Dyeing Nylon With Natural Dyes

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Abstract

Natural colors have attracted the attention of the entire world, because of their non-hazardous nature. In the present study, nylon fabric was dyed with three natural dyes derived from Onion (*Allium cepa*), Lac (*Laccifer Lacca*) and Turmeric (*Curcuma longa*) using various mordants by two different techniques (viz. open bath and HTHP dyeing methods). HTHP dyeing has been found to give better results as compared to the open bath dyeing. Good washfastness (4) was obtained with all three natural dyes. Comparative higher ratings (4-5) for lightfastness were achieved in the case of Onion and Lac, as compared to that of Turmeric (3-4).

Introduction

Nylon is commonly dyed with disperse and acid dyes. However, in recent years, a considerable amount of interest has been generated for the use of natural dyes in dyeing nylon and other synthetic fibers. The world is becoming increasingly aware of environmental issues, such as the Green House (GH) effect, ozone layer depletion, water pollution and waste disposal problems.

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. This has led to the desire to turn to a the traditional/more natural way of life, (i.e.—biological (organic) farming, natural food, etc). 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.[1-2]

The contemporary textile processing industry is getting more and more inquiries regarding "Dyeing with Natural Dyes" and, therefore, the subject of natural colors has assumed a great significance. Recently, an exhaustive review on the subject of natural dyes in textile applications has been published by Taylor.[3]

An increasing realization, that the intermediates and chemicals used in synthetic dyes are toxic and hazardous to 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 in many developing countries is presently facing the impact of German Ban on 118 specified azo dyes based on 20 carcinogenic aryl amines including benzi-

Table I: Effect of mordants on CIELAB values and fastness properties for dyeing of nylon with onion in open bath.

Mordants 10% (owf)	DE*	L*	a*	b*	C*	h*	LF	WF	SF
Control	0.00	63.86	15.78	35.88	39.19	66.26	2-3	34	4
Alum	4.39	63.74	14.80	40.16	42.80	69.76	2-3	3-4	4
Copper sulfate	18.95	57.88	14.82	53.83	55.83	74.61	4	4-5	4
Ferrous sulfate	14.19	52.41	8.58	31.58	32.72	74.80	3-4	4-5	4
Stannous chloride	17.11	67.57	11.55	52.03	53.30	77.48	3-4	4	4
Tannic acid	2.78	62.84	14.18	33.84	36.69	67.26	3	3 4	4
Harda	2.68	63.86	13.51	34.45	37.00	68.59	3	3-4	4
Aluminium sulfate	7.85	65.18	13.52	43 21	45.21	72.21	2-3	3	4

Control = Dyed shades without mordanting
LF = Light fastness (on O to 8 scale), WF = Washing fas~ess (on O to 5 scale), and
SF = Sublimation fastness (on O to 5 scale)

Table II: Effect of mordants on CIELAB values and fastness properties for dyeing of nylon with onion in HTHP.

Mordants 10% (owf)	DE*	L*	a*	b*	C*	h*	LF	WF	SF
Control	0.00	40.99	13.87	27.52	30.81	63.25	3	4	4
Alum	3.43	42.87	14.13	30.38	33.50	65.06	3	4	4
Copper sulfate	7.94	34.15	12.82	23.63	26.88	61.51	4	4-5	4
Ferrous sulfate	13.39	31.59	8.65	19.54	21.36	66.12	4	4-5	4
Stannous chloride	8.86	44.53	17.02	35.00	38.92	64.06	2-3	4	3-4
Tannic acid	9.36	33.67	11.86	22.04	25.03	61.71	3	4	4
Harda powder	2.43	39.25	15.10	26.35	30.37	60.19	3	3-4	4-5
Aluminium sulfate	1.97	40.07	-0.55	1.23	0.68	61.17	2-3	4	3-4

Control = Dyed shades without mordanting
LF = Light fastness (on O to 8 scale), WF = Washing fas~ess (on O to 5 scale), and
SF = Sublimation fastness (on O to 5 scale)

dine. 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.[4]

Acid dyes, in general, are more resistant to ozone fading than disperse dyes and, hence, the latter almost entirely have been replaced by acid dyes in the nylon carpet dyeing industry.[5] The poor resistance to ozone fading of disperse dyes has been attributed to their relatively small molecular size and lack of ionic character.[6] Ozone is a strong oxidizing agent that destroys the chromophores of certain type of dyes. For example, ozone can attack the anthraquinone ring of a dye molecule and convert it into colorless phthalic acid derivatives.[7]

The literature survey indicates that there is hardly any work reported in the field of dyeing of nylon with natural dyes. The present study was undertaken to standardize the process of dyeing of nylon with natural dyes, such as Onion (*Allium cepa*), Lac (*Laccifer Lacca*) and Turmeric (*Curcuma longa*) and to evaluate the fastness properties of the dyed substrates dyed with the same.

