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Colour Fastness of Cashmere Wool Dyed with *Phyllanthus muellerianus* Natural Dye

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Abstract: The quantitative dye content of *Phyllanthus muellerianus* leaves grown in Enugu State University of Science and Technology, Agbani was evaluated. After successive solvent extraction with acetone and water (50/50 [V/V]) of powdered dry leaves, partitioning with n-hexane, chloroform, ethyl acetate and n-butanol were carried out. Two main dyes were obtained from the ethyl acetate fraction which was identified as anthocyanin and chromene. The dyeing condition for wool with the dye were carried out at 100°C for 1 hour in dye bath containing 3% dye, 8% sodium sulphate and 4% acetic acid at liquor ratio 1:50. The post mordanting was done with various metallic salts. The colour strength of the dye on the wool were found to be highly dependent on the type of mordant, optimum results being obtained with $CuSO_4$ and $K_2Cr_2O_7$. The K/S of the dyed wool revealed that $CuSO_4$ have more stable colour strength than $K_2Cr_2O_7$, tin chloride and alum. Copper sulphate mordanted samples have good light and washing fastness than $K_2Cr_2O_7$ (rating 4.2)
Keywords: *Phyllanthus muellerianus*; cashmere wool; colour fastness; mordants; light fastness; wash fastness.

INTRODUCTION

The use of natural dyes such as madder, flame of the forest, indigo, etc. on textiles has become a matter of significant importance because of growing alarm of the threats of synthetic dyes on the environment worldwide. In the recent few decades synthetic dyes have been seriously criticized and high valued natural dyes are preferred and also for their availability and good market value [1, 2].

Most natural dyes are based on vegetable origins which are agro-renewable, bio-degradable and environmentally friendly. Natural dyes produce compatible vibrant colours creating a palette that blends with each other to give various shades to colour natural fibers and art works due to the nature, and structure of the dye, the compatibility of mordants and the conditions of dyeing methods [3].

Natural dyes find application chiefly for colouration of textiles, food, drugs, and cosmetics and for colouration of wood carvings, candle, leather, paper etc [4].

Because of prolonged wearing of dyed fabrics, the toxic chemicals used in making them are often absorbed into the skin especially when the body is warm and skin pores have opened to allow absorption. Some individual are sensitive to these chemicals and therefore develop rashes when their bodies come in contact with dyed garments [5]

Recent research has focused on the adults response/reaction to these chemicals ranging from skin rashes, headaches, trouble concentrating, nausea, diarrhea, fatigue, dizziness, muscle and joint pain, difficulty breathing, irregular heart beat and/or seizures while in children they include red checks and ear, dark circles under the eyes, hyperactivity and behavior or learning problem due to their presumed hazard to humans and the environment [6, 7].

The aim of this thesis is to develop a simple and efficient dye and dyeing method that will benefit society by making use of available and environmentally-friendly materials. Because the chemical composition of these natural dyes varied, several types of natural dyes are applied to fabrics with different mordants. In this perspective, the proposed technology is based on a bottom-up method i.e. isolate, identification and find application for the *Phyllanthus muellerianus* dye. In this line, the main objective of the present study is based on the use of varying concentration of cupric sulphate, potassium dichromate, potassium alum and Tin chloride as mordants for

fixation of the dye to the wool fabrics and to experimentally determine the effect of mordant concentration on the colour strength and colourfastness to wash and light.

MATERIALS AND METHODS

Materials

Mature *Phyllanthus muellerianus* leaves collected from Agbani near Faculty of Applied Natural Sciences of Enugu State University of Science and Technology were used in this investigation. Mature leaves samples were harvested on August 2015 and taken immediately to the laboratory where they were dried in a subdued sunlight and ground into powder form with the aid of Kika Grinding Mill (Model MP300-20 A11 basic). The powder form was used to extract the corresponding dyes. Acetone, n-hexane, chloroform, ethyl acetate, n-butanol, copper sulfate hydrate ($CuSO_4 \cdot 5H_2O$, potassium dichromate ($K_2Cr_2O_7$), potassium hydroxide (KOH), acetic acid (CH_3COOH), alum and tin chloride, and Iron (II) sulfate hydrate © *CIRAT-4, 2010* (FeSO4·7H2O, MW: 278.03, Aldrich) were used without further purification. Thin Layer Chromatography (TLC) was performed on silica gel 254 plates (Merck) with UV (254 nm) visualisation whereas chromatographic separations were conducted on silica gel using columns.

Methods

Extraction of Plant dyes

The powdered leaves material (1.5 kg) was percolated in acetone and water (50/50 v/v) at the ratio of 1:3 for 48 hours and filtered. The acetone was distilled out of the filtrate and the aqueous filtrate remaining was further partitioned successively with n-hexane, chloroform, ethyl acetate and n-butanol. Each partition were concentrated using rotary evaporator. The concentrate was dried at 65^{o}C in a Pickstone Thermostatic Oven series 30/300 (Model BD/AL) to a constant weight [8].

