



Extraction, Characterization, and Application of Natural Dye Derived from Hibiscus Sabdariffa on Cotton & Wool

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ABSTRACT

The widespread use of synthetic dyes in the textile industry has led to significant environmental and health concerns due to their toxicity and non-biodegradability. This paper explores the potential of Hibiscus sabdariffa as a sustainable alternative by focusing on its extraction, characterization, and application as a natural dye on cotton and wool fabrics. The dye was extracted using a Soxhlet extraction method with methanol at a 1:4 solid-to-solvent ratio for 48 hours, followed by concentration, filtration, rinsing, and drying for 8 hours. The yield of the dye was 0.5 percent from 900 g of dried petals. Characterization involved UV–Visible spectroscopy, which revealed an absorption maximum at 520 nm, indicative of anthocyanins, and FTIR analysis, confirming the presence of phenolics, flavonoids, and carbohydrate-based compounds. The dye was applied to cotton and wool fabrics using three mordants: aluminium sulfate, ferrous sulfate, and copper sulfate. The dyeing was carried out at a controlled temperature of 100°C. The exhaustion rates showed that wool absorbed more dye (35.90–39.11 percent) compared to cotton (22.00–26.40 percent), with aluminium sulfate providing the highest exhaustion for both fabrics. Fastness tests indicated that cotton fabrics dyed with aluminium sulfate had wash fastness ratings of 4–5, while wool fabrics had ratings of 4. The study highlights that Hibiscus sabdariffa offers a viable, eco-friendly dye with moderate to good fastness, paving the way for more sustainable textile practices.

Keywords: Dyes, synthetic bio-degradable, fabrics, anthacyanins cotton.

INTRODUCTION

Dyes are substances, typically organic compounds, which are used to impart color to materials by dissolving in medium like water or other solvents and bonding with material's molecules. Dyes are classified in various ways, including by their origin (natural and synthetic), their chemical structure (such as azo or anthraquinone dyes), and their application method (including acid dyes, basic dyes, direct dyes reactive dyes and vat dye) in Textile dyes by Iqbal, (2008). The plants, minerals, and animals are the major source of natural dyes, almost non-substantive and can be used with the help of a mordant (metallic salt). There is an edge to natural dyes as compared to synthetic dyes, as they are soft, shiny and comforting for human eyes Samanta and Konar, (2011).

Synthetic dyes in the textile industry have posed significant threats to human health, and people are becoming increasingly aware of their environmental consequences. Natural dyes are biodegradable, non-toxic, and environmentally safer than synthetic dyes (Maria et al., 2010). As a result, researchers have identified and used many plant, mineral, and animal sources in fabric dyeing, highlighting the reuse of waste materials and the shift toward environmentally friendly alternatives (Colchester, 2007).

The low toxicity of natural dyes is increasing their demand compared to their synthetic counterparts. Synthetic dyes are hazardous, harmful, and carcinogenic. Natural dyes can serve as alternatives to limit the harmful effects linked to synthetic dye production. These dyes are non-toxic and environmentally safe, mainly used in health-focused applications like food coloring, infant textiles, and leather dyeing. The textile industry's heavy use of synthetic dyes has significantly reduced the use of natural dyes. But over the last decade, natural dye use



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has grown worldwide. This increase is largely due to rising demand across sectors, pushed by new environmental regulations in several countries.

Natural dye offers a broad range of shades with strong colour fastness (Ekrami et al., 2011). This has led to ongoing global research on using plant-based dyes in textiles and reviving natural dyes as substitutes for synthetic ones (Acguah et al., 2012).

The use of plant-based dyes can be traced back to ancient times, where natural products such as roots, berries, and leaves were used to dye fabrics and textiles (Alegbe & Uthman, 2024). The history of plant-based dyes in Nigeria is deeply intertwined with the country's rich cultural heritage and its diverse flora (Okoro, 2011), and trade relationships with neighboring regions and distant lands.

