Exploring the Environmental Sustainability of Plant-Based Dyes from *Megathyrsus Maximus* Leaves: Extraction and Application on Textile

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DOI: https://doi.org/10.51584/IJRIAS.2025.100800006

Received: 21 July 2025; Accepted: 27 July 2025; Published: 29 August 2025

ABSTRACT

The intriguing use of natural dyes has grown rapidly owing to the fact that natural dyes are environmentally friendly. The objective was to extract and apply natural dyes on textile fabrics that offer viable alternatives to synthetic dyes. The Soxhlet extraction process using ethanol and an aqueous solution of optimal solvent (ethanol/water, 140:60 v/v) was employed to extract dyes from the plant. Concentration of the extract was then carried out using rotary evaporation at 40°C to obtain the crude dye extract. Among the two solvents used for extraction, the aqueous solution of the optimal solvent extract of the plant dye showed an absorbance value of 1.607 at a temperature of 80°C with a 27.475 extraction yield, while ethanol had an absorbance value of 1.804 at the same temperature with a 30.4 extraction yield within the wavelength of 200-800 nm. The crude extract was simultaneously applied to the dyeing of cotton and wool fabrics with mordants such as ferrous sulphate, alum, and copper sulphate. The amount of natural dyes exhausted and fixed on the fabrics was evaluated at different times using UV-visible spectrophotometry. The rate and degree of dye fixation on the fabrics increased notably by prolonging the dyeing time. The perspiration fastness, light fastness, and wash fastness properties were evaluated; the results reveal fair to very good with a rating range (2-4) for both wash and perspiration fastness, while for light fastness, moderate to very good with a rating range (4-6) on the dyed fabrics. The colourfastness grades of these fabrics depict an excellent result. Thus, the application of *M. maximus* dye extract on other textile substrates such as nylon, polyester, and acrylics is therefore recommended for further study.

Keywords*:* Natural dye, Extraction, Application, *M. maximus*, Fastness properties

INTRODUCTION

Natural dyes are derived from animals, plants, and minerals, with dyes from plants as the main source (Ferreira *et al.,* 2004). The demand for natural dyes has increased currently over the world due to available information about their beneficial traits (Aggarwal, 2021). A recent study on the potential dye-yielding plants revealed that indigenous dye-yielding plant species impart fascinating colour shades on fabrics (Wanyama *et al.,* 2011). There has been an increasing tendency towards the use of sustainable and environmentally friendly natural dyes (Shahid-Ul-Islam and Sun, 2017). The pursuit of dyes from nature for textiles is on the rise due to the awareness of the adverse effect of some synthetic dyes on the environment and human health (Iqbal and Ashiq, 2007). Currently, many commercial dyers have started using natural dyes as an alternative source to synthetic dyes (Patel, 2011). With regard to this problem, researchers have focused their efforts on exploring natural dye sources for environmental sustainability and as an alternative to synthetic dyes (Maleki and Barani, 2019). On this note, natural dyes from *M. maximus offer promising*

ISSN No. 2454-6194 | DOI: 10.51584/IJRIAS |Volume X Issue VIII August 2025

potential as an environmentally friendly alternative. The plant is commonly referred to as guinea grass, a major pantropical grass used for pasture, silage, and hay (Simon *et al.,* 2005). It is a large, fast-growing perennial grass with a broad morphological, ranging in height from 0.5 to 3.5 m with stems of 5 mm to 10 mm diameter (Cook *et al.,* 2005). The leaves are blade-shaped and contain more alkaloids and saponins (Adekunle *et al.,* 2005). The objective was to extract and apply the natural dyes on textile fabrics such as wool and cotton. Due to the relative low exhaustion of natural dyes, mordants such as ferrous sulphate, copper sulphate and alum are 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). Natural dye extracts from *M. maximus* leaves exhibit a better performance on the fabrics as a result of pigments, alkaloids, saponins, flavonoids etc. that are present, which are responsible for dyeing the cotton and wool fabrics (Ndiku and Ndule, 2015). Therefore, the plant presents an opportunity to investigate its potential for the extraction of dye and its application on fabrics such as wool and cotton, thereby offering a sustainable and economic solution.

