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Environmental Friendly Dyeing of Silk Fabric with Natural Dye Extracted from *Cassia singueana* Plant

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Abstract

Introduction and application of eco-friendly natural dyes on textile coloration have significant importance on the reduction of environmental pollution. Due to this reason many scientists are doing research work on natural dye application on textile materials in the past few years. Having this in mind, this investigation was concerns with the dyeing of silk fabric with colorant extracted from bark of Hambo Hambo *(Cassia singueana)* plant in the presence of Aloe Vera as natural mordant. Aqueous extraction method was employed for obtaining coloring components. The extraction parameters have been studied and optimum dye extraction condition was examined under UV-Visible spectroscopy which recorded its maximum absorption as (A=3.9). Silk fabric was dyed with the extracted natural dye without mordant and in the presence of synthetic and natural mordants using different mordanting techniques. The color strength, CIE $L^*a^*b^*$ values and fastness properties of the dyed samples were analysed according to the international standards. The Washing fastness, rubbing fastness and light fastness obtained from the directly dyed without mordant and dyed using with Aloe Vera and Copper sulphate ($CuSO_4.5H_2O$) mordants was almost similar which was recorded in the range of very good to excellent. The results of this investigation led to the conclusion that there is possibility of dyeing of silk fabric using natural dye extracted from *Cassia singueana* plant directly without mordant and dying in the presence of Aloe Vera with acceptable range of fastness properties of dyed silk fabric.

Keywords: Extraction; Mordents; Natural dye; *Cassia singueana* plant; Silk; Enviromental friendly dyeing; Aloe vera

Introduction

The Textile industry is one of the biggest consumers of synthetic dyes [1]. Basically, synthetic dyes are produced from cheap petroleum and coal-tar sources Pervaiz et al. and Devi et al. which are injurious to health, destroying eco-system and toxic to aquatic biodiversity [2-4]. Due to the current Environmental consciousness, the researcher's attention has been shifted to the use of natural dyes for dyeing textile materials in the past few years [5,6].

Demand of natural dyes is increasing continuously as their production and application does not require strong acids and alkalies [6,7]. Natural dyes are believed that, they have better biodegradability, have wide variety non-toxic, non-carcinogenic, easily available and renewable, eco-friend and generally higher compatibility with the environment; provide a wide range of beautiful shades with acceptable levels of color fastness [8-13]. As a result, it is the best alternative using natural colorants in textile application as compared with synthetic dyes [14]. Coloring components can be getting from roots, barks, leaves, fruits and flowers of plant sources. Natural dyes are not only release medicinal properties but also improve the aesthetic value of the product and they are unique and eco-friendly [15]. The extract of this plant were used in northern Nigeria for the treatment of anti-microbial and acute malaria attack [16,17]. The aspect of producing textile products without impacting on the ecological balance, affecting both human and environmental health, is an important focal point to be pursued. Therefore, to address some of these issues this study was aimed to dye silk fabric with eco-friendly natural dye and natural mordant extracted from bark of *Cassia singueana* plant and Aloe vera respectively.

Cassia singuena plant belongs to the class *Leguminosae* and family *Caesalpinioideae* which have an antimicrobial and antiplasmodial activity [17]. It has also composition of Phenols, Saponins, Tannins and anthraquinones and those chemical compositions of tannins and anthraquinones indicates that it can be used as coloring material as shown the structure in Figure 1 [16-18].

Tanned leather materials were dyed using extracted natural dye from *Cassia singueana* plant in the presence of natural mordants

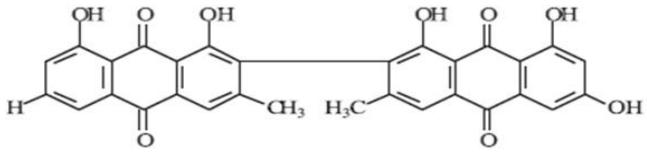

Figure 1: Typical Chemical structure of Tannins and anthraquinones of *Cassia singueana* Plant.

obtained from Aloe vera and mango bark resulted Effective results of color fastness and color strength [19]. Aloe Vera juice was also selected as natural mordant to standardize the dyeing effect of Mari gold dye on natural and synthetic fibers which is investigated by Nilani et al. [20]. This Aloe vera was also used as mordant in dyeing of tanned leather materials with natural dye studied by Berhanu and Ratnapandian and also can be used as an antimicrobial finish of textiles as mentioned by the author Athiban et al. [19-21].