For centuries, indigenous communities in Nigeria harnessed the vibrant colors found in various plant species to create dyes for textiles, crafts, and ceremonial purposes Ejiogu et al., 2017). This practice not only served practical needs but also played a significant role in expressing cultural identity and artistic traditions. Nigeria's geographical and ecological diversities have contributed to a wide array of plant species suitable for dye extraction (Lawal & Muhammad 2012).

The history of plant-based dyes in Nigeria has experienced shifts due to various factors. The introduction of synthetic dyes during the colonial period led to a decline in the use of traditional plant-based dyes because the former offered greater color variety, consistency, and efficiency in application. As a result, the traditional knowledge and techniques associated with plant-based dyeing began to fade over time. However, in recent years, there has been a resurgence of interest in traditional dyeing methods and sustainable practices (Ejiogu et al., 2017). Artisans, designers, and cultural preservationists are working to revive the use of plant-based dyes in Nigeria, not only for their aesthetic value but also for their eco-friendliness and culturally significant attributes. This revival also supports local economies by promoting cultivation and sustainable harvesting of dye plants.

Although African plant species have long been utilized for extracting bio active compounds used in traditional medicine, pest control, and food preservation, their potential in natural dye production remains under-explored (Mayunga, 2007). Africa boasts an abundance of indigenous flora with high pigment-producing capabilities, yet most of these species have not been fully investigated or harnessed for textile coloration (Alegbe & Uthman, 2024). Majority of natural dyes need a chemical in the form of metal salts to create an affinity to the fibers and pigment. These chemicals are called mordants. Common mordants used are alum, chrome, copper sulphate, ferrous sulphate etc. (Siva, 2007), Mahangade et al., 2009, Samanta and Agarwal, 2009). Hisbiscussabadariuffalinn (Zobo) is a shrub belonging to the family malvaceae. The leaf is reported to contain protein, fat, carbohydrate, fibre, calcium, phosphorus, iron, thiamine, riboflavin, miancin and ascorbic acid (Watt and Breyer, 1962).

This study focuses on extracting natural dye from Hibiscus sabdariffa and applying on cotton and wool fabrics. Additionally, the spectral properties of the dyed materials were analyzed and reported.

Methods and materials

Materials: The materials used include; Hibiscus sabdariffa petals to be used as the primary source of natural dye will be collected from a local market in Karu, Nassarawa state. Cotton and wool fabric to be dyed will be sourced from a market in Karu, Nassarawa.

Sample preparations: Dried hibiscus flowers will be collected and surface impurities will be removed. The petals will be ground into a fine powder using a mechanical grinder and then sieved to remove coarse particles, thereby increasing the surface area for extraction.

Extraction of dye: The extraction method used in this study was adapted from Yakasai et al. (2022) with slight modification. Roselle petals were collected, air dried for five days, stored in polyethylene bags, and ground into fine powder. Fifty grams of the powder was mixed with 200 ml of methanol at a 1:4 solid to





solvent ratio instead of the 1:6 ratio used by Yakasai et al., (2022) Extraction was carried out in a Soxhlet apparatus for 4–8 hours. The extract was concentrated using a rotary evaporator to remove excess solvent and further dried to powder form. The percentage yield was calculated.

Characterization: The dye extract will be characterized using Fourier Transform Infrared (FTIR) Spectroscopy, and UV-Visible Spectrophotometry.

Fourier Transform Infrared (FTIR) Analysis: FTIR analysis identifies functional groups that makes up the

chemical components of the dye extract which will be carried out using FTIR Agilent Technologies (Cary 360) carried out at the NARICT Research Institute in Zaria. All absorption bands will be expressed in cm⁻¹.

UV-Visible Spectrophotometry: The electronic absorption properties of the dye extract will be analyzed using a UV-Vis Spectrophotometer (PEC Medical USA UV 752) at Bingham University, Karu. The absorbance spectra will be recorded in the range of 200–800 nm using a quartz cuvette.

