

Analysis of dyeing properties of different lyocell fabrics natural dyed and mordanted at various concentrations

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Abstract

The use of natural dye has increased globally in order to prevent the environment from pollution. Thus, the natural dye extract from *Isatis tinctoria* leaves which produces a vibrant indigo shade and alum was chosen as the natural mordant. A novel attempt was made by choosing *Nelumbo nucifera* and *Citrus × sinensis* the two different lyocell fabrics for the study of colourfastness effects at various concentration of dye and mordant. Here the dye concentrations were taken in the ratio of 1:20 and 1:40, mordant at 10% and 20%, the values of colour efficiency (K/S) value and colour fastness (Light, Washing & Rubbing) was analysed. From the observation made on the colour depth on the natural dyed fabrics *Citrus × sinensis* has shown better result.

Keywords: Natural dye, *Isatis tinctoria*, Lyocell fabrics, *Nelumbo nucifera* and *Citrus × sinensis*

1. Introduction

1.1 Natural Dye

Natural dyes are among the oldest known methods for dyeing fabrics, and their usage for textile decorating has a long history in India. From the 15th to the 19th centuries, Gujarati and Deccan textiles with black prints and resist dyes captivated Europeans and their homes. The very early dyes were found by accident using berries and fruits, but with the experimentation and progressive scientific improvement the process of dying fabrics with natural colours has resulted in a highly sophisticated art [1-9]. Synthetic dyes were created in the West during the 19th century, but it was subsequently revealed that some of them had harmful effects on human skin and lungs. Environmentalists thus began looking for alternatives to synthetic colors, and in recent years. [2]

No matter how great the fabric's composition, color is a key component that draws attention to it. As a result, the industrial manufacturing and use of synthetic dyestuffs for textile dyeing has grown into a massive industry. Bright colors and a wide variety of colorfastness have been made

possible by synthetic dyestuffs. However, the ecology is now gravely concerned about their harmful nature. [1]

1.2 *Isatis tinctoria* L.

The herbaceous biennial or short-lived perennial species *Isatis tinctoria* L. (woad) is greyish, upright, simple below, and branching above. It is essentially hairless to hairy.[3]

By optimizing the indigo extraction process from woad cauline leaves, this study aims to increase the value of woad in a variety of application domains, including dyeing. Because it enables the quick synthesis of indigo utilizing a traditional installation and a non-toxic, ecologically friendly solvent—water—the historical infusion technique was highlighted.[4]

The plant *Isatis tinctoria* (fig 1) is used as a dye. The plant's leaves are used for dying, and the color was caused by peptide glycosides. [5]



Figure 01 – *Isatis tinctoria* L. plant

1.3 Lyocell fabrics

Because of its greater strength, solid crystalline shape, total non-toxicity, biodegradability, environmentally benign, non-polluting manufacturing technique, and close closeness to cotton, lyocell fabric has gained a lot of popularity among researchers in recent years. The impact of both natural and synthetic dyes on lyocell fabric is demonstrated in this study. [6]

Cellulosic fibers known as lyocell fibers are created when cellulose is regenerated into fiber form from a solution in N-methylmorpholine-N-oxide (NMMO)⁹. Like other cellulose, lyocell fibers absorb moisture and decompose naturally. They are almost as strong as polyester and have a greater dry strength than other cellulose. When wet, they also maintain 85% of their strength.

Lyocell fibers may fibrillate under specific circumstances, allowing for the creation of textiles with intriguing aesthetics. There are also non-fibrillating variants. Clothing textiles are the primary use for lyocell fibers. [7]

For millennia, people all throughout the world have utilized lotus fiber, a special kind of substance. Because of its exceptional sustainability, durability, and adaptability, this natural fiber is perfect for a variety of uses, including high-end fashion accessories, home goods, and apparel. Lotus fiber is regarded as a premium material and is frequently more costly than other textiles because of the labor-intensive nature of the production process. However, it is a viable substitute for synthetic fibers in the fashion sector due to its natural and ecological qualities. [8]

