeting time 35 min 3 to 4 grade of dry rubbing fastness of silk fabric is achieved. The 55 °C temperature on runs 1, 2, 3, 4, 13, 14 and 15

(13, 14 and 15 runs are the middle value of experimental design). The silk fabric achieved good dry rubbing stability at 55 °C temperature. The highest treated time was 65 min receiving a 5 grade and the lowest time was 35 min receiving a 4 grade. Excellent dry rub fastness properties of silk fabrics were achieved when the temperature was 65 °C. According to runs 7, 8, 11 and 12, the highest dyeing time of dry rubbing fastness is 5 grade at 65 min and 4 grade g at the lowest time of 35 min.

In the case of lyocell fabric, the minimum temperature in these runs 5, 6, 9 and 10 is 35 °C. In maximum time 4 grade is obtained in 65 min and in fleeting time 35 min 3 grade of dry rubbing fastness of silk fabric is achieved. The 55 °C temperature on runs 1, 2, 3, 4, 13, 14 and 15 (13, 14 and 15 runs are the middle value of experimental design). The lyocell achieved good wet rubbing stability at 55 °C temperature. The highest treated time was 65 min receiving 5 grade and the lowest time was 35 min receiving 4 to 5 grade. The excellent dry rubbing fastness properties of lyocell fabrics were achieved when the temperature was 65 °C. According to runs 7, 8, 11 and 12, the highest dyeing time of dry rubbing fastness of lyocell fabric is 5 grade at 65 min and 4 to 5 grade at the lowest time of 35 min. The silk fabric with *Citrus limon* mordant lyocell fabric also achieved good dry rubbing fastness properties. The dry rubbing fastness of fabrics silk and lyocell is greatly affected by time and temperature. With the passage of time and increasing temperature, the characteristics of dry rubbing fastness increase. The concentration of the dye does not affect the fastness property of the wet rub. The concentration of the dye has minimal effect on the rubbing stability of the silk and lyocell material.

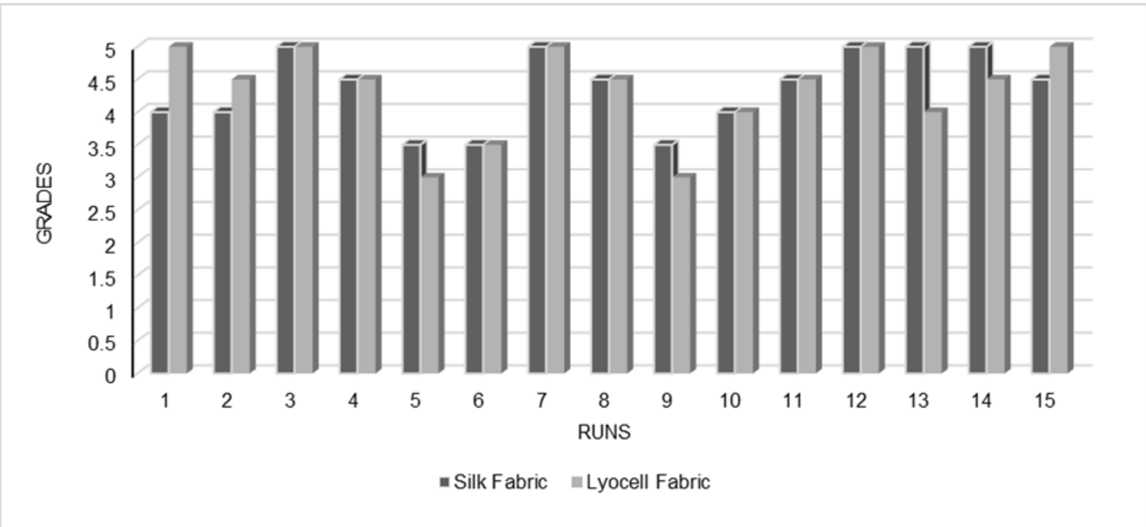


Figure 6. Dry rubbing fastness property of silk and lyocell fabric mordanting with *Citrus limon* mordant

Wet rubbing fastness property of silk and lyocell fabric mordanting with Citrus limon mordant

It was discovered that the various variables of research design dye concentration, time and temperature externally affected the wet rubbing fastness of silk and lyocell fabrics. The silk and lyocell fabric was treated with the *Citrus limon* mordant with a meta-mordanting process. The Box-Behnken experimental design has been used for the sample design. Figure 7 shows the results of wet rub fastness of silk and lyocell fabrics according to grayscale.