The outermost dry papery skins of Onion are the best source of the color, which contain flavonoid quercetol, kaempferol, quercetin-3-glucoside and some tannin. Lac is the resinous protective secretion of the tiny Lac insect, which is a pest on a number of plants. The insects secrete a thick resinous fluid, which envelops their bodies, forming a hard continuous encrustation over the twigs. The twigs are harvested and the encrustation scraped off, dried and processed to yield shellac or the dye.[8] Turmeric is historically one of the most famous and the brightest of naturally occurring yellow dye. It also possesses antibacterial property as well as medicinal value. It is extracted From the fresh or dried rhizomes of Turmeric.

Experimental

Materials

A 100 % nylon woven fabric, medium weight, scoured and bleached was used for dyeing.

Acetic acid and sodium hydroxide were used for adjusting the acidic and alkaline pH respectively. The sources of the natural dyes were from Onion (*Allium cepa*), Lac (*Laccifer lacca*) and Turmeric (*Curcuma longa*).

Table III: Effect of mordants on CIELAB values and fastness properties for dyeing of nylon with Lac in HTHP.

Mordants 10% (owf)	DE*	L*	a*	b*	C*	h*	LF	WF	SF
Control	0.00	42.16	17.88	4.08	18.34	12.87	2-3	4	4
Alum	13.43	40.94	30.40	8.80	31.64	16.14	2-3	4	4
Ferrous sulfate	12.09	38.73	6.58	1.48	6.74	12.67	3-4	4	4
Stannous chloride	23.76	46 72	33 04	15.80	41.19	22.55	2-3	4	4

Control = Dyed shades without mordanting
LF = Light fastness (on O to 8 scale), WF = Washing fas~ess (on O to 5 scale), and
SF = Sublimation fastness (on O to 5 scale)

Table IV: Effect of mordants on CIELAB values and fastness properties for dyeing of nylon with turmeric in open bath.

Mordants 10% (owf)	DE*	L*	a*	b*	C*	h*	LF	WF	SF
Control	0.00	70.46	15.85	89.30	90.69	79.93	2	3-4	4
Alum	2.13	80.87	1.38	42.80	42.82	88.15	2	4	4
Copper sulfate	8.61	77.42	5.82	41.01	41.42	81.92	3	4	4
Ferrous sulfate	16.16	68.58	9.52	28.05	29.62	71.26	3	4	4-5
Stannous chloride	3.36	81.85	-0.98	46.94	46.96	91.20	2	34	4
Tannic acid	6.88	76.63	-2.90	40.33	40.43	94.11	2-3	3-4	4-5
Harda powder	1.41	81.32	-4.29	39.33	39.56	96.22	2-3	3-4	4
Aluminium sulfate	1.28	81.94	0.10	42.48	42.48	89.86	2	4	4

Control = Dyed shades without mordanting
LF = Light fastness (on O to 8 scale), WF = Washing fas~ess (on O to 5 scale), and
SF = Sublimation fastness (on O to 5 scale)

Table V: Effect of mordants on CIELAB values and fastness properties for dyeing of nylon with turmeric in HTHP.

Mordants 10% (owf)	DE*	L*	a*	b*	C*	h*	LF	WF	SF
Control	0.00	53.36	7.72	44.33	44.99	80.12	2	4	4
Alum	4.70	53.64	9.09	48.48	49.32	79.37	2	4	4-5
Copper sulfate	4.78	51.68	9.91	46.45	47.49	77.96	3	4	4
Ferrous sulfate	7.66	47.96	9.52	43.55	44.58	77.67	3	4	4-5
Stannous chloride	16.92	39.71	10.76	38.66	40.13	74.45	2	3-4	4
Tannic acid	2.37	55.03	12.17	54.95	56.28	77.51	2	3-4	4-5

Control = Dyed shades without mordanting
LF = Light fastness (on O to 8 scale), WF = Washing fas~ess (on O to 5 scale), and
SF = Sublimation fastness (on O to 5 scale)

The following mordants were used for mordanting

Aluminium Potassium Sulfate [$K_2SO_4.Al_2(SO_4)_3.12H_2O$]
Copper Sulfate [$CuSO_4.2H_2O$]
Ferrous Sulfate [$FeSO_4$]
Stannous Chloride [$SnCl_2.2H_2O$]
Tannic Acid
Harda Powder (*Chebulic myrabolan*)

Extraction of Dyes

Onion Dye

The outermost dry papery skin of Onion was removed and boiled with water for one hour. The extract was cooled to room temperature (25°C) and filtered. The filtrate was then used as the solution.

