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Dyeing of nylon fabric with natural dye extracted from waste leaves of *Terminalia catappa* locally known as tropical almond tree

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Abstract
Many natural resources which are being wasted indiscriminately or thrown away as a waste product contain useful dyes and pigments. In the present study, natural dye extracted from the waste leaves of *Terminalia Catappa* (tropical almond) and its application on nylon fabric pretreated with eco-friendly and noneco-friendly mordants have been carried out successfully. Different shades with excellent to good fastness properties have been obtained.

Keywords Natural dyes, *Terminalia catappa*, mordant, dyeing, eco-friendly, fastness

Introduction
Nylon is commonly dyed with disperse and acid dyes. However, in the recent years, a considerable amount of interest has been generated for the use of natural dyes in dyeing nylon and other synthetic fibers.
Until the latter half of the 19th century people were using natural dyes [1] for colouring the textile fibre after invention of synthetic dyes, natural dyes are not used because of the advantage of synthetic dye over natural dye in respect of application, colour range, fastness properties, and availability. Some synthetic dyes are hazardous, carcinogenic and also release vast amount of pollutant in the environment during their manufacturing [2-9].
Synthetic dyes are not good due to their toxic effect; and it creates allergic reaction to skin and also creates pollution. Thus revival of natural dyeing technique as one of the alternative is being emphasized for this purpose. Many natural resources which are being wasted indiscriminately or thrown away as waste product contain useful dye and pigment. Earlier studies have revealed that the waste contain many flavones which can be effectively used as dyes [10-11].
The use of synthetic dyestuffs during their application in the dyeing and printing industries has been criticized due to introduction of contaminants into the environment.
With a belief that "All natural things are good for life on the Earth". In line with this trend, there is now an ever increasing lobby for using natural coloring matters for textile substrates both natural and synthetic [12-13].
An increasing realization, that the intermediates and chemicals used in synthetic dyes are toxic and hazardous o human health as well as to the environment, has led to the revival of interest in the non-toxic eco-friendly coloring materials. Serious efforts are now being made to boost the use of natural dyes and to identify more raw materials and to standardize the recipes for their use. The textile industry in India and if many developing countries is presently facing the impact of German Ban on 118 specified azo dyes based on 20 carcinogenic aryl amines including benzidine. These dyes have also been, banned in India. The banned dyes include 26 acid dyes and 6 disperse dyes used in dyeing/printing of nylon. The industry is, therefore, in need of safe alternatives. The use of natural dyes can be one of the substitute alternatives for many hazardous synthetic dyes [14].
In the present study, we review the studies carried out so far on the application of natural dyes on nylon from waste leaves of Tropical almond (*Terminalia catappa*).

Source of images: Google Images

Photograph 1: Tropical almond (*Terminalia catappa*)

It is a large, spreading tree now distributed throughout the tropics in coastal environments. It grows to about 35 meter tall, with an upright, symmetrical crown and horizontal branches. During the dry season (autumn), the leaves turn into colours of red, copper, gold. The tree usually sheds all its leaves twice a year in January-February and July-August. The tree first drops its leaves when it reaches 3-4 years old and then every year. See Photograph-1.

The leaves are large, 15–25 centimeters (5.9–9.8 inch) long and 10–14 centimetres (3.9–5.5 inch) broad, ovoid, glossy dark green and leathery. Before falling, they turn pinkish-red or yellow-brown, due to pigments such as violaxanthin, lutein, and zeaxanthin. The leaves contain several flavonoids (like kaempferol or quercetin), several tannins (such as punicalin, punicalagin or tercatin), saponines and phytosterols. Due to this chemical richness, the leaves and also the bark are used in different traditional medicines for liver diseases, dysentery and diarrhea. It is also reported that the leaves contain agents for prevention of cancers (although they have not demonstrated anticarcinogenic properties) and antioxidant as well as anticlastogenic characteristics. see figure-1. The leaves are found to have strong anti-bacterial properties and promote fish breeding, curing sick fish and speed up healing of damage. The dried leaves act as a "black water extract" which gradually turns the water brown like tea and effectively reduces the pH levels in water, releasing organic compounds such as humic acids, flavanoids and tannins into the water which absorb harmful chemicals [15].