With GOTS (Global Organic Textile Standard) certification, the plant is the most environmentally friendly material in the world since it is grown in dirty water without the use of chemicals during manufacture. It is biodegradable and organic. It helps to improve rural and tribal workers' employment. Lotus fabrics exhibit the wonderful blend of silk and linen, which has the qualities of being wrinkle-resistant and breathable. Additionally, it is biodegradable, eco-friendly, organic, handcrafted, UV resistant, has high moisture absorption, air permeability, softness, comfort, and the capacity to absorb free radicals produced by the body while inhibiting the creation of fat. Lotus fiber produces negative oxygen ions, which are beneficial for the body's immune system development. [9]

Orange Fiber produces a biodegradable fabric that can be turned in the form of compost without any environmental impact. The fabric looks and feels like silk: soft to the touch with a shiny appearance. The biodegradable yarn can be spun with any type of existing yarn. It can also be blended with other materials and be opaque or shiny according to the needs. As Orange Fiber is a cellulose fiber, it can be used in much the same way as its man-made counterparts. This fiber can be dyed, colored and printed on to create various textile products.[10]

2. Material and Methods

Materials used here two lyocell woven fabrics and natural dye *Isatis tinctoria* L. leaves was collected, dried and made into a fine powder. Alum was taken as the mordant. The below tables represent the methods of this study.

2.1 Extraction of *Isatis tinctoria* L. leaves

The leaves of woad plant which produces blue shade dye are collected from the field of Kallaru, Sirumugai, Coimbatore. The collected leaves were set to dry in shade for 10 days before crushing. After the leaves are dried and the colour turned slightly to plea green, they were grind into a fine powder before taking it to the dye bath.



Figure 2: *Isatis tinctoria* L. leaves

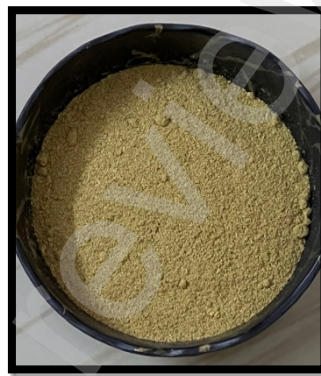


Figure 4: *Isatis tinctoria* L Powder Selection

of fabrics

The fabrics selected for dyeing process are *Nelumbo nucifera* and *Citrus × sinensis* lyocell fabrics. *Nelumbo nucifera* specifies 96 EPI, 76 PPI and 150 GSM, where *Citrus × sinensis* 96 EPI, 76 PPI and 180 GSM.



Figure 5: *Nelumbo nucifera* fabric

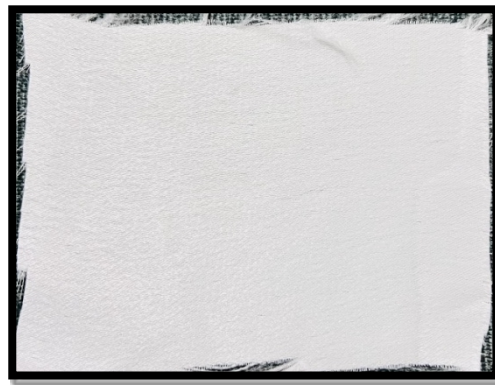


Figure 6: *Citrus × sinensis* fabric

2.3 Pre-Treatment Process

All of these contaminants are eliminated during pre-treatment, which also makes the fabric brighter and more absorbent, making following treatments simpler. 3% NaOH (V/W) and 3% Na₂CO₃ (V/W) were used for the scouring procedure, and the treatment was run for 60 minutes at 100°C. For 60 minutes, the bleaching procedure was conducted using H₂O₂ at 100°C.









2.4 Pre- mordanting

The selected fabrics *Nelumbo nucifera* and *Citrus × sinensis* were pre-mordanted with 10% and 20% (V/W) Alum. At 95° C, the treatment procedure lasted for 40 minutes. To ensure that the dyeing process was completed successfully, the samples were next dried and cold washed to get rid of any non-bonded surface particles.