In general, the dyeing silk fabric with extracted natural dye from *Cassia singuana* plant and extracted mordant from Aloe Vera will give the following advantages; 1) since no synthetic dyes or chemicals are added during dyeing it is possible to say this is fully environmental friendly dyeing method, 2) Since the *Cassia singuana* plant and Aloe vera has property of anti-malarial and anti-microbial activity respectively, this dyed silk fabric will behave anti-malarial and anti-microbial at the same time.

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Materials and Methods

Materials and chemicals

The bark of *Cassia singueana* plant and Fresh leaves of Aloe vera as shown in Figures 1 and 2 were collected manually from Northern Ethiopia (Tigray). Bleaching (degummed) silk fabric was obtained from mark. Formic acid (CH_2O_2) and Copper sulphate ($CuSO_4.5H_2O$) were used to control the pH of the dye bath and used as synthetic mordant for comparison respectively.

Equipment and testing devices

Main Equipment and Testing Devices used throughout this work were; Mechanical grinder, Eco-infrared dyeing machine, Oven dryer, pH meter used to check pH, digital electronic balance was used to measure weight of materials and chemicals, Perkin Elmer UV/VIS Spectrometer was used for measuring color absorption, COLOR-EYE 3100-was used for measuring reflectance, K/S and CIE $L^*a^*b^*$ and Fastness (Wash fastness, Light Fastness and Rubbing fastness) testing devices. All those Equipment and Testing Devices were obtained from Bahir Dar University-EiTEX.

Methods

Dye extraction and optimization: Aqueous extraction method was employed for extraction of coloring components from the bark of *Cassia singueana* plant. Pervaiz et al. extracted natural dyes from bark of different plants with temperature (90°C) and time 60 min [22]. Plant parts were collected and washed thoroughly with water to remove soil and dust particles [22,23]. The washed plant materials were shade dried until the moisture is reduced as much as possible. The dried bark was grinded and sieved with 0.5 particle size to obtain the fine powder. The powder was immersed in distilled water with using different combinations of bark concentrations of (20 g/l, 40 g/l and 60 g/l) for 24 hours. The solution was subjected to temperatures of (45, 75 and 95°C) and time (30 min, 45 min and 60 min) respectively. The dye solution was filtered and its absorption was tested using UV-visible spectroscopy (Model lambda 25). The temperature, concentration and time at which attain maximum absorption was taken as an optimum condition for extraction of natural dye from *Cassia singueana* to extract the dye solution from the plant nine experiments were conducted which was decided and calculated by the statistical Minitab software (Figure 2).

Determination of percentage of dye yield: The dye percent of yield was calculated using equation (1)

$$percent\ of\ dye\ yield = \frac{mass\ of\ plant\ leaves - mass\ of\ dried\ precipitate}{mass\ of\ plant\ leaves} \times 100$$

Figure 2: Typical picture of Cssia *singueana* Plant.

Mordant extraction from Aloe vera leaf: Fresh leaves of Aloe vera was collected and washed thoroughly, the outer green surface was peeled off and the inner white mass was collected and crushed to semi solid form using electronic agitator. The crashed semi solid form of Aloe vera becomes filtered. According to Nilani et al. natural mordant was extracted from Aloe Vera leaf by crashing 150 g/l of whit inner part of Aloe vera leaf and heated 50-100°C for 1 h (Figure 3) [20].

Mordanting methods: Natural dyes typically have lower affinities to textiles synthetic dyes. As a result they often required mordants [24]. Three different mordanting techniques (i.e. Pre mordanting, Post Mordant and simultaneous mordanting) were carried out as shown in Figure 3. Silk fabric samples were treated with the extracted natural mordant (Figure 4).

