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Optimization of solvents extraction and application of dye from *Saccharum officinarum* leaves on textile

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Abstract
In recent years, there has been growing interest in finding sustainable and ecofriendly alternatives in the textile industry. This research has focused its effort on exploring *S. officinarum* as natural dye source that offer viable alternatives to synthetic dyes. Two different extraction solvents, namely, ethanol and aqueous solution of optimal solvent (ethanol/water, 140:60 v/v) were employed to extract dye from the plant. Of the two solvents used, aqueous solution of the optimal solvent extract obtained had the optimum absorbance value 1.54 at temperature 80 ^{0}C with 26.71 percentage recovery while ethanol with the absorbance value 1.86 at temperature 80 ^{0}C with percentage recovery 29.95 respectively within the wavelength 200-800 nm. The dye extract was simultaneously applied to the dyeing of cotton and wool fabrics with mordants such as ferrous sulfate, alum and copper sulfate. Percentage exhaustion in the dye bath was evaluated using UV-Visible Spectrophotometry. The rate and degree of dye fixation on the fabrics increased significantly by elongating the dyeing time. The light, wash and perspiration to fastness of the dyed fabrics were evaluated. The results demonstrated good to very good with a rating range of (3-4) for wash fastness, good to excellent with a rating range of (3-5) for perspiration fastness and moderate to excellent with a rating range of (4-7) for light fastness. The colour fastness grades of the wool and cotton fabrics dyed with natural dyestuff and mordants exhibited a remarkably results. All these impressive results are important evidence that the plant which is not economically evaluated, can be utilized in the textile industry.

Keywords: Natural dye, extraction, application, textiles, *S. officinarum, mordants*, UV-VIS spectrophometer, exhaustion, fastness properties

Introduction

Natural dyes, derived from plants, insects and mineral have been used to colour products in many areas such as food, textiles, etc., from the inception till now (Dayioglu *et al.*, 2016; Kilinc *et al.*, 2015; Shukla and Vankar 2013) [11, 17, 23]. The use of natural dyes for textile dyeing purposes gradually decline to a large extent after the discovery of synthetic dyes in1856, owing to cheapness and its easily availability (Siva, 2007 and Ali *et al.*, 20ll) [24]. There is a serious concern on the incessant usage of synthetic dyes in textile industry as they incur water pollution and waste disposal problems which may affect the ecosystem (Ali *et al.*, 2013) [6]. In response to these concerns, researchers have focused their efforts on discovery natural dye sources that are environmentally sustainable and offer viable alternatives to synthetic dyes (Maleki and Barani, 2019) [18]. One such potential natural dye source is *S. officinarum* leaves, is a long and narrow leaves with pointed tip and a waxy coating that helps to present water (Parihk and singh, 2015). The leaves are important source of various policosanols and D-003. The plant leaves are also reported to have some phenolic compounds like flavonoids, a colouring matters which HPLC micro fractionation of methanolic extract of the leaves was successfully done and various flavones -O- and -C- glycosides (39 - 40, 46 - 47, 53 - 54) were identified (Amandeep *et al.*, 2015) [7]. This study investigated the potential of *S. officinarum* leaves as a source of natural dye and its application in dyeing textile fabrics such as wool and cotton. Due to the relatively low exhaustion of natural dyes, mordants such as ferrous sulfate, alum and copper sulfate is usually employed to improve the colour strength and fastness, and to obtain multiple shades (UI-Islam *et al.*, 2018; Adeel *et al.*, 2018; Barani, 2018) [2, 8]. Natural dye from *S. officinarum* leaves exhibit a better performance on the fabrics as a result of pigments, flavonoid and other pigments that are present which are responsible for dyeing the cotton and wool fabrics (Ndiku and Ndule, 2015) [20]. Therefore, *S. officinarum* leaves presents an opportunity to explore its potential for extracting natural dyes with its application on fabrics, thereby offering a sustainable and economically viable solution.