In the case of silk fabric. The lowest temperature in these runs 5, 6, 9 and 10 °C is 35 °C. In maximum time, 4 grade is achieved in 65 min and in smaller time 35 min achieved 3 grade of Wet rubbing fastness of silk fabric is obtained. The 55 °C temperature on runs 1, 2, 3, 4, 13, 14 and 15 (13, 14 and 15 runs are the middle value of experimental design). The silk fabric achieved good wet rubbing stability at 55 °C temperature. The highest treated time was 65 min receiving 4 to 5 grade and the lowest time was 35 min receiving 4 to 5 grade. Excellent wet rubbing fastness properties of silk fabrics were achieved when the temperature was 65 °C. According to runs 7, 8, 11 and 12, the highest dyeing time of wet rubbing fastness is 4 to 5 grade at 65 min and 4 to 5 grade g at the lowest time at 35 min.

In the case of lyocell fabric, the minimum temperature in these runs 5, 6, 9 and 10 is 35 °C. In maximum time 3 grade is obtained in 65 min and in fleeting time of 35 min 2 to 3 grade of wet rubbing fastness of silk fabric is achieved. The 55 °C temperature on runs 1, 2, 3, 4,13, 14 and 15 (13, 14 and 15 runs are the middle value of experimental design). The lyocell achieved good wet rubbing stability at 55 °C temperature. The highest treated time was 65 min receiving 5 grade and the lowest time was 35 min receiving 4 to 5 grade. Excellent wet rub fastness properties of lyocell fabrics were achieved when the temperature was 65 °C. According to runs 7, 8, 11 and 12, the highest dyeing time of wet rubbing fastness of lyocell fabric is 5 grade at 65 min and 4 grade at the lowest time at 35 min.

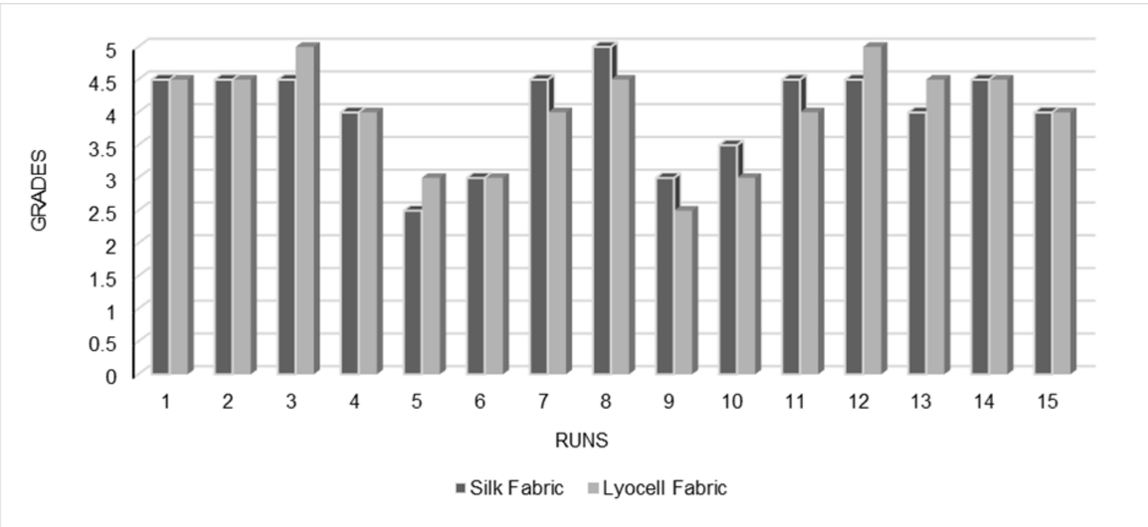


Figure 7. Wet rubbing fastness property of silk and lyocell fabric mordanting with *Citrus limon* mordant

The good wet rubbing fastness was also achieved by silk fabric with *Citrus limon* mordant lyocell fabric. As the temperature and time increased, the wet rubbing fastness of silk and lyocell fabrics enhanced significantly. The wet rubbing fastness of lyocell and silk fabric is enhanced significantly with time and temperature. About the consistency of the wet rubbing fastness of silk and lyocell fabric, the concentration of the dye doesn't matter. Dye concentration has a negligible effect on the rubbing stability of silk and lyocell.