Material and Methods

Fallen leaves of tropical almond are big in size and easy to collect. Pinkish-reddish leaves were collected and dried at room temperature, ground and sieved.

Extraction of dye

100 gram dry powder was taken in 1 litre water and allowed to stand for overnight. Next day the mixture was boiled for 30 minute and then filter with cotton cloth and with simple filter paper to get a clear solution. See Photograph-2 the weight of dry powder after dye extraction was taken to know the concentration of dye by using following formula. pH of dye solution was measured. (Weight of the dry powder after extraction was 80 grams and pH of the dye solution was acidic 4-5)

Dry leaf of Tropical Almond | Dry leaves powder of Tropical Almond | Dry leaves of Tropical Almond

Photograph 2: Dry leaf, dry leaves powder and dye extract of tropical almond

Spectral analysis of dye extract

IR spectra of dye extract were obtained by using-IR Spectrometer Perkin Elmer Spectrum GX Range: 10,000 cm-1 to 370 cm-1; ATR accessory for reflectance measurement; IR Quant software; Spectrum search software. Spectra were recorded in 4000-400 cm^{-1} by filling the solid dye extract under a form of thin film of potassium bromide (KBr) spectral pellets. See graph-1 UV-VIS absorption spectra of dye extract were obtained by using a LAMBDA 19 UV/VIS/NIR spectrophotometer at Data interval 1.0000 nm, Scan speed 240.00 nm/min, Slit width 5.0000nm, Smooth band width 8.00nm. See graph-2

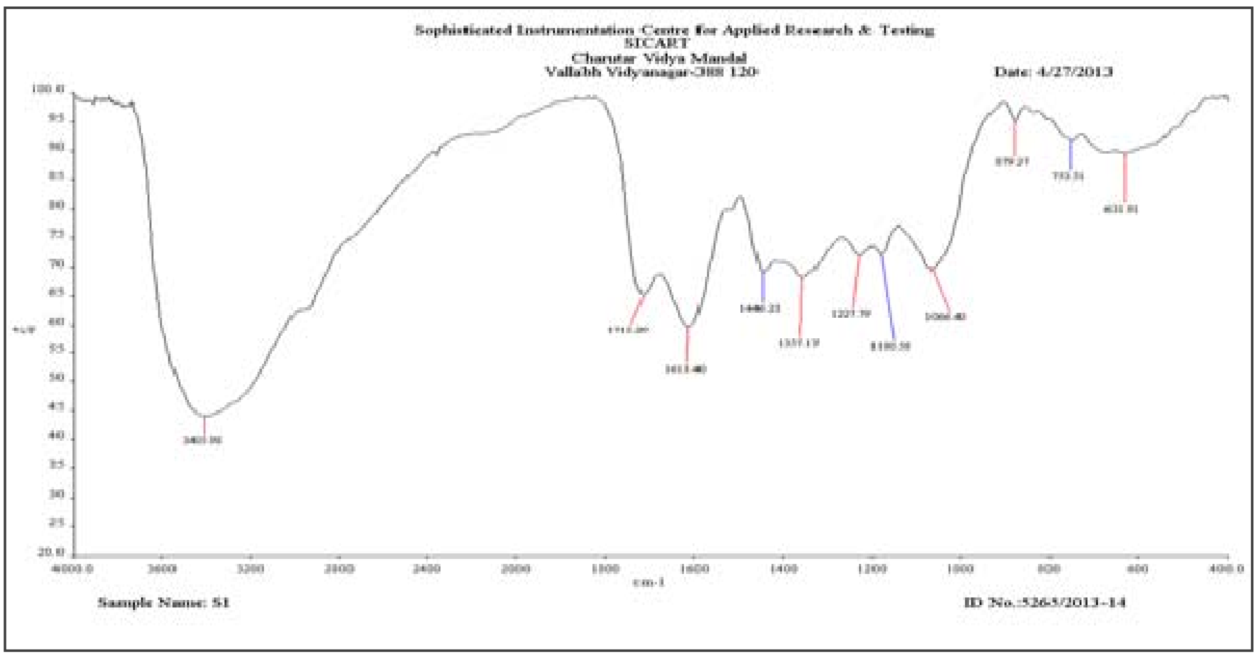
Dyeing procedure
Nylon fabric samples were scoured in mild detergent solution and dried in shade then weighed accurately and further soaked in water and treated with 10% mordant solution for 30 minutes at room temperature. Mordants used were eco-friendly and non-eco-friendly for comparison only as shown in table-1 Dye solution was taken on the weight of fabric for 10% shade and M:L = 1:30 was maintained The dye bath was warm to which mordanted fabric sample was entered and the temperature was increased up to 85 to 90º C) for 25-30 minute. The pH of dye bath was measured. The sample was allowed to cool in the dye bath then washed in cold water, squeezed and dried in shade.

