EFFECTS OF MORDANTING METHODS OF DYE FROM *VERNONIA AMYGDALINA* ON COTTON FABRICS COLORATION

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ABSTRACT

The study investigated the effects of different methods of application of selected mordants on dyeing woven cotton with natural dyes from the leaves of *Vernonia amygdalina.* The effects analyzed are color fastness to; light, washing, wet and dry rubbing and color characteristics on CIELab color coordinates. The aqueous extraction method was used to extract the dye. Some selected mordants were used for dyeing *viz*; alum, potassium dichromate, ferrous sulphate, iron water and ash water. The mordanting methods used includes; pre-mordanting, simultaneous mordanting and post-mordanting. In the control dyeing without the use of mordants, very good fastness were registered with the following fastness ratings; for washing (4R), dry rubbing (5), wet rubbing (5) and light (5). The natural dye is therefore a substantive dye since it is very fast without mordants. The use of mordants improved color fastness to light from ratings of (5) to (6) for most mordants with post-mordanting methods registering the best results. Multiple colors were produced with different mordants therefore the plant leaves possess polygenetic dye. Post-mordanting method registered the best color strengths K/S values and it exhibited the best color saturation values C. In all cases post-mordanting method gave the best color characteristics with the most brilliant colors on fabrics and good color fastness with all mordants. However, there was no observable effect of mordanting methods on the yellowness or greenness of color.

Keywords: *Vernonia Amygdalina*, post-mordanting, color fastness, substantive dye

INTRODUCTION

In Uganda, plants have traditionally been utilized as a source of colorants for dyeing mats, ropes and other home based materials for a long time. These plants are a potential source of natural dyes since Uganda is a home of thousands of largely unknown and undocumented plants. The majority of natural dyes are from plant sources namely; roots, berries, barks, leaves, seeds, woods and other organic sources such as fungi and lichens (Ferreira *et al*., 2004). Dyes of natural origins are great for color appreciation as any variation in the concentration of dye, mordant, type of water, soil and climate give variations in colors. With the invention of synthetic dyes in 1856, the prominence of natural dyes slacked because the synthetic dyes had some advantages over natural dyes like color fastness, good reproducibility of shades, brilliance of color and easy to use (Anderson, 1971). The synthetic dye stuffs are suspected to cause allergies, and are carcinogenic and detrimental to human health (Singh and Singh, 2002). Ironically, Germany that discovered azo dyes became the first country to ban certain azo dyes in 1996 then later followed by Netherlands, India and some other countries (Patel, 2011). Natural dyes derived from plants have recently gained economic advantage over synthetic dyes because of their non-toxic and biodegradable nature (Samanta and Agarwal, 2009). Due to the current eco-consciousness, researchers' attention has been shifted to the use of natural dyes for dyeing textile materials (Bechtold, 2006).

Most natural dyes need chemical species called mordants for binding the dye to fabrics to improve color fastness. Mordants help in binding of dyes to fabric by forming a chemical bridge from dye to fiber thus improving the staining ability of a dye with increasing its fastness properties (Padma, 2000). Some of these mordants are chemical agents which are not eco-friendly therefore it is important to use natural dyes with eco-friendly mordants to reap their health and environment benefits. The color fastness and characteristics of natural dyes on fabrics are influenced by the mordanting method applied whose effects vary with the source of the dye.

Vernonia amygdalina is a shrub of family Asteraceae characterized by bitter leaves, traditionally used for treating fever. Both the roots and the leaves are used in phyto-medicine to treat fever, hiccups, kidney disease and stomach discomfort (Hamowia and Saffaf, 1994). The plant has acquired relevance recently, having been proven in human medicine to possess potent antimalarial and antihelminthic properties (Abosi and Raseroka, 2003).

In the present study, dyes from the leaves of *Vernonia amyygdalina* were used to dye plain woven cotton fabrics by application of various selected mordants and mordanting methods. In each case color fastness to washing, rubbing, light and color characteristics were determined.

MATERIALS AND METHODS

Materials

Desized, scoured and bleached plain weave cotton fabric (220 ends/dm, 180 picks/dm, 120 g/m^2) from Nyanza Textiles Limited was used. Fresh leaves of *Vernonia amygdalina* were collected from Busitema University compound, Busia-Uganda and brought to textile lab. Grey scale and blue dyed silk were used for color fastness rating.