It was observed that both temperature and time had a significant effect on the fastness properties of silk and lyocell fabrics. The fastness properties of both fabrics changed with time as the temperature increased. The dyeing temperature of silk and lyocell fabric affects the friction fastness property because a higher temperature allows the dye molecules to penetrate deeper into the fabric, which in turn provides greater colour fastness and improves fastness to rubbing. Furthermore, higher temperatures can cause more friction and colour transfer between the surfaces of the clothing. In addition, when the dyeing temperature and time were increased, the dye penetration degree increased, resulting in higher colour fastness and improved rubbing fastness. As a result, it indicated that temperature changes had a significant impact on the rubbing fastness properties of silk and lyocell fabrics [26].

Dyeing time also significantly affects the fabric's rub fastness property, as a longer dyeing time allows dye molecules to penetrate the fabric; as time increases, the penetration of dye molecules into the fabric increases, resulting in greater colour fastness. Therefore, both dyeing temperature and time affect the fastness properties of silk and lyocell fabrics.

It has been discovered that increasing the dyeing temperature and time improves the fastness properties of silk and lyocell fabrics significantly.

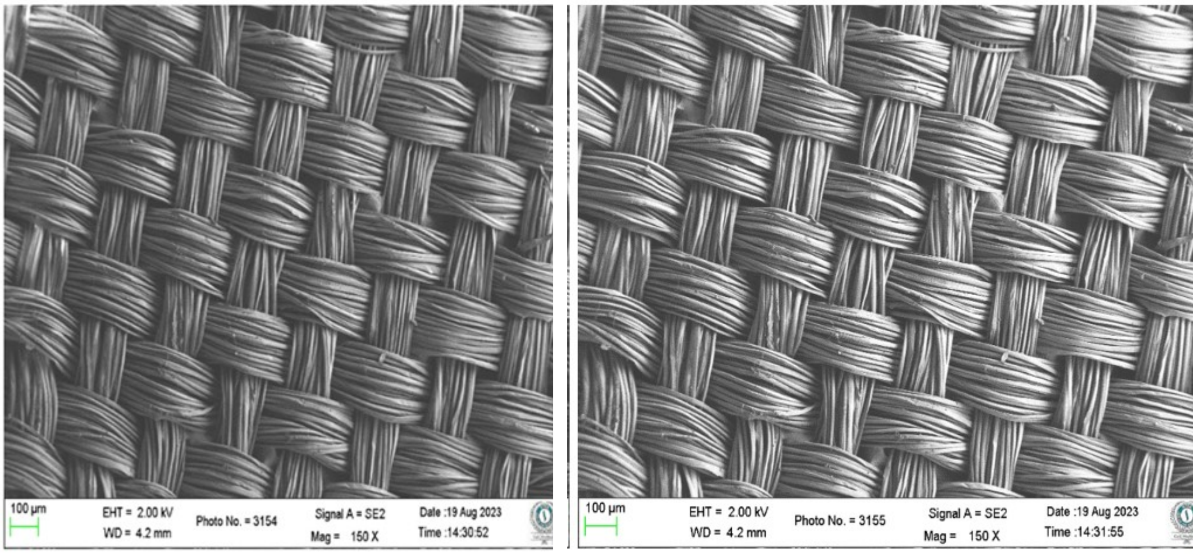
Protein fibres were most easily dyed because of the many reactive functional groups present. Under acidic, neutral, or slightly basic conditions, they can be coloured in a wide range of ways. Protonated amino groups, or NH, are formed when amino groups in protein fibres are exposed to an acidic environment. They can form acids and mordant dyes as well as other ion-containing pigments in this state.

Mordants were used in textile dyeing to improve the fastness of silk and lyocell fabrics by helping fix the dye molecules on the fibres. These mordants help bind the dye molecules more strongly to the fibres and prevent them from being washed out as easily. The combination of dyes, prolonged dyeing, and high temperatures helps improve the rubbing fastness properties of silk and lyocell fabrics. Mordants were chemicals that were used to make the dye molecules stick more strongly to the fabric, creating a more permanent and reliable bond. Chemical and natural mordants were used in this research, which has given better results for rubbing fastness [27].

a) b)

