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Evaluation of dyeability of Cotton and Silk Fabrics Dyed with Henna Extract

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Abstract: Henna (Lawsonia inermis L.) dyes were known for a long time, and were used for decoration and food purposes. They were recently used for dyeing fabrics after the revolution of environmental protection. Their natural colors are distinctive and have no effect on the environment, enabling them to compete with synthetic dyes. The objective of this paper is to evaluate extraction and application of dyes from henna into textile materials to maintain a clean environment with cheap inputs. The dye was extracted from Henna leaves using (water, ethanol and water \ ethanol) media at extraction time (6, 12 and 24 hours). The extracted dyes were applied to cotton and silk using an exhaustion method. The dyeability and some physical properties of the dyed fabrics, such as absorbance, reflectivity, dye strength (k/s), exhaustion % and color fastness were evaluated. The best results were obtained when using water \ ethanol medium for 12-hour extraction. The highest dye strength for cotton (k/s= 6.8) and for silk (5.8), the maximum dye exhaustion for (cotton 80%) and for (silk 75%). Color fastness rates for washing, light and rubbing for both cotton and silk were "very good to excellent". It was recommended that, according to the challenges facing the environment, there is a necessity to return to natural dyes from Henna for dyeing textiles because, their dyeability properties are good, they provide safety for humans and the environment, the extraction of dye as well as its application to fabric are easily applied at home without the need for sophisticated equipment.

***Index Terms*: Solvent -Extraction -Henna -Color fastness -Exhaustion%, Natural textiles.**

I. INTRODUCTION

Dyeing was known as early as in the Indus Valley period (2600-1900 BC), since the ancient humans applied the art of dyeing in the colouring of leather, clothing and bedding. Dyes are of great importance in providing textiles with colors where man has always been interested in, to increase luster and value consequently; marketing opportunities increase (Ali, 2007), (Mansour et al., 2013).

In the past, dyes were extracted from natural sources (minerals, animals or plants) until the first synthetic dye "Mauveine" was discovered by the English chemist "William Perkin" in 1856, (Hunger, 2003), (Najawi, 1986). Chemists succeeded in preparing a number of new dyes that do not exist in nature, and there is a high demand for coal tar as a raw material for a large number of new dyes (Anod, 2012). In recent decades, the technology of manufacture of synthetic dyes has witnessed a huge boom which has led to increased production of dyes of various types. However, this progress has resulted in environmental pollution.

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Some intermediate organic matters involved in dye synthesis have carcinogenic effects, the estimated wasted dye is 15% which is discharged as effluent and therefore affects soil, water, humans, animals and plants. Synthetic dyes are therefore non-environmentally friendly and result in numerous environmental hazards (Ali, 2007), (Susan, 1982), (Mamatha G., and N. Goutham, 2018), (Lichtfouse E. et al., 2013).

Due to increased environmental awareness, as well as the greater emphasis on a cleaner and greener production process, about the organic value of natural products: consumers have renewed their interest for use and preference of natural dyes. Recently, most commercial dyes and textile export houses have begun to reconsider the use of natural dyes for dyeing and printing of textiles to target specialized markets, (Rahman Bhuiyan, et al., 2018), (Kumar, 2011). Therefore, research has focused on natural dyes, due to their advantages such as: low toxicity and skin sensitivity, renewable sources, traditional dye technology can be rebuilt; safer if used in food industry, biodegradable and compatible with the environment. Despite these drawbacks, natural dyes have the following disadvantages: lack of fastness when exposed to light and washing, high economic cost, have a limited range of colors, lack of technical knowledge and technology of their use, some of the mordants used for fixing dyes are harmful to some fabrics and may cause allergies, (Redwan, 2014).

