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STUDY ON NATURAL DYEING OF SILK YARNS USING FERMENTED TURMERIC RHIZOMES WITH THEOBROMA CACAO FRAGRANCE

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SUMMARY

This study explores the use of a natural colouring method on silk strands. Before and after dyeing, the chemical composition changes in silk yarn are inspected using a Fourier-Transform Infrared Spectroscopy (FTIR) study. The outcomes showed that colourants and fragrances could be introduced to silk yarns without compromising their basic assembly. Physical examination of the yarns revealed that the natural dyeing method consumed no effect on breaking strength, only moderately increased elongation and colourfastness for staining, and barely altered other characteristics like nep count and unevenness. The significance of the naturally dyed fabrics increased as a result of the study's significant findings regarding the decrease of thin spots and the long-lasting retention of the Theobroma cacao scent.

Key words: *natural dye, silk, natural dyeing, biodegradable, aromatic, sustainable, breaking strength, FTIR, colour fastness.*

INTRODUCTION

Yarns made of natural fibers—from shrub, animal, or mineral sources—are polished by weaving after being converted into filaments. Because of their special qualities and sustainable origins, these fibers—basically cellulose-based compounds in a lignin and hemicellulose matrix of micro-fibrils—require to attract increased attention [1]. The proportion of fibre length to diameter is commonly used to evaluate the quality of natural yarns; longer and finer fibres are normally associated with higher quality. Thermal comfort, tactile softness, reduced electrostatic charge, hygro-scopicity and superior insulation, are only a few advantages of natural yarns [2]. Natural fibre yarns are increasingly being used in a variety of applications, from materials to sophisticated composite materials, as a result of these qualities as well as their sustainable origin [8].

Natural dyeing is an environmentally friendly procedure in which colours are extracted from natural sources, often plant matter, by heating them in water [6]. Fibres or textiles are then dyed with the resultant dye solution; the length of soaking time determines how intense the colour is. Compared to synthetic dyes, which are made from petrochemical sources using risky chemical procedures and frequently contain carcinogenic ingredients, this method is typically thought to be safer for human use and more environmentally friendly. Sericin (17-25% weight and Fibrin (75-83% by weight) are the dualistic main protein components of raw silk. A layer of sericin contains two fibroin filaments. Amazing qualities including breathability, flexibility, absorbency, heat regulation, rapid drying, and sheen are all displayed by silk yarns. To bring out the full shine and beauty of silk, however, de-gumming must be done before dyeing to eliminate the sericin, which gives the fabric strength but dulls its look [3][13].

Natural colourants derived from natural materials in our atmosphere, such as insects, minerals like iron ore, plants, or plant components, are known as natural or organic dyes [4]. The bulk of natural dyes is made from organic colours that come from plants, such as leaves, wood, berries, roots, and barks as well as other biological sources like fungi. In addition to being biodegradable and biocompatible colourants, natural dyes give the material being used a lot of useful qualities. Natural dyes' overall colour fastness to light and washing is lower than that of well-chosen and applied synthetic dyes, and they typically fall short of buyer expectations. Natural dyes are environmentally safe, skin-friendly, aesthetically beautiful, non-toxic, biodegradable, and allergic.

By heating natural materials plant matter in water, pigments may be extracted, making natural dyeing an eco-friendly technique [6]. Fibres or textiles are then dyed with the resultant dye solution; the length of soaking time determines how intense the colour is [12]. Compared to synthetic dyes, which are made from petrochemical sources using risky chemical procedures and frequently contain carcinogenic ingredients, this method is typically thought to be safer for human use and more environmentally friendly.