Alum, Ferrous Sulphate, potassium dichromate, Ash water and Iron water were mordants used in dyeing. The following instruments were used; Analytical balance, Color data spectraflash photometer, manually operated Crockmeter, and Lauder-0-meter.

Fabrics pretreatment processes

Pieces of plain woven cotton fabrics with dimensions of (8 x 10 cm) each with an average weight of 1.41 g were scoured by washing them in sodium carbonate solution (0.5 gpl) and non-ionic detergent (Tweet® 80, 2 gpl) at 50ºC for 25 minutes keeping the material to liquor ratio at 1:40. The scoured fabrics were thoroughly washed with tap water and dried at room temperature.

Extraction of dye

Dye was extracted from fresh leaves of *Vernonia amygdalina* by aqueous method as described by Deo and Roshan (2004). The leaves were soaked in distilled water (400 cm^3) in a beaker for four hours. The soaked plant materials were heated at 60°C for 30 min and temperature gradually raised to boiling temperature (90°C) and maintained at the boiling temperature for one hour to yield a dye extracts. The dye extract was left to stand for 30 min at ambient temperature and filtered. The colored crude dye solution (150 cm^3) was diluted with distilled water (50 cm^3) and immediately used for dyeing.

Dyeing the cotton fabrics

Cotton fabrics were dyed using pre-mordanting, simultaneous and post-mordanting methods.
The scoured fabrics were soaked in clean water for 30 min prior to dyeing process.

In dyeing with pre-mordanting method, the pretreated cotton fabrics were soaked in a solution containing 10 % on weight of fabrics (o.w.f) of a mordant, at 60°C for 30 min with material-to-liquor ratio of 1:20. The samples soaked with mordant solutions were dyed as described in the method of Kumaresan *et al*. (2011).

Dyeing with simultaneous mordanting method was done according to the method described by Katy (1997). The pretreated fabrics were put in a beaker (250 cm^3) containing 10% on weight of fabric (o.w.f) of mordant and 20% o.w.f of sodium sulphate. The pH of each dye solution were maintained between 6 and 8 with 2 - 5% w/w of acetic acid (40% solution) monitored by test papers during the dyeing process. A material to liquor ratio (LR) of 1:20 was used in all dyeing. This was done separately using each of the selected mordants.

In post-mordanting dyeing of cotton fabrics, the pretreated fabrics were dipped in a dye bath (150 cm^3) and after 10 minutes, 20% on weight of fabric (o.w.f) of sodium sulphate was added. The dyeing was carried out for one hour at 50°C with intermittent stirring. The dyed samples were removed from dye bath and squeezed to remove excess dye. The dyed fabrics were then soaked in a mordant solution (100 cm^3) containing 10 % on weight of fabric (o.w.f) of a mordant at 60°C for 30 minutes with material to liquor ratio of 1:20 as described by (Kumaresan *et al*., 2011).

In all the above three methods, solutions for each of the selected mordants were separately made. The dyed samples were washed with soap solution (2 gpl) at 50°C for 10 minutes, rinsed with tap water and dried at room temperature.

Evaluation of color fastness

Wash fastness of the dyed samples were analyzed as per the AATCC (American Association of Textile Chemists and Colorists) test method 61, 2 (A) using a Launder-o-meter. The wash fastness rating was measured using standard grey scale for loss of shade depth and staining.

Color fastness to dry rubbing and wet rubbing fastness were tested as per AATCC test method 8 using a manually operated Crockmeter and grey scale.

Color fastness to light was evaluated with AATCC test method 16 specifications in coordination with AATCC test method181using MBTF Fade-o-meter. The fading of each sample was observed against the fading of blue wool standards (1-8).

Color measurements

Color development and dye absorption potential of cotton fabrics were evaluated in terms of CIELab color coordinates; L (lightness), a (redness or greenness of color), b (yellowness or blueness of color), C (chroma) and H (hue angle), and K/S (color strength) values as described by Sule (1997).

RESULTS AND DISCUSSIONS

Variable color shades were formed on the cotton fabrics dyed with extracts from the leaves of *Vernonia amygdalina*. The color variations were with respect to the mordants used and the variations in shades were due to mordanting methods employed.