Materials and Methods

Chemicals and Reagents

Laboratory grade ferrous sulfate ($FeSO_4$), alum KAl $(SO_4)_2$, and Copper sulfate ($CuSO_4$) at concentration of 0.1 M into 100 ml were used as mordants. Solvents such as ethanol (C_2H_5OH) and de-ionize water was used for the dye extraction. Reference detergent A soap (3 g/l) was used to wash the fabrics. 3.0 g/l, Sodium sulfate (Na_2SO_4) was added as exhausting agent. Acetic acid (2 %) was added to the dye bath to considerably reduce the amount of metal mordants in the spent bath.

Soap (5 g) in 1000 ml of distilled water and Soda ash (2 g) was prepared in 1000 ml distilled water for wash fastness test.

Fastness to perspiration test, the acidic solution consists of sodium chloride (5 g/l), disodium hydrogen orthophosphate dehydrates (Na_2HPO_4, 2.5 g/l) and histidine monohydrochloride monohydrate. The pH of the solution was adjusted to 5.5 while the alkaline solution consists of $C_6H_9O_2N_3.HCl.H_2O$ (0.5 g/l) and is adjusted to pH 8 using

0.1 N sodium hydroxide (NaOH). The liquor ratio for the test was 20:1, all of which were of analytical grade and obtained from Merck (Darmstadt, Germany).

Plant Material

The fresh leaves of *S. officinarum* were harvested from their matured plant. The leaves were further identified and authenticated as *Saccharum officinarum* in the Department of Biological Sciences, Abubakar Tafawa Balewa University, Bauchi, Nigeria and given a voucher number (ATBU DBSH:2497) which was deposited at the Departmental Herbarium. All the procedures and collection of plant material was done in accordance with local and national guidelines and regulations. The fresh leaves were washed, chopped into smaller pieces and air dried for three weeks. The leaves were further reduced to powder using grinding machine to facilitate better dye extraction using solvent.

Extraction of Dye (Solvent extraction)

In total extraction, 20.0 g of the pulverized plant material was poured into 250 ml flat bottom flask in the inner part of the soxhlet extractor and 200 ml of ethanol was added and heated under reflux with a condenser using heating mantle at various temperature of 50 ^{0}C, 60 ^{0}C, 70 ^{0}C, 80 ^{0}C, 90 ^{0}C, 100 ^{0}C which varies with time extraction to generate absorbance. Similarly, experiment was conducted with aqueous solution of optimal solvent (140:60, ethanol/water v/v) to compare the extraction power of the solvents (Jinasena *et al.*, 2016) [16]. The process automatically repeat itself and the solvent in the extract was distilled off to obtain the dye extract. A plot of absorbance against temperature was carried out in each case of extraction as shown in Figure 2-3

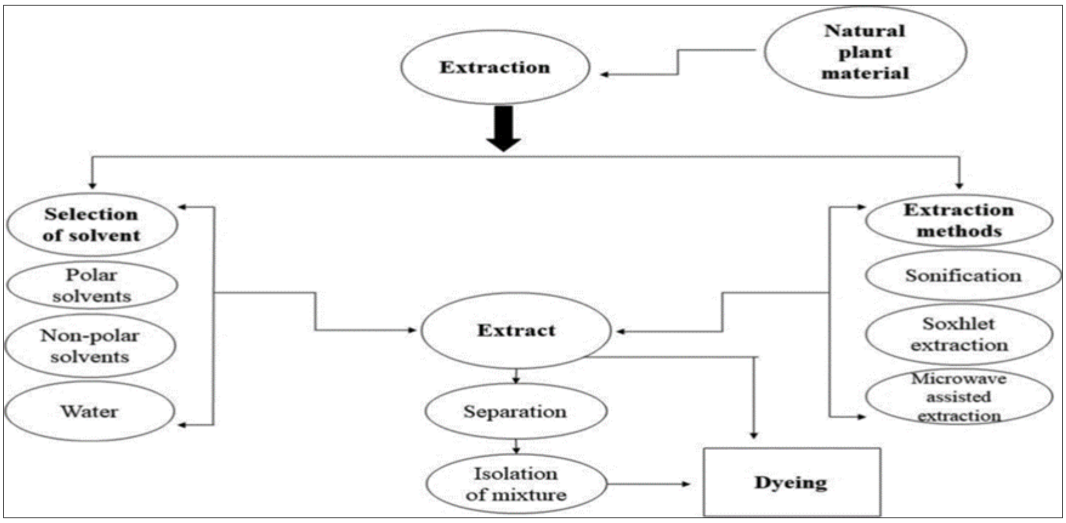

Fig 1: Schematic flow chart of natural dye extraction procedure

UV-Visible Analysis

A JENWAY 6405 UV/visible Spectrophotometer was used for all spectrophotometric measurements. All measurements were carried out using quartz cells 10-mm at room temperature (25±2 ℃) using standard procedures (Jack *et al.,* 2020) [15] to detect the changes in their absorption (200-800 nm) were noted.