Isolation of the plant dye

The ethyl acetate fraction was of interest and further purified by fractionating with n-hexane, chloroform, ethyl acetate and n-butanol respectively and concentrated. The ethyl acetate concentrate was subsequently purified by fractionating three more times with n-hexane, chloroform, ethyl acetate and n-butanol respectively and the ethyl acetate final fraction was labelled EA-1 (plant dye).

The ethyl acetate fraction was evaporated in vacuo. and the residue purified by column chromatography (eluted with gradient of n-hexane increasing the polarity with ethyl acetate). Eight fractions were collected and TLC analysis conducted. The two combined fraction were tested for the presence of flavonoids.

Dying of fabrics

Wool (Cashmere) samples were dyed using a dye bath containing 3 % dye, 8 % sodium sulphate and 4 % acetic acid at liquor ratio of 1:50. The dyeing of wool was performed at acidic pH by adding the required amount of acetic acid (CH_3COOH). The temperature was gradually raised from room temperature to boiling and maintained at the boiling point for 1hr [9-12].

Mordanting

The post mordanting were done by using different mordents, namely copper sulfate, potassium dichromate, alum and tin chloride with concentrations varied between 2.5, 5, 7.5, 10, 12.5 and 15 % at optimized dyeing conditions using 1% acetic acid. The process of dyeing was started at 60 ^{o}C and slowly increased to boil with gentle stirring and continued for 1 h [13, 11, 12].

Washing Fastness

This accelerated laundering test is to evaluate the washfastness of textile which are expected to withstand frequent laundering. The staining of fabric experiment was performed in a thermostatic water bath with shaker. (AATCC Test Method 61-1972). The set of standards and the specimens (5 x 10 cm) to be tested were stapled along the edge of the test specimen and in contact with the face of the standard. For the IA, IIA and IIIA tests, the appropriate amount of detergent solution (preheated) designated number of stainless steel balls were added to each beaker and clamped with cover respectively [15-19].

Colourfastness to light: Daylight

This test evaluates the colourfastness to light of dyed textiles exposed to sunlight for 24 hours a day for 22 days. The light fading experimentwere performed in an exposure cabinet with window glass of clear flat glass sheet of 6 mm thick (AATCC Test Method 16C-1971). The set of standards and the specimens to be tested were mounted on the cardboard as shown in plate 1, with the exposure area measuring 3 x 3 cm adjacent to an unexposed area having the same dimension. The specimens and standards were simultaneously exposed to light under the same condition [10, 16-19].

RESULTS AND DISCUSSIONS

Effect of test solution IA and 11A condition to washing/ staining fading of dye from wool fabric

The results of the fading experiments to determine the wash/staining colour fastness effect indicate clearly that test IA and IIA has little or no effect on the dyed fabrics mordanted with $K_2Cr_2O_7$ and $CuSO_4$ respectively.

Table-1: Effect of test solution IA and 11A condition to washing/ staining fading of dye from wool fabric

Mordant concentration (%)	Fading with test solution 1A and condition	
	$CuSO_4$	K_2CrO_7
2.5	4.5	4.5
5	4.5	4.5
7.5	4.5	4.5
10	4.5	4.5
12.5	4.5	4.5
15	4.5	4.5

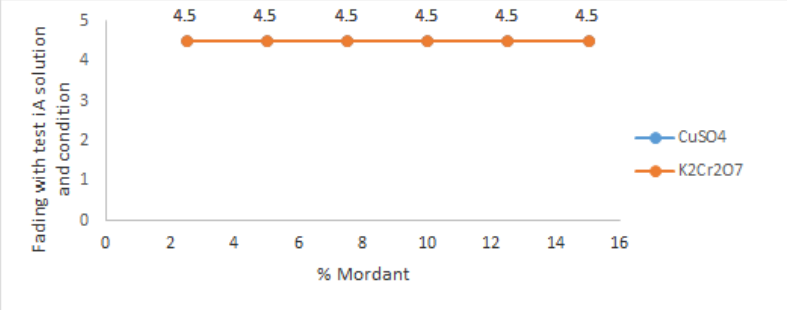

Fig-1: Effect of test solution IA and 11A condition to washing/ staining fading of dye from wool fabric

In the result of test IIIA where 100 numbers steel balls with higher concentration of detergent was used, there was therefore, faster fading of the wool fabric mordanted with $CuSO_4$ (57%) than in the $K_2Cr_2O_7$ (92%) mordanted. This greater degree of fading obtained with $CuSO_4$ mordanted could be attributed to higher temperature, concentration and number of steel balls involved.