MATERIALS AND METHODS

Chemicals and Reagents

Laboratory-grade ferrous sulphate, alum, and copper sulphate at a concentration of 0.1 M in 100 ml were used as mordants. Solvents such as ethanol (C_2H_5OH) and deionised water were used for the dye extraction. Reference detergent A soap (3 g/L) was used to wash the fabrics. Sodium sulphate (Na_2SO_4) of 3.0 g/l was added as an 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) were prepared in 1000 ml of distilled water for the wash fastness test.

For the fastness to perspiration test, the acidic solution consists of sodium chloride (5 g/L) and disodium hydrogen orthophosphate dehydrate (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 the solution was adjusted to pH 8 using 0.1 N sodium hydroxide (NaOH). The liquor ratio for the test was 20:1, all of which was of analytical grade and obtained from Merck (Darmstadt, Germany).

Plant Material

The fresh leaves were further identified as *Megathyrsus maximus* from the Department of Biological Science, Abubakar Tafawa Balewa University, Bauchi, Nigeria, and were given a voucher number (ATBU DBSH: 2498), which was deposited at the Departmental Herbarium. All the procedures and collection of plant material were done in accordance with local and national guidelines and regulations. The fresh leaves were washed, chopped into smaller pieces, and air-dried for weeks. The leaves were further reduced to powder form to facilitate better dye extraction using solvents.

Extraction of Natural Dyes (Solvent extraction)

Natural dyes were extracted from the plant using Soxhlet extractor. The powdered sample (20 g) was weighed and poured into a 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 a heating mantle at various temperatures of 50°C, 60°C, 70°C, 80°C, 90°C, and 100°C to generate absorbance. A similar experiment was conducted with an aqueous solution of optimal solvent (140:60, ethanol/water v/v) to compare the extraction power of the solvents (Jinasena *et al.,* 2016). The process automatically repeated itself and was concentrated. A plot of absorbance against temperature was carried out in each case of the extraction as shown in Figure 2-3

ISSN No. 2454-6194 | DOI: 10.51584/IJRIAS |Volume X Issue VIII August 2025

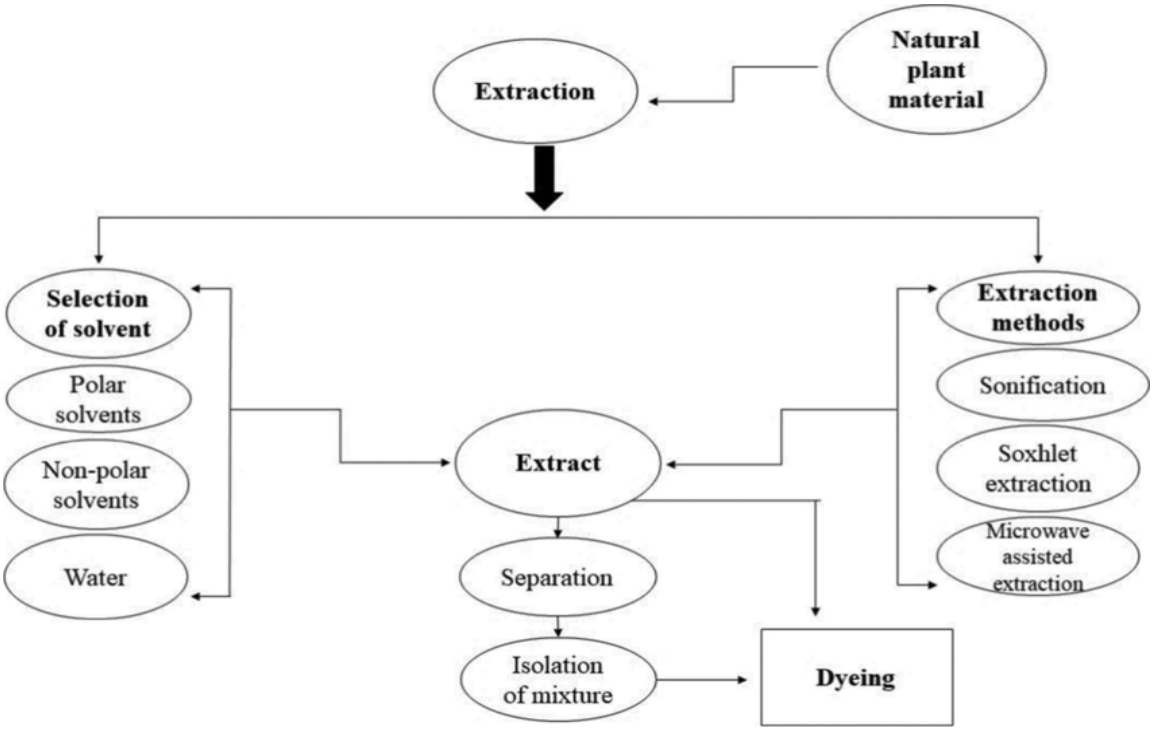

Figure 1 Schematic flow chart of natural dye extraction procedure

Source:www.researchgate.net/figure/Schematic-flow-chart-of-natural-dye-extraction (2023)

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) 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 were washed thoroughly with water and air-dried at room temperature.