Preparation of the fabrics

The cotton and wool were treated in a soap solution of (3 g/l) at 60 ^{0}C for 30 minutes and washed thoroughly with water and air-dried at room temperature.

Preparation of stock solution

Ferrous sulphate, copper sulphate and alum were prepared at concentration of 0.1 M into 100 ml of distilled water, it was then transferred to volumetric flask.

The volume required from each stock solution was calculated based on the formula

$v = p \times w / c$

Where: P = percentage shade

W = weight of fabric

C = concentration of stock solution

Mordanting

In this experiment, the process of mordanting was conducted simultaneously with the dye, referred to as meta-mordanting. The aim of meta-mordanting was to enhance the adsorption of the dye and ensure a strong bond between the dye and the fabrics. The commonly used mordant, such as ferrous sulfate, copper sulfate and alum were selected. Initially, the cotton and wool fabrics were immersed in warm water (approximately 46 ℃) for 30 minutes to relax the fabrics, which would make the fabrics more receptive to mordanting and dyeing. Subsequently, the specific mordanting procedure was carried out based on the information found in the literature (Geetha and Judia Harriet Sumathy, 2013) [14].

Dyeing Procedure

The dyeing procedures were carried out in accordance with the general dyeing method (Geetha *et al.,* 2013) [14]. The extracted solution of the extract was applied to cotton and wool fabrics with mordants. The fabrics were cut into several pieces each weighing 1.0 g. The dye extract and mordants such as ferrous sulphate, copper sulphate and alum were used in the dyeing process. In each case, a few drops of acetic acid (2 %) was added to the dye bath to considerably reduce the amount of metal mordants in the spent baths

The pre-treated cotton and wool fabrics (1.0 g) each were dyed in separate dye baths with the dye extract. The dyeing was carried out using ferrous sulfate, copper sulfate and alum as mordants. The dyeing was conducted at a material to liquor ratio of 1:50 using a shade of 2 % and 6 % on the weight of fabric. The dyeing was carried out at 40 ^{0}C for 1 hour and continues for further 1 hour and 1 ml of the solution was taken at 0, 10, 20, 30, 40, 50, 60 and 120 minutes at different temperature in standard laboratory dye master and it was used to generate absorbance value. After half of dyeing time, Sodium sulfate (Na_2SO_4) of 3.0 g/l was added as exhausting agent. At the end of the dyeing time the samples were removed, washed and dried.

Percentage (%) Exhaustion of dye extract

The absorbance of the extracted dye was determined before and after dyeing at the maximum wavelength (316 λmax) using JENWAY 6405 UV/visible Spectrophotometer (Murugan and Parimelahagan, 2014) [19]. The percentage exhaustion was calculated using the expression below:

% $Exhaustion$ = absorbance before dyeing – absorbance after dyeing x 100 absorbance before dyeing

Colour Measurement

Colour wash to fastness

The wash fastness test was carried out using heating mantle and International Organization for Standardization (ISO) wash fastness test No.3. Soap solution was prepared containing 5 g of soap in 1000 ml of distilled water and sodium carbonate (Soda ash) was prepared containing 2 g in 1000 ml distilled water. The dyed samples fabrics was sandwiched between un-dyed cotton and wool fabrics and subjected to the wash fastness test. The composite specimens were separately agitated in 100 ml beaker containing the soap solution (15 ml), sodium carbonate (10 ml) plus distilled water to give a liquor ratio of 50 ml. The cotton and wool fabrics pieces were immersed in the washing solution and heated for 30 minutes at 40 ^{0}C to 60 ^{0}C. The composite specimens were then removed, rinsed and the components separated and dried. The assessment of colour changed and staining of the dyed specimens was assessed with the appropriate grey scale rating between 1-5.