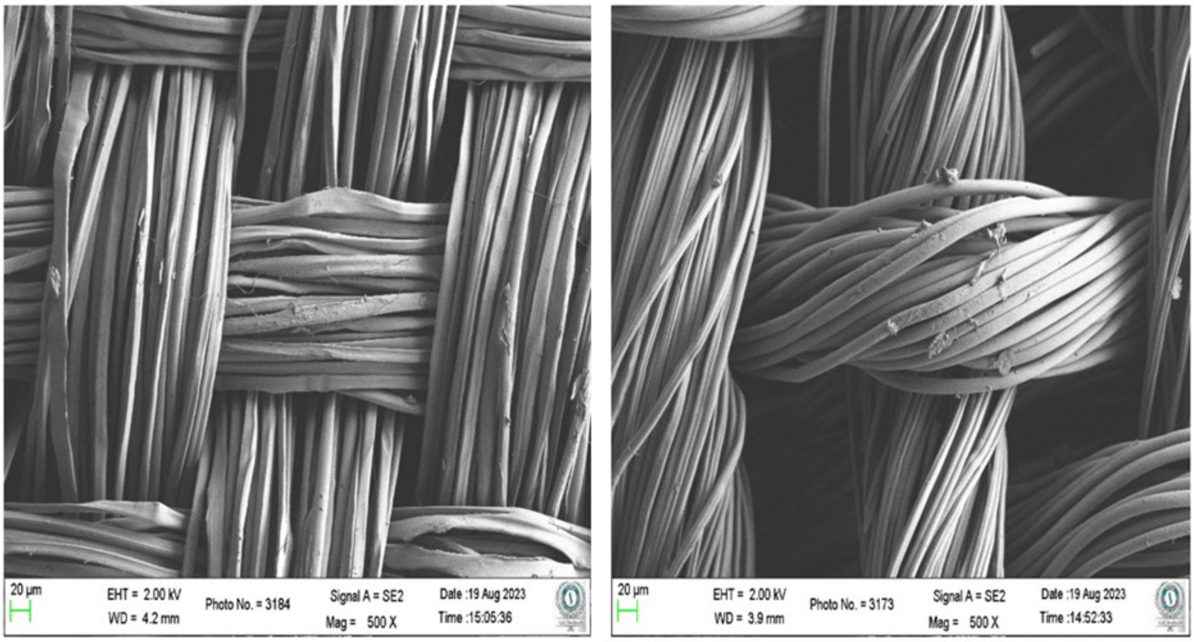
c) d)

Figure 8. a) Powder form of *Tinospora cordifolia*, b) extract dye by utilizing open dye bath method, c) lemon juice for mordanting, d) testing dyed silk fabric rubbing fastness property on crock meter



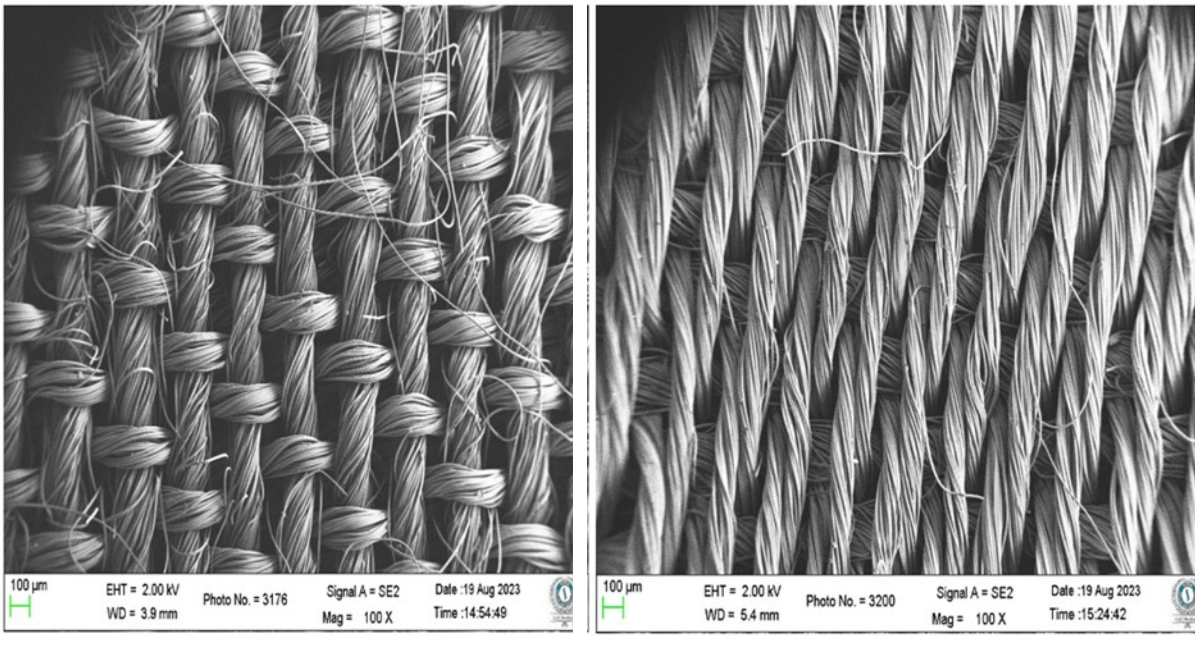
a) b)