(Crews, 1987) extracted natural dyes from henna, turmeric, alfalfa and saffron plants, and carried out different chlorometeric measurements. The study has shown that most of these dyes fade quickly. (Ali, 2009) Extracted natural dyes from henna leaves with water (in neutral and alkaline media), to improve extraction and dyeing conditions of cotton treated with alum and sulphate as mordants. It was found that dyes extracted in alkaline medium have a better dye strength, and medium to good fastness properties than those extracted with neutral water. It was concluded that there is no significant improvement in color fastness, however natural dyes from henna have a potential to work when mixed with synthetic dyes. (Yusuf, 2011) studied the effect of tin chloride on dyes extracted from dried henna and its effect on dyeing of woollen yarns in terms of dyeability, and color fastness. Dye strength is very good for all dyed samples. The color fastness to light, washing and rubbing is quite satisfactory. (Nazari, 2017) studied the dyeing of wool with henna extract, its chemical composition, and color fastness properties. He also applied henna dyes to cellulose, proteinine and synthetic fibers. Some improvement in color fastness properties was detected through chemical modification of textile fibers**.** Many studies have been conducted on the potential health risks of henna; these studies have shown that henna has low health risks (Probu, 1998).

(Sivarajasekar, 2018) was extracted dyes from henna leaves using extraction media of alkali, ethanol, hot water, and enzymes. Silk, cotton and wool materials were dyed and tested. Based on concentration, the efficiency of enzyme extraction was found to be roughly equal to that of ethanol.

II. CHEMICAL COMPOSITION OF HEENA

Henna leaves contain 0.5- 0.6% glycosides , the most important of which are Lawson of basic chemical molecule of type 2 - hydroxy 1, 4 - naphthoquinone and its molecular structure is ($C_{10}H_6O_3$) as in figure 1, which is responsible for the dye brownish color. Henna contains non-lawson compounds in small quantities such as naphthalene, coumarins, xanthones, flavonoids, gallic acid and steroid, tannins (5-10%), sugar and resin content (about 1%), (Yusuf, 2017).

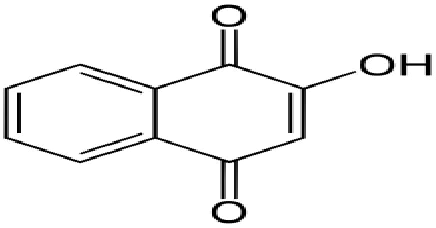

Fig 1: Chemical composition of henna

Many researchers have been conducted on extraction of henna with water in an alkaline medium or ethanol, and its application to natural textiles. However, many of the results obtained suffer from the lack of color consistency of textile materials when exposed to light and washing, and they need mordants to fix the dye.

This paper aims at evaluation and extraction of henna dye in neutral water, ethanol and ethanol\water media, and its application into natural textiles in the presence of alum as a mordant. According to my best knowledge this aspect is still in need of more research. The paper aims at extracting natural dyes from henna using conventional methods, and to study its application into cotton and silk fabrics, using simple methods that can be used at the home, and to study the dyeability of textiles, such as: color strength, exhaustion% and color fastness, and to evaluate the effect of extraction time and media in dyeability in a comparative manner, in such a way that to encourage and localize the use of natural dyes for the purpose of preserving clean environment.

III. MATERIAL AND METHODS

A household Henna (Lawsonia inermis L.) leaves was collected from Wad Medani / Gezira State. Henna was thoroughly cleaned with distilled water from impurities and lingering soil, and dried at room temperature, and was then grinded and sieved.

Cotton and silk fabrics were used according to the following specifications:

- Silk cloth (pillow - bleached) from German industry, purchased from the German market, of density 148 ends\in^2, 108 picks\ in^2.
- Cotton cloth (100% plain weave, scoured and bleached), from Sudanese industry (72 ends\in^2, 51 picks\ in^2).
- All the chemicals used for extraction and dyeing were (laboratory reagent purposes):
- Ethanol, detergent, sodium carbonate, potassium aluminium sulphate (Alum).
- For application of dyes on fabrics and evaluation of color characteristics and color fastness, the following devices and equipment were used:
- Pad mangles model LA-110 (Japan). Laboratory scale exhaustion machine model LA-215 (Japan). Spectrometer model S-725 (China). Colorimeter model LA-315 (Japan). pH meter, crock meter model LA-375. Launder-o-meter model LA-355 (Japan). Fadometer with xenon arc lamp model XF- 15N (Japan).