Colour fastness to light

The light fastness was assessed by exposing the fabrics to the Xenon Arc Lamp of a Fedo meter, according to the conditions of AATCC Test Method 1.6 E-1990 (AATCC, 1990; ISO 1994). A small piece of the dye weighing 0.25 ± 0.02 g was cut and mounted on pattern cards (Blue Wool Scale). The exposed side was labeled E and the unexposed side labeled UE. The cards were placed in a Fedo Meter; the light turned on and left for about 72 hours so that an appreciable colour change exists with respect to the unexposed side. The conditions for the test were: black panel temperature 63 ^{0}C, dry bulb temperature, 43 ^{0}C; relative humidity 30 %. After testing, the samples were rated against standard blue wool scale.

Colour fastness to perspiration

The test measures the resistance of the colour of textile fabrics of all kinds to perspiration in all forms. Perspiration was carried out under acidic and alkaline solutions; the acidic solution consists of sodium chloride (5 g/l), disodium hydrogen orthophosphate dehydrates (Na_2HPO_4 2.5 g/L) and the pH of the solution was adjusted to 5.5 while the alkaline solution consists of histidine, monohydrochloride monohydrate ($C_6H_9O_2N_3.HCl.H_2O$ 0.5 g/l) and was adjusted to pH 8 using 0.1 N sodium hydroxide (NaOH). The liquor ratio for the test was 20:1. Composite sample was made by sand-witching the dyed sample measuring (2×3 cm) between two pieces of un-dyed bleached cotton and wool fabrics measuring (2×3 cm), the composite specimen was thoroughly wetted in this solution (acidic and alkaline) at room temperature for 30 minutes. At the end of 30 minutes the composite specimen was removed from the solution and the composite sample placed between two glasses plates measuring about 7.5×6.5 cm under a force of about 4.5 kg. The apparatus containing the treated composites was then placed in a perspirometer at 37 + 2 ^{0}C for 4 hours. After 4 hours, the specimens were removed from the perspirometer and dried at room temperature (Clark *et al.*, 2023) [23]. The change in colour of the specimen was assessed using grey scale.

Results and Discussion

Results

Extraction Recovery

The extraction recovery was calculated, this was done by measuring the solvent efficiency to specific components from the original material (Murugan and Parimelahagan, 2014) [19]. This will be defined as amount of extract recovery in mass compared to the initial amount of whole plant. This is presented in percentage (%). Extraction recovery (%) = Initial weight – Final weight × 100 Initial weight

Table 1: Extraction recovery of *S. officinarum* using ethanol

Temp 0C	Initial weight (g)	Final weight (g)	Extraction recovery (%)
50	20	16.874	15.63
60	20	16.106	19.47
70	20	15.380	23.6
80	20	14.010	29.95
90	20	17.252	13.74
100	20	17.220	10.9

Table 2: Extraction recovery of *S. officinarum* using aqueous solution of optimal solvent

Temp °C	Initial weight (g)	Final weight (g)	Extraction recovery (%)
50	20	17.154	14.23
60	20	16.680	16.6
70	20	15.750	21.25
80	20	14.658	26.71
90	20	17.112	10.94
100	20	18.320	8.4

Solvents extraction of natural dye at different temperature

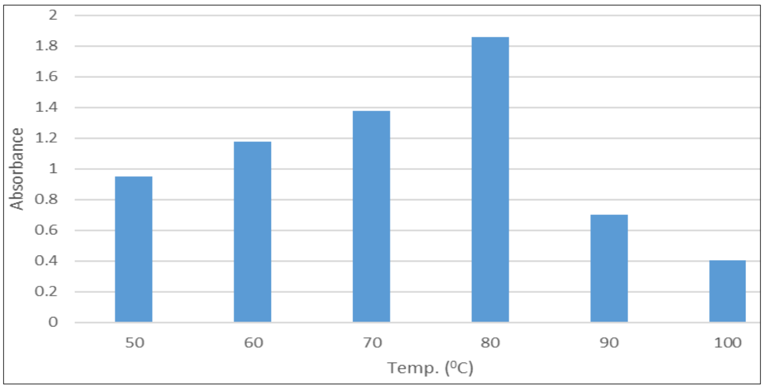

Fig 2: Effects of different temperatures on absorbance of dye extract from *S. officinarum* using ethanol as solvent at λmax 316