Figure 9. a) Silk fabric without mordents, b) Silk fabric with mordents



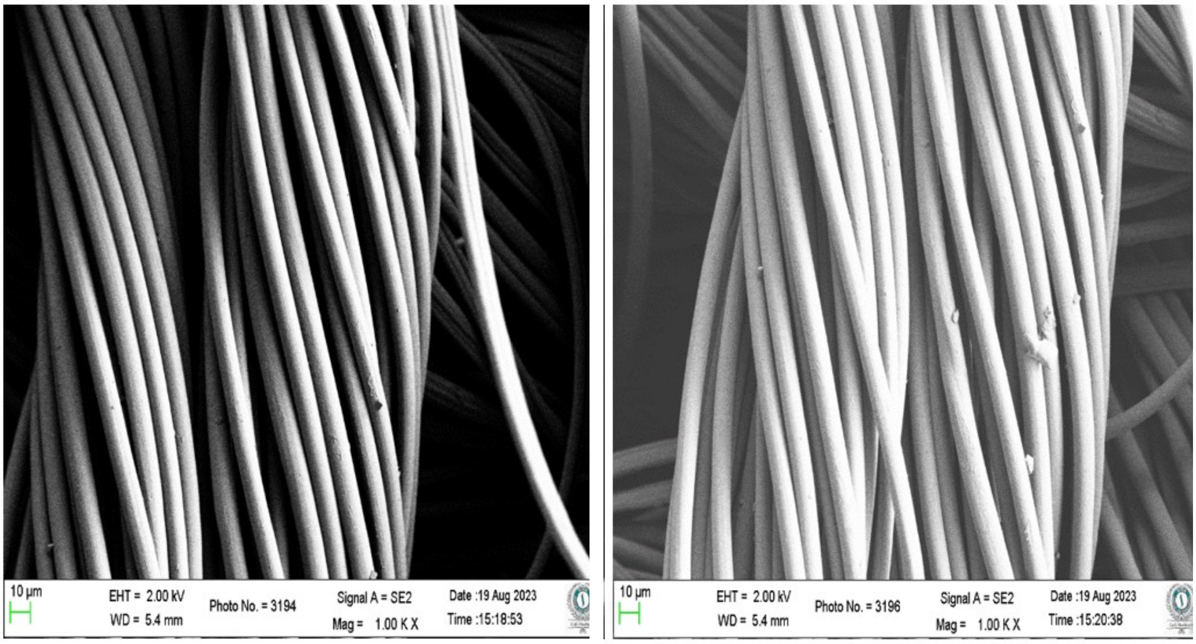
a) b)

Figure 10. a) Silk fabric without mordants, b) Silk fabric with mordants



a) b)

Figure 11. a) Lyocell fabric without mordents, b) Lyocell fabric with mordents



a) b)

Figure 12. a) Lyocell fabric without mordents, b) Lyocell fabric with mordents

Dye uptake

A colourimeter was used to measure how much the dye solution absorbed before and after dying silk and lyocell fabrics. Equation 1 was used to figure out how much dye was taken up [28].

Table 5: Dye uptake of dyed silk and lyocell fabric

S. No.	Fabric	Fabric weight (g)	Dye uptake (mg/g)
1	dyed silk Fabric	3.5	120.19
2	meta-mordanting with C.L. (silk fabric)	3.5	154.27
3	meta-mordanting with Alum (silk fabric)	3.5	149.35
4	dyed lyocell fabric	3.5	115.12
5	meta-mordanting with C.L. (lyocell fabric)	3.5	133.23
6	meta-mordanting with Alum (lyocell fabric)	3.5	145.32

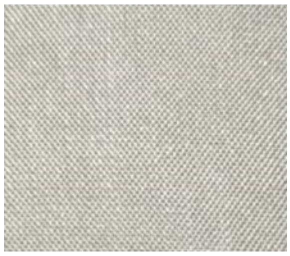
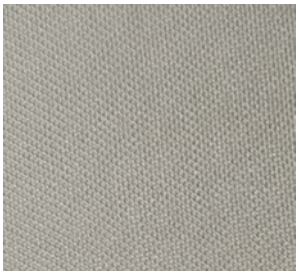
*C.L. denotes *Citrus limon*

For the dyed silk fabric, the recorded dye uptake is 120.19 mg/g, indicating a moderate affinity of silk for the dye employed in the experiment. The application of meta-mordanting with *Citrus Limon* (Silk Fabric) results in a substantial enhancement of dye uptake to 154.27 mg/g. This outcome highlights the effectiveness of *Citrus Limon* as a mordant, significantly improving the fixation of dye within silk fabrics. Similarly, meta-mordanting with Alum (Silk Fabric) yields a favourable dye uptake of 149.35 mg/g, further demonstrating the positive impact of mordants on enhancing dye absorption and retention in silk.

