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Alternative mordants for wool dyeing using leaves of *Punica granatum* and *Lantana camara*

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Abstract

Application of bio-mordants in fabrics dyeing is a sustainable and ecological way to preserve the environment from textile industries wastes. Considering the use of potential plant-based mordants as an alternative to metallic mordants, this study deals with the comparison between the use of *Punica granatum* and *Lantana camara* leaves and alum as mordant. Two different mordanting techniques namely simultaneous and post mordanting were applied along with the dyeing process using leaves of *Psidia altissima* as source of dye. It is a Malagasy endemic plant species commonly used by craftsmen to get green color on silk. Few tests were carried out in order to evaluate the fastness and fixing properties of the involved mordants on wool knitted fibers. The washing fastness of the dyed and mordanted samples were assessed by comparing their color coordinates. The results from this study showed that leaves of *Punica granatum* and *Lantana camara* can be used in natural mordanting process to dye wool in range of green to brownish color with *Psiadia altissima* dye. Their wash fastness values are more interesting compared to alum. Thus, processes from this study will conduct to an ecofriendly technology of textile dyeing.

Keywords: Bio-mordant, natural dyeing, textile, mordanting, tannins, *Psidia altissima*, *Punica granatum, Lantana camara*

1- Introduction

According to a World Bank estimate, 17% to 20% of industrial water pollution comes from textile dyeing. This pollution is caused by the use of large quantities of chemicals in dyeing processes, notably synthetic dyes, fixatives and mordants [1]. These inputs are generally products of chemical synthesis and are discharged into the environment, even though they may present a danger to the environment [2] [3]. They also contain heavy metals that are harmful to human health [4]. Being aware of the dangers generated by synthetic dyes and bites, people become more and more concerned about environmental issues and started to find out safe approaches as alternatives [5]. In natural dyeing processes, mordants play an essential role in obtaining a wide spectrum and nuance of colors. Mordants are chemicals that contribute in conferring properties on textiles that improve color stability and fastness by creating complexes between dyes and fibers [3] [6]. Thus, application of bio-mordant in natural dyeing is also a promising challenge to ensure an eco-friendly process in textile dyeing. Considered renewable, biodegradable and environmentally friendly, the use of bio-mordants for natural textile dyeing has received more attention due to growing environmental awareness as a replacement for hazardous synthetic metal products which themselves are pollutant. [7] [8] [9].

Therefore, this issue of looking for natural mordants is investigated in this paper. Madagascar is home to hundreds of identified dyeing plants among which can dye without mordant due to the presence of chemical components having a fixation property [10]. Metal hyper accumulating and tannin-rich plants are source of biomordant [11]. *Punica granatum* is rich in tannins that are used in many applications including its uses as mordant [12] [13]. Some studies have reported the use of peels of pomegranate as mordant in dyeing wools [14] [15]. According to the surveys we carried out among local dyers of silk and raffia, they use ashes as mordant especially those from leaves of *Lantana camara.* In this study, tannins from *Punica granatum* and mordants from *Lantana camara* leaves ash and extract have been used for the mordanting tests. In addition to their use in the treatment of skin diseases, *Psidia altissima* is among the wide range of dye plant used to dye silk and produce the green hue [10]. It is an endemic Malagasy plant and belongs to the family of Asteraceae. Hence, we carried dyeing tests on woolen fibers using *Psidia altissima* to investigate on the effect of plant-based bio-mordants and mineral mordant alum. The solidity test of dyes on the fabrics have been assessed by applying the wash fastness test.

2- Materials and methods:

The experimental part includes only water as solvent from extraction to dyeing in order to reflect production of bio products.

4-1- Materials:

Commercial knitted woolen yarns have been used for the experiments. Fresh leaves of *Psidia altissima*, *Punica granatum* and *Lantana camara* were collected in the region of Analamanga Madagascar under the research authorization N° 109/23/MEDD/SG/DGGE/DAPRNE/SCBE.Re. Then, they were dried at room temperature for few days. Specification of each raw materials are represented in **Table 1.**

4-2- Methods:

4-2-1- Extraction process:

The maceration technics has been used for the extraction in order to get a concentrated liquid extract and to determine the extraction yields of the plant based biomordants. 190g and 150g of *Punica granatum* and *Lantana camara* were respectively extracted with distilled water in ration 1/6 (w/v) at 70°C. This process lasted for 2 hours and was repeated three times.