Color fastness of the dye without the use of mordants

This was a control experiment to determine the color fastness of the dye without application of any mordant. As can be noticed in **Table 1** below, color fastness of the dye on the fabrics range from very good to excellent in the range of (4) and (5) against grey scale standard of 1-5 for washing and rubbing and respectively. A good light fastness grade of (5) against standard blue dyed wool of 1-8 was determined.

Table 1. Color fastness of the dye applied on cotton fabric without mordant

Wash fastness		Rub fastness				Light fastness
		Dry		Wet		
CC	CS	CC	CS	CC	CS	
4R	3	5	4-5	5	4	5

CC=Color change, CS=Color staining:
Remarks: *=stronger, *R=Stronger and Reddish, W=Weaker, R=Reddish.

A slight staining of grade (3) was determined in washing with very slight dry and wet rub staining of (4-5) and (4) respectively. Generally the color fastness obtained without application of mordant is suitable for dyeing cotton fabrics. From this piece of information it can be concluded that this dye is classified as a substantive dye. 'Substantive Dyes' are those that dye

the fibers directly and 'Adjective dyes' are those dyes mordanted with a metallic salt (Gulrajani & Gupta, 1992).

Effects of mordants and mordanting method on colour fastness

In dyeing the cotton fabrics some selected mordants were used viz; alum, potassium dichromate, ferrous sulphate, iron water and ash water. All these mordants were applied on the fabrics using pre-mordanting, simultaneous mordanting and post mordanting methods separately**. Table 2** below contains data for color fastness to washing, rubbing and light for the selected mordants using different mordanting methods. Color change and staining were determined for washing, dry and wet rub fastness.

Table 2: Color fastness of dyed cotton fabrics with

Mordant	**Method of Mordanting**	**Wash fastness**		**Rub fastness**				**Light fastness**
				Dry		**Wet**		
		CC	**CS**	***CC***	***CS***	***CC***	***CS***	
Alum	PREM	**4-5**	**4-5**	***5***	**4-5**	***5***	**4**	**5**
	SM.	**4W**	**4-5**	***5***	**4**	**5**	**3-4**	**5**
	POM	**4-5**	**4-5**	***5***	**4-5**	***5***	**3**	**4**
Ferrous sulphate	PREM	**4-5R**	**4-5**	***5***	**4-5**	***5***	**4**	**5**
	SM	**4R**	**4-5**	***5***	**4-5**	***5***	**4**	**6**
	POM	**4R**	**3-4**	***5***	**4-5**	***5***	**3**	**5**
Potassium dichromate	PREM	**4-5**	**4-5**	**5**	**4-5**	**5**	**4**	**6**
	SM	**4-5W**	**4-5**	**5**	**4-5**	**5**	**4-5**	**5**
	POM	**4-5**	**3-4**	**5**	**4-5**	**5**	**4**	**5**
Iron water	PREM	**4-5R**	**4-5**	**5**	**4-5**	**5**	**3-4**	**6**
	SM	**4-5R**	**3-4**	**5**	**4-5**	**5**	**3**	**6**
	POM	**4-5R**	**4-5**	**5**	**4-5**	**5**	**4**	**6**
Ash water	PREM	**4-5**	**4-5**	**5**	**4-5**	**5**	**4**	**5**
	SM	**4 W**	**4-5**	**5**	**4-5**	**5**	**4**	**4**
	POM	**3-4 R**	**3-4**	**5**	**4-5**	**5**	**4**	**4**

CC=Color change, CS=Color staining:
Remarks: *=stronger, *R=Stronger and Reddish, W=Weaker, R=Reddish.
PREM=Pre-mordanting, SM=Simultaneous mordanting, POM=post-mordanting

Alum mordant produced no observable change in color fastness properties in comparison to that without a mordant this is true for all mordanting technique except post mordanting method which exhibited a reduced light fastness from (5) to (4). The mordanting methods used had no positive effect on the color fastness properties in this case. However, there was a slight increase in staining of (3-4) and (3) for simultaneous mordanting and post-mordanting methods respectively.