Compared to synthetically dyed silk yarns, naturally dyed silk yarn cost is significantly expensive due to the complex and labor-intensive process of extracting natural dyes and dyeing methods, which often requires more time and focused techniques, making it a costly production method than using synthetic dyes [10]. Sourcing and preparing natural dyes from naturally available sources like plants, and insects can be knowingly expensive than readily availing synthetic dyes. So, it is concluded that naturally dyed silk yarns are to be priced considerably higher than synthetic dyed silk yarns. Given that natural dyes are more environmentally friendly and organic than synthetic dyes, such as acid dyes, the cost of natural dyes can be somewhat lower than that of synthetic dyes. Scalability, when compared to synthetically dyed silk yarn, naturally dyed silk yarns generally experience a very slight amount of increase in volume due to the dyeing process itself, which can sometimes cause a minor fiber swelling, this change is usually negligible and not readily noticeable to the naked eye, also the factor is that natural dyes often produce softer, more muted colors compared to the synthetic dyes. Durability, when compared to synthetic dyes silk yarns, naturally dyed silk yarns sometimes be considered more durable than synthetic dyed silk yarns in terms of their inherent fiber quality, the colourfastness of natural dyes is lower, whereas the color may fade faster over time comparison to artificial dyes. However, natural dyes can be more environmentally friendly & have certain beneficial properties like anti-bacterial effects depending on the sources of dyes used. Some natural dyes may have additional properties like anti-microbial or UV protection depending on the sources used. Natural dyes are becoming more and more popular as people's interest in organic and other natural clothing options has grown, as has their knowledge of chemical sensitivities.

The substrate is submerged in a medium, often water that contains auxiliary chemicals and dyestuffs as part of the dyeing process. Adsorbed onto the surface of the substrate, dyes slowly diffuse into the fibre, leveling out and fixing there. Natural dyeing provides several benefits, including using renewable resources, producing vivid and unique colours without leaving stains, preserving traditional craft expertise, and having a low environmental impact. Because of these qualities, natural dyes are entirely eco-friendly and biodegradable, which is consistent with sustainable textile production methods.

Complex mixtures made entirely of natural ingredients, such as fruits, trees, herbs, leaves, seeds, roots, and animal extracts, are known as natural fragrance oils. Perfumes isolate and recombine individual components from a variety of natural aromatic materials to generate complex smells. These oils are entirely plant-based and are derived without the use of chemicals. These oils are useful in aromatherapy for purposes including stress relaxation, anxiety reduction, and muscle pain relief because they frequently have wellness and therapeutic qualities. When these oils are added to products, their appealing scents improve their sensory appeal. Fragrance finishing is a cutting-edge technique in the textile industry that infuses textile substrates with enticing fragrances, adding substantial consumer value. This organic method of making fragrances fits nicely with purchasers' increasing desire for ecological and health-conscious goods.

Compared to natural dyeing, synthetic dyeing generally results in significantly more water wastage, where some studies indicate that synthetic dyeing uses 5 to 10 times more water per unit of yarn or fabric [5], due to the additional chemical processes involved in creating synthetic dyes and their fixations. Synthetic dyeing often requires larger volumes of water for the dyeing process itself, as well as for rising and cleaning due to the complex chemical reactions involved. But natural dyes, derived from plants and other natural sources, often require less water as the dyeing process is generally simpler and less chemically intensive. While both synthetic and natural dyeing can produce wastewater, synthetic dyes are considered more polluting due to the presence of harmful chemicals that can be difficult to remove. Along with these beneficial characteristics, dyes are associated with environmental hazards, particularly those concerning the water ecosystem [7].
Amount of Energy consumption in the process of natural dyeing compared to synthetic dyeing process, generally, synthetic dyeing consumes significantly less energy compared to natural dyeing, as the process of extracting and preparing natural dyes requires more processing time, making it energy intensive, however, the energy consumption of synthetic dyes can be high due to the chemical production process involved in creating the dyes themselves, which are often derived from petroleum. Typically synthetic dyes use less energy during the dyeing process compared to natural dyeing due to dye fixation being faster and less pre-treatment, whereas, natural dyeing requires more energy due to the time takes in sourcing, preparing, and mordanting and longer dyeing times [14]. While the dyeing process itself might use less energy with synthetic dyes, the environmental impact can be higher due to the energy-intensive production of synthetic dyes, which involves harmful chemicals.