Protein fabrics were dyed with Morus Alba plant bark extract using Aloe Vera as natural mordant and different synthetic mordants which was reviewed by Shoba K et al. [25]. Natural dye with Aloe Vera mordant shows a good dyeing effect and color fastness on the animal fibers. Srivastavaa S, Chopraa A, et al. were extracted natural dye from *Rheum emodi* and applied on protein fabric which was mordanted with different natural mordants with MLR of 1:20 and temperature of 70-80°C for 60 min [26]. Therefore by taking those literatures as a reference, the mordanting MLR, concentration, temperature and time was selected randomly and designed as shown in Table 1.

Key: CPRM (Pre mordanted with copper sulphate), CPOM (Post mordanted with copper sulphate), SMC (Simultaneously mordanted with copper sulphate), APRM (Pre mordanted with Aloe Vera), APOM (Pre mordanted with Aloe Vera), SMA (Simultaneously mordanted with Aloe Vera), SM (Simultaneously mordanting), POM (Post mordanted), PRM (Pre mordanting).

Dyeing procedure: Natural dyes were extracted from different plants and applied to tanned leather in the presence of mordants and without mordant under identical conditions to find out whether the extracted dye shows affinity to tanned leather in the absence of mordant [11]. Win et al. was also extract natural dye from mango bark and applied on Protein fibers with temperature, time and MLR of 50-80°C, 60 min and 1:25 respectively [23]. The dyed material gives good color fastness property and attractive color shade. Natural dye was extracted from *Odina wodier* plant bark and applied on wool fabric with different temperature such as 40, 60 and 80°C for 1 hour which is done by Saravanan P et al. [12]. It has been noted that, the K/S value was good at 80°C.

Figure 3: Typical picture of Aloe Vera leaf.

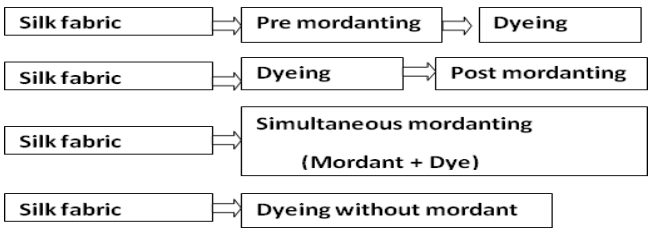

Figure 4: Typical mordanting techniques.

Sangeetha K et al. extracted natural dye from lemon leaves and applied on Silk fabric using different natural mordants with MLR (1:20), Temperature (60-90°C) and time for dyeing and mordanting (30 and 60 min) respectively [27]. Therefore, similar condition were employed for dyeing of silk fabric using an extract of bark of *Cassia singueana* plant and natural mordant with dyeing temperature of 25, 40, 55 and 70°C, time of 20, 30, 45 and 60 min and pH of 5.5, 5, 4.5, 4 and 3.5 were randomly selected as shown in Table 2. Level of pH for the extracted dye and silk fabric was cheeked by using pH meter. The temperature, pH and time at which the dyed silk fabric attained highest K/S value were selected as optimum dyeing condition. Four experiments were selected through random selection method and conducted under each dyeing and mordanting methods as shown in Table 2. The weight of each specimen which was dyed with the extracted dye was 30 grams. All necessary calculations was carried out based on the weight of the sample and decided MLR (Table 2).

Evaluation of dyed silk fabric sample: Color fastness (Wash fastness, rubbing fastness light fastness) properties of the dyed silk fabric samples were determined under the optimized conditions according to ISO 105-CO3, ISO 105-X 12 methods and ISO 105B02 method respectively.

Results and Discussion

Optimization of natural dye extraction from *Cassia singueana* plant

Natural dye solution was extracted from the bark of *Cassia singueana* plant at different bark powder concentration, temperature and time. The absorption of resultant solution was determined using UV-Visible spectroscopy (Lambda 25) and optimum extraction condition was obtained with Maximum dye solution absorption of 3.9) at bark powder concentration of 60 g/l, temperature of 95°C and time of 60 min as shown in Figures 5 and 6.