Colour fastness properties
Colour fastness properties mainly sunlight, washing, perspiration (acid & alkali), rubbing (dry & wet) was carried out using standard methods [16].

Colour strength
Colour strength of dyed cotton fabric was measured in terms of L, a, b colour scale by using hunter lab instrument (Hunterlab Easy Match QC) Colour flex EZ 45/0, Reflectance. Mode

Result and Discussion
Terminalia catappa leaves extract contain several flavonoids, tannins, saponines and phytosterols. Due to this chemical richness leaves extract was used in this study to dye nylon.

Characteristics of Tropical Almond leaves extract
The colour was extracted in water photograph 2 without using any chemical reagent. pH of the extract was measured and it was acidic (5.1). Colour of the extract was dark brownish yellow. In order to get exact reproducibility in shade it is important to determine the percentage soluble matter which was calculated and was found to be 2%.
Characterization of functional group in Tropical Almond leaves extract was recorded with the help of FT- IR spectra in the range of 4000-400 cm^{-1} by filling the solid dye extract under a form of thin film of KBr spectral pellets. IR is given in graph 1



Graph 1: IR of leaves extract Tropical Almond

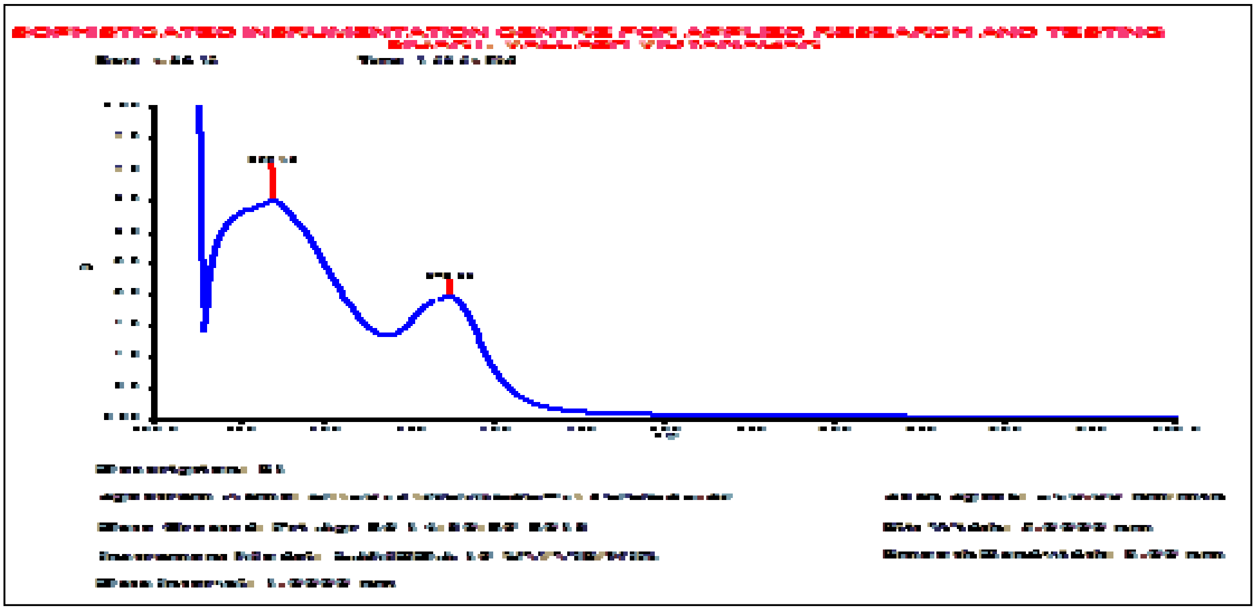
Table 1: Characterization of functional group in leaves extract of tropical almond