Table-2: Effect of test solution IIIA and condition to washing/ staining fading of dye from wool fabric

Mordant concentration (%)	Fading with test solution 111A and condition	
	$CuSO_4$	K_2CrO_7
2.5	4	2.5
5	4.5	2.5
7.5	4.5	3
10	4.5	3
12.5	5	3
15	5	3

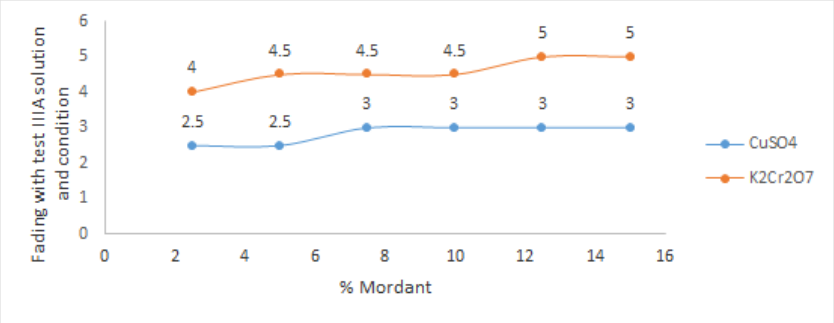

Fig-2: Effect of test solution IIIA and condition to washing/ staining fading of dye from wool fabric

Effect of exposure to light on fading of dye from fabric after 528 hours (22 days)

Difference in fading of dyed wool fabric on exposure to light in 'exposure cabinet. The potassium dichromate ($K_2Cr_2O_7$) mordanted wool fabric exposed to light in the 'exposure cabinet' lost about 2/3 (67%) of their colour after 528 hours (22 days) while copper sulphate ($CuSO_4$) mordanted wool fabric lost less than 1/5 (20%) of their colour after 528 hours (22 days) exposure to light. The fading of dyed wool fabric mordanted with $K_2Cr_2O_7$ become more noticeable after 192 hours exposure (8 days). The difference in effect of colourfastness to light of dyed wool fabric between the two mordants ($K_2Cr_2O_7$ and $CuSO_4$) is quite big.

Table-3: Effect of exposure to light on fading of dye from fabric after 528 hours (22 days)

Time (h)	Fading to staining with10% calcium hydroxide	
	Average of $CuSO_4$	Average of K_2CrO_7
0	4.7	4.5
24	4.7	4.4
48	4.7	4.4
96	4.7	4.3
192	4.5	4.1
288	4.3	3.9
384	4.3	3.4
528	4.2	2.7

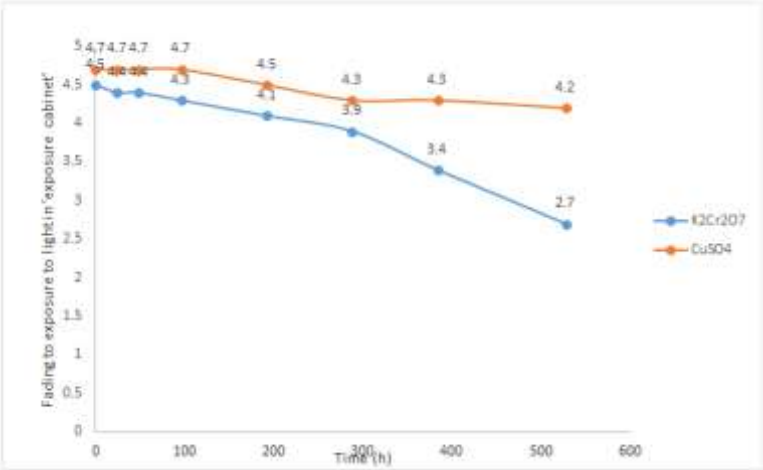

Fig-3: Effect of exposure to light on fading of dye from fabric after 528 hours (22 days)

This is probably is due to the fact that fading depends to a considerable extent upon the state in which the dye exists in the fabric. The physical state of the dye, that is, the degree of aggregation frequently has a substantial effect on the dye light fastness, in fact, many dyes owe their high fastness properties to their highly aggregation state. There may also be a surface effect where the dye on the surface is first attacked by the action of the light and the decomposed dye on or near the surface then forms an umbrella to shade the dye below. This would decrease the fading of the dye. This probably is the case with $CuSO_4$ mordanted wool fabric.

At the end of the exposure period (528 days), AATCC Grey scale (Make: American Association of Textile Chemists and Colorists, ISO International Standard R105/I, Part 2 1978) was used to determine the change in colour of the dyed specimen and the degree of staining of the two pieces of the adjacent fabrics. The ratings of 1 to 5 was used in which 1 indicates very poor and 5 indicates excellent [20, 21, 22]

CONCLUSIONS

The mordanted wool fabric has very good wash/staining fastness for test solutions 1A and 11A, indicating resistance to normal domestic washing of the $K_2Cr_2O_7$ and $CuSO_4$ mordanted wool fabric respectively.

In test solution 111A (having higher concentration of detergent and 100 numbers of steel balls), the amount of dye faded was more in $CuSO_4$ mordanted wool (57%) than $K_2Cr_2O_7$ (92%).

The potassium dichromate mordanted wool fabric faded (decomposed) about 2/3 (67%) of their colour after 528 hours (22 days) while copper sulphate mordanted wool fabric lost less than 1/5 (20%) of their colour for the same period. Therefore, copper sulphate mordanted wool fabric have a superior light fastness properties than potassium dichromate mordanted wool fabric.

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