Shifting the focus to lyocell fabric, the dye uptake is slightly lower at 115.12 mg/g compared to dyed silk fabric. However, meta-mordanting with *Citrus Limon* (Lyocell Fabric) leads to a notable increase in dye uptake, recording 133.23 mg/g. This emphasizes the positive role of *Citrus Limon* in facilitating dye fixation even in lyocell fabrics. Additionally, the utilization of meta-mordanting with Alum (Lyocell Fabric) results in a dye uptake of 145.32 mg/g, further confirming the beneficial influence of Alum on enhancing the dyeing process for this fabric type.

These findings underscore the significance of mordanting agents like *Citrus Limon* (lemon) in augmenting dye uptake and retention. The results collectively suggest that both *Citrus Limon* and Alum mordants contribute significantly to enhancing dye absorption and fixation properties in both silk and lyocell fabrics [29-30].

Table 6. Dyed and without dyed fabric samples

Fabric	Alum mordanting fabric	Lemon mordanting fabric	Undyed samples
Silk			
Lyocell			

CONCLUSION

The current study compared the rubbing fastness properties of mordants on silk and lyocell fabrics, such as *Citrus limon* extract and potassium aluminium sulfate. In this research, silk and lyocell fabrics were dyed with giloy stem extract. In this investigation, different variables such as dye concentration, time, and temperature had the most noticeable impact on the rubbing fastness properties of silk and lyocell fabric. The research results showed that higher dyeing temperatures and longer times of dyeing significantly increased the rubbing fastness of mordants on both silk and lyocell fabric. Because dyeing time and temperature were such heavily influenced variables in the dyeing process, they had a

https://doi.org/10.31881/TLR.2023.105

significant impact on rubbing fastness. This research found that both potassium aluminium sulfate and C*itrus limon* improve the excellent rubbing properties of silk and lyocell fabrics. At low temperatures and times, both fabrics achieved an average rubbing fastness grade, but at high temperatures and times, both fabrics achieved excellent wet and dry rubbing fastness properties. *Citrus limon* mordant was discovered to have excellent wet and dry rubbing properties comparable to potassium aluminium sulfate, which is why natural mordants can be used to improve the fastness property of textiles. The results of this research suggest that natural mordant *Citrus limon* is an effective way to improve the fastness properties of textiles. The research on enhancing rubbing fastness through *Tinospora cordifolia*, *Citrus limon* extract, and potassium aluminium sulfate presents a promising trajectory for the future of textile dyeing. Through rigorous experimentation, interdisciplinary collaboration, and a holistic perspective on sustainability, this study's impact could extend beyond colour fastness enhancement, shaping the textile industry's evolution towards a more environmentally conscious and technologically advanced future.

Author Contributions

Conceptualization – Rani J, and Guru R; methodology – Rani J, and Guru R; formal analysis – Rani J, and Guru R; investigation – Rani J, and Guru R; resources – Rani J, and Guru R; writing-original draft preparation – Rani J, and Guru R; writing-review and editing – Rani J, and Guru R; visualization – Rani J, and Guru R; supervision – Rani J, and Guru R. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

Funding

This research received no external funding.

Acknowledgements

Special thanks for providing all testing facilities in the School of Design Department at Mody University of Science and Technology, Lakshmangarh, Rajasthan, India.

REFERENCES

[1] Vankar PS. Natural Dyes for Textiles Sources, Chemistry and Applications. Cambridge: Woodhead Publishing; 2017. Chapter 1A, Newer Natural Dyes for Cotton; p. 1-15. https://doi.org/10.1016/B978-0-08-101274-1.00001-X

[2] Kumar P, Kamle M, Mahato DK, Bora H, Sharma B, Rasane P, Bajpai VK. *Tinospora cordifolia* (Giloy): Phytochemistry, Ethnopharmacology, Clinical Application and Conservation Strategies. Current Pharmaceutical Biotechnology. 2020; 21(12):1165-1175. https://doi.org/10.2174/1389201021666200430114547

[3] Vankar, PS. New Trends in Natural Dyes for Textiles. Cambridge: Woodhead Publishing; 2019. Chapter 1, Newer Natural Dyes for Various Textile; p. 1-24.https://doi.org/10.1016/C2017-0-03994-0