MATERIALS AND METHODS

Preparation of Dyes

(b) (c) (a)

Figure 1. (a) Turmeric Rhizomes (Crucuma Longa), (b) Lemon (Citrus Limon), (c) Alum (Potassium Aluminium)

As shown in Figure 1, three essential ingredients were utilized in the natural colouring process: *Curcuma longa* (turmeric rhizomes), potassium aluminum sulphate (alum), and citrus limon (lemon). Fermented turmeric rhizomes, which are created using the under-part of the stem of the *Curcuma longa* plant, were used to extract the primary dye. Fresh lemon juice & ratio potash alum was used as auxiliary agents during the process of fermentation & drawing out operations to improve the colour output and fixing qualities. To maximize the extraction and colouring processes, natural and mineral ingredients were chosen.

In a sealed earthenware pot, alum, lime water, and turmeric rhizomes are united in a special fermentation process to create the dye. To create a crimson or pink dye, this pot is at that point buried underground for a long time, usually six months. Longer fermentation times—up to 24 months—produce darker maroon or red colours that resemble the pigment of kumkum. The mixture goes through intricate biochemical changes during this underground phase that are impacted by temperature, moisture, and soil pressure. The fermented material that results, which has certain characteristics of termite mound material, is then removed, sieved to remove impurities, and used for dyeing. This age-old technique produces a rich, long-lasting colourant by utilizing microbial and natural geochemical processes.

Alum and lime water are used as mordants in the dyeing process, while Theobroma cacao, or chocolate, is used to improve smell. The silk threads are put through mechanical and physical tests to determine baseline qualities before dyeing. Un-dyed and dyed silk yarns, both samples are analyzed using FTIR after the dyeing process. By identifying and contrasting the peak values and chemical bond compositions of the treated and untreated samples, this analytical method sheds light on the molecular alterations brought about by the natural dyeing procedure.

Silk Yarn Dyeing Using Natural Dye and Fragrance Oil

A sophisticated natural dye bath consisting of wood ash solution, sodium carbonate (Na2CO3), Theobroma cacao essential oil, and fermented turmeric rhizome residue (0.15% w/v) was used to dye degummed silk threads. The yarn was saturated first, and then it was submerged into a 30L dye bath that was first boiled up to 70º Celsius & then kept at 80 degrees. Three stages of dyeing took place: a 10-minute first stage using turmeric dye and chocolate aroma, a 15-minute time after adding Na2CO3 (which served as an electrolyte), and a concluding 10-minute period involving a solution of wood ash. Yarns were maintained completely submerged and stirred occasionally during the 35-minute procedure to guarantee even dye absorption and produce the required kumkum-like or light red colouration.

The silk strands were dyed and then washed in four steps. A "Softener-Triple S," a proprietary mixture of lemon water and solidified coconut oil, was used for the last wash after the first two were completed with ordinary tap water and the third with diluted lemon water. After being diluted in thirty liters of water, this softener provided the yarns with a smooth, glossy finish. To provide predictable results, great care was taken to maintain constant temperatures & immersion times during the dyeing processing and washing operations.

Treated silk strands were permitted to air dry in the shadow after the washing process. Ten distinct tests were used to evaluate the olfactory persistence over a long length of time. This analysis showed that the aroma of Theobroma cacao had penetrated the yarn structure deeply and had lasted for several months. This extended preservation of smell points to a possible synergistic interaction between the cacao essential oil, mordants, and natural dye ingredients, which may be made possible by the special molecular structure of silk fibroin. Additional quantitative examination of fragrance persistence and its relationship to dye fixation may shed light on the processes underlying aroma retention in textiles that are naturally dyed.

Figure 2. Natural Dyed and Fragrance-finished Silk Yarn

FTIR Analysis

Using (FTIR), the molecular interactions & the chemical character of uncoloured and naturally coloured silk strands infused with scent were examined. To guarantee continuous contact with ATR crystal, the yarn samples are washed with the use of ethanol, allowed to air dry, & then cut into 5mm segments. In order, to increase the signal-to-noise ratio, 64 scans were co-added to each sample of spectra obtained in, the mid-infrared region (4000-400 cm-1), and at the firmness of 4cm-1. Raw data was transformed into absorbance spectra by applying a Fourier transform technique to the resulting interferograms.