Lac Dye

Lac powder was mixed with a required amount of water and boiled for one hour. The extract was cooled to a room temperature (25°C) and filtered. The filtrate was then used as the dye solution for dyeing.

Turmeric Dye

Turmeric powder was boiled with water for one hour. The content was cooled to the room temperature (25°C), filtered and the filtrate was used for dyeing.

Mordanting

The pre-mordanting method was used in the present work. For mordanting, pots were prepared with the required amount of mordant (on the weight of fabric basis) and nylon fabric was introduced into the pot at room temperature (25°C). The temperature of the bath was raised to boil and mordanting was continued for 45 min. at boil. The fabric was then removed, squeezed evenly and dyed in the respective dye solutions without intermediate washing or drying.

Dyeing

Dyeing of nylon without using mordant has been treated as the control. Dyeing of the mordanted fabric was carried out in the open bath as well as in the HTHP dyeing machine (Parikh Elecronics, Bombay). Material-to-liquor-ratio (M.L.R.) of 1:40 was mentained for both open and HTHP dyeings. Control Dyeing was also carried out using both the methods of dyeing. For all the dyeings, 1% extracts of Onion and Turmeric, and 2% extract of Lac were used.

Open Bath Dyeing

Dye pots were prepared with addition of required amount of dye solution and other additives. Mordanted fabric was put into dye solution at room temperature (25°C). The temperature was then raised to the boil and dyeing was continued for another 45 min. at the boil. After dyeing, cold wash was given with water followed by dye-fixation treatment with 2 g/1 Fixanol PN (ICI (India) Ltd., Bombay) for 15 min. at 45°C under acidic conditions. After a cold wash, soaping was carried out with 2 g/1 Auxipon NP® (Auxichem, Bombay), the non-ionic soap. Finally, fabric samples were washed thoroughly and dried.

HTHP Dyeing

The same method, as mentioned, was followed, except that the dyeing was carried out in beakers in the HTHP dyeing machine (Parikh Elecronics, Bombay). Here, dyeing was carried out at 130°C for 45 min. in the closed pots under pressure.

Color Measurement

The various color changes such as depth, tone, λ_{max}, etc. were measured with a Spectraflash SF300 (Datacolor International, USA) in terms of K/S values and CIE L*a*b* data with illuminant D65 at 10° observer.

Fastness Determination

Washfastness tests were carried out according to ISO II method. Lightfastness and sublimation fastness were evaluated by standard test methods, using Suntest CPS (Heraeus, Germany) and Sublimation Tester (Electronic and Engineering Co., Bombay).

Table VI: Effect of mordants on λmax for dyeing of nylon with various natural dyes.

Mordants	λmax		
	Onion	Lac	Turmeric
Control	400	460	420
Alum	400	460	430
Copper sulfate	400	-	400
Ferrous sulfate	400	460	400
Stannous chloride	400	520	420
Tannic acid	400	-	430
Harda powder	400	-	440
Aluminiurn sulfate	400	-	430

Results and Discussion

Figure 1 shows the effect of dyebath pH on depth of shade (expressed as K/S) of nylon dyed with the three natural dyes viz. Onion, Lac and Turmeric. In general, all three natural dyes showed a higher dye uptake under acidic conditions as compared to the alkaline pH. Beyond the neutral pH on the acidic side, in general, there was sharp increase in color values. The highest depths of shade were obtained with Turmeric at pH 5 with both methods of dyeing viz. open bath and HTHP dyeing.

Dyeing of Lac in HTHP gave maximum color under stronger acidic conditions. Similarly, in the case of dyeing nylon with Onion, a higher dye uptake at acidic pH was obtained. Although, HTHP dyeing with Turmeric under

Figure 1–Effect of pH of dyebath on dyeing of nylon with natural dyes.