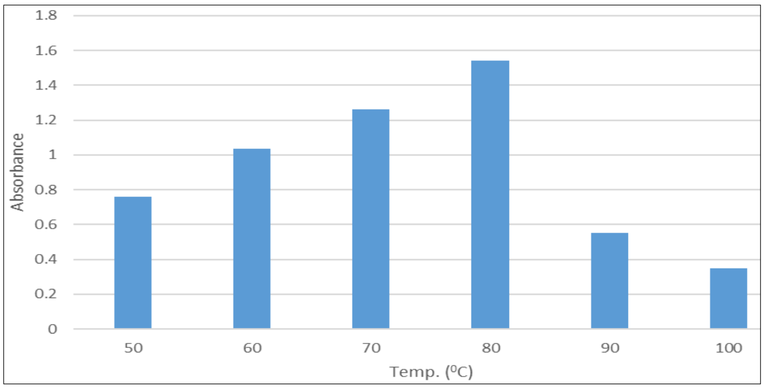

Fig 3: The effect of temperature on absorbance of dye extract from *S. officinarum* using aqueous solution of the optimal solvent at λmax 316

Percentage exhaustion of dye extract

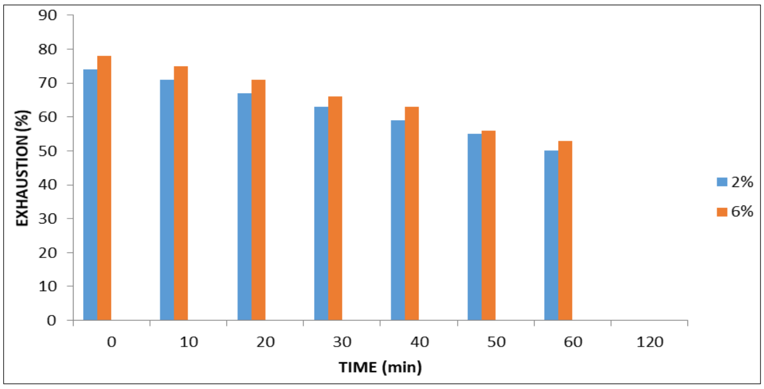

Fig 4: Percentage (%) exhaustion of dye extract from *S. officinarum* on cotton fabric at various minute and at λmax 316

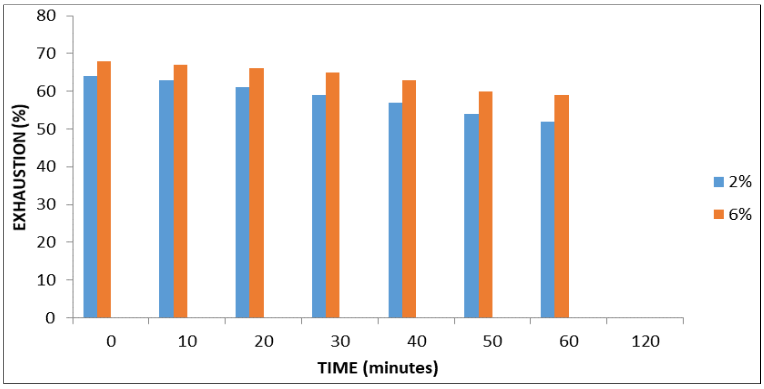

Fig 5: Percentage (%) exhaustion of dye extract from S. *officinarum* at various minute on wool fabric and at λmax 316

Fig 6: Cotton and wool fabrics dyed with dye extract and test for wash fastness

Fig 7: Cotton and wool fabrics dyed with dye extract and test for perspiration fastness