Physical Testing of Yarns

A Uster Tester 5-S800 was used to assess the evenness and defects of the yarn, and a Testometric M350-5CT tensile testing apparatus was used to assess the strength characteristics. The samples were acclimatized for 24 hours at 27±2oC & 65±2% relative humidity before testing. Neps (+200%), Thick areas (+50%), Thin places (-50%), and Unevenness (%) were measured on 1000 m of the testing yarn at the speed of 400 m/min in compliance with ASTM D1425/D1425M. In compliance with ASTM D2256/D2256M, elongation (%), yarn tenacity (RKM), and breaking strength (gf) was measured using a testo-metric machine equipped, with the crosshead speed of 500 mm/minute & gauge length of 500 mm.

RESULT AND DISCUSSION

Analysis of FTIR of Both Un-dyed and Dyed Silk Yarns

(FTIR) was used to investigate the molecular structures and chemical contents of silk strands that were uncoloured and those that had been treated with natural dyes and smells. This methodical technique created emission spectra or high-resolution infrared absorption throughout a large spectrum range simultaneously, proposing improved performance associated with dispersive spectrometers, which measure intensity over a restricted number of wavelength bands one after the other. An interpretable spectrum is produced from the raw interferogram data using a calculated procedure termed the "Fourier transform." Both raw (untreated), organic dyed, and aromatic-infused fragrance yarns of silk strands were subjected to FTIR analysis to interpret the changes in chemical bonding and arrangement caused by the dyeing and the fragrance infusion progressions. Our spectroscopic investigation targeted to, recognize specific absorption bands linked with, the structure of the yarn of silk & a few changes caused by the treatments to provide information on the interactions of molecules amongst the fibres of silk and applied natural compounds of chemicals.

Table 1. Un-dyed Silk Yarns FTIR Examination

S.No.	Link/Bond	Bond category	Specific bond category	Absorption of peak (cm^{-1})	Approximate value of the undyed sample	Appearance
1	C=O	Aldehyde/ketone	Cyclic 5-membered	1750	1743.65	
2	N–O	Nitro compounds	Aliphatic	1540	1527.62	Stronger
3	C–O	Alcohols	Tertiary	1150-1200	1165.00	Average
4	C–H	Vinyl	Monosubstitutedalkenes	990	972.12	Strong
5	C–H	Aromatic	Meta-disub.benzene	750-800	794.67	Strong
6	C–H	Vinyl	Cis-disubtituted alkenes	670-700	686.66	Strong
7	C–X	Chloroalkanes	Any	540-760	648.08	Feeble - average
8	C–X	bromoalkanes	Any	500-600	555.50	Average - strong
9	C–X	Iodoalkanes	Any	500	493.78	Average - strong
10	C–X	Iodoalkanes	Any	500	439.77	Average - strong

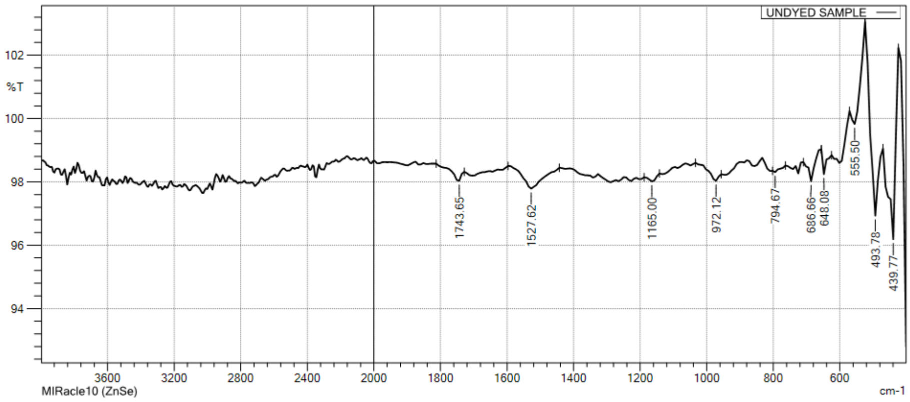

Figure 3. Analysis of FTIR - Silk Yarns (Un-dyed)