Preparation of stock Solution

Mordants such as ferrous sulphate, copper sulphate, and alum were prepared at a concentration of 0.1 M into 100 mL of distilled water, which was then transferred to a 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 with the dyes, referred to as simultaneous mordanting. The aim of simultaneous mordanting was to enhance the adsorption of the dyes and ensure a strong bond between the dyes and the fabrics. The commonly used mordants, such as ferrous sulphate,

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copper sulphate, and alum, were selected. Initially, the cotton and wool fabrics were immersed in warm water (approximately 46°C) 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).

Dyeing Procedure

The dyeing procedure was carried out in accordance with the dyeing method adopted by Shariful Islam *et al.* (2020). The pretreated cotton and wool fabrics (1.0 g) each were dyed in separate dye baths with the dye extracts. The dyeing was carried out using mordants such as ferrous sulphate, copper sulphate, and alum. In each case, a few drops of acetic acid (2%) were added to the dye bath to considerably reduce the amount of metal mordants in the spent bath. Dyeing was conducted at a material-to-liquor ratio of 1:50 using a shade of 2% and 6% on the weight of the fabric. The dyeing was carried out at 40°C for 1 hour and continued for a further 1 hour, and 1 ml was taken at 0, 10, 20, 30, 40, 50, 60, and 120 minutes in a standard laboratory dye master, and it was used to generate absorbance values. After half of the dyeing time, Sodium sulphate (Na_2SO_4) of 3.0 g/l was added as exhausting agent and at the end of the dyeing time, the samples were removed, washed, and dried.

Percentage of Exhaustion of Dye extract

The absorbance of the dyes was determined before and after dyeing at the maximum wavelength (665 λmax) using a JENWAY 6405 UV/visible Spectrophotometer. The percentage of exhaustion was calculated using the expression below:

$$\text{Percentage of } Exhaustion = \frac{\text{absorbance before dyeing} - \text{absorbance after dyeing}}{\text{absorbance before dyeing}} \times 100\%$$

Colour Measurement

Colour Fastness to Wash

The wash fastness test was carried out using a heating mantle and the International Organization for standardization (ISO) wash fastness test No. 3. A 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 of distilled water. The dyed sample fabrics were sandwiched between undyed cotton and wool fabrics and subjected to the wash fastness test. The composite specimens were separately agitated in a 100 ml beaker containing the soap solution (15 ml), sodium carbonate (10 ml), and distilled water to give a liquor ratio of 50 ml. The cotton and wool fabric pieces were immersed in the washing solution and heated for 30 minutes at 40°C to 60°C. The composite specimens were then removed and rinsed, and the components were separated and dried. The assessment of colour change and staining of the dyed specimens was assessed with the appropriate grey scale rating between 1 and 5.

Colour Fastness to Light

The lightfastness was assessed by exposing the fabrics to the Xenon Arc Lamp of a Fedometer, 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 labelled E and the unexposed side labelled UE. The cards were placed in a Fedo Meter; the light was turned on and left for about 72 hours so that an appreciable colour change existed with respect to the unexposed side. The conditions for the test were: black panel temperature 63°C, dry bulb temperature 43°C, and relative humidity 30%. After testing, the samples were rated against the standard blue wool scale.

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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) and disodium hydrogen orthophosphate dehydrate (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 the solution was adjusted to pH 8 using 0.1 N sodium hydroxide (NaOH). The liquor ratio used for the test was 20:1. A composite sample was made by sandwiching the dyed sample measuring 2×3 cm between two pieces of undyed 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 was placed between two glass 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°C for 4 hours. After 4 hours, the specimens were removed from the perspirometer and dried at room temperature. The change in colour of the specimen was assessed using a grey scale rating between 1 and 5.