2.6 Process of Dyeing

The selected two fabrics were treated under two different concentrations of dye and mordant. The concentration of dye was at 20% and 40%, whereas alum concentration was at 10% and 20%. The dyeing process was carried out at 80°C for 60 minutes in the traditional dye bath. After which the fabric was taken out and washed thoroughly in cold water. The below table 1 represent the effect in the shades of the fabric at difference concentrations of dye and mordant.

Table 1: Effects of two concentrations of dye and mordant on lyocell fabrics

Lyocell fabrics	Dye Concentration	Mordant at 10% Concentration	Mordant at 20% Concentration
<i>Nelumbo nucifera</i>	1:20		
<i>Nelumbo nucifera</i>	1:40		
<i>Citrus × sinensis</i>	1:20		
<i>Citrus × sinensis</i>	1:40		

3. Characteristics of the study

3.1 Colour Strength

In this study, the Kubelka-Munk equation (equation (1)) was used to calculate the color strength (K/S) of the samples.

$$K/s=(1-R^2)/2R-----(1)$$

The materials' reflectance spectra and the CIE L*, a*, b*, C*, and ho color coordinates were measured using a Texflash spectrophotometer (Data color Co., Switzerland). With the exception of

the specular component, all color parameters were examined using the CIE 1964 10o standard observer under illuminant D65.

3.2 Colorfastness to wash, light and rubbing

Before performing this color fastness test, the samples were washed at 40°C using 5 g/L soap for 30 minutes. In accordance with ISO 105-C06: 1994 (E), ISO 105-B02:1994 (E), and IS766L1988, the samples' wash, light, and rubbing fastness were assessed. For light fastness, the samples were exposed for a whole day to an air-cooled xenon arc lamp. The degree of staining and color change was used to gauge wash fastness. In a similar vein, rubbing fastness controls how much color can be wiped off of colored textiles and onto other surfaces.

4. Results and Discussion

The *Nelumbo nucifera* and *Citrus × sinensis* samples were dyed using *Isatis tinctoria* L. and Alum as mordant which was taken under two concentrations ie., 20% & 40% and 2% & 4% respectively. The results are shown in below Tables.

4.1 Spectrophotometer analysis

The below table 2 represents the colorimetric values of the selected fabrics which are dyed at different percentages of dye and mordant concentrations.

Table 2: Colorimetric values of *Nelumbo nucifera* and *Citrus × sinensis* samples at various dye and mordant concentrations

Samp le code	Samples	Dye %	Mordant %	L*	RF L%	K/s value
S1	<i>Nelumbo nucifera</i>	20	10	33.46	16.01	40.29
S2	<i>Nelumbo nucifera</i>	20	20	40.48	15.56	42.31
S3	<i>Nelumbo nucifera</i>	40	10	29.75	5.75	81.56
S4	<i>Nelumbo nucifera</i>	40	20	28.94	5.38	83.34
S5	<i>Citrus × sinensis</i>	20	10	41.41	10.5	66.51
S6	<i>Citrus × sinensis</i>	20	20	38.49	9.32	68.42
S7	<i>Citrus × sinensis</i>	40	10	31.53	4.38	128.32
S8	<i>Citrus × sinensis</i>	40	20	28.43	4.11	129.29