Dyeing: The Dyeing was carried out using a simultaneous mordanting method adapted from Ishegbe et al. (2014). A stock solution was prepared from the dye extract. The dye bath was prepared using a liquor ratio of 1:50 at 3 % shade on the weight of fabric (o.w.f). The volume required from each stock solution was calculated based on the formula,

$$V = \frac{P \times W}{C}$$
 ----- Equation 1

Where: P = percentage shade, W = weight of fabric, C = percentage concentration of stock solution

Scoured cotton and wool fabrics were cut into several pieces each weighing 0.4 g and 0.5 g respectively. Three different mordants (1g each) which includes Aluminuim sulfate, Ferrous sulfate and Copper sulfate were used in the dyeing process as shown in figure 2.

Determination of % Exhaustion: The % exhaustion of the dye were determined before and after dyeing at the maximum wavelength (y_{max}) using a UV-Vis Spectrophotometer (PEC Medical USA UV 752) at Bingham University Karu. The percentage exhausion was calculated using

% E =
$$\frac{A1 - A2}{A1}$$
 x 100----- Equation 2

Where; % E = % Exhaustion, A1 = Absorbance before dyeing, A2 = absorbance after dyeing

Colour Fastness Test: This test measures how well a dyed fabric resists fading from washing, rubbing, and hot pressing. It helps assess the durability and quality of the extracted dye on the fabric.

Wash Fastness: Wash fast testing will follow the ISO 105-C06 standard. Dyed samples will undergo washing under controlled conditions with a soap solution. Color change and staining on adjacent fabrics will be evaluated using a gray scale.

Dye Extraction: The dye was extracted from the dried calyces of Hibiscus sabdariffa using the Soxhlet extraction method as described by Yakasai et al. (2022). A total of 900 grams of the plant material was crushed, sieved, weighed and placed in a thimble inserted into the Soxhlet extractor. Methanol was used as the extraction solvent at a sample to solvent ratio of 1 to 4, giving a total of 3600 ml for 4-8hours. After extraction, the extract was concentrated using a rotary evaporator to remove most of the solvent. The concentrated extract was then washed, filtered and air dried to obtain a powder. A total of 4.5 grams of crude dye powder was recovered from the 900 grams of hibiscus sample. The percentage yield was calculated as:

% Yield =
$$\frac{4.5}{900}$$
 x 100 = 0.5%----- Equation 3

Table 1: Summary of Dye Extraction Process.



D .	T7.1
Parameter	Value
Weight of dried hibiscus	900 g
Volume of methanol used	3600 ml
Extraction method	Soxhlet
Solvent removal method	Rotatory evaporator
Drying method	Air dried
Weight of dye extract	4.5 g
Percentage yield	0.5%

The percentage yield of the Hibiscus sabdariffa dye extract was 0.5 % (from 900 g of dried calyces), which is much lower than the 81.1 % reported by Yakasai et al. (2022) using ethanol as the extraction solvent at a 1:6 ratio. The difference in yield may be due to variations in solvent type, solvent ratio, extraction time, and drying method. Egbujor et al., (2019) obtained 3.5, 3.0, 2.6 and 1.4g in ethanol, methanol acetone and N-hexane extract respectively from 50g of Hibiscus Sabdariffalinn (Zobo).

Figure 1: Dye extract



Table 2: Physical Properties of Dye Extract

Dye code	Empirical	Molecular	Melting	Yield	Color of
	formula	mass (g/mol)	point	(%)	crystal
HS dye extract	$C_{26}H_{29}O_{15}$	581.50 g/mol	130-135°C	0.5	Black

Application of Dye on Cotton and wool: The dye extracted from Hibiscus sabdariffa was applied to cotton and wool fabrics using a simultaneous mordanting method adapted from Ishegbe et al. (2014) with slight modifications. Aluminium sulfate, copper sulfate, and ferrous sulfate were used in the dye baths. A 3% shade and a liquor ratio of 1:50 were maintained for all dyeing procedures.