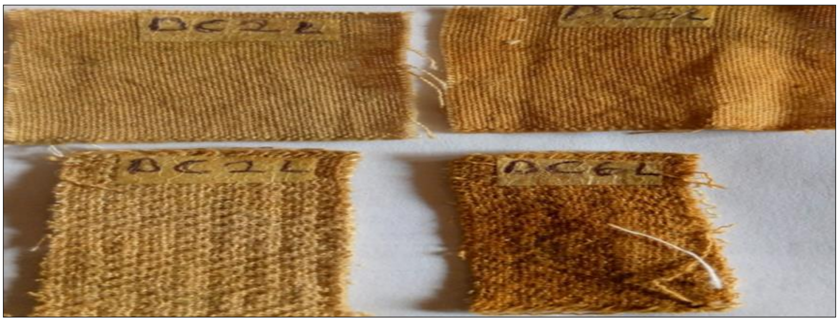

Fig 8: Cotton and wool fabrics dyed with dye extract and test for light to fastness

Fastness Properties

Table 3: Colour fastness to wash

Fabrics	Sample code	Colour change	Staining
Cotton	SO 2%	4	3
Cotton	SO 6 %	3/4	3
Wool	SO 2 %	3	2
Wool	SO 6 %	3/4	3

Key: SO- Dye concentration, 1- Very Poor, 2- Fair, 3- Good, 4- Very Good, 5- Excellent

Table 4: Colour fastness to perspiration test

Fabrics	Sample code	Acid	Alkaline	Staining
Cotton	SO 2 %	3	4	3
Cotton	SO 6 %	4	4	3
Wool	SO 2 %	4	4	4
Wool	SO 6 %	4/5	4	3

Key: SO- Dye concentration, 1- Very Poor, 2- Fair, 3- Good, 4- Very Good, 5- Excellent

Table 5: Colour fastness to light

Fabrics	Sample code	Colour change
Cotton	SO 2%	4
Cotton	SO 6 %	5
Wool	SO 2 %	5
Wool	SO 6 %	7

Key: SO- Dye concentration, 1-very poor, 2-poor, 3-fair, 4-moderate, 5- good, 6-very good, 7-excellent, 8-outstanding

Discussion

Solvents extraction at different temperature

The results of solvents extraction of dye from *S. officinarum* using two different extraction solvents, namely, ethanol and aqueous solution of the optimal solvent (ethanol/water, 140:60 v/v) were employed to extract dye from the plant sample at different temperature. The results obtained from the graphs (Figure 2-3) above showed the relationship between absorbance and temperature changes. The results revealed that of the two solvents used for the extraction of dye from *S. officinarium*, ethanol at temperature 80 ^{0}C was observed to have maximum absorbance value of 1.86 with 29.95 percentage recovery while aqueous solution of optimal solvent at temperature of 80 ^{0}C had maximum absorbance value of 1.54 with 26.71 percentage recovery within the wavelength 200-800 nm. This increased of dye extract was due to the effect of temperature, increase of temperature may be caused by an increase in pigment molecule diffusivity and pigment solubility. The results revealed that temperature 80 ^{0}C has the highest yield which agreed with the results obtained in literature by Abd Razak *et al.*, (2011) [1] which suggested that natural organic dyes are stable at temperature less than 80 ^{0}C. Therefore, temperatures more than 80 ^{0}C probably caused a decrease in the dye extract due to chemical structure degradation of pigments. Therefore, the optimal temperature 80 ^{0}C has the highest yield using both solvents which suggested that dye extract of this plant are stable at temperature less than 80 ^{0}C.

Percentage (%) Exhaustion of dye extract

The absorbance of the dye extract was determined before and after dyeing at the maximum wavelength (316 λmax) using JENWAY 6405 UV/visible Spectrophotometer are presented in the Figure 4-5. The percentage exhaustion of the dyed fabrics was carried out in eight different time (0, 10, 20, 30, 40, 50, 60 and 120 minutes) in the dye bath at temperature different temperature. Dye solution of 1 ml was taken from the dye bath to generate absorbance using UV-Visible Spectrophotometry. The amount of dye extract that was exhausted and fixed on the fabrics was evaluated. The rate and degree of dye fixation on the fabrics increased significantly by elongating the dyeing time. The decrease in absorbance in the dye bath to zero after dyeing with the fabrics at 120 minutes owning to high temperature that increase the kinetic energy of dye molecules, allowing for better penetrating and binding. Also the high degree of exhaustion in the dyed bath with cotton fabric was due to the fact that cotton composed of cellulose, a polysaccharide with hydroxyl (-OH) groups that form hydrogen bonds with water and dye molecule allowing them to penetrate the fabric; while that of wool was due to keratin, a protein with amino (-NH_2) and carboxyl (-COOH) groups, that form ionic bonds with dye molecules, allowing them to bind to the fabrics.