Ferrous sulphate mordant produced a slight improvement in light fastness for a simultaneous mordanting method from rating (5) to (6) compared to that obtained without a mordant. The pre-mordanting and post-mordanting methods registered no change in fastness against that without mordant application. However, post-mordanting method had more staining of rating (3-4) for washing and (3) for wet rubbing a result which is inferior to all other mordanting methods and the control without mordant. Perhaps this is a result of the deeper shades developed.

Potassium dichromate mordant with pre-mordanting gave improved light fastness of rating (6) compared to the control without mordant with rating of (5). Simultaneous mordanting and post mordanting methods registered fastness rating of (5) showing no improvement over the control. Post-mordanting registered more staining of rating (3-4) and (4) for washing and wet rubbing respectively. The pre and simultaneous-mordanting methods had very faint staining of ratings (4-5) and (4).

The application of iron water mordant registered a good and improved light fastness of (6) for all mordanting methods better than the control test of rating (5). Wash fastness grade was (4-5R) for all mordanting methods, meaning the color become reddish on washing and faster than in the control with rating (4R). In pre-mordanting method registered a slight staining of (3-4) in wet rubbing washing and dry rubbing had negligible staining of (4-5). Simultaneous mordanting registered a significant staining in washing and wet rubbing with ratings of (3-4) and (3) respectively. Post-mordanting method registered negligible staining of rating (4-5) for washing, wet and dry rubbing.

The use of ash water as mordant registered no positive contribution to color fastness with all mordanting techniques. Reduced light fastness grade of (4) was registered for simultaneous and post mordanting methods. Post-mordanting method registered increased staining of ratings (3-4) for washing and a weak wash fastness of (3-4R) in comparison to the control test. Ash water generally had no positive contribution on fastness and staining, it is the poorest of all the selected chemical species used as mordants.

In terms of color fastness rating obtained with the mordants used, post-mordanting method exhibited the best results with exception of ash water, which is isolated with negative overall effect. In terms of fastness strength, mordants with best ratings registered are; iron water, potassium dichromate, ferrous sulphate and alum in that order. This good wash fastness is as a result of the presence of strong metal coordination complexes formed inside the internal fiber structure between the mordant and dye fibers (Jothi, 2008). The iron species produced redder and deeper shades and alum produced greener and yellower shades that varied with the method of application of mordant on the cotton fabrics. A high quality colored fabric possesses an acceptable amount of color fastness rating of at least 3.0 on a five point grey scale (Kadolph, 2005). Results of this study therefore demonstrates that dye from the leaves of *Vernonia*

amygdalina, with light fastness rating of range (6) and (5) with and without mordants is far above the minimum performance requirements for application on cotton fabrics.

Color characteristics based on CIELab color coordinates of dyed cotton fabrics.

The dyed fabrics were assessed for their color coordinates as reported in **Table 3** and the results are discussed with the aid of color space diagrams and surface color value graphs. Where L*=Lightness, a*=red-green, b*= yellow-blue,C*=chroma H^{o} = Hue angle, CV-SWL= Color value at single wave length and it is equivalent to K/S (Color strength) in terms of value.

Table 3:The CIELab color coordinates of cotton fabrics dyed with crude dye extracts from *Vernonia amygdalina*.

Mordant	**Mordanting Method**	**L***	**a***	**b***	**C***	**h^{o}**	**CV-SWL**
Potassium dichromate	PREM	81.75	-1.39	13.63	13.7	95.82	1.53
	SM	81.34	-1.51	14.96	15.03	95.78	1.502
	POM	70.46	-0.61	26.79	26.8	91.3	3.972
Alum	PREM	78.83	-3.87	19.78	20.15	101.07	2.299
	SM	81.61	-3.7	23.91	24.19	98.81	2.259
	POM	74.73	-6.71	41.09	41.64	99.28	25.464
Ferrous sulphate	PREM	54.17	-0.9	13.51	13.54	93.79	8.037
	SM	57.92	-1.54	9.29	9.41	99.45	4.804
	POM	43.92	-2.44	17	17.18	98.17	66.571
Iron water	PREM	64.65	-1.36	10.96	11.04	97.06	3.216
	SM	59.42	-1.28	11.12	11.19	96.57	4.904
	POM	69.91	-0.57	11.06	11.07	92.96	2.355
Ash water	PREM	83.43	-3.49	13.53	13.97	104.46	1.705
	SM	78.07	-1.88	11.22	11.37	99.52	1.574
	POM	81.88	-3.03	14.61	14.92	101.7	1.896
No mordant/control	-	78.57	-3.44	19.71	20.01	99.89	3.013

