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Process Optimization for Application of Tea Extract (TANNIC Acid) Using Henna (Lawsonia Inermis) Dye on Cotton Fabric

Meena Batham, Bhupinder Kaur* & Suveen Kaur
Department of Fabric and Apparel Science, Institute of Home Economics, University of Delhi, New Delhi

Abstract
Natural dyes and mordants are increasing in demand due to growing environmental awareness among people. This study is focused on application of tannins extracted from tea as mordant on cotton fabric and dyed with henna. The extraction of tannins was done using a hydrous extraction process. For the extraction commercially available fresh and used tea leaves were used. Different parameters were studied to optimize the condition of mordanting cotton fabric with tea extract. Concentration, time and temperature were varied and then finalized based on visual evaluation and color fastness properties of these fabrics when dyed with henna. Both pre mordanting and post mordanting methods were used. The results produced with fresh tea tannin were better than fabrics mordanted with used tea tannin. Also the fastness properties of the henna dyed fabrics were found to be average to good.

Keywords: *Dye, Mordant, Natural dye, Tannin, Tea*

1. Introduction

India has been considered as the initiator of the art of textile dyeing with natural colorants. This is evident from Ajanta, Ellora, Sithannavasal and Mithila wall paintings. It is also mentioned in Vedas that the main colorants used were red, yellow, blue, black and brown. The craftsmen used lac, safflower and madder for red, cutch for brown, indigo for blue and turmeric and saffron for yellow. Moreover, the Egyptian pyramids are said to have overdone with natural colorants and so are the Spanish caves of Altamira and Elcatillo as well as the French Pyrene caves of Niaux (Prabhu, 2012).

2. Review of literature

There are various studies showing extraction of tannins from natural sources, using them as mordant and use of henna as dye.

Khasnabis (2015) in her study of determining tannin content in different types of tea prepared the extract using the hydrous extraction process. The solution containing tea and distilled water was heated in a magnetic stirrer at 70ºC for 5 minutes. The solution was then filtered. The filtrate was centrifuged at 10000 rpm for 15 minutes. Tsujimura (2014) had prepared a fine powder of green tea and extracted tannins at ordinary temperature using ethyl acetate with 10% water. The prepared solution was evaporated and further treated with various chemicals such as lead acetate, sulphuric acid, ether, chloroform and a few more. After treatment with these chemicals, the extracted was evaporated and powdered tannic acid was obtained. Hoyos-Martinez (2018) did research on different ways of extraction of tannins from various sources which narrowed down to seven different ways. These methods include extraction with organic solvents, hot water, ionic liquids, supercritical fluids, subcritical water, microwave and ultrasound. Muramatsu (1986) prepared a crude catechin mixture for his study on field rats. The mixture was prepared from spray dried Indian green tea powder. The tea was dissolved in hot water and extracted

three times with ethyl acetate, which was later evaporated. The resulting concentrated solution was freeze dried to yield a crude catechin mixture.

Ding (2017) used a pre-mordanting method in which he used 300ml tannic acid solution (1.5g/l) at 70ºC. The fabrics were agitated for 2h while the temperature dropped to room temperature. The fabrics were then rinsed and placed in a solution containing a mixture of aluminum sulphate and soda ash. This solution was agitated for 2h and the fabrics were dried and used for further dyeing. Rahman (2016) on dyeing cotton with basic dyes mordanted the samples with tannic acid. The fabrics were immersed in a boiling tannic acid solution of 4% concentration and maintained at 60ºC for 2h. The samples were then squeezed and added to a 2% tartar emetic bath for 30 minutes for tannic acid fixation. The samples were squeezed and used for dyeing.

Togo and Komaki (2009) in dyeing cotton with lac dye mordanted fabric in tannic acid solution (0-5 g/l). The fabric was entered in 50ml of this solution at 60ºC and boiled. The heating continued for 10 minutes. The fabric was rinsed with deionized water and dried for later use in dyeing. Ali *et al* (2010) used tannic acid as mordant for dyeing cotton samples with *Acacia nilotica* bark. The samples were treated in a 4% tannic acid solution with ML ratio of 1:15 at 60ºC for 1 hour. The samples were rinsed in cold water, dried and used for dyeing.