RESULTS AND DISCUSSION

Results

Extraction Yield

The extraction yield was calculated; this was done by measuring the solvent efficiency to specific components from the original material (Murugan and Parimelahagan, 2014). This will be defined as the amount of extracts recovered in mass compared to the initial amount of the whole plant. This is presented in percentage (%).

$$\text{Extraction Yield (\%)} = \frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}} \times 100$$

Table 1: Extraction yield of *M. maximus* dye using ethanol and mass (20 g) of raw material

Temperature (°C)	Mass of Extracted Material (g)	Extraction Yield (%)
50	16.014	19.93
60	15.384	23.08
70	14.656	26.72
80	14.92	30.4
90	17.22	13.9
100	17.842	10.79

Table 2: Extraction yield of *M. maximus* dye using aqueous solution of optimal solvent and mass (20 g) of raw material

Temperature (°C)	**Mass of Extracted Material (g)**	**Extraction Yield (%)**
50	16.602	16.99
60	15.872	20.64
70	15.280	23.60
80	14.505	27.475
90	17.790	11.05
100	18.296	8.52

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Extraction of dye from *M. maximus* at different Temperatures

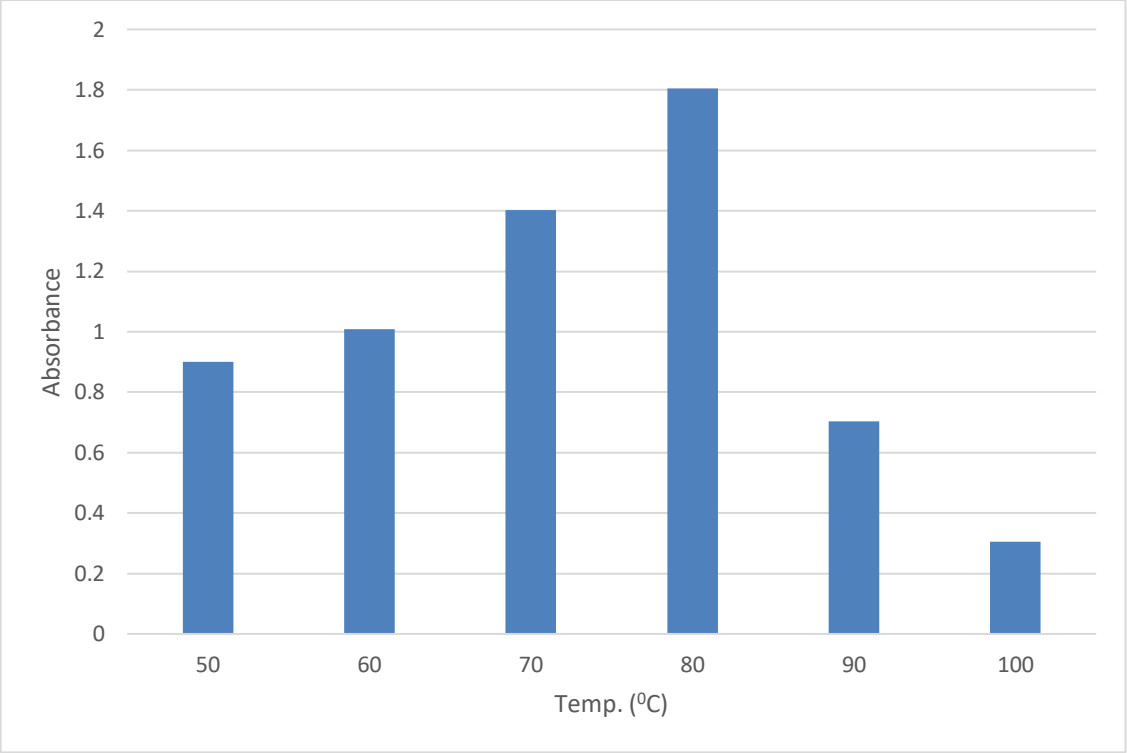

Fig 2: Effect of different temperatures on absorbance of dye extract from *M. maximus using* ethanol as a solvent

