Extraction of Tannin as a Natural Mordant from Tea Leaf for Dyeing of Cotton Cloth

Khin Htay Win[1], Thidar Khaing[2], Htun Htun Naing[3], Yinn Kay Khaing[4]

Abstract

Tea leaf is consumed by more than two thirds of the world's population and tea is the most popular beverage after water. This drink has been consumed for thousands of years and this long safety record presents its medicinal properties and achieve chemical constituents such as tannins, caffeine, enzymes and essential oils. In this research, tea leaf was selected for extraction of tannin. Firstly, the preliminary detection of phytochemical compounds present in the tea leaf was investigated. The mineral content of tea leaf was analyzed by Energy Dispersive X-ray Fluorescence (EDXRF) spectroscopy. Extraction of tannin as a natural mordant from tea leaf was made by using hot aqueous method. The qualitative analysis of tannin was carried out with various reagent. The extracted tannin was used as a natural mordant alone and in combination with metal mordant for cotton. It was also used together with natural dyes namely the beetroot and the leaves of Hin-nu-new. The dye solution of two samples were extracted by using aqueous method. The pre-mordant fabric on dyeing gave better colour strength than the dyeing obtained without mordanting. Finally, rubbing fastness and washing fastness tests were done to predict the performance of dye in cotton cloth.

Keywords -Tea leaf, EDXRF, tannin, pre-mordant, rubbing fastness, washing fastness

Introduction

The tea plant, *Camella sinensis*, is a member of the Theaceae family, and black, Oolong, and green tea are produced from its leaves. Tea plant is small evergreen shrub cultivated reach the height of 7 to 8 feet. Tea fruit is a smooth, flattened, rounded, trigonous three-celled capsule; seed is solitary in each cell and has size of a small nut.

Tea contains caffeine, essential oils, enzymes, tannins and phenolic compounds. Green tea is produced from steaming fresh leaves at high temperatures, thereby inactivating the oxidizing enzymes and leaving the polyphenol content intact. The polyphenols found in tea are more commonly known as flavanols or catechins and comprise 30 - 40 percent of the extractable solids of dried green tea leaves. Green tea polyphenols have demonstrated significant antioxidant, anti-carcinogenic, anti-inflammatory, and antimicrobial properties in numerous human, animal, and in vitro studies. [4]

Tannins are defined as naturally occurring water soluble poly phenolic compounds of high molecular weight (about 500–3000) containing phenolic hydroxyl groups to enable them to form effective cross links between proteins and other macromolecules. Tannin is an astringent vegetable product found in a wide variety of plant parts such as bark, wood, fruit, fruit pods, leaves, roots and plant galls. They are largely used in medicine, preparation of inks, preservation of lather, food industry and in the dyeing process The use of mordant in dyeing not only increase the dye and colour fastness but the use of different mordant on a natural dye can yield different colours and shades [3].

[1] Lecturer, Department of Chemistry, University of Mandalay khinhtaywin.6mu@gmail.com
[2] Lecturer, Department of Chemistry, University of Mandalay
[3] Lecturer, Department of Chemistry, University of Mandalay
[4] Associate Professor, Department of Chemistry, University of Myitkyina

There are two types of dyes: natural dyes and synthetic dyes. Natural dyes are biodegradable, non-toxic and generally have higher compatibility with the environment when compared with their synthetic counterparts. Natural dyes are dyes or colorants derived from plants, invertebrates or minerals. Almost all parts of the plants like root, bark, leaf, fruit, wood, seed, flower, etc produce dyes.

Although, natural dyes have several advantages, there are limitations as well. Tedious extraction of colouring component from the raw material, low colour value and longer time make the cost of dyeing with natural dyes considerably higher than with synthetic dyes. Some of the natural dyes are fugitive and need a mordant for enhancement of their fastness properties.

The objectives of this study were to extract tannin from tea leaf and its application as a natural mordant alone and in combination with metal mordant, namely copper sulphate for dyeing cotton, using natural dyes namely beetroot and the leaves of Hin-nu-nwe.