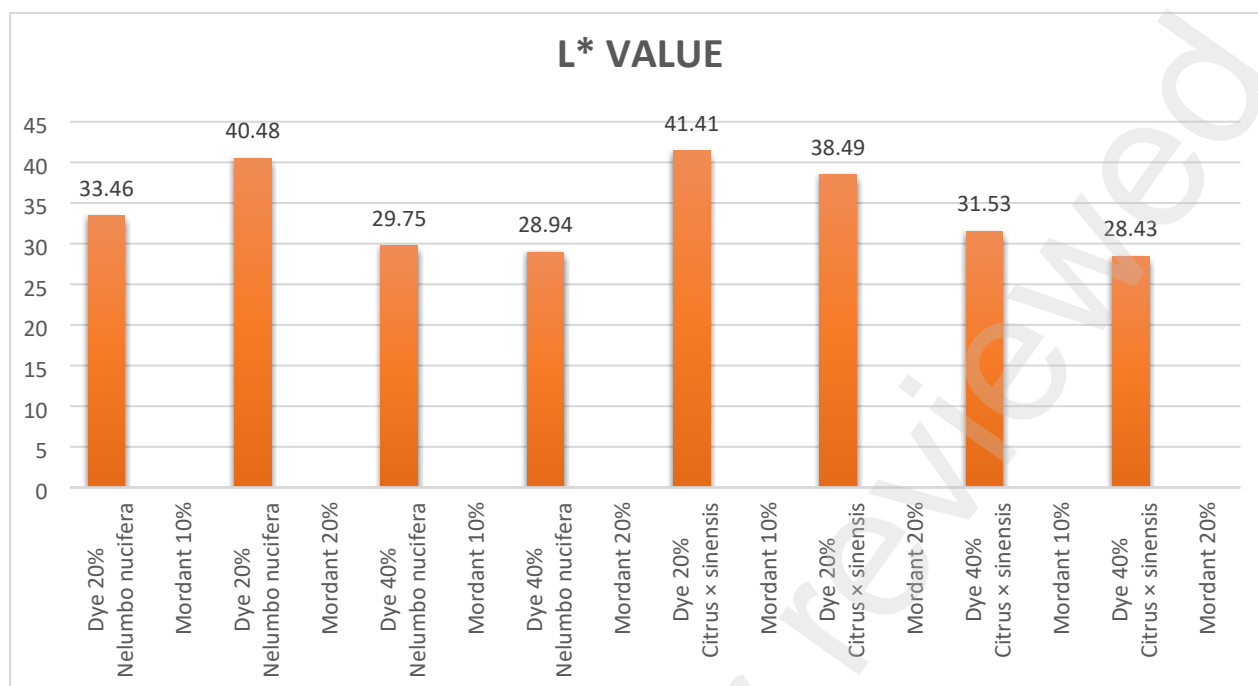


Figure 7: L* Value of *Nelumbo nucifera* & *Citrus x sinensis*

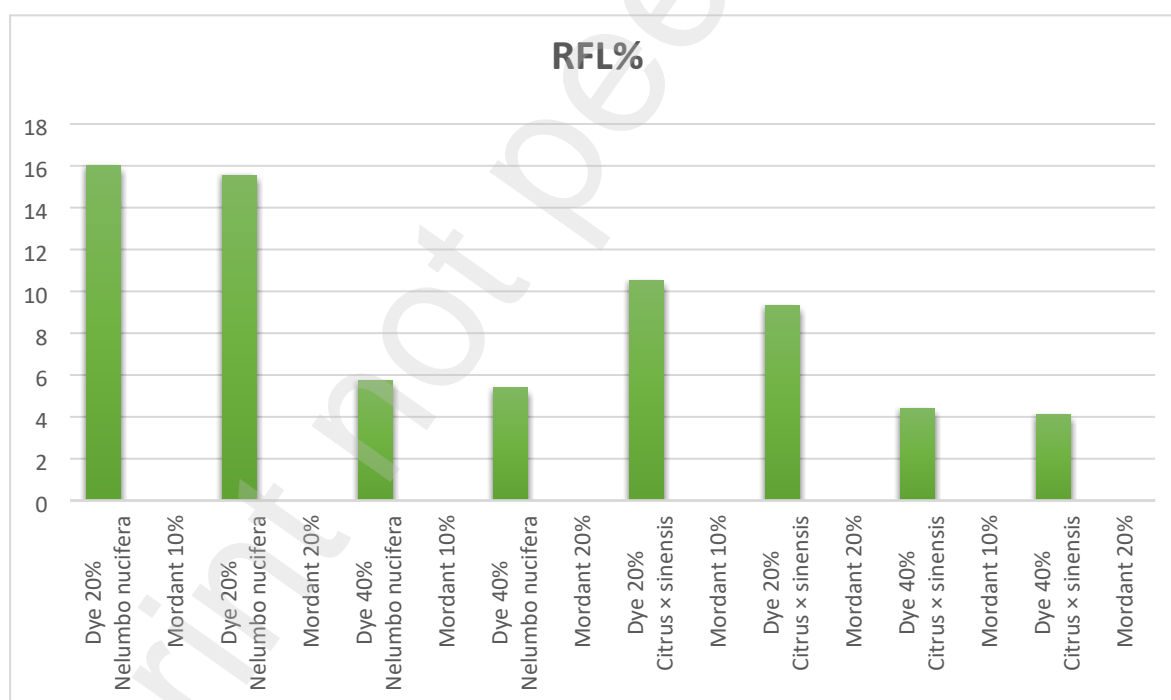


Figure 8: RFL% of *Nelumbo nucifera* & *Citrus x sinensis*

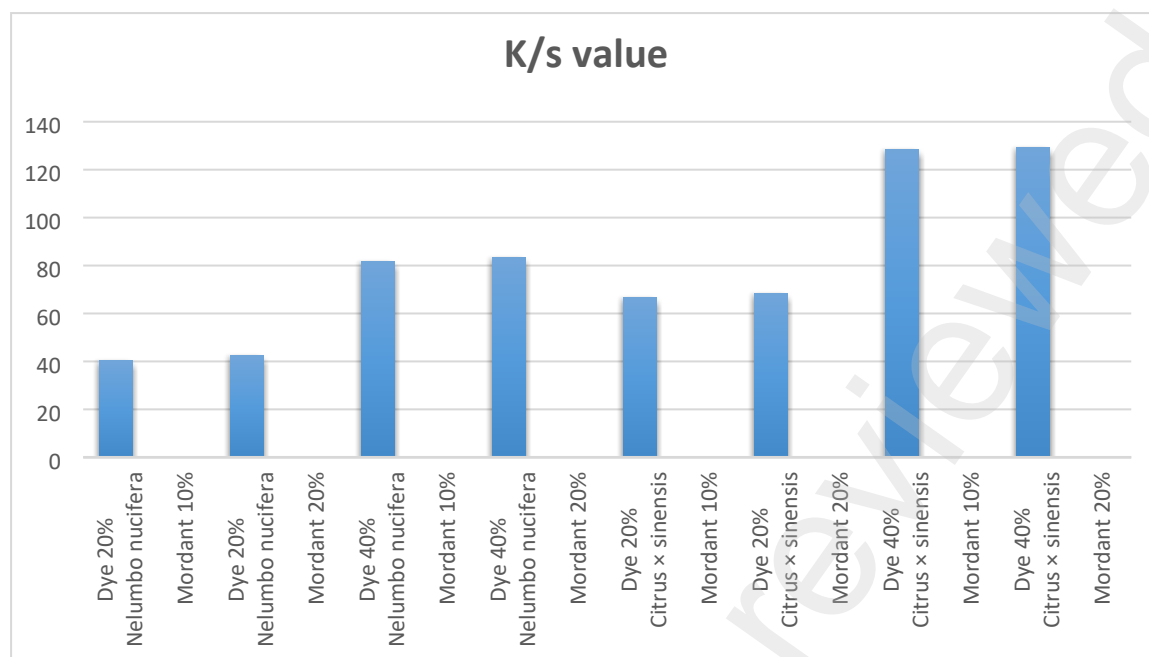


Figure 9: K/s Value of *Nelumbo nucifera* & *Citrus x sinensis*

Table 2 has shown the L*, K/S and RFL % . It has indicated that lower value of L* has darker shades and higher value of L* has lighter shade for Lyocell fabrics. *Citrus x sinensis* lyocell fabrics dyed with 40% dye concentration and 20% mordant and has the lowest L* values yielding the darkest shade and. *Citrus x sinensis* lyocell fabrics dyed with 20% dye concentration and 10% mordant and has the highest L* values yielding the lightest shade . Further, the reflectance value of *Citrus x sinensis* lyocell fabrics dyed with 40% dye concentration and 20% mordant is the lowest and *Citrus x sinensis* lyocell fabrics dyed with 20% dye concentration and 10% mordant is s the highest. The highest color value (K/S) was obtained with *Citrus x sinensis* lyocell fabrics dyed with 40% dye concentration and 20% mordant. The lowest color value (K/S) was obtained with *Nelumbo nucifera* lyocell fabrics dyed with 20% dye concentration and 10% mordant.