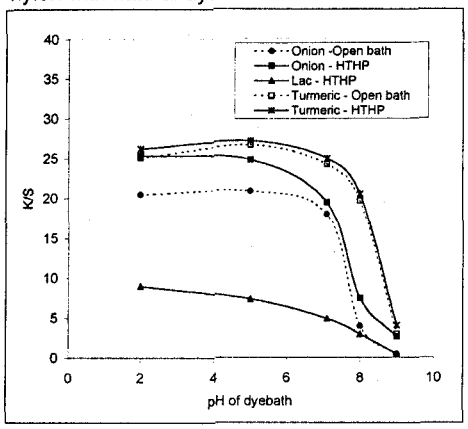


Figure 2–Effect of pH of dyebath on dyeing of nylon with natural dyes.

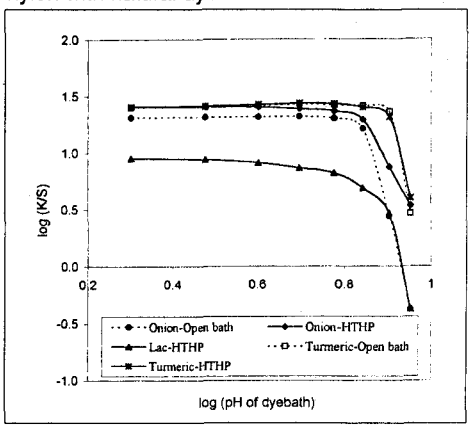


Figure 3–Effect of mordants on color values of dyeing of nylon with onion in open bath and HTHP.

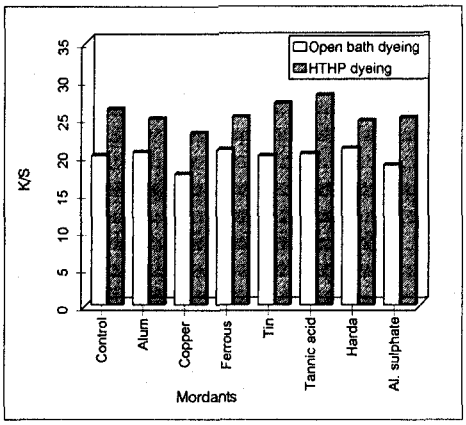
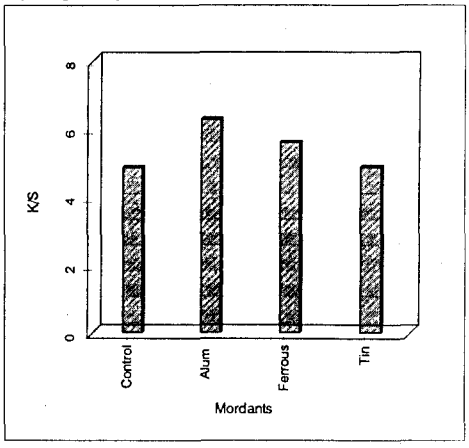


Figure 4–Effect of mordants on color values of dyeing of nylon with Lac in HTHP.



acidic conditions produced dull shades, it gave very bright yellow shades during open bath dyeing. It is also clear that the dyeing of nylon with Onion, Lac and Turmeric followed an almost similar type of absorption curve in both methods of dyeing (i.e.—Langmuir type of absorption curve, which is characteristic of acid dyes on nylon). It may be due to the adsorption of the dye molecules at the positively charged amine and amide groups.[9]

Since Turmeric is very sensitive to the alkaline conditions, the natural fibers like cotton, silk and wool dyed with Turmeric turned red when washed with alkaline soaps. But the nylon samples dyed with Turmeric did not turn red in the alkaline soap solutions. Thus, this is the additional advantage of dyeing of nylon with Turmeric dye. It also shows the difference in the mechanism of dyeing of nylon with Turmeric dye as compared to other fibers like cotton, silk and wool where the dye molecules are held by ionic attraction on these fibers. It is, therefore, concluded that the natural dyes like Turmeric are dyed on nylon by solid-solution mechanism.

Therefore, higher dye uptake on the nylon in acidic conditions could be attributed to (a) Structural features of the dye and the fiber, where the electrostatic forces between the positively charged end amino groups of the fiber and the dye molecules could be expected to play a dominant role in the increased dye adsorption, and (b) Solid solution dyeing, where all the three natural dyes behave like disperse dyes and gave higher dye uptake under the acidic conditions.

Onion and Turmeric exhibit similar behavior in both the methods of dyeing and gave the shades of comparable K/S values (20.55 and 25.34 for Onion-Open bath and HTHP, respectively, and 26.63 and 27.16 for Turmeric-Open bath and HTHP, respectively). Lac, however, showed much lower K/S values (8.96 for Lac-HTHP), although the nature of the curve remained the same as those for Onion and Turmeric.