Botanical Description

Scientific Name : *Camellia sinensis* L.
Family Name : Theaceae
Local Name : Letphet
English Name : Tea plant
Part used : Leaf

Figure 1. Plants of Tea

Family name : Amaranthaceae
Botanical name : *Amaranthus viridis* L.
English name : Spinach
Myanmar name : Hin-nu-nwe
Part used : Leaves

Figure 2. Plant of *Amaranthus viridis* L.

Family name : Amaranthaceae
Botanical name : *Beta vulgaris*
English name : Beetroot
Myanmar name : Tha-gyar-moanlar
Part used : Rhizome

Figure 3. Beetroot

Materials and Methods

Materials

Tea leaves were collected from Taungyi Township, Shan State. Tea leaf was made into powder by grinding machine and then stored in plastic container before use. Beetroot and Hin-nu-nwe were collected from local market, Mandalay Township. Cotton cloth was purchased from Yadanabon Market, Mandalay. Commercial grade reagents were used for research work.

Method

Preliminary Phytochemical Screening

Preliminary phytochemical tests were carried out on tea leaf powdered according to the reported methods [2]. Tea leaf sample was tested by phytochemical screening to determine the presence or absence of organic constituents.

Determination of Mineral Contents

Mineral contents of tea leaf was measured at Department of Chemistry, Monywa University by applying EDXRF spectroscopy method.

Extraction of Tannin

The finely powdered tea leaves (10 g) was extracted with water (120 ml) at boil for 1 hour and after cooling it was filtered through a fine muslin cloth and the filtrate was collected separately. The remaining residue was extracted three more times, in order to complete the extraction. The total extract (200 ml) was heated to boil and was allowed to stand overnight and filtered again. The clear filtrate was concentrated in a water bath and treated with saturated brine solution. A brownish coloured precipitate thus obtained was filtered and dried in an oven.

Qualitative Analysis for Tannin

The qualitative analysis was carried out by treating 0.5% solution of the above tannin product with various reagents such as gelatine, aqueous ferric chloride, lead acetate, copper sulphate solution, dilute hydrochloric acid solution and dilute sulphuric acid solution. The colour change after the addition of reagent was observed [6].

Extraction of Dyes Solution from Beetroot and the Leaves of Hin-nu-nwe

In this research work, dye solution was extracted from beetroot and the leaves of Hin-nu-nwe by using aqueous method. Distilled water only was used for extraction solvent. 10 g of fresh sample was boiled with 300 ml of distilled water about 30 mins. And then, the dye solution was cooled for a few minutes and filtered. Finally, the filtrates were used in dyeing process [9].

Figure 4. Beetroot dyed solution

Figure 5. Hin-nu-nwe dyed solution

Bleaching of Cotton Cloth

Firstly cotton cloth was washed with tap water containing 2 drops of detergent and soaked in a bath made of four parts cold water to one part vinegar for at least 30 minutes. Then, it was taken out of the sink, run under cold water and wring out of a bit of excess water.

Figure 6. Bleaching of Cotton Cloth

Mordanting

Pre-mordanting technique was used for this study. Cotton cloth was treated with 2% tannin solution on weight of fabric at (70ºC) for 45min, keeping the material to liquor ratio 1:40. The tannin treated cotton was further treated with 1% of copper (II) sulphate solution at 70ºC for 45 min, keeping the material to liquor ratio as 1:40. Cotton cloth, only mordanted with tannin and in combination with mineral mordant, were squeezed and subjected to dyeing [5].

Dyeing Method

Cotton cloth with and without pre-mordanting were introduced into the beetroot dye and the leaves of Hin-nu-nwe dye solution at room temperature at a liquor ratio of 1:40 and slowly the temperature was raised to 70ºC. After dyeing, the cotton was lift out and squeezed by hand. They were rinsed in cold water to remove unfixed dyes and dried in air. Rinsing process was done by one time [1].

Determination of Rubbing Fastness and Washing Fastness of Dyeing Sample

The dried sample was sent to Development Center for Textile Technology, Ministry of Industry, Yangon, Myanmar to measure the color fastness to rubbing test and color fastness to washing test [8].

Results and Discussion

In this section, the result obtained from the experimental work such as phytochemical tests, mineral contents, qualitative analysis of tannin, dyeing process and determination of rubbing fastness and washing fastness of dyeing sample were discussed.