Sr No.	Absorbency peak Obtained in cm^{-1}	Intensity	Characterization of Functional Group	Inference From IR spectra
1	3406	Strong, broad (3200-3600)	O-H stretching	it can be concluded that there was a quercetin and related compounds present in the dye extract which are known as flavonoids compound and most of the yellow colours are due to hydroxy and methoxy derivatives of flavones and isoflavones [17]
3	2950	Strong 2850-3000	C-H stretching	
4	1713	Strong	C=O stretching	
5	1613	Variable, medium-weak, multiple bands, (1400-1600)	-C-H bending, C=C aromatic stretching	
6	1446	Variable, medium-weak, multiple bands,(1400-1600)	-C-H bending, C=C alkenes and aromatic stretching	
7	1357	Variable, medium-weak, multiple bands, (1400-1600)	- C=C aromatic stretching	
8	1227	medium-weak (1080-1360)	C-O stretching	
9	1180	medium-weak (1080-1360)	C-O stretching	
10	1066	Strong (1050-1150)	C-O and C-O-C stretching	
11	879	Strong (675-1000)	=CH- bending	
12	631	Strong (450-650)	C-O-H bending	

IR spectra showed that -OH group is in the region of wavelength absorption 3406 cm^{-1} with a very strong and broad signal. Cluster C = C are in the catchment area with a wavelength of 1613 cm^{-1}.In the catchment area 1446 cm^{-1} indicates the presence of C=C of aromatic group and the absorption band 1243-1091 cm^{-1} indicate the presence of C-O groups, and a catchment area with a wavelength of 807-675 cm^{-1} indicates a =CH group.

The Fourier Transform Infra-Red (FT-IR) spectra were recorded for isolated samples in the range of 4000 - 450 cm^{-1}. The pronounced peaks belonging to the vibration of 3366 cm^{-1}, 1650 $cm^{-1}$and 679 cm^{-1} in the spectra corresponds to the characterized peaks of flavonoids. [17]

The UV absorption specra is given in graph 2



Graph-2 UV of tropical almond leaves extract

UV-Visible spectra for quercetin (spectrum) exhibits two absorption bands at 256 and 374 nm referring to conjugations. [18]

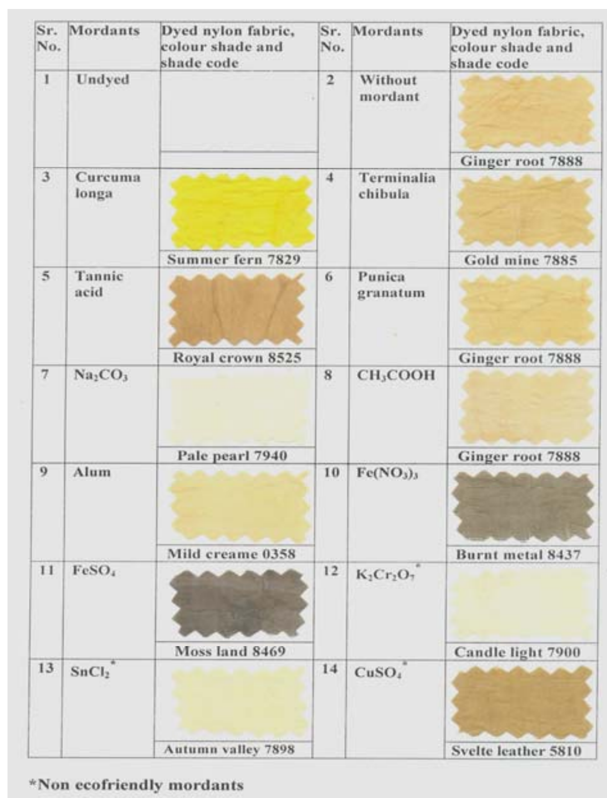
UV-VIS spectra obtained from dye extracted from Tropical Almond leaves exhibits two absorption bands at 269 and 372 nm referring to conjugations in the B-ring and A-ring, respectively which is related to quercetin.