Type of mordant	Sample no.	Method	MLR	Conc. (o.w.m)	Temperature	Time	Sample name
Copper sulphate		PRM	01:25	3%	60°C	60	CPrm
		POM				min	CPom
		SM			According to dyeing parameters		SMC
Aloe Vera	1	PRM	-	10 g/l	70°C	60 min	APrm1
	2			20 g/l			Aprm2
	3			30 g/l			Aprm3
	4			40 g/l			Aprm4
	1	POM	-	10 g/l	70°C	60 min	APom1
	2			20 g/l			Apom2
	3			30 g/l			Apom3
	4			40 g/l			Apom4
	1	SM		10 g/l	According to dyeing parameters		SMA1
	2			20 g/l			SMA2
	3			30 g/l			SMA3
	4			40 g/l			SMA4

Table 1: Designed mordanting methods using different parameters.

Dyeing conditions	pH	Temperature (°c)	Time	Input sample code	Output sample code
Dyeing without mordant	5.5	25	20	Degummed silk fabric	Dwm1
	5	40	30		Dwm2
	4.5	55	45		Dwm3
	4	70	60		Dwm4
Pre-Mordanting with Aloe Vera	5.5	25	20	Aprm	DAPrm1
	5	40	30		DAPrm2
	4.5	55	45		DAPrm3
	4	70	60		DAPrm4
Post-Mordanting with Aloe Vera	5.5	25	20	APom	DAPom1
	5	40	30		DAPom2
	4.5	55	45		DAPom3
	4	70	60		DAPom4
Simultaneous mordanting with Aloe Vera	5.5	25	20	Bleached silk fabric	SMA1
	5	40	30		SMA2
	4.5	55	45		SMA3
	4	70	60		SMA4
Pre-Mordanting with copper sulphate	3.5	60	60	CPrm	DCPrm
Post-Mordanting with copper sulphate	3.5	60	60	CPom	DCPom
Simultaneous mordanting with copper sulphate	3.5	60	60	Degummed silk fabric	SMC

Table 2: Different combinations of parameters used to dye silk fabric with the extracted natural dye.

Performance test results of dyed silk fabric specimens

Color strength (K/S) value, reflectance (%) and CIE L*a*b* value: Color developed and color strength on dyed silk fabric samples was evaluated in terms of their CIE L*a*b* coordinates and K/S values. The L* (lightness/darkens), a* (redness/greenness), b* (yellowness/blueness) value of dyed silk fabric samples obtained from all dyeing and mordanting methods was lied in dark grey to light grey, red and yellow direction respectively. All dyed silk fabric samples are in the red direction which was reflected in a* and yellow direction in b* as shown in the CIE L*a*b* color space. The value of b* (yellowness) obtained from dyed samples which are pre mordanted with Aloe Vera was higher than the other dyed samples. The darkness of direct dyed silk fabric samples was higher than the samples mordanted with Aloe Vera clearly shown in Table 3.

In the directly dyed silk fabric samples, the maximum dye absorption (K/S) value and minimum reflectance (%) was obtained from sample 4 (Dyed silk fabric without mordant 4) which was dyed at temperature of 70°C, time of 60 min and pH of 4 as shown in Table 3. This sample was taken as optimum condition for dyeing of silk fabric with the extracted dye.

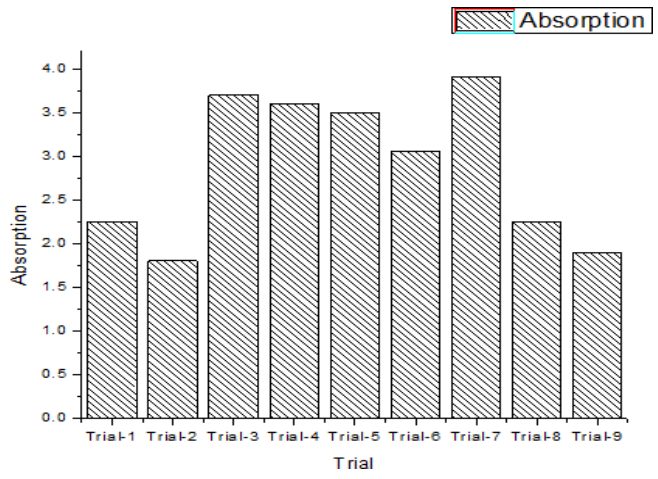

Figure 5: Maximum absorption value of extracted dye solution from each trial at 400 nm.