Preliminary Phytochemical Tests

Preliminary phytochemical tests were done to investigate the presence or absence of chemical constituents in tea leaf. According to the phytochemical examinations alkaloids, steroids, flavonoids, polyphenol, saponins, reducing sugar, glycoside and tannins were found to be present in the sample. Among these compounds, tannins play an important role in dyeing process because it can be used as a natural mordant to sustain coloring matter permanently.

Mineral Contents of Tea Leaf

The mineral contents of tea leaf were determined by using EDXRF at Department of Chemistry, Monywa University.

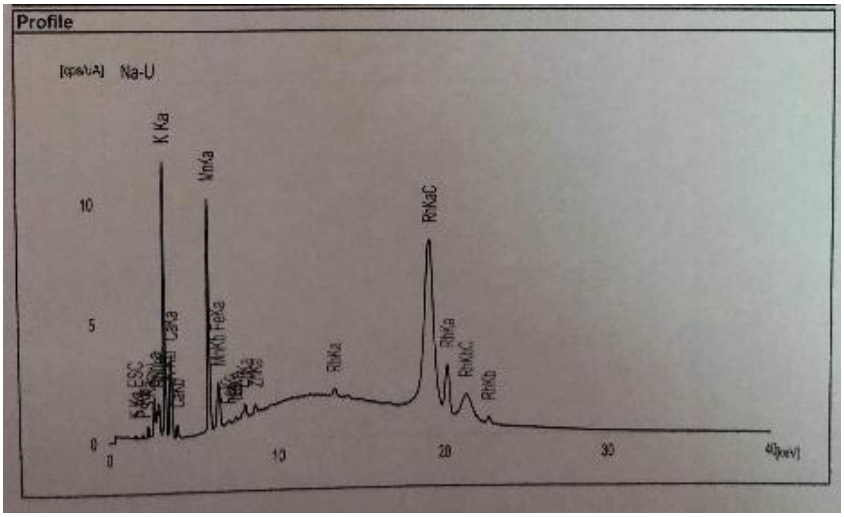

Figure 7. EDXRF Spectrum of Tea Leaf

Table (1) Elemental compositions of the Tea Leaves

Element	Symbol	Result Abundance
Potassium	K	0.967
Calcium	Ca	0.171
Phosphourus	P	0.170
Sulfur	S	0.164
Manganese	Mn	0.080
Iron	Fe	0.005
Copper	Cu	0.002
Zinc	Zn	0.001
Rubidium	Rb	0.001

According to the EDXRF result, the tea leaf do not contain any toxic heavy metals.

Qualitative Analysis of Tannin

In order to investigate the class of tannin obtained from tea leaf, a set of qualitative experiments were carried out with various reagents and their results are given in Table 2. It produced dirty white precipitate with 2 % gelatin, a dark green precipitate with aqueous ferric chloride and dark red solids with dilute HCl solution. It clearly confirms that tea leaf extract is condensed tannins.

Table (2) Qualitative Analysis of Tannins

Reagents	Observation
2% Gelatin	Dirty white precipitate
5% Aqueous ferric chloride	Dark green precipitate
10% Lead acetate	Pinkish precipitate
Copper (II) sulphate solution	Faint green
Dilute hydrochloric acid solution	Dark red solids
Dilute sulphuric acid solution	Flesh coloured precipitate

Dyeing Process

The dyeing process includes three major steps; first begins the extraction of colouring matter from beetroot and the leaves of Hin-nu-nwe, second is mordanting the cotton cloth and the last is dyeing. Three different color of dyeing products were obtained. The color of dyeing cotton cloth are shown in following Table (3).

Table (3) Color of Dyeing Cotton Cloth with Without Mordant, 2 % Tannin and 2 % Tannin + 1 %$CuSO_4$

Dye Solution	Without Mordant	2 % Tannin	2 % Tannin + 1 % $CuSO_4$
Beetroot			
Leaves of Hin-nu-nwe			

Rubbing Fastness Properties of Dyeing Products

The sample has to be tested in the experimental condition. The fabric/sample has to be air conditioned at least 8 hours by standard climate (20°C/ 65 %. relative humidity) without washing the fabric. The air conditioned specimen has to be rubbed with the dry and wet cotton rubbing cloth in warp, weft or diagonal direction of face side. The results are shown in Table (4).