From IR and UV data of dye extracted from Tropical Almond leaves it can be stated that there was a quercetin related compound present in the dye extract which has a flavonoid compound and most of the yellow colours are due to hydroxy and methoxy derivatives of flavones and isoflavones.

Experiment was carried out using various eco-friendly mordant and non-eco-friendly mordant (for comparison only). pH of the dye bath was 5-6 without using mordant. By using different mordant (10%), pH of the dye bath varied from acidic (1.8) to basic (10.5). (Table-2)

Dyeing of unmordanted fabric, resulted in cream yellow shade. Dyeing with mordanted fabric resulted in different shades of yellow, black and grey. which are also shown in plate-4.5.

It was also observed that by using non eco-friendly mordant few shades obtained matched with eco-friendly mordant shades so it is advisable to use more and more eco-friendly mordants.



Sr. No.	Mordants	Dyed nylon fabric, colour shade and shade code	Sr. No.	Mordants	Dyed nylon fabric, colour shade and shade code
1	Undyed		2	Without mordant	Ginger root 7888
3	Curcuma longa	Summer fern 7829	4	Terminalia chibula	Gold mine 7885
5	Tannic acid	Royal crown 8525	6	Punica granatum	Ginger root 7888
7	Na_2CO_3	Pale pearl 7940	8	CH_3COOH	Ginger root 7888
9	Alum	Mild creame 0358	10	$Fe(NO_3)_3$	Burnt metal 8437
11	$FeSO_4$	Moss land 8469	12	$K_2Cr_2O_7$*	Candle light 7900
13	$SnCl_2$*	Autumn valley 7898	14	$CuSO_4$*	Svelte leather 5810

*Non ecofriendly mordants

Plate 4.5: Shade card of nylon fabric sample dyed with Tropical Almond leave extract with and without mordants, Colour shade and code number

Colour shade, colour strength and colour fastness of nylon fabric dyed with extract of Tropical Almond

Various colours were obtained which varied from yellow to grey and black, these colour mainly depended on the mordant used. Dyeing of unmordanted nylon fabric was resulted in golden yellow shade. Dyeing with mordanted fabric resulted in different shades of yellow, grey and black. The colour shades obtained with ecofriendly mordents was compared with those shades obtained by non-eco-friendly mordents. The shade card is presented in plate 4.5 the presence of mordanted sample in the dye bath influenced the pH of the dyebath. pH of the dye bath was 5-6 without mordant. By using various mordant (10%), pH of the dye bath varied from acidic (1.8) to basic (10.5). refer table-2

Table-2: pH of the dye bath, colour strength and fastness properties of dyed nylon fabric with Tropical Almond leaves extract

Sr. No.	Name of the mordant	pH of dye bath	Colour strength			Fastness properties of dyed fabrics					
			L	a	b	Li	Wa	PA	PB	DR	WR
1	undyed		46.94	3.12	-14.24						
2	Without mordant	5.6	37.97	0.99	9.31	5	5	5	5	5	4
3	Curcuma longa	5.6	43.20	-5.88	21.32	4 c	5	5	5	5	4
4	Terminalia chibula	4.2	35.52	0.14	11.33	5	5	5	5	5	5
5	Tannic acid	4.5	38.54	2.17	9.26	5	5 cd	5	5	5	4
6	Punica Granatum	5.1	38.58	-1.20	10.46	5 c	5	4-5	5	5	4
7	Na_2CO_3	10.5	45.07	-0.78	0.41	4	5	4-5	3-4	5	4
8	CH_3COOH	3.4	39.42	-0.53	7.72	5 c	5	5	5	5	4
9	Alum	2.8	40.32	-1.47	8.84	5	5	4	5	5	3-4
10	$Fe(NO_3)_3$	1.8	27.27	-0.33	3.19	5	5 cd	4	4	5	4
11	$FeSO_4$	4.0	24.13	0.16	0.83	5	5 cd	4	4	4	3
12	$K_2Cr_2O_7$	5.7	44.70	0.70	0.15	5	4	4-5	3	4	4
13	$SnCl_2$	2	44.69	-2.42	5.84	5 c	5	5	4-5	4	4
14	CuSO4	3.5	32.48	0.93	8.62	5	5	5	4-5	4	3-4