IV. METHODS

Laboratory methods and chemical analysis were carried out in accordance with the Japanese Standards (JIS) or ISO tests.

Methods of extraction of dyes

Henna dyes were extracted using water, ethanol and ethanol/water at different intervals of time as follows: Three samples each 5 g of henna powder were dissolved separately in 100 ml of distilled water, and left at room temperature for 6, 12 and 24 hours. The three dye samples were then disposed of to dissolve the insoluble materials, then each stored in a clean glass bottle; these steps were repeated using ethanol and ethanol / water 50:50.

Dyeing procedure

Bleached cotton and silk fabric Samples each of 5 gram were dyed separately in a dye bath of henna dye (L: R 1:50) according to extraction media and time using exhaustion methods. Each specimen was immersed in a dye bath composed of Henna extract (5%) and potassium aluminium sulphate (alum) (15g/l). Dyeing was carried out at 80°C for, 60 min. Thereafter, cotton fabrics were rinsed thoroughly in tap water followed by soaping soap (2g/L), Na_2CO_3 (3g/L), liquor ratio (1:40), at 95°C, for 5 min. The samples were rinsed and dried. The above dyeing steps were carried out using ethanol, water, and ethanol\water extract for different intervals of time, for cotton and silk dyeing under the same conditions described.

Color fastness tests

Dry and wet rubbing fastness of the dyed cotton and silk were tested according to ISO 105-X12 method using the crock meter and the grey scale for assessing change in

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color and for staining. Wash fastness of the samples dyed under the prescribed conditions, was tested according to ISO 105-CO3 method with a launder tester using the grey scale for assessing change in color and for staining. The samples were washed in standard soap solution at 60 C^{o} for 30 min. The liquor to material ratio was kept (L:R 1:50). Light fastness was tested according to ISO 105-BO2 method. The dyed cotton and silk fabrics were exposed to xenon arc lamp for 24 hour at standard testing conditions using the blue scale. All tests were performed for henna-dyed cotton and silk. The color fastness rates were evaluated according to the type of solvent used and the extraction time (6, 12, and 24 hours) (Anon, 1990).

Measurement of dye absorbency, reflectivity, and dye strength

The absorbency of dye solution was measured using spectrometer after being calibrated using distilled water, ethanol, and water\ethanol according to the material to be tested. The wavelength was set from 400nm up to 700nm and the readings were recorded. Reflectance and dye strength values were calculated using the following equations:

A = log10 (1 \ R) : R = 1 \ 10A , Where: A= absorbance, R= reflectance according to of Beers and Lambert Law, (Bassam, 2010)

K \ S = $(1 - R)^2$ \ 2R , Where K \ S: dye strength, R: reflectance according to Kubelka and Mank equation (Al-Najawi, 1986).

V. RESULTS AND DISCUSSION

The following Figure 1 shows the influence of the extraction media and the extraction time on the final result of the hue of henna dye. Figure 1 is shown in Appendix. As can be observed from figure 1 when water is used, the color obtained is green, as time of extraction increases from 6 to 12 hours the depth of color increases, but it decreases as the extraction time is increased to 24 hours. While extraction with ethanol it is noticed that the color has changed to yellowish red. Comparatively the deepest color was obtained at 12-hour extraction time using ethanol / water.

Results of fabric dyeing

The influences of the extraction media and time on the final shade of dyeing of cotton and silk fabrics are shown as in figure 2. Figure 2 is shown in Appendix. From figure (2) it is observed that dark colors can be obtained from henna extracted at 12 hours and applied to cotton and silk materials for all the three extraction media. At extraction time of 6 hours a relatively low depth of shade was obtained, this might be due to the insufficient extraction time. At 24-hour extraction time a relatively low depth of shade was also obtained, possibly due to color bleeding and migration from fabric to dye bath.