PREM= pre-mordanting, SM= simultaneous mordanting, POM= post-mordanting

As can be noticed from **Table 3,** color strengths recorded for post-mordanting method were; (66.571), (25.464), (3.972) and (1.896) for ferrous sulphate, alum, potassium dichromate and ash water respectively. The post mordanting method gave the best color strength measured for all the mordants mentioned. However, with iron water the greatest color strength of (4.904) was recorded for simultaneous mordanting methods. Pre-mordanting method registered the following color strength; (8.037), (3.216), (2.299), (1.705) and (1.53) for ferrous sulphate, iron water, alum, ash water and potassium dichromate respectively. In the control test without the application of mordants, the measured color strength of (3.013) which is a little stronger than when mordants are used in some cases. The pre-mordanting method gave the second best color strength across all mordants from the color strength data.

It can be noted in **Table 3** that, post mordanting method exhibited the best color strength except with iron water. Post mordanting method is therefore the method to be adopted in the application of dyes from the leaves of *Vernonia Amygdalina* on cotton fabrics.

Dyes from *Vernonia amygdalina* produced lighter shades (L > 70) with alum, dichromate and ash water. Ferrous sulphate and iron water produced deeper shades with L (57.92-43.92) and (59.42-69.91) for ferrous sulphate and iron water respectively. Post- mordanting method produced lower L values hence deeper shades with the mordants used.
Shades formed by post-mordanting with alum exhibited the highest degree of color saturation C (41.64) on cotton. The net effect of its high value of chroma and lightness is reflected in its high brilliant appearance.
Fabrics dyed with *Vernonia amygdalina* exhibited only negative "a" value for all the mordanting methods as shown in **Table 3**. The values of "a" and "b" placed the dyed fabric with *Vernonia amygdalina* extract in the yellow-green quadrant of the color space diagram as shown in **Figure 2**.

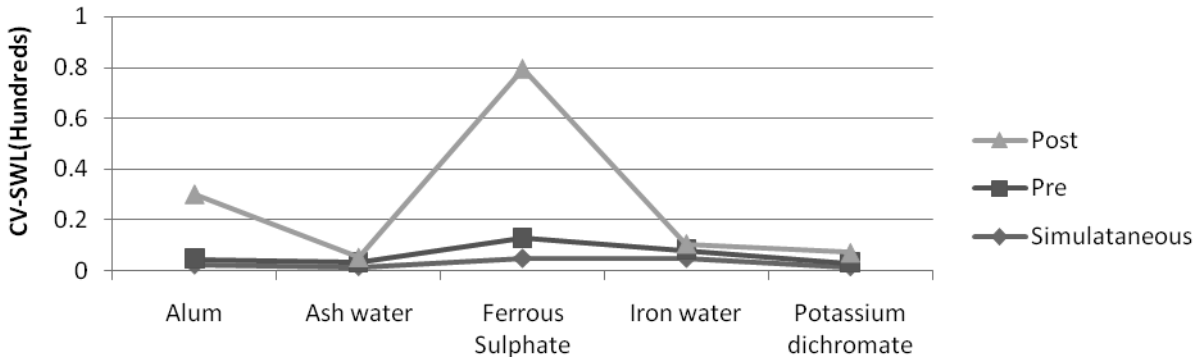

Figure 1.Plot of the surface color value of the dyed fabrics with the selected mordants for pre, simultaneous and post mordanting methods.

Post-mordanting of *Vernonia amygdalina* dye with alum gave the highest "b" value (41.09) (yellowness). The values of "b" (yellowness-greenness) were greater than those of "a" (yellowness-redness) on the dyed fabrics demonstrating that the shades were more yellowish than red in appearance. The hue angles (H) is between (91.3) and (104.64) across all mordanting methods shows that the shades are closer to yellow than green as shown in **Figure 2** below.