Mordanting and dyeing procedure: Dyeing was carried out using a simultaneous mordanting method. A stock solution of the dye was prepared at a mass concentration of 0.025 g/cm³. The amount of dye stock used for each dye bath was calculated using the formula:

$$V = \frac{P \times W}{C} \quad ---- \quad Equation \quad 4$$

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Where: V = Volume of dye stock to use (cm³), P = Percentage shade (3%), W = Weight of fabric (g), C = Concentration of dye stock (0.025 g/cm³)

For **cotton**, 0.4 g of fabric was dyed using:

$$V = \frac{3x0.4g}{0.029g/cm^3} = 48cm^3$$
 ----- Equation 5

For **wool**, 0.5 g of fabric was dyed using:

$$V = \frac{3x0.5g}{0.029g/cm^3} = 60cm^3$$
 ----- Equation 6

In both cases, the calculated dye stock volume was diluted with 25 ml of distilled water. Exactly 1g of the mordants (aluminium sulfate, copper sulfate, and ferrous sulfate) was added directly to the dye bath before heating. The dye bath was gradually heated. The fabric samples were introduced into the dye solution at 40°C. The temperature was then raised to 100°C and maintained for 60 minutes. After 1 hour, the fabrics were removed, rinsed thoroughly in distilled water and air dried.

Figure 2: Applied dye on cotton and wool before and after





Applied dye on cotton and wool before and after

The dyed samples were photographed after washing and drying to document the colours obtained for each fabric and mordant type. For easy reference, each sample was given a short code. WC represents wool dyed with copper sulfate mordant, WA is wool dyed with aluminuim sulfate, and WF is wool dyed with ferrous sulfate. CC represents cotton dyed with copper sulfate mordant, CA is cotton dyed with aluminuim sulfate, and CF is cotton dyed with ferrous sulfate. These codes appear under the samples in the photographs and are used in the discussion of their colour, exhaustion values, and fastness test results.



Percentage exhaustion of dye

The effectiveness of the dye uptake was measured by UV-Visible spectrophotometry at a wavelength of 520 nm. The absorbance of the dye bath before and after dyeing was recorded. These formula was used to calculate the percentage exhaustion of dye.

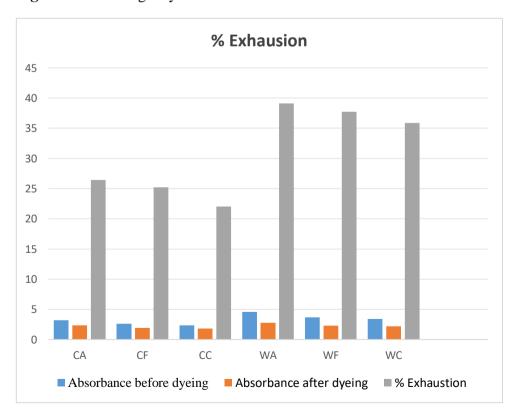
% E =
$$\frac{A_1 - A_2}{A_1} \times 100$$
----- Equation 7

Where: % E = % Exhaustion, A1 = Absorbance before dyeing, A2 = absorbance after dyeing

Table 3: Percentage Dye Exhaustion of Cotton and Wool at 520 nm

		T		
Fabric and mordant	A1 before dyeing	A2 after dyeing	% Exhaustion	
CA	3.181	2.341	26.40	
CF	2.593	1.939	25.22	
CC	2.354	1.836	22.00	
WA	4.589	2.794	39.11	
WF	3.701	2.304	37.75	
WC	3.406	2.184	35.90	

Figure 3: Percentage Dye Exhaustion of Cotton and Wool at 520 nm



In this study, percentage exhaustion values ranged from 22.00 to 26.40 % for cotton and from 35.90 to 39.11 % for wool, with aluminium sulfate producing the highest exhaustion for both fabrics. Ramprasath et al. (2017) reported lower dye uptake values for cotton dyed with percentage exhaustion below 25 % for all mordants

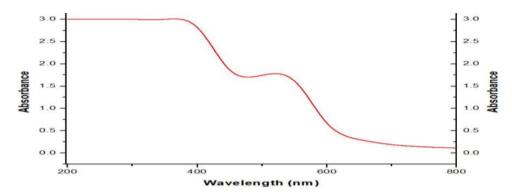


tested, and similarly higher uptake on protein fibres compared to cotton. This indicates that, consistent with their findings, wool in this study exhibited greater affinity for the dye than cotton, likely due to its protein structure and higher number of active sites for bonding with anthocyanin molecules.