Color Fastness to rubbing

As can be observed from Figure:3 it is revealed that rubbing fastness rate of the dyed material increases with the increase of extraction time from 6 up to 12 hours, thereafter it decreases at 24 hours extraction time. Rubbing fastness rate is affected by the extraction medium. Water extraction has shown the least rubbing fastness rate while, the fabric dyed with (water \ ethanol) extract has recorded the highest rate of rubbing fastness. The best results of rubbing fastness for cotton and silk were obtained when henna dyes are extracted at 12 hours using (water \ ethanol) medium.

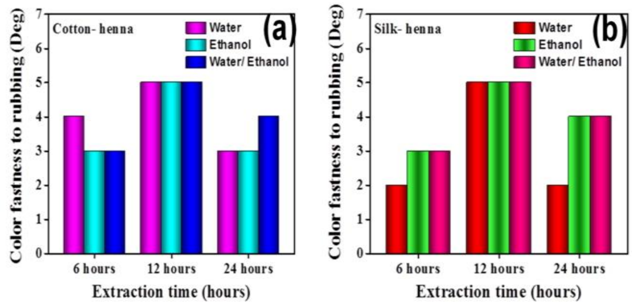

Fig 3: Influence of extraction media and time on rubbing fastness of cotton and silk

Color Fastness to washing

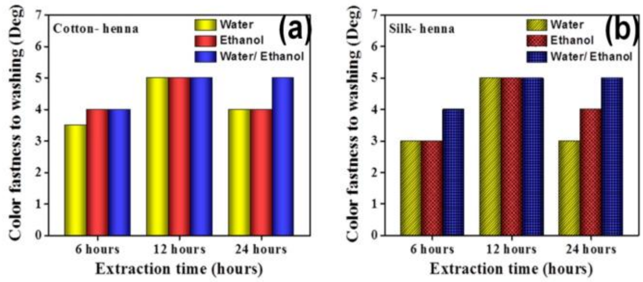

Fig 4: Influence of extraction media and time on washing fastness of cotton and silk

As in Figure: 4, it can be revealed that the color fastness to washing of cotton and silk dyed with henna increases when the extraction time increases from 6 to12-hours while, it starts to decrease to a minimum when the dye extraction is conducted with (water \ ethanol) at 24 hours. The best color fastness results were obtained when extraction of dye was carried out for 12 hours using (ethanol \ water).

Color Fastness to light

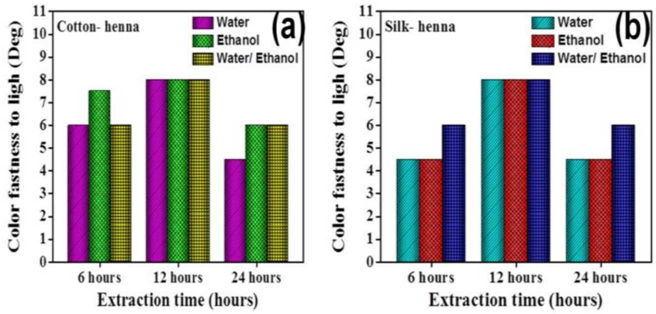

Fig 5: Influence of extraction media and time on light fastness of cotton and silk

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Based on the results obtained in Figure (5) it can be revealed that the color fastness to light for silk and cotton dyed with henna extract increases with the increase of extraction time from 6 to 12 hours, and then it decrease as the extraction time is increased to 24 hours. The best results of light fastness were obtained when using dyes extracted with (water \ ethanol) at the extraction time of 12 hours, this can be interpreted by the fact that some active chemical components of henna are water soluble, while the other components are soluble in ethanol.

VI. RESULTS OF ABSORBANCE, REFLECTANCE AND DYE EXHAUSTION% OF THE DYE SOLUTION

From Table 1, it can be observed that the absorbance of henna dye solution prior to dyeing is higher than that after dyeing of cotton, as a consequence of the transfer of dye to the surface and into the fabric. The highest absorbance value (2.76) of the dye solution is observed when using (ethanol \ water) at extraction time of 12 hours, which indicates that henna dye solution contains more absorbing molecules of dye, which is consistent with its higher dye strength. When comparing the values of absorbency of the dye solution after dyeing to that before dyeing, lower values are obtained for the dye solution remained after dyeing, the least of which is obtained when using ethanol \ water solvent for extraction at 12 hours, this means that lower dye molecules are remained in the dye bath, therefore it will justify higher dye strength and higher dye exhaustion% for cotton.