Hasan *et al* (2015) did their research on applying henna on cotton and silk fabric. The mordants used in the study were tartaric acid, copper sulphate and alum. During the optimization process they concluded that a dye bath of 4% henna (o.w.f.) maintained at 70ºC at a pH of 7 for 45 minutes is best suited for dyeing. Bhuyian *et al* (2018) during their research on dyeing polyester with natural dye henna found out that dyeing polyester at a higher temperature of 130ºC with a material-to-liquor ratio of 1:40 is optimum for dye exhaustion. The concentration of dye used was 20% o.w.f. After dyeing the samples were washed in 1gpl soaping agent at 60ºC for 10 minutes and rinsed in cold water.

Bhuyian (2017) in his review regarding chemistry and application of henna stated that henna on cellulosic fiber produces best results in neutral to slightly alkaline medium at 70ºC maintained for 100 minutes with a ML ratio of 1:30. Ebrahimi and Gashti (2015) did their research on extraction of natural dyes from henna, pomegranate rind and *Pterocarya fraxinifolia*. The dyeing process was started at 40ºC which was raised to boil in 20 minutes and held at boil for 1 hour. MLR was maintained at 1:40 and pH was kept at 9 by addition of sodium hydroxide. Yusuf *et al* (2011) in studying dyeing with henna and madder with and without tin chloride as mordant prepared two sets of samples. Both mordanted and un-mordanted samples were soaked in water for 30 minutes and then immersed in a dye bath with liquor ratio 1:40. The pH of the bath was maintained at 7 and temperature at 91-93ºC. Dyeing was done for 1 hour.

3. Methodology

3.1 Material used

Fabric selected for the study was cotton. Plain weave cambric of 80 GSM was used. Black tea of the brand Taj Mahal was used for extraction of tannins to be used as mordant in the study. Both fresh and used tea samples were made of this tea only. The tea was acquired from a local vendor in the local market. *Lawsonia inermis* (henna). The dye was obtained from the west zone of Delhi, from a local park in the form of leaves and was then prepared in powder form manually.

3.2 Method

- **To extract tannic acid from commercially available tea leaves and used tea leaves -** Extraction of tannins from tea leaves using hydrous extraction process.
- **To optimize the process of mordanting on cotton fabric -** Pre-treatment of the fabric selected -Desizing of cambric fabric. Application of mordant on the fabric – Pre-mordanting and Post-mordanting.
- **To study the effect of tannic acid as mordant (fresh and used tea) on developed shades using standard henna dye -** Determining the best process to apply henna dye on the mordanted fabric.
- **To compare the color strength and fastness properties of the dyed cotton fabrics -** Analyzing the wash and rub fastness of the dyed fabric and determining the color strength of the dyed samples.

4. Results and Discussion

4.1 Pre -treatment of the fabric selected

The pre-treatment of the fabric was done by desizing of the cambric fabric. This was done so as to remove any kind of starch or impurities present on the surface of the fabric and thus preparing it for dyeing. The iodine test was done to check the

presence of starch on the desized fabric. The results of the iodine test demonstrated that when a few drops of iodine were put in the water on boiling, the water did not turn blue. Hence, it can be concluded that the starch was removed completely from the fabric.

4.2 Application of tannin as mordant on fabric

The extracted tannin solution from tea leaves was used as mordant on cotton fabric. Both pre mordanting and post mordanting methods were used.

4.3 Optimizing mordanting parameters

The conditions of mordanting were standardized by studying the effect of time, temperature of mordanting bath and concentration of mordant used. The desized cotton fabric samples were mordanted with the tea tannin extract. Difference in parameters showed visual differences in appearance of the mordanted fabric samples. The samples kept in the tea bath for a lesser period of time, entered at lower temperature or with a lesser amount of tea in the solution showed lighter brown tints whereas the increase in these conditions resulted in medium browns. However, the parameters for optimizing the mordant bath were not selected on the appearance of mordanted samples but based on the visual evaluation as well as fastness results once these samples were dyed with henna. Results of various parameters are discussed below;

Concentration of tea leaves- Different concentrations of tea leaves were taken i.e. 3g, 5g and 7g. Time and temperature at which the samples were mordanted were kept constant i.e. 30 min at 100ºC. The shades obtained on fabrics were as shown in table 1.1.