Table 2. Dyed silk yarns FTIR examination

S.No.	Link/Bond	Bond category	Specific bond category	Absorption of peak (cm^{-1})	Approximate value of dyed sample	Appearance
1	C–H	Alkynes	Any	3300	3271.27	Average
2	C=C	With benzene ring	Dienes	1625	1620.21	Strong
3	N–O	Nitro compounds	Aromatic	1520	1519.91	Lower if conjugated
4	C–O	Ethers	Aromatic	1220-1260	1226.73	
5	C–O	Alcohols	Tertiary	1150-1200	1172.72	Average
6	C–O	Alcohols	Primary	1040-1060	1041.56	Strong, broad
7	C–H	Vinyl	Cis-disubstituted alkenes	670-700	686.66	Strong
8	C–X	Chloroalkanes	Any	540-760	601.79	Weak - average
9	C–X	bromoalkanes	Any	500-600	555.50	Average - strong
10	C–X	bromoalkanes	Any	500-600	509.21	Average - strong
11	C–X	Iodoalkanes	Any	500	478.35	Average - strong
12	C–X	Iodoalkanes	Any	500	447.49	Average - strong

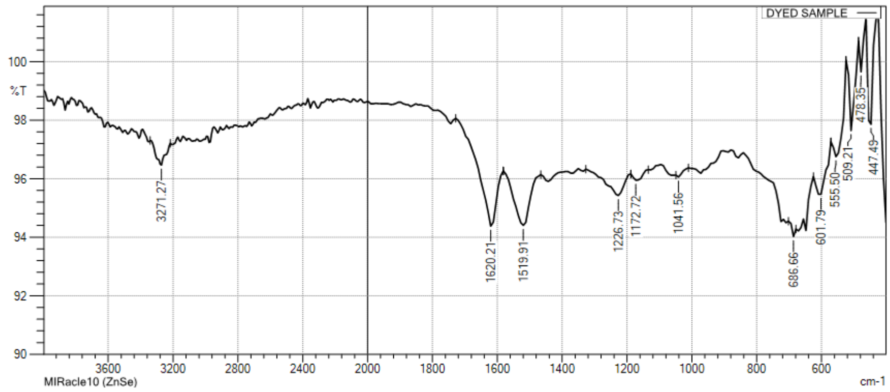

Figure 4. Analysis of FTIR - silk yarns (Dyed)

The FTIR examination of about undyed and dyed silk strands, as shown in Table 1,2 and Figure 2, 3, 4 showed notable changes in chemical composition as a result of the natural dyeing technique that included fermented turmeric rhizomes, lemon water, alum, and fragrance oil of Theobroma cacao. Consistent with earlier research on silk fibroin structure, undyed silk strands had distinctive peaks linked to the protein structure of silk fibroin, such asN-O bonds (1527.62 cm^{-1}), (1743.65 cm^{-1}) C=O stretches and the different links of C-H. A more complex spectrum was displayed by the coloured sample, suggesting that the dye and aroma chemicals were well incorporated [15].

New peaks, likely generated by turmeric chromophores, occurred at 3271.27 cm^{-1} (alkynes) & 1620.21 cm^{-1} (aromatic C=C). The preservation of points in the fingerprint region (500-1500 cm^{-1}) in both samples recommends that the basic structure of silk yarn be conserved after natural dyeing, which is critical for fibre integrity.

The effective binding of dye molecules to silk fibres, conceivably via vander Waals interactions and hydrogen bonding is indicated by the dyed sample's increased spectrum complexity, especially in the 3000–3500 cm^{-1} region [9].

These findings show that silk yarn's basic protein structure can be preserved while its chemical makeup can be successfully altered by natural dyeing techniques. Important information on the chemical interactions of natural dyes, aroma compounds, and silk fibroin can be gained from the detected changes in FTIR spectra.