[4] Joshi S, Midha V, Rajendran S. Grapeseed Oil (Vitis vinifera L.) Treatment on Polyester-Based Fabrics to Develop Antibacterial and Physiologically Comfortable Health Care and Hygiene Textiles. Journal of The Institution of Engineers (India): Series E. 2021; 102:339-351. https://doi.org/10.1007/s40034-021-00226-0

[5] Joshi S, Midha V, Rajendran S. Investigation of Durable Bio-polymeric Antimicrobial Finishes to Chemically Modified Textile Fabrics Using Solvent Induction System. Tekstilec. 2021; 64(1):55–69. https://doi.org/10.14502/Tekstilec2021.64.55-69

[6] Yates CR, Bruno EJ, Yates MED. *Tinospora cordifolia*: A review of its immunomodulatory properties. Journal of Dietary Supplements. 2022; 19(2):271-285. https://doi.org/10.1080/19390211.2021.1873214

[7] Sharma H, Singh AK, Borad S, Deshwal GK. Processing stability and debittering of *Tinospora cordifolia* (giloy) juice using ultrasonication for potential application in foods. LWT. 2021; 139:(110584). https://doi.org/10.1016/j.lwt.2020.110584

[8] Krupanidhi S, Peele KA, Venkateswarulu TC, Ayyagari VS, Bobby MN, Babu DJ, Narayana AV, Aishwarya G. Screening of phytochemical compounds of *Tinospora cordifolia* for their inhibitory activity on SARS-CoV-2: an in silico study. Journal of Biomolecular Structure and Dynamics. 2021; 39(15):5799-5803. https://doi.org/10.1080/07391102.2020.1787226

[9] Rani J, Singh S. Antimicrobial Properties of Herbal Dyes of Indian Medicinal Plants.Textile and leather review. 2022; 5:199-222. https://doi.org/10.31881/TLR.2022.16

[10] Midha V, Joshi S, Kumar S. Performance of chemically treated jute geotextile in unpaved roads at different in situ conditions. Journal of The Institution of Engineers (India) Series E. 2017; 98:47-54. https://doi.org/10.1007/s40034-017-0093-0

[11] Sharma P, Pant S, Poonia P, Kumari S, Dave V, Sharma S. Green Synthesis of Colloidal Copper Nanoparticles Capped with *Tinospora cordifolia* and Its Application in Catalytic Degradation in Textile Dye: An Ecologically Sound Approach. Journal of Inorganic and Organometallic Polymers and Materials. 2018; 28:2463-2472. https://link.springer.com/article/10.1007/s10904-018-0933-5

[12] Shree P, Mishra P, Selvaraj C, Singh SK, Chaube R, Garg N, Tripathi YB. Targeting COVID-19 (SARS-CoV-2) main protease through active phytochemicals of ayurvedic medicinal plants – Withania somnifera (Ashwagandha), *Tinospora cordifolia* (Giloy) and Ocimum sanctum (Tulsi) – a molecular docking study. Journal of Biomolecular Structure and Dynamics. 2022; 40(1):190-203. https://doi.org/10.1080/07391102.2020.1810778

[13] Das SC, Uddin MA. Extraction and Characterization of Antimicrobial Surgical Suture from the Bast of Tinospora Cordifolia. Journal of Natural Fibers. 2022; 19(15):11235-11245, https://doi.org/10.1080/15440478.2021.2022560

[14] Mohapatra D, Pratap R, Pandey V, Dubey PK, Agrawal AK, Parmar AS, Sahu AN. *Tinospora cordifolia* Leaves Derived Carbon dots for Cancer Cell Bioimaging, Free radical Scavenging, and Fe3+ Sensing Applications. Journal of Fluorescence. 2022; 32(1):275–292. https://doi.org/10.1007/s10895-021-02846-6

[15] Chowdhury P. In silico investigation of phytoconstituents from Indian medicinal herb '*Tinospora cordifolia* (giloy)' against SARS-CoV-2 (COVID-19) by molecular dynamics approach. Journal of Biomolecular Structure and Dynamics. 2021; 39(17):6792-6809. https://doi.org/10.1080/07391102.2020.1803968

[16] Murugesan S, Kottekad S, Crasta I, Sreevathsan S, Usharani D, Perumal MK, Mudliar SN. Targeting COVID-19 (SARS-CoV-2) main protease through active phytocompounds of ayurvedic medicinal plants – Emblica officinalis (Amla), Phyllanthus niruri Linn. (Bhumi Amla) and *Tinospora cordifolia* (Giloy) – A molecular docking and simulation study. Computers in Biology and Medicine. 2021; 136(104683):1-13. https://doi.org/10.1016/j.compbiomed.2021.104683