4.2 Colourfastness analysis

The below table 3,4 & 5 represents the colourfastness grade to light, rubbing and washing of the selected fabrics which are dyed at different percentages of dye and mordant concentrations.

Table 3: Colourfastness to light grade of *Nelumbo nucifera* and *Citrus x sinensis* samples

Sample code	Samples	Dye %	Mordant %	Light fastness
				CC
S1	<i>Nelumbo nucifera</i>	20	10	4

S2	<i>Nelumbo nucifera</i>	20	20	5
S3	<i>Nelumbo nucifera</i>	40	10	6
S4	<i>Nelumbo nucifera</i>	40	20	7
S5	<i>Citrus × sinensis</i>	20	10	6
S6	<i>Citrus × sinensis</i>	20	20	6
S7	<i>Citrus × sinensis</i>	40	10	7
S8	<i>Citrus × sinensis</i>	40	20	9

CC-Colour change ;

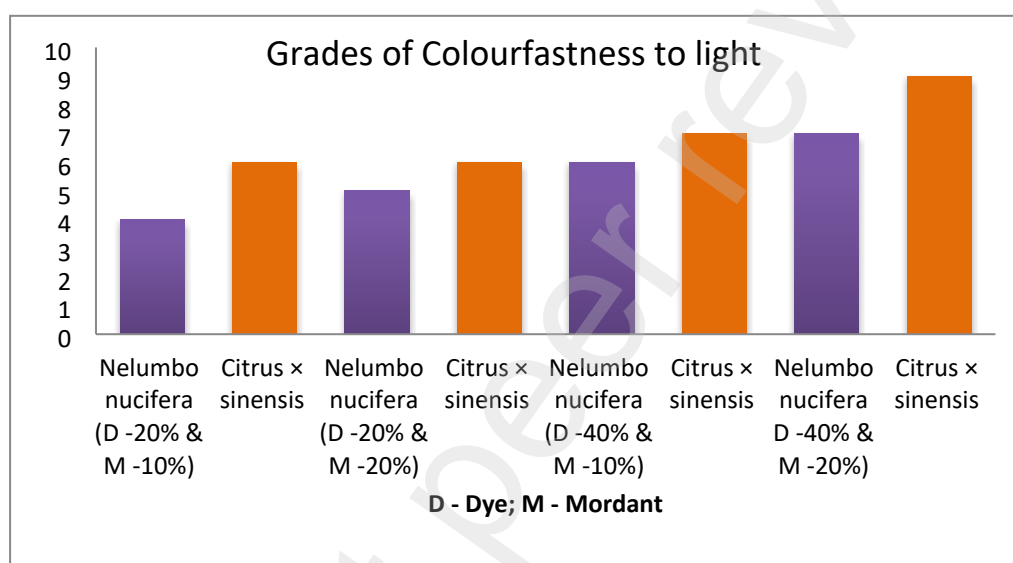


Figure 9: Grades of Colourfastness to light of *Nelumbo nucifera* & *Citrus × sinensis*

Table 4: Colourfastness to rubbing grade of *Nelumbo nucifera* and *Citrus × sinensis* samples

Sample code	Samples	Dye %	Mordant %	Rubbing fastness	
				CC	CS
S1	<i>Nelumbo nucifera</i>	20	10	3	3-4
S2	<i>Nelumbo nucifera</i>	20	20	3	4
S3	<i>Nelumbo nucifera</i>	40	10	4	4-5
S4	<i>Nelumbo nucifera</i>	40	20	4-5	4-5
S5	<i>Citrus × sinensis</i>	20	10	4	4-5
S6	<i>Citrus × sinensis</i>	20	20	4	5
S7	<i>Citrus × sinensis</i>	40	10	4-5	4-5
S8	<i>Citrus × sinensis</i>	40	20	5	5