Li=Light, WA=Washing, PA=Acid Perspiration, PB=Alkali Perspiration, DR=Dry Rubbing (Dry crocking), WR=Wet Rubbing (Wet crocking), DC= Dry Cleaning, c= Colour Change, cd= Colour Change to Dark

Term for fastness properties except crocking: 5=Excellent, 4=Good, 3=Fair, 2=Poor, 1= Very Poor

Degree of Crocking or staining of fabric: 5= No Staining, 4= Slightly Stain, 3= Noticeably Stain, 2=Considerably Stain, 1=Heavily Stain

Colour fastness of nylon

Sunlight fastness property of unmordanted and premordanted fabric sample was studied. Excellent (5) result was obtained for all dyed fabrics except curcuma longa. For fabric premordanted with curcuma longa, slight change in colour of the fabric was observed.

Washing fastness properties of dyed nylon fabric, results were excellent while in case of tannic acid, ferric nitrate and ferrous sulphate colour of the fabrics changed to dark.

Acid and alkali perspiration fastness properties of unmordanted and mordanted dyed fabric samples were carried out and results obtained for acidic perspiration were good to excellent for all samples. For alkaline perspiration the result also obtained was good to excellent, except for dyed sample pre-mordanted with sodium carbonate and potassium dichromate, it was poor to good.

The results obtained for dry rubbing fastness was good to excellent for all dyed nylon fabric, while for wet rubbing fastness it was fair to good.

L,a,b Colour Scale

The L,a,b colour scale is more visually uniform than the XYZ colour scale. In a uniform colour scale, the differences between point plotted in the colour space correspond to visual difference between the colour plotted. The L,a,b colour space is organized in cube form. The L axis runs from top to bottom. The maximum for L is 100, which would be a perfect reflecting diffuser. The minimum for L would be zero which would be black. The a and b axis have no specific numerical limits. Positive a is red. Negative a is green. Positive b is yellow. Negative b is blue. Below is a diagram of the hunter L,a,b colour space. Which are also shown in Figure-2.