[17] İşmal ÖE, Yildirim L. Metal mordants and biomordants. In: ul-Islam S, Butola BS, editors. The Impact and Prospects of Green Chemistry for Textile Technology. Cambridge: Woodhead Publishing; 2021. p. 57-82. https://doi.org/10.1016/B978-0-08-102491-1.00003-4

[18] Gong K, Rather, LJ, Zhou Q, Wang W, Li Q. Natural dyeing of merino wool fibers with Cinnamomum camphora leaves extract with mordants of biological origin: a greener approach of textile coloration. The Journal of The Textile Institute. 2020; 111(7):1038-1046. https://doi.org/10.1080/00405000.2019.1698228

[19] Klimek-Szczykutowicz M, Szopa A, Ekiert H. *Citrus limon* (Lemon) Phenomenon—A Review of the Chemistry, Pharmacological Properties, Applications in the Modern Pharmaceutical, Food, and Cosmetics Industries, and Biotechnological Studies. Plants. 2020; 9(1):119. https://doi.org/10.3390/plants9010119

[20] Micó-Vicent B, Viqueira V, Ramos M, Luzi F, Dominici F, Torre L, Jiménez A, Puglia D, Garrigós MC. Effect of Lemon Waste Natural Dye and Essential Oil Loaded into Laminar Nanoclays on Thermomechanical and Color Properties of Polyester Based Bionanocomposites. Polymers. 2020; 12(7):1451. https://doi.org/10.3390/polym12071451

[21] Yusuf M, editor. Handbook of Renewable Materials for Coloration and Finishing. United States: Wiley; 2022. 67 p. https://doi.org/10.1002/9781119407850

[22] Srinivasan S, Sadasivam SK. Biodegradation of textile azo dyes by textile effluent non-adapted and adapted Aeromonas hydrophila. Environmental Research. 2021; 194:110643. https://doi.org/10.1016/j.envres.2020.110643

[23] Yusuf M. Synthetic Dyes: A Threat to the Environment and Water Ecosystem. In: Shabbir M, editor. Textiles and Clothing. United States: Wiley; 2019. p. 11-26. https://doi.org/10.1002/9781119526599.ch2

[24] Yusuf M, Shabbir M, Mohammad F. Natural Colorants: Historical, Processing and Sustainable Prospects. Natural Products and Bioprospecting. 2017; 7:123–145. https://doi.org/10.1007/s13659-017-0119-9

[25] Park YH, Kang IS, Bae SH. Changes in the Dyeing Property of Tencel Blended Fabrics as Susceptible Functional Fiber through Chitosan Finishing. Journal of the Korean Society of Clothing and Textiles. 2007; 31(12):1672- 1681. https://doi.org/10.5850/JKSCT.2007.31.12.1672

[26] Jaglan J, Jaglan S, Jaglan P, Jaglan A. Inductively coupled plasma optical emission spectroscopy based toxicological risk assessment of cadmium and lead in Tinospora cordifolia. Pharmacological Research - Modern Chinese Medicine. 2023; 7:100246. https://doi.org/10.1016/j.prmcm.2023.100246

[27] Yusuf M, Islam S-ul, Khan MA, Mohammad F. Investigations of the colourimetric and fastness properties of wool dyed with colorants extracted from Indian madder using reflectance spectroscopy. Optik. 2016; 127(15):6087-6093. https://doi.org/10.1016/j.ijleo.2016.04.084

[28] Singh M, Vajpayee M, Ledwani L. Eco-friendly surface modification of natural fibres to improve dye uptake using natural dyes and application of natural dyes in fabric finishing: A review. Materialstoday Proceeding. 2021; 43(5):2868-2871. https://doi.org/10.1016/j.matpr.2021.01.078

[29] Tsatsaroni E, Liakopoulou-Kyriakides M. Effect of enzymatic treatment on the dyeing of cotton and wool fibres with natural dyes. Dyes and Pigments. 1995; 29(3):203-209. https://doi.org/10.1016/0143-7208(95)00044-G

[30] Dulo B, Somer TD, Phan K, Roosen M, Githaiga J, Raes K, Meester SD. Evaluating the potential of natural dyes from nutshell wastes: Sustainable colouration and functional finishing of wool fabric. Sustainable Materials and Technologies. 2022; 34:e00518. https://doi.org/10.1016/j.susmat.2022.e00518