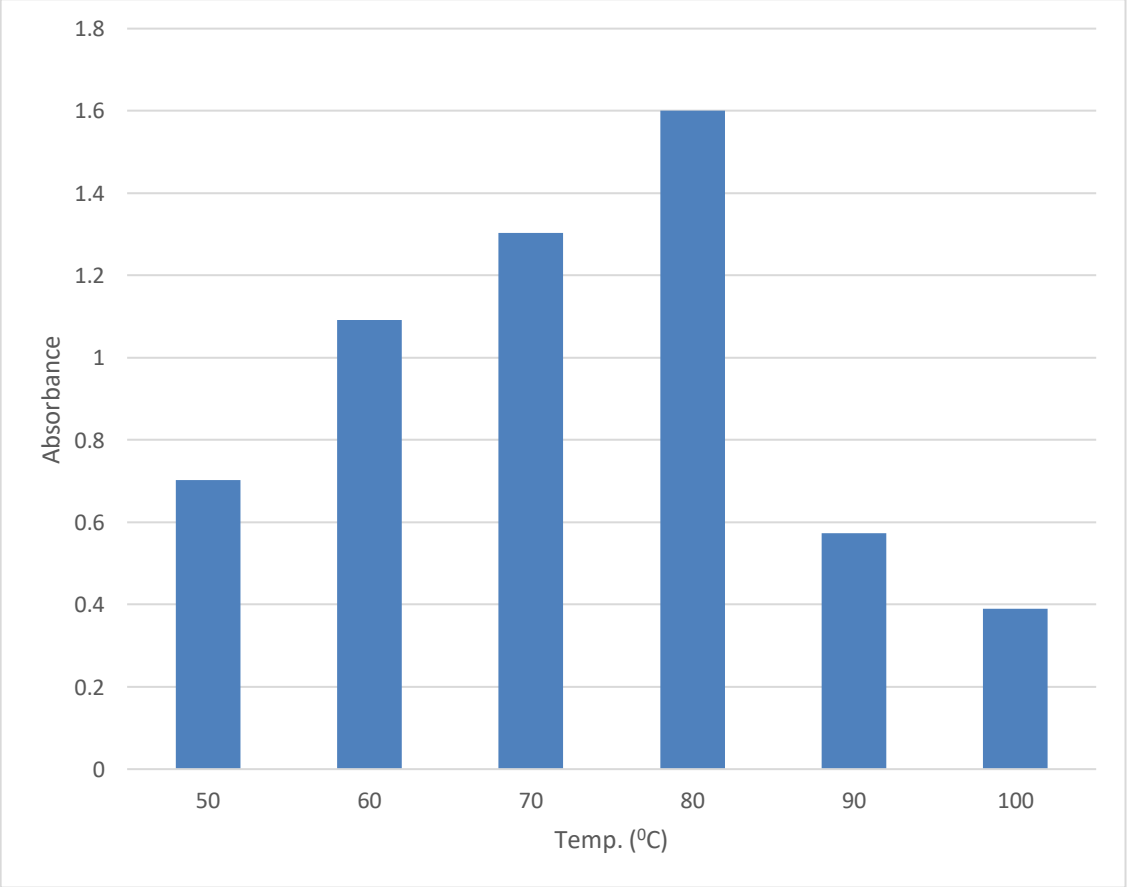

Fig 3. Effect of different temperatures on absorbance of dye extract from *M. maximus using* aqueous solution of optimal solvent

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Percentage of Exhaustion of Dye extract