Table 1.1 Concentrations of fresh tea leaves used for mordanting

Concentration	Time—30 min, Temperature - 100ºC	
	Pre-mordanting	Post-mordanting
3g		
5g		
7g		

As per above shade card, shades produced by pre-mordanting the samples are brighter and lustrous than the shades produced by samples that were post-mordant. It was also observed that out of the three concentrations taken, the sample mordanted with 5g tea produced better results than the samples mordanted with 3g and 7g tea.

Temperature - Finalized concentration of tea leaves was taken and then the samples were treated at different temperatures i.e. room temperature, 70ºC and 100ºC. Time duration for the treatment process was kept constant at 30 min. The shades obtained on fabrics were as shown in table 1.2.

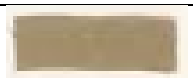
Table 1.2 Temperatures used for mordanting process with fresh tea leaves

Temperature	Concentration - 5g, Time—30 min	
	Pre	Post
Room temperature		
70ºC		
100ºC		

As per above shade card, it was observed that the pre-mordanted samples gave better shades than the post-mordanted samples at different temperatures of mordanting. It was also observed that out of the three temperatures at which the samples were entered in the mordanting bath, i.e. room temperature, 70ºC and 100ºC, the sample mordanted at 100ºC produced the brightest shade.

Time -Concentration of tea leaves and temperature were kept constant using 5g tea leaves at 100ºC for different time durations of 15min, 30 min and 45 min. The shades obtained on fabrics, shown in table 1.3.

Table 1.3 Time duration of mordanting with fresh tea leaves

Time	Conc- 5g, Temp - 100ºC	
	Pre	Post
15 min		
30 min		
45 min		

As per above shade card, it was evaluated that the shades produced by post – mordanting the samples are duller than the shades produced by pre-mordanting the samples. On comparing the samples further, it was observed that the sample mordanted for 30 min produced better shade than the samples mordanted for 15 min and 45min. Although the shades produced by mordanting the sample for 30 and 45 min were similar, 30 min was chosen as the time for mordanting due to its economic viability.

After visually evaluating the results of color shades obtained with different parameters, a set of conditions were finalized for optimizing the mordanting process by use of tannins extracted from tea leaves for both pre mordanting and post mordanting.

After visually evaluating the results of color shades obtained with different parameters, a set of conditions were finalized for optimizing the mordanting process by use of tannins extracted from tea leaves for both pre mordanting and post mordanting (table 1.4 and 1.5).

Table 1.4 Final recipe for mordanting with fresh tea leaves

Concentration – 5g Time – 30min, Temperature 100ºC		
Control	Pre mordanting	Post mordanting
		

Table 1.5 Mordanting with used tea leaves

Control	Pre mordanting	Post mordanting
		

4.4 Studying the effect of tannic acid as mordant (fresh and used tea) on developed shades using standard henna dye

On pre-mordanting the samples with tea extract and then dyeing with henna, the shades produced were tints of khaki-green color rather than orange-brown color. The cotton fabric pre-mordanted with used tea tannin solution by opting standardised set of conditions produced a very light khaki-green shade.

On dyeing the samples first with henna without any mordants and then post mordanting with tannins extracted from fresh tea, the color produced were shades of khaki-brown. The dyed samples were somewhat green before immersing them in tannin solution for post mordanting. The khaki brown shades achieved after post mordanting might be due to the brown color of the tea extract. Although, cotton fabric dyed henna and post mordanted with tannin extracted prepared by used tea resulted in very light brown color.

4.5 Comparison of the fastness properties of the dyed cotton samples

Further, the comparison was done of the fastness properties of the dyed sample (shown in table 1.6 and 1.7).

Table 1.6 Colour Fastness of fabrics mordanted with fresh tea tannin extract and dyed with henna

	Wash			Rub		Light
	Original fabric	Second fabric	Wash fastness	Color change	Staining	
Pre	4.5	4	4.5	4.5	4	3.5
Post	4	4	4	4	4	3

Table 1.7 Colour Fastness of fabrics mordanted with used tea tannin extract and dyed with henna

	Wash			Rub		Light
	Original fabric	Second fabric	Wash fastness	Color change	Staining	
Pre	3.5	3	3	3.5	3	3
Post	3	3.5	3	3.5	3	3

5. Conclusion - As per above results, it is possible to mordant cotton fabric with commercially available tea leaves and then dyed with henna. Products made with these ingredients will be fully natural and could bring the consumer a high level of satisfaction.

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