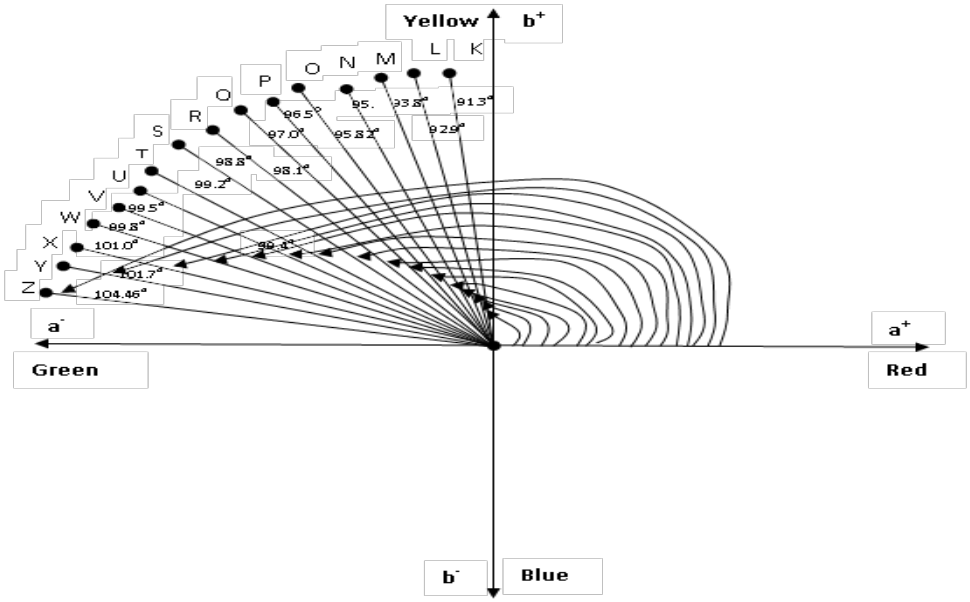

Figure 2: Location of colored fabric samples in the a-b color space diagram.

Key: PREM= pre-mordanting, SM= simultaneous mordanting, POM= post-mordanting
K: POM with Potassium dichromate. L: POM with Iron water. M: PREM with ferrous sulphate. N: SM with Potassium dichromate. O: PREM with Potassium dichromate. P: SM with Iron water. Q: PREM with Iron water. R: POM with ferrous sulphate. S: SM with alum, T: POM with Alum, U: SM with ferrous, V: SM with Ash water, W: No mordant (control), X: PREM with Alum, Y:POM with Ash water, Z: PREM with Ash water.

Control fabrics also gave a hue angle H (99.89) falling within the range of the mordanted fabrics. The mordanting methods used did not portray any influence on the yellowness or greenness of color shades formed.

CONCLUSIONS

Dyes extracted from the leaves of *Vernonia amygdalina* yielded various colors with different mordants this therefore means that dye from the leaves of this plant species is polygenetic. The cotton fabrics dyed without the use of mordants exhibited a good wash fastness of (4), a very good dry fastness of (4-5), wet rubbing fastness of (4) and a good light fastness of (5). The dye is therefore substantive dye, which possesses good color fastness without the use of mordants. The application of mordants yielded various colors with different mordants and shades that varied with the mordanting methods without any observable improvement in wash and rubbing fastness. The formation of multiple colors with different mordants classifies the dye as polygenetic dye. Light fastness improved from (5) to (6) with the application of mordants especially with iron water, ferrous sulphate and potassium dichromate. However, there was no significant improvement in light fastness with the application of alum.

Color characteristic of the dye on cotton fabrics was enhanced by the application of mordants and the method of application of the mordants. For most of the mordants used, post mordanting method gave the best color fastness and strength on the fabrics the same mordanting method

gave averagely better color fastness with most mordants with exception of ash water. The ash water also yielded poor color fastness and color characteristics with all mordanting methods it is therefore not suitable for dyes from the leaves of *Vernonia amygdalina* plant species for cotton fabrics.

ACKNOWLEDGEMENT

We would like to acknowledge the Management of Busitema University for funding this study. Gratitude also goes to Dr. P.A.G. Wanyama of Kyambogo University for general guidance. Recognition goes to Mr. Elisha Obella the Academic registrar of Busitema University for his continued encouragement in conducting research for generation of knowledge. Technical support was rendered by Professor B.K. Behera of Indian Institute of Technology.

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