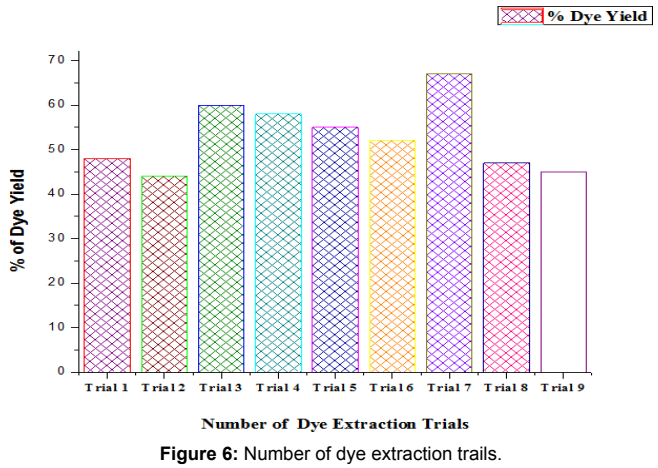

Figure 6: Number of dye extraction trails.

In dyed silk fabric which was pre mordanted with Aloe Vera obtained maximum color strength (K/S) 7.01 Sample 4 (Dyed after pre mordanted with Aloe Vera) which is nearly similar result with the simultaneously mordanted with copper sulphate. Silk fabric sample at which simultaneously mordanted with copper sulphate gives highest K/S value of 10.42. The dyed silk fabric samples in which attained maximum K/S value has been selected from each group of dyed samples and compared each other. As a result, the sample which was directly dyed sample 4 (DWM4) was attained maximum color strength (K/S) value with 10.50 next to sample which was simultaneously mordanted with copper sulphate as compared with other mordanted samples with Aloe Vera as shown in Figure 5 above.

In general it was observed that, the best K/S value was obtained from the samples dyed at temperature 70°C, time 60 min and pH of 4 in all samples which were mordanted with natural extracted mordant from Aloe Vera.

All silk fabric samples were dyed with an extracted natural dye keeping various pH values as 5.5, 5, 4.5, and 4. It was observed that, the color strength (K/S) value was good at pH of 4 as clearly shown in Tables 2 and 3.

Dyebility of silk fabric with the extracted natural dye was evaluated by dying different samples of silk in the presence of Aloe Vera leaf and copper mordants. Maximum K/S (color strength) was got from dyed fabric samples at maximum temperature of 70°C and time of 60 min. This is due to the increments of kinetic energy leads to increase diffusion of dye molecules inside the silk at high temperature and increase diffusion rate of dye molecules in to the leather structure at long dyeing time.

Fastness test results: The color fastness (Washing fastness, rubbing fastness and light fastness) properties of dyed silk fabric samples were tested according to the standard ISO methods of ISO 105-CO3, ISO 105-X 12 and ISO 105B02 methods respectively. The performance of Washing fastness, rubbing fastness and light fastness of the dyed silk fabric specimens were presented in Table 4.

In most of the dyed fabric samples, the performance of fastness property was rated within the range of 3-5 except some dyed without mordant and post mordanted samples with Aloe Vera such as DWM1, DWM3 and simultaneous mordanted (Sma1 and Sma2) shows fair (below 3) results. For the light fastness test results, all dyed silk fabric samples showed good to excellent (6-5 &5,7) except some samples which were directly dyed without mordant and samples which were post mordanted with Aloe Vera showed fair (3-3/4) test results. As the mordant concentration increase the fastness properties of the dyed silk fabric samples also increase in all mordants and mordanting techniques as shown in Table 4.

Samples pretreated with Aloe Vera juice and directly dyed silk fabric samples showed very good to excellent rubbing and washing fastness, and good to very good light fastness property was recorded. Poor rubbing fastness results which is below 3 was obtained from premordanted (Daprm1 and Daprm2), Dapom1 and Dapom2.

In general, natural dye extracted from bark of *Cassia singueana* plant could be used for commercial purpose in the acceptable color fastness range of international standards for (Washing fatness, Rubbing fastness >3 and Light fastness>5) as shown in Table 4.