4-2-2- Dyeing and mordanting:

Dry and fresh leaves of *Psidia altissima* were cut separately into small pieces and extracted with water for 1 hour at 90°C with a ratio 1/10 (w/v). The solution was next filtered with filter paper in order to remove the plant residue. The extraction of the mordant solution for the other plant materials were done with the same process as the extraction of the dye solution but with a gradient of concentrations. Apart from the use of its aqueous solution, *Lantana camara* has been reduced to ash as it is the method used by dyers for dyeing with *Psidia altissima*. Two mordanting methods were used for this study: simultaneous mordanting and post mordanting. All fabrics were cut into 1g samples and rinsed with water to ensure the non-presence of any dirt particles. **Figure 1** illustrates all steps followed from dyeing to rinsing.

Several tests have been done in order to investigate the influence of the two mordanting plants and alum on the color variation, intensity and fastness of the woolen knitted fabrics. The characteristics parameters of each test are shown in **table2** and **table 3.**

We have also prepared *Psidia altissima* dye baths at two different concentrations but using only *Lantana Camara* and alum as mordant, in order to observe the influence on color: C1= 1/10 and C2=2/10, (w/v)

4-2-3- Wash fastness test:

Wash fastness was evaluated for this stage to test the fastness of the dye on the fibers after five washes. This involved washing the fabrics with a local soap at pH=8 in accordance with ISO 105-A05:1996 "Textile - color fastness testing - Part A05: instrumental evaluation of color change for gray scale determination". Next, the appropriate L*a*b* coordinates were recorded by measuring each fabric using the color reader. L* represents lightness (L=0 means black and L=100 means white). Scale of redness (a^{*-ve}) /greenness (a^{*-ve}) is represented by a* and scale of yellowness (b^{*+ve}) /blueness (b^{*-ve}) is represented by b*. [16]

ΔE is the total color difference which is intended to be single number metric for pass/fail decisions and in the CIELAB system ΔE is given by the following equation:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

ΔE is measured between a scale of 0 to 100. According to Zachary Schuessler, these are the standard perception ranges [17]:

- <= 1.0: Not perceptible by the human eye
- 1-2: Perceptible through close observation
- 2-10: Perceptible at a glance
- 11-49: Colors are more similar than the opposite
- 100: Colors are exactly the opposite

Although, ΔE<2 indicates the best wash fastness properties.

3- **Results:**

5-1- Results of extraction:

The yields of aqueous extractions with *Punica granatum* and *Lantana camara* were 19% and 20% respectively.

5-2- Results of dyeing and mordanting:

The dyeing experiments were carried out with four types of mordant: aqueous solution of *Punica granatum*, aqueous solution of *Lantana camara*, ash of *Lantana camara* and alum. It was hard to obtain the green color after dyeing without mordant with fresh and dry leaves of *Psidia altissima*, even with a twofold increase in the concentration of the dye solution. However, a green color change was observed immediately after the first wash.

This change in color and the wash fastness values are illustrate in **table 4**.

5-2-2- *Punica granatum* as mordant:

The tests produced different types of color. The hue varied from dark yellow to brown for fibers dyed and mordanted with *Punica granatum*. The results of the experiments on mordant concentration variation are summarized in **Table 5.**

Dyeing tests with Punica granatum were also carried out to confirm the presence of tannins and measure their ability to bind to the fiber. Data on wash fastness are shown in **Table 6**.

5-2-2- *Lantana camara* as mordant:

Mordanting with the aqueous extract of *Lantana camara* leaves gave green hues. The use of *Lantana camara* ash gave a dark shade to the fiber. We did not obtain the green color by dyeing with the dry, fresh material without mordant, even when increasing the concentration of the dye solution twice. However, a green color change was observed immediately after the first wash.

5-2-3- Alum as mordant:

Mordanting with alum gave the wool a yellow color, which became more intense after washing. **Table 8** shows the results of this experiment part.

4- Discussion:

6-1-Dyeing and mordanting

Different colors of dyes were obtained according to the nature of the mordant. This is due to the nature of mordant: tannin or other metallic extract of the plant.