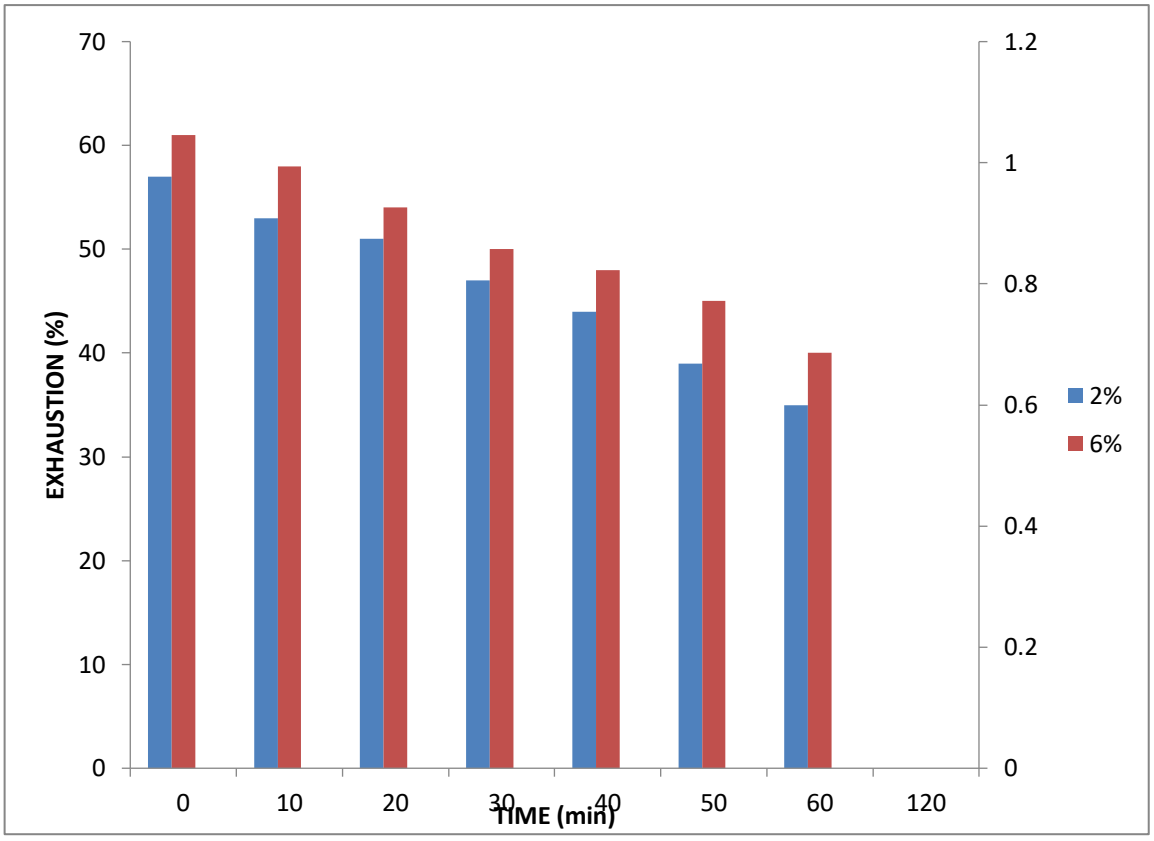

Fig. 4: Percentage exhaustion of dye extract from *M. maximus* at different minutes on cotton fabric and at λmax 665