The reflectance of henna dye solution before the dyeing process is lower than that after dyeing of cotton fabric as a result of transference of dye molecules to the surface and into the fabric. Reflectivity of henna dye solutions before dyeing have recorded lower values compared to that obtained after dyeing for all the dye solutions extracted at different periods of time. At 12 hours extraction times, the reflectivity of dye solutions has shown lower values, compared to the rest of other solutions of dye extracts.

It can be noticed that the exhaustion% of henna dye is higher on using a 12-hour extracted dye for all the dye extracts. The solvent (ethanol \ water) has given the highest exhaustion (80%), which is compatible with the high dye strength of the extracted dye and the high affinity of cotton towards henna dye.

From Table 2, it can be noticed that the absorbance of dye solution before dyeing is higher than that after dyeing, which confirms that dye molecules were transferred to the surface and into silk fabric. The maximum absorbance of dye solution (2.76) extracted with ethanol / water at 12 hour extraction is relatively higher compared to the rest of the dye extracts at different extraction conditions which indicates a high dye strength However, when we compare absorbance of dye solution after silk dyeing to that after cotton dyeing, it can be observed that relatively higher absorbance values of the remaining dye solution are obtained after silk dyeing than that after cotton dyeing, due to low affinity of silk to henna dye and high substantively of cotton.

It can be revealed that reflectance of henna dye solution prior to dyeing is lower than that after dyeing of silk, due to transfer of dye particles into the fabric. When dye is extracted at 12 hours, lower values of reflectance are obtained compared to the rest of dye solution extracted at (24, and 6 hours). It can also be observed that the reflectance of henna dye solution after dyeing of silk is higher than reflectance of residual dye bath after dyeing of cotton fabric which, might be due to the high affinity of cotton to henna dye.

It can be observed that the exhaustion (75%) of henna dye extracted with ethanol\ water at 12 hours is comparatively higher than the dye extracts at different conditions, which can be strongly correlated to the higher dye strength of henna extract and its high affinity to fabric. Therefore, the exhaustion% of the cotton is higher than that of the silk fabric due to the relatively lower affinity of silk to henna dye and higher affinity of henna to cotton fibres. Table 1 and 2 are shown in Appendix.

Dye strength

Table 3: Influence of extraction media and time on the dye strength of dyed cotton.

Extraction media	Water	Ethanol	Water\ Ethanol
Dye strength / Extraction time	K\S	K\S	K\S
6 hours	4.3	4.1	6.1
12 hours	5.9	5.4	6.8
24 hours	5.8	5.2	6.7

As in table: 3 It can be revealed that k\s values of cotton dyed with henna increases with the increase of extraction time, until it reaches its maximum value at 12 hours, then becomes constant at 24 hour extraction, comparatively higher dye strength is obtained for cotton dyed with henna extracted with (ethanol / water), this can be justified by; some dye components are water soluble, while the others are ethanol soluble.

Table 4: Influence of extraction media and time on the dye strength of dyed silk.

Extraction media	Water	Ethanol	Water\ Ethanol
Dye strength of fabric / Extraction time	K\S	K\S	K\S
6 hours	4.1	4.0	5.1
12 hours	5.2	4.5	5.8
24 hours	5.1	4.3	5.7

The results obtained in Table 4 disclose that with increasing the extraction time of henna from 6 to 12 hours, the K/S values are enhanced, which reflects the positive impact of extraction time, on improving the k\s of silk fabrics. The maximum k\s value is obtained when extraction is conducted with water \ ethanol at 12 hours extraction time. On comparison of the k\s values of dyed cotton and silk at the same extraction media and time, it can be said that cotton fabric dyed with henna can give higher k\s values than silk, this is due to its relatively high exhaustion%, obtained as in table 1 and table 2, in addition to that cotton has high affinity to henna dye relative to silk fabric.