As regards the mechanism of the dyeing of these three natural colors, it seems that they are governed by one and the same mechanism. When curves of log of K/S verses log of pH of dyebath were constructed (Figure. 2), it can be seen that all the three dyes gave a very good co-relation between the two parameters on the log-log scale, although the curve for Lac is shown separately due to its weak shades obtained. The remaining curves are almost overlapping for both Turmeric and Onion irre-

spective of the method of dyeing.

The pre-mordanting method of dyeing of nylon, in general, gave darker shades and, therefore, it should be reflected in the L* values and it should give higher DE* values. But DE* also takes care of the component a* (redder-greener) and b* (yellower-bluer). If there is no change in a* and b* values due to mordanting, then a darker shade due to mordanting is expected, which should give higher values of DE* compared to the control (i.e.—a shade produced without using a mordant).

The data of the effect of various mordants on CIELAB values and fastness properties of shades of nylon dyed with Onion in open bath and HTHP dyeing have been given in Table I and II, respectively.

From Table I, it can be noted that mordanting with copper in open bath dyeing gave a maximum total color difference (DE* = 18.95) as compared to the control, which is mainly attributed to the lower values of L*, which is followed by tin (DE* =17.11). However, in the case of HTHP dyeing of Onion, the higher DE* was given by iron (DE* = 13.39), which is somewhat lower than even the highest value for open bath dyeing. It may be due mainly to lower values of L* and a* obtained in this case.

The rest of the mordants gave marginal DE* values. The C* represents Chroma and indicates the saturation value of the color and h* represents the hue angle (hue angles for red, yellow, green and blue are 30°, 90°, 180°, and 270°, respectively).

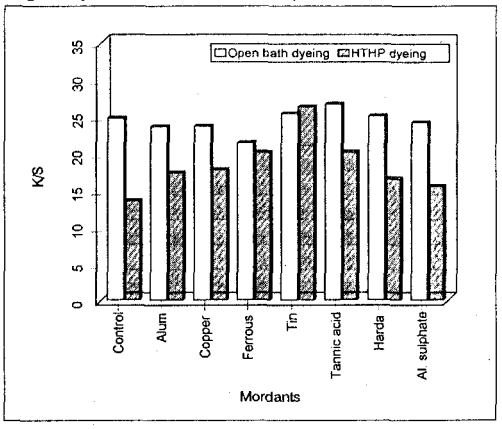
When the comparison was made between the control and the pre-mordanted shades, it was observed that alum gave a reddish mustard shade, which when compared with the control, turned out to be darker, stronger and greener, while copper produced a much brighter mustard color which was darker, stronger and yellowish as compared to the control.

Iron gave a very dark but bluish shade, while tin produced lighter, stronger and greenish shades. Harda and tannic acid gave mustard shades, which were darker with lower saturation values, while aluminium sulfate gave lighter and greener shades with high saturation values.

Table II describes the effect of various mordants on CIELAB values and fastness properties for dyeing of nylon with Onion in HTHP. When the dyeings after mordanting were compared with the control, it is clear that iron showed a highest total color difference (DE* = 13.39) followed by tannic acid (DE* = 9.36), tin (DE* = 8.86) and copper (DE* = 7.94), while other mordants gave marginal color difference.

The higher DE* values given by copper, iron and tannic acid are due mainly to lower values of L*, a* and b* and that of tin is mainly due to higher values of L*, a* and b*.

Figure 5–Effect of mordants on color values of dyeing of nylon with turmeric in open bath and HTHP.



All the dyed samples were subjected to the determination of wash, light and sublimation fastness properties. Mordanting with copper and iron showed a slight improvement in the light fastness (from 2-3 to 4), while the rest of the mordants had no effect on light fastness. It is also known that, the stronger the dye-fiber bond, the easier is the transfer of the excitation energy from the dye molecule to the fiber macromol-

ecular chain.

Thus, the dye-fiber bond serves as a bridge for transferring the excitation energy between the two components of the dye fiber ambient system. If this bond promotes energy transfer, the light fastness increases. The increase in the lightfastness after mordanting with iron and copper may be due to the easier formation of the dye-metal-fiber bond. Washing (4) and sublimation fastness (4) of all the dyeings were quite good.