Fastness Properties

Colour Fastness to Wash

Wash to fastness of dye extract from *S. officinarium* are presented in Table 3 and Figure 5. The natural dye extract was applied on cotton and wool simultaneously with mordants. The cotton fabric dyed with OS 2 % and OS 6 % concentration revealed a rating of (4) and (3/4) which indicated very good and good to very good performance on the fabric. Also, with OS 2 % and 6 % OS concentration, a rating of (3) which agreed witzh the results obtained by Clark *et al.* (2023) [23] and a rating of (3/4) for wool fabric which showed a good and very good performance on the wool fabric. Thus, the results indicate that dye extract from *S. officinarum* exhibited a better wash fastness performance on both fabrics.

Fastness to Perspiration test

The perspiration to fastness on cotton and wool fabrics dyed with *S. officinarum* extract was evaluated under acidic and alkali conditions, as shown in Table 4 and Figure 6. The fabrics (mordanted with ferrous sulfate, copper sulfate and alum) at 2 % and 6% concentration on dyed fabrics had a rating of (4) which was in agreement with Shariful Islam *et al.* (2020) [22] indicate a very good performance on both fabrics for alkaline perspiration. Also, a very good to excellent fastness with a rating of (4/5) was observed on dyed wool for acidic perspiration with very light staining on adjacent fabrics at 6 % concentration. Finally, a good fastness with a rating of (3/4) at 2 and 6 % concentration was observed on cotton fabric. These results indicate that the acidic and alkali extract from *S. officinarum* dye extract can produce fabrics that are resistant to perspiration in different environments.

Colour Fastness to Light

Colour fastness to light test was conducted on the dyed fabrics to evaluate their resistance to light exposure using Xenon Arc Lamp of a Fedo meter. Dye extract at SO 2 % and 6 % concentration on cotton and wool had a rating (5) which was in agreement with Shariful Islam *et al.* (2020) [22] revealed good fastness to light. However, at SO 2 % concentration on cotton received a rating (4) and at SO 2 % on wool received a rating of (7) on the blue wool scale after 48 hours (Table 5 and Figure 7). This indicates that dye extract on wool fabric exhibited a better light performance than cotton. Thus, cotton and wool fabrics can be effectively dyed using *S. Officinarum* dye extract. The presence of flavonoids in the dye extract, as mentioned by Adeoye (2021) [3], may be responsible for this successful dyeing process. Flavonoids are phenolic compounds that can form hydrogen bonds with carboxyl groups present in protein fibres such as wool (Agarwal and Patel, 2002) [4]. Additionally, Burkinshaw and Kumar (2009) [9] suggested that the characteristics of mordants like ferrous sulfate, copper suphate and alum play a more significant role in determining the fastness properties of natural dyes.

Conclusion

Natural dye was extracted from *S. officinarum* leaves using ethanol and aqueous solution of optimal solvent. The present results showed that temperature at 80 ^{0}C had the maximum absorbance value for the two solvents within the wavelength 200-800 nm. Temperatures above 80 ^{0}C probably caused a decrease in the dye extract due to chemical structure degradation of pigments in the plant. The dye extract was applied on the cotton and wool fabrics simultaneously with ferrous sulfate, copper sulfate and alum as mordants. The percentage exhaustion of the dye extract was determined, the rate and degree of dye fixation on the fabrics increased significantly by elongating the dyeing time which resulted to decrease in absorbance in the dye bath to zero at 120 minutes owning to high temperature that increase the kinetic energy of dye molecules, allowing for better penetrating and binding. The natural dye at different concentration of dye extract with mordants exhibited a better performance on the fabrics as a result of some pigments, such as tannins, indigoid, and flavonoid (Ndiku and Ndule, 2015) [20] etc. which are responsible for dyeing the fabrics. Thus, the dye showed improvement on cotton and wool fabrics with the application of mordants.

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