Method	Sample code	Reflectance (%) at 400 nm	K/S at 400 nm	CIE L* a* b * system		
				L*	a*	b *
Directly dyed without mordant	DWM 1	7	3.2	52.5	22.3	25
	DWM2	7.4	3.2	51.25	20.8	24.5
	DWM 3	5.43	6.53	45.5	25.25	26.48
	DWM4	3.06	10.5	38.46	27.4	22.09
PRM with Aloe Vera	DPRMA 1	9.87	3.04	55.3	15.6	22.1
	DPRMA 2	7.05	4.22	50.04	19.60	23.07
	DPRMA3	5.40	5.01	48.32	23.02	26.26
	DPRMA4	6.78	7.28	49.07	25.5	28.05
POM with Aloe Vera	DAPom1	11.26	2.14	57.74	13.82	20.18
	DPOMA 2	6.68	4.55	52.58	19.66	23.29
	DPOMA3	6.21	4.97	49.68	24.06	27.07
	DPOMA 4	5.18	5.12	48.74	17.03	25.90
SM with Aloe Vera	SMA1	6.82	4.43	49.96	15.40	21.86
	SMA2	5.60	5.61	47.72	16.58	23.18
	SMA3	5.56	5.65	50.92	16.81	26.20
	SMA4	5.10	5.83	44.26	17.92	24.72
PRM with copper sulphate	DCPrm	3.63	8.82	36.91	10.99	16.79
POM with copper sulphate	DCPom	3.90	8.22	39.00	16.54	21.27
SM with copper sulphate	SMC	3.03	10.42	39.00	16.57	15.96

Table 3: Color strength (K/S) value, reflectance (%) and CIEL*a*b* values of dyed silk fabric samples with the extracted dye and using different mordants at 10°C observer.

Type of mordant	Method of mordanting	Sample code	Mordant concentration	Rubbing fastness		Light fastness	Wash fastness	
				Dry	Wet	24 hr	Acidic	Basic
Nil	Dyed without mordant	Dwm1	Nil	3/4	3	4-5	3	3
		Dwm2	Nil	4	3	5	3	3/4
		Dwm3	Nil	3/4	3/4	5	3/4	4
		Dwm4	Nil	5	4	6	4	5
Aloe Vera juice	PRM	DAPrm1	10 g/l	3	2	5	3	3/4
		DAPrm2	20 g/l	4	2	5-6	3/4	3
		DAPrm3	30 g/l	4/5	4	5-6	4	4/5
		DAPrm4	40 g/l	5	4/5	6	4/5	5
	POM	DAPom1	10 g/l	3	2/3	5	3	4
		DAPom2	20 g/l	3	2/3	4	3/4	3/4
		DAPom3	30 g/l	4	4	5-6	3/4	3/4
		DAPom4	40 g/l	4/5	4	6	4	4/5
	SM	SMA1	10 g/l	4/5	2/3	5	3	3
		SMA2	20 g/l	4	3	5-6	4	3
		SMA3	30 g/l	4	4	6	4	3/4
		SMA4	40 g/l	4/5	2	6	4/5	4/5
Copper sulphate	PRM	DCPrm	3 % (o.w.m)	4/5	5	6-7	4	4/5
	POM	DCPom	3 % (o.w.m)	5	4/5	7	4/5	5
	SM	SMC	3 % (o.w.m)	5	5	8	4/5	5

Table 4: Color fastness properties test result of dyed leather samples with bark extract and different mordants.

Conclusion

The present work shows that, barks of *Cassia singueana* plant can be used as a dye for coloring of silk fabric in the presence of natural mordant extracted from Aloe Vera. *Cassia singueana* plant is grown throughout Ethiopia, Nigeria, Egypt and some areas of India and it is an easily available plant makes the raw material cheap. As some authors studied the chemical composition of this plant, it has antimicrobial and anti-mosquito property in addition to use as natural colorant. This plant extract for coloration of silk fabric would give the benefits of reducing import of synthetic dyes and minimize environmental pollution. The washing, light and rubbing fastness of all dyed silk fabrics without and using natural mordant were quite good. The dye has good scope in the commercial dyeing of Protein fibers.

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