The effect of mordants on color values of nylon dyed with Onion in open bath as well as HTHP is shown in Figure 3. Although, mordanting has a marginal effect on the color values, it gave a variety of shades ranging from dull yellow to light brown in open bath dyeing and mustard to dark coffee brown in HTHP dyeing. For a given dye concentration, HTHP dyeing gave darker shades, compared to the open bath dyeing on nylon.

Figure 4 shows the effect of mordants on color values of nylon dyed with Lac in HTHP. Lac did not show any appreciable dyeing on nylon in the open bath method. Among the mordants used in Lac dyeing, alum showed the highest color value (K/S=6.46) followed by iron, while, tin gave marginal increase in the color value as compared to the control, which happens to be 4.85.

The effect of various mordants on CIELAB values and fastness properties for dyeing of nylon with Lac in HTHP is given in Table III. Among all the dyeings, the samples dyed with iron and alum showed a marginal decrease in the L values, indicating darkening of shades as compared to the control dyeing.

The highest total color difference values obtained with tin ($DE^* = 23.76$) followed by alum ($DE^* = 13.43$) are due mainly to the higher values of a^* and b^*; that of iron ($DE^* = 12.09$) is due mainly to the lower values of a^* and b^*. Mordanting with alum produced a bright strawberry shade, which was a little lighter, stronger and redder; iron gave a blackish brown, which was darker, very weaker and greener; tin gave a yellowish strawberry shade, which was a little lighter, redder with high saturation value as compared to the control.

The fastness ratings given in Table III indicate that the highest rating for light fastness was noticed with iron (from 2-3 to 3-4), while other samples showed no change in lightfastness. Washfastness and sublimation fastness ratings for all the samples were good-to-excellent (4-5).

Table IV gives the effect of various mordants on CIELAB values and fastness properties for dyeing of nylon with Turmeric in open bath. Iron showed a maximum color difference followed by copper and tannic acid, which is attributed to the lower values of a^* and b^*.

The effect of mordants on CIELAB values and fastness properties in HTHP dyeing is shown in Table V. In this case, dyeing with tin showed a higher total color difference ($DE^* = 16.92$). The high value is due mainly to the lower values of L^* and b^* and higher values of a^* as compared to the control.

When all the samples were subjected to light, wash and sublimation fastness evaluation, it was observed that all samples showed good washing fastness (4) and good-to-excellent sublimation fastness (4-5). The samples mordanted with copper and iron and tin showed a fair light fastness rating (3), while those mordanted with the rest of the mordant studied gave poor lightfastness (2).

Figure 5 gives the effect of mordants on color values of nylon dyed with Turmeric by both dyeing techniques. In the case of open bath dyeing, the mordants-tannic acid (K/S = 26.52), tin (K/S = 25.23) and harda (K/S = 24.90)-showed slightly higher color values as compared to the control (K/S = 24.63), whereas, other mordants (for alum K/S = 23.47; copper K/S = 23.50; iron K/S = 21.30, Aluminium sulphate K/S = 24.02) gave a slight decrease in the color values as compared to the control (K/S = 24.63). On the other hand, all the mordants studied gave higher color values, when nylon was dyed in HTHP dyeing machine. In the case of open bath the shades obtained with mordanting were bright and were in the range from yellow to reddish-yellow, while HTHP produced duller shades.

Table VI gives the data on the effect of mordants on λ_{max} values of dyeing of nylon with the three natural dyes studied. Different natural dyes showed different λ_{max} values corresponding to the mordants used.

Compared to dyeing of polyester with the above three natural dyes, dyeing of nylon showed higher color values and higher total color difference10, possibly due to lower Glass Transition Temperature (Tg) and more open structure of nylon fiber as compared to the polyester.

Conclusions

The natural dyes Onion, Lac and Turmeric studied in this work, showed a higher dye uptake under acidic conditions. The dyeing of nylon in HTHP showed a better dye uptake than that of open bath dyeing. All the dyes used gave a variety of shades after mordanting with different mordants. The shades produced differed from mordant to mordant. Mordanting with iron gave dull shades, while alum and tin gave brighter shades. Higher ratings for light fastness was conferred with copper and iron mordanting (4 for Onion, 3-4 for Lac and 3 for Turmeric) as compared to the control (2-3). The wash fastness and sublimation fastness of all the dyeings with mordants were quite good (4-5). The mechanism of dyeing may be solid-solution which is preceded by the and electrostatic attraction between dye molecules and positively charged end amino groups of the nylon fiber in the acidic medium. □ □ □

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