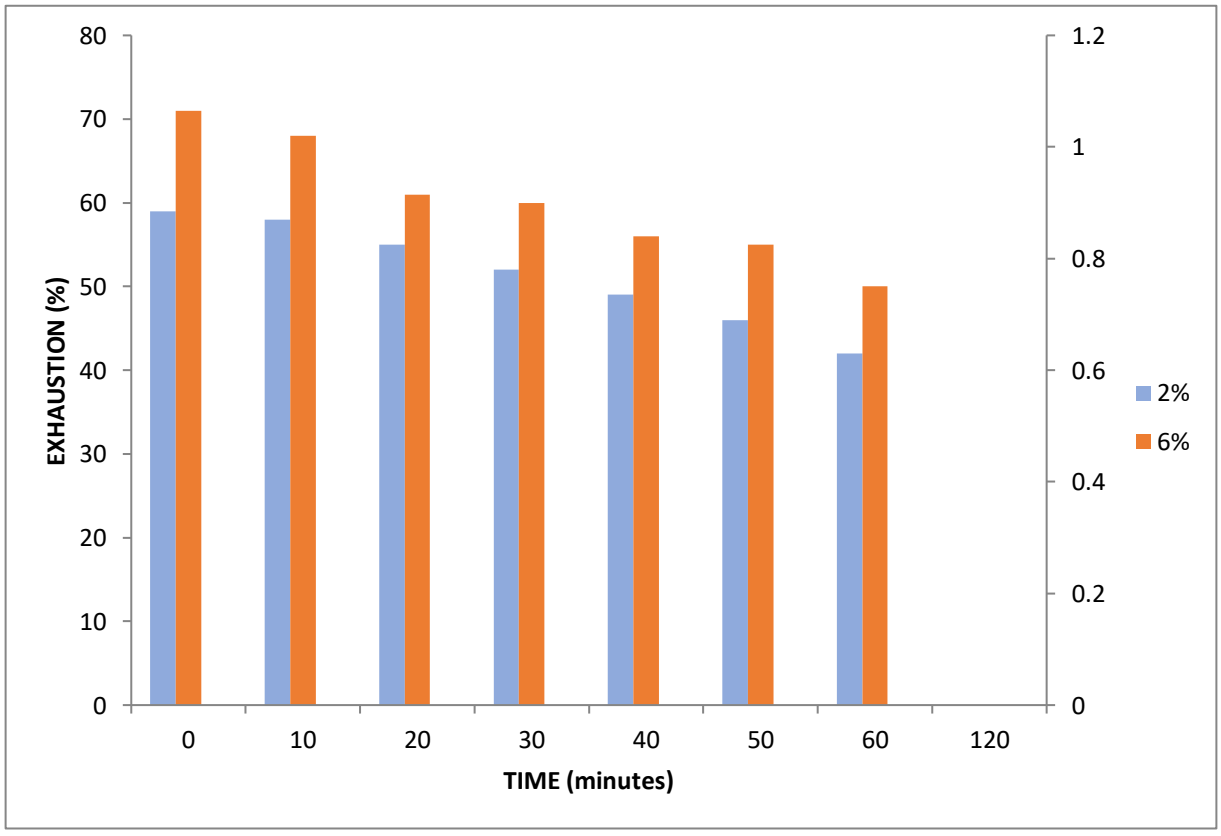

Fig. 5: Percentage exhaustion of dye extract from *M. maximus* at different minutes on wool fabric and at λmax 665.

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

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

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

Fastness Properties

Table 3: Colour Fastness to Wash

Fabrics	Sample Code	Colour Change	Staining
Cotton	SO 2%	3	3
Cotton	SO 6%	3/4	3
Wool	SO 2%	2	2
Wool	SO 6%	2/4	4

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

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Table 4: Colour Fastness to Perspiration Test

Fabric	Sample Code	Acid Resistance	Alkaline Resistance	Staining
Cotton	SO 2%	2/3	3/4	3
Cotton	SO 6%	3	4	3
Wool	SO 2%	3	4	3
Wool	SO 6%	4	4	4

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%	4
Wool	SO 2%	5
Wool	SO 6%	6

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

DISCUSSION

Determination of optimum Solvent Extraction

The extraction of dyes from *M. maximus* leaves using two different extraction solvents, namely, ethanol and an aqueous solution of optimal solvent (ethanol/water, 140:60 v/v) at different temperatures, is presented in Figure 2-3. The results from the graphs showed the relationship between absorbance and temperature changes. The study revealed that of the two solvents used, ethanol at a temperature of 80°C, which was in agreement with Abd Razak *et al.* (2011), was observed to have a maximum absorbance value (1.804) with a 30.4 extraction yield, while an aqueous solution of the optimal solvent (ethanol/water, 140:60 v/v) at the same temperature had an absorbance value (1.607) with a 27.475 extraction yield within the wavelengths of 200-800 nm. Ethanol as an organic solvent has been extensively used to extract natural organic dyes from various plant species (Peschel *et al.,* 2006). The results show that the highest dye yield was obtained at a temperature of 80°C, which suggested that some natural organic dyes are stable at temperatures less than 80°C. The increase in dye concentration is as a result of an increase in temperature, which may be caused by an increase in pigment molecule diffusivity and pigment solubility; these properties are related to the increase in the internal energy of molecules, which increases the extract concentration (Cacace and Mazza, 2003). Temperatures more than 80°C probably caused a decrease in the extract concentration due to chemical structure degradation of pigments. Thus, a temperature of 80°C proves to be the optimum temperature for extraction with high yield.

Determination of Percentage Exhaustion

The dye extract absorbance values were determined before and after dyeing at the maximum wavelength (665 lambda max) using the JENWAY 6405 UV/visible Spectrophotometer and are presented in Figure 4-5. The percentage of exhaustion was carried out at 0, 10, 20, 30, 40, 50, 60, and 120 minutes in the dye bath at temperatures of 45, 48, 53, 58, 61, 66, 70, and 98°C. The liquor solution (1 ml) was taken from the dye bath at 10-minute ’intervals at different temperatures to generate absorbance. The amount of dyes that was exhausted and fixed on the fabrics was evaluated; it was observed that an increment in time from 0 to 120 minutes gradually increased the transfer of dye molecules from the dye bath onto the fabrics, which was in agreement with Broadbent (2001). Exhaustion of dye is a function of time, so as time increased, the rate of

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uptake of dye on the fabrics increased while the rate of dyeing decreased. A decrease in exhaustion at 120 minutes implies that the dye extract in the spent bath has been used up by the fabrics as the temperature increases.