Color variation depends on the plant used, the concentration of mordants, the concentration of the dye solution and the mordanting method: simultaneous mordanting or post-mordanting. Moreover, the use of Punica granatum led to darker coloration of the wool while alum brightened the fibers color. This fact is proved by the values of L* from the table (*Punica granatum*: 50.92, alum: 63.09). In our experiments, dyed and mordanted fibers were initially pale and became darker after washing. This color change is thought to be due to the increase in pH during washing with soap. In other words, there is a change in the electron configuration (resonance) of the natural dye molecules. Tannins are neutralized in an alkaline environment, leading to the appearance of good hue [18].

6-2-Wash fastness

6-2-1- Without mordant:

Results obtained for the mordanted and non-mordanted dyed fabrics are different. The color of dyed wool without mordant is less intense than those dyed with mordant. It is confirmed by the color of the fabrics. The high values of ΔE of the non-mordanted dyed wools (C1=11,4 and C2=18.24) shown in the table 4 reveal the great change in color of the fabrics after five washings: from beige to light green pale. This may due to the increase of the pH in the washing bath (pH=8) but also due to the fact that the color is hardly fixed on the fiber at the same time.

6-2-2- Mordanting with *Punica granatum*:

Wool fabrics mordanted with the extracts from the two plants source of bio-mordant reveals higher intensity of color than those dyed without mordants. The presence of tannin in the aqueous extract of *Punica granatum* influences the color into brown. At a first sight, there is a slight difference of color between the two mordanting methods, whether the initial color or the final one (Table 6). Therefore, obtaining the same result with both methods is not obvious. Post mordanting with pomegranate produced brighter color compared to simultaneous mordanting. Moreover, it gives dark shades to the fabrics in term of lightness values (L* are lower than

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simultaneous mordanting). However, simultaneous method represents better wash fastness (ΔE values are lower). Regarding to all the ΔE values in table 6, the concentration may influence the wash fastness: the higher the concentration is, the better the wash fastness.

6-2-3- Dyeing with *Punica granatum*:

Aminoddin and *al*. have already worked on fallen leaves of *Punica granatum* as source of natural dye for mordant-free dyeing of wools. They demonstrate that pomegranate fallen leaves are potential for dyeing wool without mordant and high fastness were obtained with the optimum pH and dyebath temperature 4 and 100°C respectively [19]. This fact is closely confirmed in our experiments but it differs in the chosen parameters. Table 6 demonstrates that the fastness may depend on the concentration of the pomegranate extract input: C1 has a better wash fastness than C2.

6-2-4- Mordanting with *Lantana camara*:

In general, use of *Lantana camara* as mordant helps to obtain the scale of green colors. It happens especially from the first wash.

Based on results represented in table 4, the concentration of the dye solution may influence the variation of color intensity at first sight for the case of *Lantana camara* and alum. Whereas the ΔE values of mordanting with *Lantana camara* at higher concentration of *Psidia altissima* are better than the less concentrated dyeing solution. The aqueous extract of *Lantana camara* would then be able to better fixe dyes at a lower concentration. The concentration of mordants in the dye bath plays a role in the color intensity and the wash fastness on fabrics. The more concentrated the mordant solution, the more intense the color.

Ash of *Lantana camara* cannot fixe dyes on the wool when it is applied simultaneously with the dyeing process (table 7). This is then the reason why dyers use ash of *Lantana camara* just after the dye bath.

6-2-5- Mordanting with Alum:

Alum always gave a range hue of yellow while using *Psidia altissima* as source of dyes. All the ΔE values of the less concentrated solutions of *Psidia altissima* are higher than those more concentrated. This fact may conclude that fixing site of the mordant are not sufficient for the molecule of dyes at the given concentration. A huge shift of color was observed on the fibers mordanted with alum. The color became intense and this explain the high value of ΔE.