Table (4) Results of Rubbing Fastness of Dyed Cotton Cloth

Dye Solution	Type of mordant	Rubbing Fastness 500 g; 100 Times	
		Cotton Cloth	
		Dry Mark	Wet Mark
Beet Root	Without Mordant	4	3
	2 % Tannin	4	3
	2 % Tannin + 1 % $CuSO_4$	4	2-3
Leaves of Hin-nu-nwe	Without Mordant	4	3
	2 % Tannin	4	3
	2 % Tannin + 1 %$CuSO_4$	4	3

The change in color of the sample was compared with standard scale. Cotton cloth are acceptable and good condition for dry test and fair condition for wet test.

Washing Fastness Properties of Dyeing Products

This test determines the loss and change of colour in the washing process by a consumer and the possible staining of other garments or lighter portion that may be washed with it. This test is used to predict the performance of any dyed or printed textile product to the common washing process using a detergent and additives.

Table (5) Results of Washing Fastness of Dye Cotton Cloth

Dye Solution	**Type of mordant**	**Washing Fastness ISO. Test 3 60°C; 30 mins**
		Change of Shade
		Cotton Cloth
Beetroot	Without Mordant	1
	2 % Tannin	2
	2 % Tannin + 1 %$CuSO_4$	2
Leaves of Hin-nu-nwe	Without Mordant	1
	2 % Tannin	2
	2 % Tannin + 1 %$CuSO_4$	2

Natural dyes are among the promising options for developing a greener textile dyeing process. However, the major drawback of the natural dyed-textiles is their poor colour fastness to light and washing fastness. Incorporation of the mordants in natural dyeing can help to improve such inferior properties and at the same time, provides a wide variety of shades of natural dyes obtained on the same textile substrates.

Conclusion

In this research work, the tea leaf was selected for phytochemical analysis and extraction of tannin as a natural mordant for dyeing of cotton cloth with natural dye. According to the phytochemical screening the tea leaf contains tannin. Tannin play an important role in dyeing process because it can be used as a natural mordant to sustain coloring matter permanently. According to the EDXRF result, the tea leaf does not contains any toxic heavy metals.

Tea leaf extract can be successfully employed as a natural mordant for dyeing cotton fabrics with natural dyes. Tea leaf mordant alone, and in combination with copper sulphate mordant and natural dyes, dyed cotton fabrics showed higher washing fastness as compared to only natural dyes dyed cotton fabrics. Therefore, the extracted tannin was found to be suitable for a mordant in dyeing process. The process is promising and should be easily industrialized, and popularized among the local natural dyes in order to obtain an eco-friendly product with value added properties.

References

[1] Ashis Kumar Samanta, Pubalina Samanta and Deepali Singhee, (2017), "Fundamentals of Natural dyeing of Textiles: Pros and Cons," Volume 2-Issue 4-February 2017.

[2] Harbone JB, Methods of Plant analysis. In: phytochemical Methods (Chapman and Hall, Landon). 1973.

[3] Katie, E, and Richard, W. (2006), "Squirrels: the animal answer guide", John Hopkins University Press. p. 91.

[4] Kulkarni, S.S, *et. al.,* "Cotton Dyeing with Natural Dye Extracted from Pomegranate Peel," *Universal Journal of Environmental Research and Technology*, 2011, *1*(2), 135-139.

[5] Nic, M., *et al.* Mordant. IUPAC Compendium of Chemical Terminology, 2005.

[6] Prabhu, K.H., and Teli, M.D, "Eco-dyeing Using Tamarindus indica L. seed coat tannin as natural mordant for textiles with antibacterial activity", *Journal of Saudi Chemistry Society*, 2011, King Saud University.

[7] Ramakrishnan, K., Selve, S.R., Shubha, R., Tannin and its analytical techniques, Indian Chemical Engineering, Section A 48 (2), 88-93, 2006.

[8] Samanta, A.K. "Application of Natural Dyes on Textiles", *Indian Journal of fibre & Textile Research*, 2009, 34, 384-399.

[9] Saravanan P. and Chandramohan G. (2011). Dyeing of silk with Eco-friendly Natural dye obtained from Barks of ficus religlosa L. Universal Journal of Environmental Research and Technology, 1 (3) : 268 - 273.