VII. CONCLUSION

Based on the results of extraction of dye from henna, dyeing of cotton and silk, and the test results of the dyed materials, the following could be concluded:

Higher dye strength of henna based on extraction medium, and time could be obtained. As can be observed, the color strength of dyed fabric increases with the increase of extraction time and reaches its maximum value at 12 hours. Further increase in extraction time up to 24 hours results in decrease of color strength. On extraction with (ethanol / water) at 12 hours, relatively higher dye strength (k/s = 6.8) is obtained. When water medium is used for extraction, the lowest dye strength is obtained because; cotton has high affinity to henna and has more porous structure than silk. The color fastness rates to washing, light and rubbing for all the dyed cotton and silk fabric samples are in the range of very good to excellent. The maximum dye exhaustion obtained is 80% for cotton and 75% for silk.

The study recommends the application and localization of henna dyes to cotton and silk as they are environmentally friendly, renewable resources available in Sudan, as well as their extraction and application does not require high technology. The paper recommends further study on different dyeing conditions and the use of other types of solvents and their mixtures to increase the efficiency and optimize the dye extraction, dye strength, and color yield of dyes from henna.

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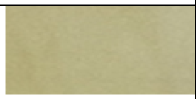
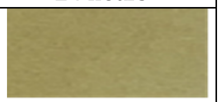
APPENDIX

Method of extraction	Dye extracted in 24 Hours	Dye extracted in 12 Hours	Dye extracted in 6 Hours
Water			
Ethanol			
Ethanol+ water			

Fig 2(a): Influence of the extraction media and time on the hue of henna dye.

Fabric	Extraction medium	Dye extracted in 6 hours	Dye extracted in 12 hours	Dye extracted in 24 hours
Cotton	Water			
	Ethanol			
	)Ethanol + Water(			
Silk	Water			

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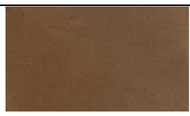
	Ethanol			
	Ethanol + Water			

Fig 2(b): Influence of the extraction media and extraction time on dyed cotton and silk

Table 1: Influence of extraction media and time on properties of the dye solution of cotton.

Extraction media	Water			Ethanol			Water \ Ethanol		
Extraction time / **Dye properties**	6 Hours	12 Hours	24 Hours	6 Hours	12 Hours	24 Hours	6 Hours	12 Hours	24 Hours
Maximum absorbance (A) before dyeing	1.63	2.21	1.59	1.87	2.31	1.78	2.03	2.76	2.01
Maximum reflectance (R) before dyeing	0.009	0.006	0.010	0.013	0.005	0.017	0.023	0.002	0.026
Maximum absorbance (A) after dyeing	1.23	1.19	1.21	1.42	1.11	1.65	1.90	1.10	1.64
Maximum reflectance (R) after dyeing	0.013	0.065	0.023	0.038	0.078	0.022	0.059	0.079	0.062
Dye exhaustion %	36.4	46.2	32.6	44.1	51.9	37.3	54.5	80.1	53.9

Table 2: Influence of extraction media and time on properties of dye solution of silk.

Extraction media	Water			Ethanol			Water + Ethanol		
Extraction time / **Propertie of dye**	6 Hours	12 Hours	24 Hours	6 Hours	12 Hours	24 Hours	6 Hours	12 Hours	24 Hours
Maximum absorbance (A) before dyeing	1.63	2.21	1.59	1.87	2.31	1.78	2.03	2.76	2.01
Maximum reflectance (R) before dyeing	0.023	0.006	0.026	0.013	0.005	0.017	0.009	0.002	0.010
Maximum absorbance (A) after dyeing	1.54	1.29	1.37	1.52	1.25	1.66	1.94	1.24	1.91
Maximum reflectance (R) after dyeing	0.029	0.051	0.043	0.030	0.056	0.022	0.011	0.058	0.012
% Dye exhaustion	35.5	41.6	43.8	38.7	45.9	41.7	54.4	75.0	52.02