7- Conclusion

Psidia altissima is an endemic plant famous to dye silk in the southern area of Madagascar to produce green color while using metallic mordant. This source of dyes produces a wide range of colors according to the used mordant and the shades of each color increase with the amount of plant material used. The present study has enabled us to develop dyeing and mordanting tests using dry leaves of *Punica granatum* and *Lantana camara* as mordant. The mordanting tests using water soluble extracts of *Punica granatum* produce a range hue from dark yellow to brown with *Psidia altissima* on wools and represent better wash fastness when it is applied simultaneously in the dyebath. *Lantana camara* can be used in two forms as mordants. It can be reduced into ash to produce dark green when applied after the dyeing process. Aqueous extract of *Lantana camara* sufficiently keep the basic color produced by *Psidia altissima* even after five washing steps. Thus, this study contributes with new alternatives of mordant through plant-based bio-mordants and sustainable processes applied to natural fibers.

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10- Tables

Table 1: Specification of plant raw materials

Raw materials	Famille	Nature	Part used	Form of the mordant
Psisdia Altissima	Asteraceae	Dye	Leaves	-
Punica granatum	Punicaceae	Mordant	Leaves	Aqueous solution extract
Lantana camara	Verbenaceae	Mordant	Leaves and bark	Aqueous solution extract and ash

Table 2: Characteristics parameters of mordanting with *Punica granatum*

Source of biomordant	*Punica granatum*					
Methods	Simultaneous mordanting			Post-mordanting		
Used biomordant	C1	C2	C3	C1	C2	C3
Ratio w/v (dry leaves/water)	150g/L	100g/L	50g/L	150g/L	100g/L	50g/L
Content of dyeing bath	2*1g wool + 60mL of dyeing solution + 60mL of mordant solution			1- 2*1g wool + 60mL of dyeing solution 2- +100mL of mordant solution		

Table 3: Characteristics parameters of mordanting with *Lantana camara*

Source of biomordant	*Lantana camara*					
Methods	Simultaneous mordanting			Post-mordanting		
Used biomordant	C1	C2	Ash	C1	C2	Ash
Ratio w/v (dry leaves/water)	100g/L	50g/L	3g/100mL	100g/L	50g/L	3g/100mL
Content of dyeing bath	2*1g wool + 60mL of dyeing solution + 60mL of mordant solution			1- 2*1g wool + 60mL of dyeing solution 2- +100mL of mordant solution		

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Table 4: Wash fastness properties of fabrics dyed without mordants

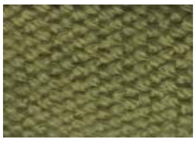
Concentration of *P. altissima*	L*	a*	b*	ΔE	Initial color	Final color
C1=1/10	58,43	1,88	27,03	**11,4**		
C1=2/10	68,79	1,44	20,64	**18,2**		

Table 5: Results of dyeing and mordanting with *Punica granatum* (Concentration of *Psidia altissima* = 1/10)

			Dyeing with *Psidia altissima* C1=1/10 (w/v)					
	Mordant	Mordant quantity	L*	a*	b	ΔE	Initial color	Final color
Simultaneous mordanting	***P. gra***	**C1**	50,92	7,20	32,59	**1,5**		
	P. gra	**C2**	49,10	6,54	29,84	**3,81**		
	P. gra	**C3**	48,98	8,00	28,42	**5,51**		
Post-mordanting	***P. gra***	**C1**	45,81	7,81	37,95	**3,55**		
	P. gra	**C2**	47,60	8,50	37,11	**5,51**		
	P. gra	**C3**	48,51	5,52	36,66	**5,73**		

Table 6: Wash fastness values of dyeing with *Punica granatum*

Punica granatum **concentrations**	**C1**	**C2**
Valeurs de solidité au lavage	1,12	2,5

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Table 7: Results of dyeing and mordanting with *Lantana camara* and alum. (Concentrations of *Psidia altissima* =1/10 and 2/10)