CC-Colour change ; CS-Colour staining

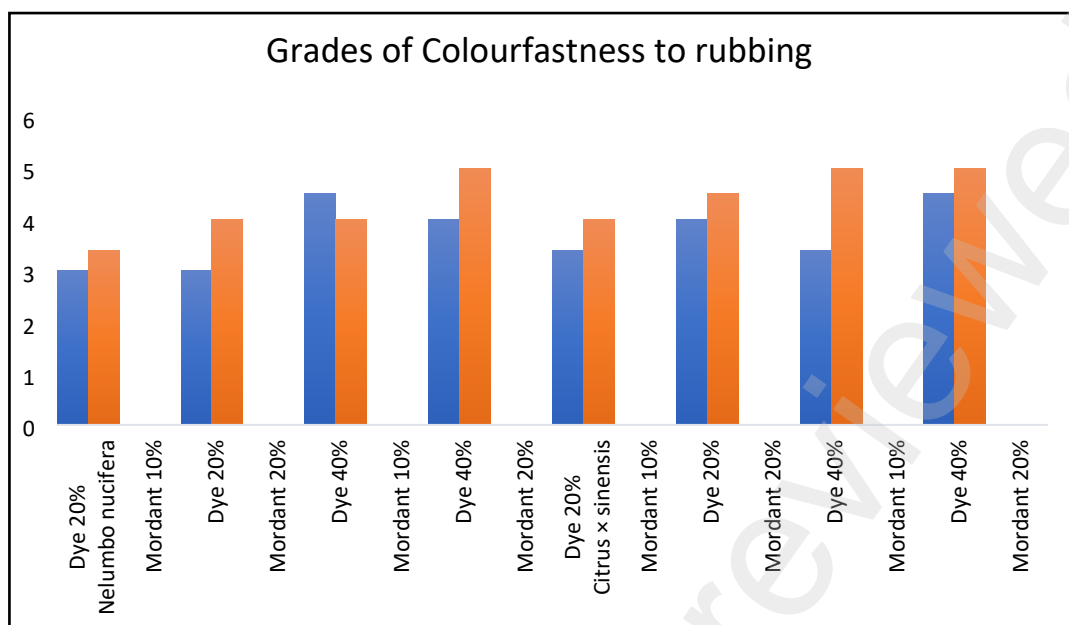


Figure 10 : Grades of Colourfastness to rubbing of *Nelumbo nucifera* & *Citrus × sinensis*

Table 5: Colourfastness to washing grade of *Nelumbo nucifera* and *Citrus × sinensis* samples

Sample code	Samples	Dye %	Mordant %	Washing Fastness	
				CC	CS
S1	<i>Nelumbo nucifera</i>	20	10	3	3-4
S2	<i>Nelumbo nucifera</i>	20	20	3	4
S3	<i>Nelumbo nucifera</i>	40	10	4-5	4
S4	<i>Nelumbo nucifera</i>	40	20	4	5
S5	<i>Citrus × sinensis</i>	20	10	3-4	4
S6	<i>Citrus × sinensis</i>	20	20	4	4-5
S7	<i>Citrus × sinensis</i>	40	10	3-4	5
S8	<i>Citrus × sinensis</i>	40	20	4-5	5