Physical Testing of Yarn

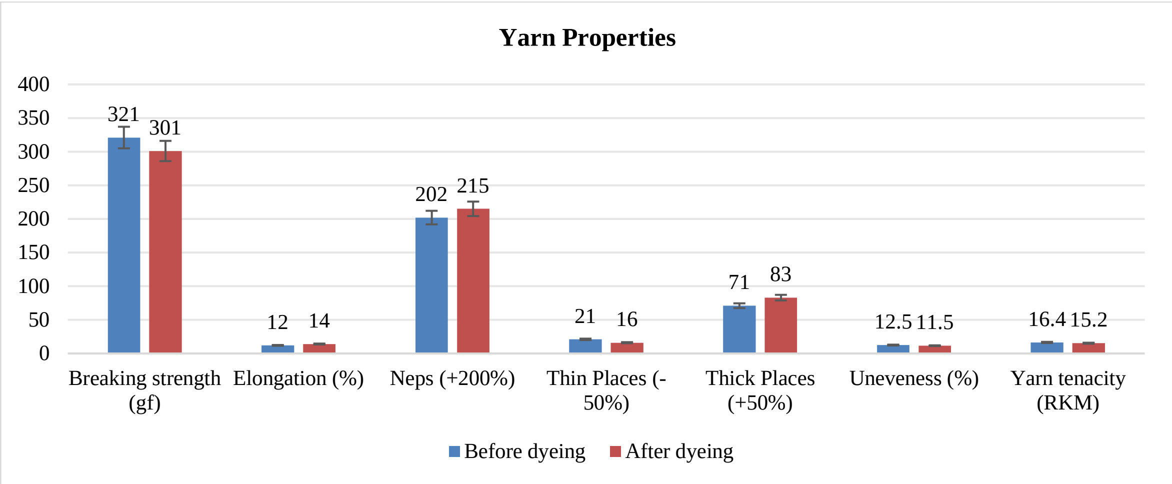

Figure 5. Dyed and Un-dyed Silk Yarn Properties

To determine, about effect of treatment on silk yarn quality, the physical characteristics of the silk yarns were compared before & after the organic dyeing procedure. The comparison of several yarn properties, of silk yarns for both un-dyed and dyed is shown in Figure 5.

The breaking strength of dyed silk yarns is somewhat lower than that of the undyed silk yarns. This decrease implies that the yarn's inherent strength was mostly preserved and that the natural dyeing procedure had little effect on its tensile characteristics [16]. The dyed yarns showed a small considerable increase in elongation. The interaction between the dye molecules and the silk yarn may be the cause of this minor increase in extensibility, which could result in better fibre mobility.

When compared to un-dyed yarn, the yarn's unevenness exhibited a slight increase. In dyed yarns, a noticeable decrease in thin areas was noted. This notable improvement raises the possibility that the yarn structure was evened out by the dyeing process, possibly as a result of treatment-induced fibre swelling [11].

In dyed yarns, there were a few more dense spots. Uneven fibre swelling during the dyeing process or localized dye particle buildup could be the cause of this little increase [17]. In dyed yarns, a little rise in neps was seen. This rise may be the result of some fibre tangling during the dyeing and washing procedures, even if it appears to be a significant percentage change. Yarn tenacity (RKM), is decreased somewhat for natural dyed silk yarns. The slight drop implies that the natural dyeing procedure had a certain effect on the tested yarn's tenacity.

These findings suggest that the general physical characteristics of the silk strands were very slightly impacted by the natural dyeing method that used fermented turmeric rhizomes, lemon water, and alum in addition to Theobroma cacao scent. The decrease of thin areas & the minor rise in dense areas and in neps were the, most notable changes. The yarn's mechanical qualities were not seriously harmed by the dyeing procedure, as seen by the maintenance of breaking strength &the little enhancement in elongation.

The aids of using eco-friendly and natural dyes, and smells likely outweigh the little rises in unevenness, dense patches, and in neps, which are visible but below adequate limits for naturally dyed silk yarns. These fluctuations in the yarn quality could be qualified to the physical properties of dyeing and washing techniques on the arrangement of fibres inside the yarn, as well as interactions between natural dye chemicals, mordant, & silk yarn structure.