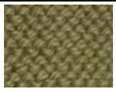
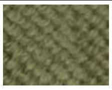
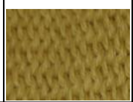
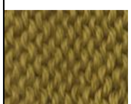
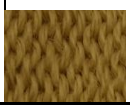
			Psidia altissima: C1=1/10 (w/v)						*Psidia altissima*: C2=2/10 (w/v)					
	Mordant	Mordant quantity	L*	a*	b*	ΔE	Initial color	Final color	L*	a*	b*	ΔE	Initial color	Final color
Simultaneous mordanting	***L. cam***	**C1**	56,26	-1,66	25,49	**3,65**			49,43	0,42	26,79	**4,72**		
	L. cam	**C2**	61,25	1,01	25,81	**5,98**			50,53	1,59	27,07	**4,41**		
	Ash of *Lantana camara*	**3g/100mL**	55,03	2,57	21,46	**8,29**			44,13	0,34	26,99	**9,13**		
Post-mordanting	***L. cam***	**C1**	53,71	0,74	26,14	**6,65**			53,71	0,54	28,62	**2,77**		
	L. cam	**C2**	57,39	2,09	27,45	**7,83**			54,27	1,90	28,03	**3,99**		
	Ash of *Lantana camara*	**3g**	55,03	2,57	21,46	**5,85**			44,13	0,34	26,99	**5,69**		

Table 8: Wash fastness of fabrics dyed with *Psidia altissima* and mordanted with alum mordant

Mordant	Concentration of *P. altissima*	Mordanting method	L*	a*	b*	ΔE	Initial color	Final color
Alum	C1= 1/10 (w/v)	Sim	63,09	4,28	34,64	**11,1**		
	C2= 2/10 (w/v)		59,84	6,09	42,42	**28,6**		
	C1= 1/10 (w/v)	Post	54,83	4,37	35,60	**18,7**		
	C2= 2/10 (w/v)		54,36	4,31	38,42	**21,8**		

11- Figures

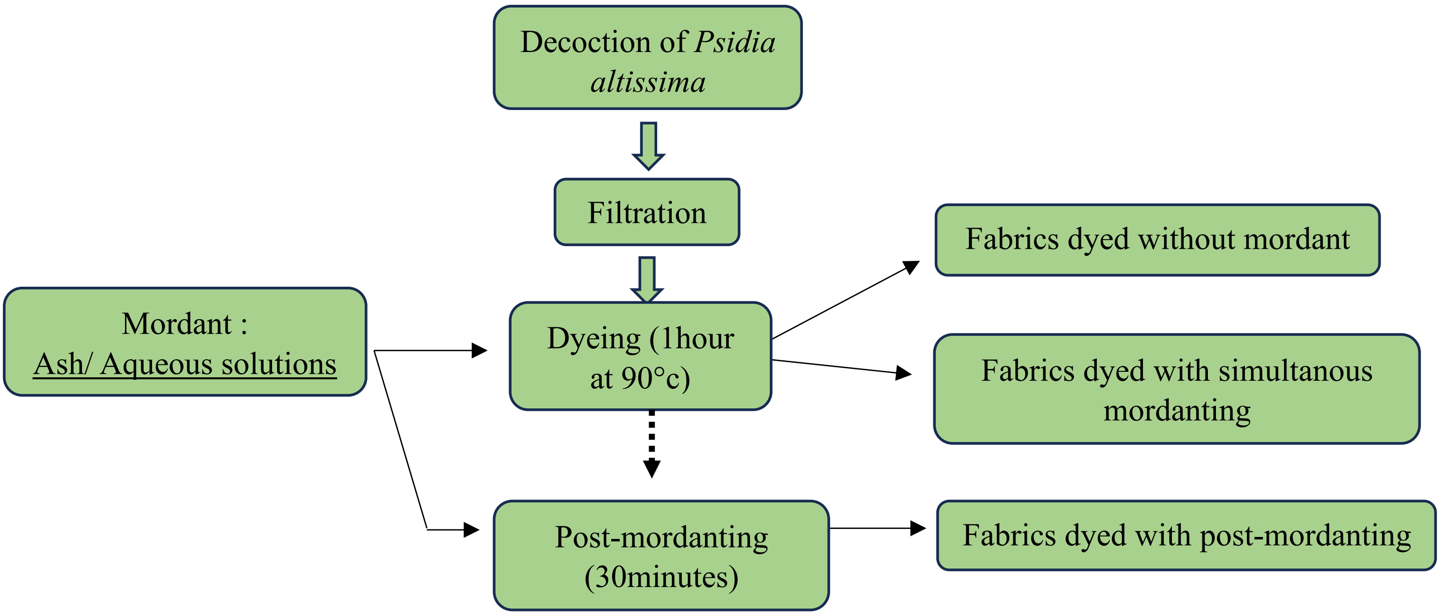


Figure 1: Dyeing and mordanting steps