CC-Colour change ; CS-Colour staining

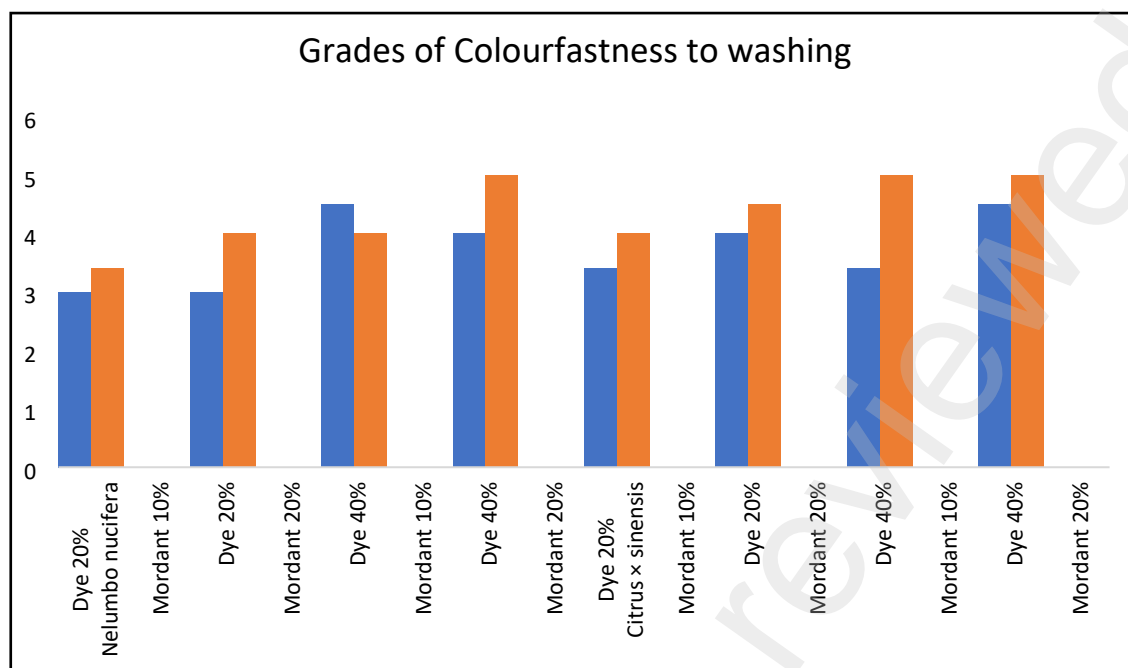


Figure 10 : Grades of Colourfastness to washing of *Nelumbo nucifera* & *Citrus × sinensis*

Ratings obtained for color fastness to rubbing (crocking) indicated that, sample S8 has the highest rating of 5 for both colour change (dye wash off) and colour stain(color transfer to adjacent undyed surfaces). In the case of color fastness to light, *Citrus × sinensis* lyocell fabrics dyed with 40% dye concentration and 20% mordant received the highest rating. Regarding wash fastness assessment, the ratings assigned to the dyed samples S3 and S8 for color staining gave the highest ratings. For the ratings related to color change, samples S3 and S8 gave the highest ratings varying between 4-5.

4.3 FT-IR Analysis



Figure 11 : FT-IR analysis of *Nelumbo nucifera* (40% dye concentration and 20% mordant)



Figure 12 : FT-IR analysis of *Citrus × sinensis* (40% dye concentration and 20% mordant)

5. Conclusion

The selected lyocell fabrics were successfully dyed using the extraction of *Isatis tinctoria* L. leaves. The fabrics were pre treated prior the dyeing process. Further, mordanting at two different concentrations 10% and 20% was carried out, also the dye concentrations 20% and 40% was chosen for each fabrics. So, totally 8 samples were taken under consideration for the evaluation. Here, spectrophotometer analysis and colourfastness to light, rubbing and washing through grading was carried out for each sample. Both the fabrics showed variation at different concentrations of dye and mordant.

Citrus × sinensis lyocell fabrics dyed with 40% dye concentration and 20% mordant and has the lowest L^* values yielding the darkest shade with lowest reflectance values. The highest color value (K/S) was obtained with *Citrus × sinensis* lyocell fabrics dyed with 40% dye concentration and 20% mordant. *Citrus × sinensis* fabric showed better grades of colourfastness to light, rubbing & washing at the concentrations D 40% and M 20%. As the result it is concluded that *Citrus × sinensis* fabric has good colour values and colour fastness properties than *Nelumbo nucifera* fabric.

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