Colourfastness

A) Staining in Wet state – 10 times in crock meter
B) Change in colour in Wet state – 10 times in crock meter

The samples are tested in colourfastness grey scale to define the dye bleed from the fabric when it is tested in a crock meter.

Table 3. Colourfastness of Naturally Dyed and Fragrance-finished Silk Yarn

Testing No.	Staining in wet state	Change in colour in wet state
1	5	5
2	5	5
3	5	5
4	4	5
5	4	4
6	4	4
7	4	4
8	4	4
9	3	3
10	3	3

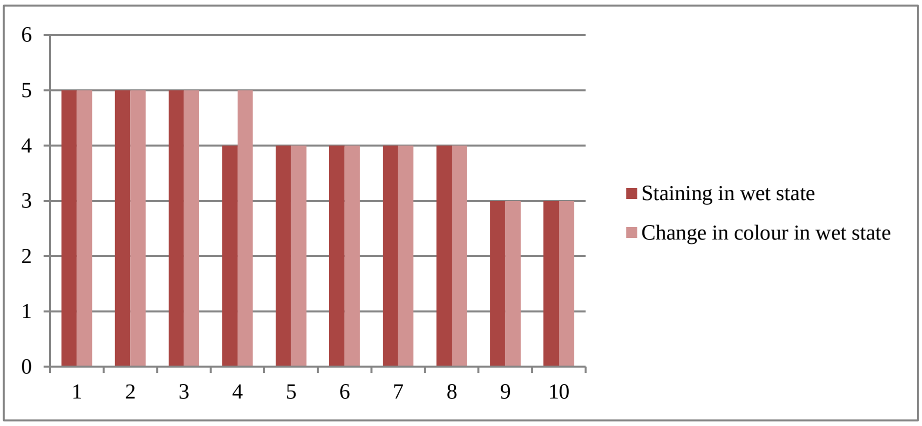

Figure 6. Colourfastness of Naturally Dyed and Fragrance-finished Silk Yarn

Table 3 and Figure 6 demonstrate the colourfastness of natural coloured yarns with particular dyes and fragrances. It is evident that the colourfastness of dyed yarns stains when wet and changes colour while wet, and that this colourfastness steadily diminishes following the rub test.

Abrasion Resistant: Silk yarn that is naturally dyed has a moderate level of abrasion resistance. When silk is turned into fabric, it may deteriorate if it comes into contact with another surface, like another fabric. But compared to other kinds of silk, spun silk fabrics are more resistant to abrasion. Because other fibres can withstand the heavy wear that silk cannot. It is only moderately resistant to abrasion, though. Since this study focuses on yarns, the natural dyed silk has abrasion resistance when turned into fabric, therefore when it is rubbed, it will cause abrasion on another surface.

Wash Durability: Although natural-dyed silk yarn can withstand a lot of washing, it should be handled carefully as it becomes weak when wet.

CONCLUSION

A promising environmentally acceptable substitute for synthetic dyes for silk yarns is the natural dyeing method that uses fermented turmeric rhizomes, lemon water, and alum in addition to the aroma of Theobroma cacao. The FTIR study showed that dye and aroma chemicals were successfully incorporated while preserving the basic silk thread. The physical examination of the yarns revealed that the natural dyeing method only minimally altered other qualities including unevenness and nep count, while having little effect on breaking strength and a little improving elongation. To find out which dyes are more effective when rubbed when wet, colour fastness analysis is also carried out. The decrease in weak spots and maintenance of yarn quality overall indicate that these natural dyeing techniques are practical ways to create environmentally friendly, aesthetically beautiful, and fragrant silk yarns. The value proposition of the ecologically dyed textiles is further enhanced by the aroma of Theobroma cacao's long-lasting preservation. Although there were a few slight compromises in the evenness of the yarn, these were probably exceeded by the advantages the natural dyeing process had for the environment and the special qualities it offered.

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