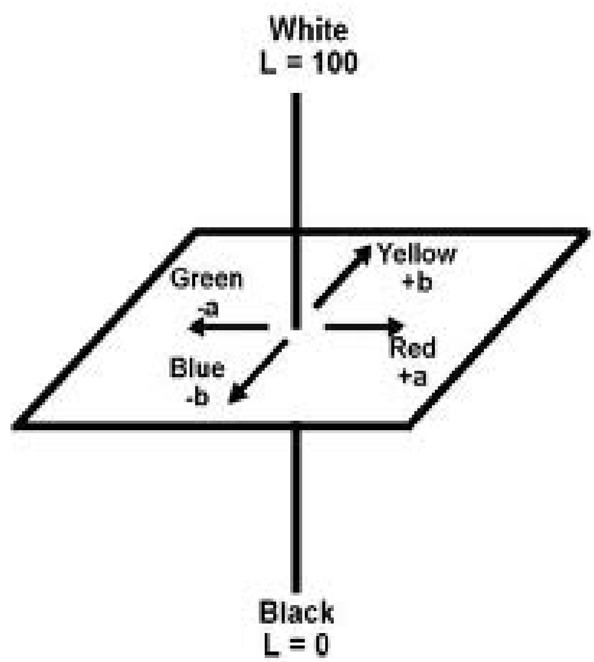
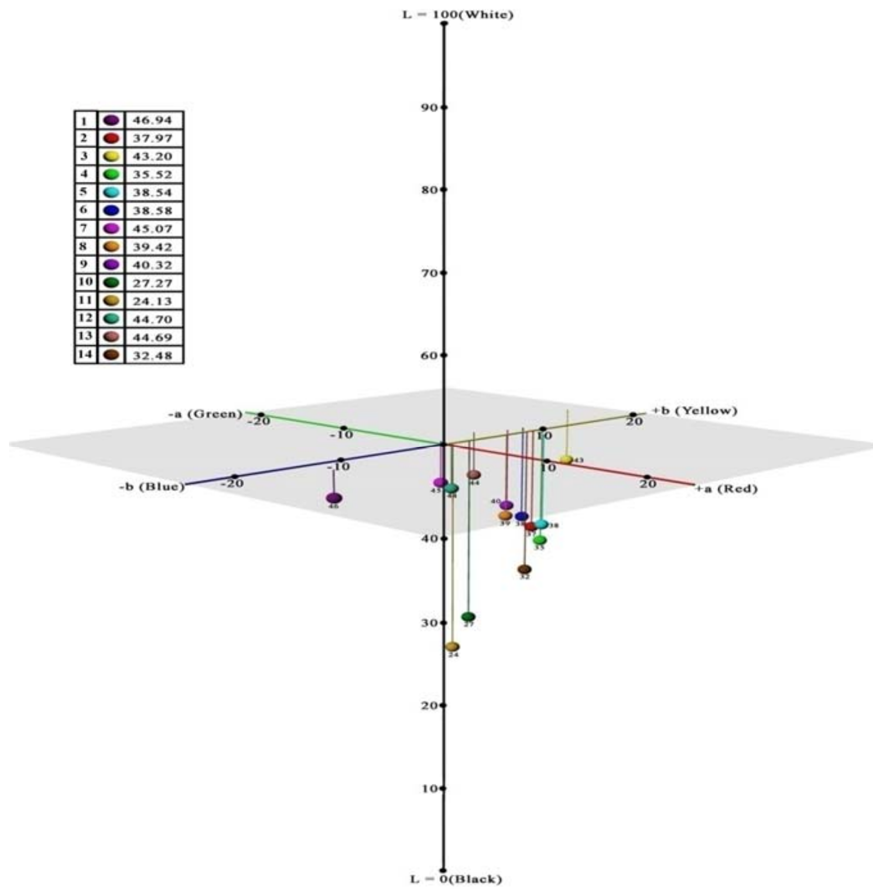


Fig 2: (L,a,b Colour Scale)



Graph-3 Colour strength of nylon fabric dyed with Tropical Almond leave extract

Colour strength of nylon

Colour strength of undyed and dyed nylon fabric was measured in terms of L, a, b colour scale. The tabulated values are given in table 4.11 and graphically presented in graph 4.10. The result was obtained in the range of 24.13 to 45.07 for 'L' value, shows lower value because of dark shade and for 'a' value it was in the positive range to negative value (0.14 to - 5.83) indicate all the colour value in green region. While in 'b' the value obtain in the range of 0.15 to 21.32

From the overall data value of L,a,b colour scale shows that colour obtained was yellow(+b), red to green (+a to -a) but near to yellow line. Most of the samples could be classed as greenish yellow or reddish yellow and black i.e. L<50

L,a,b value of ecofriendly mordanted nylon fabric was compared with L,a,b value of shades obtained by non-eco-friendly mordents, Nearly similar L,a,b were obtained by sodium carbonate and potassium dichromate.

Conclusion

Natural dyes are safe and eco-friendly and textiles dyed with natural dyes are almost free from hazardous chemicals. Red listed mordants may be either avoided or may be optimized as per eco-standard, without impairing the desirable properties (e.g. fastness) of the textiles

Tropical almond trees are easily found in the local region. Leaves fall twice in a year; these can be collected and used for dyeing variety of colours on nylon very effectively. Different shades on nylon fabrics with good to excellent fastness properties have been obtained. After extraction of dye, remaining matter can be use as fertilizer and after dyeing process all the liquid matter can be used in fish breeding. So the process of dyeing is totally eco-friendly.

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