Evaluation of Colour Fastness

This research reports on the fastness properties, including light fastness, perspiration fastness, and wash fastness of fabrics dyed with *M. maximus* extract.

Colour Fastness to Washing

Wash fastness of dye was influenced by the rate of diffusion of dye and the state of dye inside the fabric (Kanchana *et al.,* 2013). The crude extract from the plant was applied on both fabrics simultaneously with mordants. The results presented in Table 3 and Figure 6 revealed a rating of (4) and (3/4) on the dyed cotton fabric at OS 2, 6% concentration, which was in agreement with Shariful *et al.* (2020). Also, at OS 2, 6% concentration, a rating of (2) and (2/4) was observed on the dyed wool fabric, which indicated fair and very good performance with slight staining on the adjacent fabric. Thus, the results show that the crude extract exhibits a better wash fastness performance on both dyed fabrics.

Colour Fastness to Perspiration test

Perspiration fastness on dyed fabrics was evaluated under acidic and alkali conditions, as shown in Table 4 and Figure 7. The fabrics (mordanted with ferrous sulphate, copper sulphate, and alum). At 2% concentration, the dyed cotton fabric revealed a rating (acidic 2/3, alkaline 3/4). Also, at 2 and 6% concentration, a rating (acid 3, alkaline 4) was observed of both dyed fabrics with moderate and slight staining on the adjacent fabrics. Lastly, the perspiration fastness rating of the dyed wool fabric at 6% concentration reveals (acidic 4, alkaline 4), which was in agreement with Clark *et al.* (2023). These results indicate that the acidic and alkali dye extract from *M. maximus* can produce fabrics that are resistant to perspiration in different environments.

Colour Fastness to Light

A colourfastness to light test was conducted on the dyed fabrics to evaluate their resistance to light exposure using the Xenon Arc Lamp of a Fedo meter. It was seen from Table 5 and Figure 8 that light fastness with a rating of (4), which was in agreement with Shariful Islam *et al.* (2020), was observed on the dyed cotton fabric at 2.6% concentration. However, dyed wool fabric at 2% SO concentration received a rating of (5), which indicates good fastness performance with moderate fading on adjacent fabric, while at 6% SO, it received a rating of (6) on the blue wool scale after 72 hours. 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 the plant extract. The presence of alkaloids and saponins in the dye extract, as mentioned by Adekunle *et al.* (2005), may be responsible for this successful dyeing process. Additionally, Burkinshaw and Kumar (2009) suggested that the features of mordants like ferrous sulphate, copper sulphate, and alum play a more significant role in determining the fastness properties of natural dyes than the dyes themselves. The obtained dyes and their properties are the result of the formation of wool mordant dye interactions.

CONCLUSION

Crude extract was obtained from *M. maximus* leaves using ethanol and an aqueous solution of optimal solvent. This study shows that at a temperature of 80°C, both solvents used had the maximum absorbance value: 1.804 (ethanol) and 1.607 (aqueous solution of optimal solvent). The percentage of exhaustion was evaluated in the dye bath; the rate and degree of dye fixation on the fabrics increased significantly in the dye bath from 0 to 120 minutes. The result of the dye extract from *M. maximus* on wool fabric exhibits a better lightfastness performance than that of cotton fabric. As a result of the pigment being present in the plant

coupled with the metallic salt mordant, there was an improvement in the fastness properties of the fabrics (Ndiku and Ndule, 2015), etc., which are responsible for dyeing the fabrics. Additionally, cotton fabric composed of cellulose and wool (keratin), a protein that bonds with dye molecules, is also responsible for the binding of the fabrics with the dyestuff. Thus, the plant extract provides promising results in terms of colourfastness on fabrics with the application of mordants.

Conflict of Interest

The authors declare that they have no conflict of interest regarding the publication of this manuscript.

ACKNOWLEDGEMENTS

The author appreciates the Faculty of Polymer and Textile Technology, Ahmadu Bello University, Zaria, Nigeria, and also the Department of Chemistry, Abubakar Tafawa Balewa University, Bauchi, for providing all the necessary facilities used to accomplish this research work.

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ISSN No. 2454-6194 | DOI: 10.51584/IJRIAS |Volume X Issue VIII August 2025

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