Characterization of the Dye Extract

Visible Spectroscopy: The UV-Visible spectrum of the dye extract was obtained in the range of 200 to 800 nm. This was carried out using UV-Vis Spectrophotometer (PEC Medical USA UV 752) at Bingham University, Karu.

Figure 4: UV-Visible spectra of extracted dye

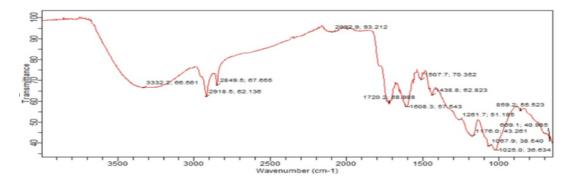


The UV-Vis spectrum of the extracted dye showed high absorbance in the UV region from 200 to 350 nm, indicating the presence of phenolic and flavonoid compounds. A peak was observed around 500 nm, suggesting the presence of anthocyanin pigments. Absorbance gradually decreased towards 800 nm, indicating minimal light absorption the visible in far region. This profile shows similarities to that reported by Ramprasath et al. (2017) for Hibiscus rosa-sinensis without mordant. In both spectra, there is a single broad visible peak in the 500-550 nm range, corresponding to anthocyanin absorption, and a pronounced UV absorption band below 350 nm linked to phenolics and flavonoids. Both show a gradual decline in absorbance towards the longer wavelengths, indicating similar pigment light absorption behaviour across the visible range. These similarities suggest comparable chromophore structures and related pigment composition between the two species. The UV-Vis results confirm that the Hibiscus sabdariffa dye extract contains bioactive compounds including phenolics, flavonoids, and anthocyanins, in agreement with previous reports. Egbujor et al., (2019) in their study also found the absorption peaks for Hibiscus sabdariffa linn (zobo), using a UV-spectrophotometer and the maximum absorptivity using ethanol, methanol and acetone solvents were found to be 2.350, 3.524 and 1.560 respectively. The highest peaks were deduced from the graph at 500xmax for Hisbiscus sabdariffa linn (zobo).

Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectrum of the dye powder was obtained in the range of 4000 to 400 cm⁻¹. This was carried out using FTIR Agilent Technologies (Cary 360) carried out at the NARICT Research Institute in Zaria.

Figure 5: FTIR analysis result showing the major peaks







and C-O-

The FTIR spectra of extract dye showed a broad OH stretching band at 3332 cm⁻¹ indicating hydroxyl groups from phenolics and alcohols. Aliphatic CH stretching bands appeared at 2918 and 2849 cm⁻¹, confirming the presence of aliphatic chains. A strong C=O stretching band at 1720 cm⁻¹ suggested carbonyl groups, likely fromflavonoids or related compounds. Aromatic C=C vibrations were recorded at 1608 cm⁻¹, indicating aromatic structures from phenolics and flavonoids. Peaks between 1260 and 1020 cm⁻¹ corresponded to C-O

C stretches, typical of carbohydrate-related compounds.

These peaks are consistent with those reported by Ramprasath et al. (2017) for hibiscus flower extracts, confirming the presence of phenolics, flavonoids, anthocyanins, and carbohydrate-related compounds in the extract.

The FTIR results confirm that the extracted dyecontains bioactive compounds including phenolics, flavonoids, anthocyanins, and carbohydrates, in agreement with previous reports.

Color fastness to Washing: Wash fastness was assessed using ISO 105-C06:2010 method. Each dyed sample was subjected to a standard washing test involving 5 grams per litre of soap solution at 60°C for 30 minutes with constant agitation. After washing, samples were rinsed thoroughly, air dried, and evaluated for any change in colour and staining on adjacent undyed fabric. Assessment was done using the gray scale for colour change and staining, with ratings from 1 (poor) to 5 (excellent).

Table 4: Color Fastness Result

Fabric	Mordant	Wash Fastness Rating	Interpretation
Cotton	Ferrous sulfate	4	Good fastness
Cotton	Copper sulfate	3	Fair fastness
Cotton	Aluminium sulfate	4/5	Between good & excellent fastness
Wool	Ferrous sulfate	4	Good fastness
Wool	Copper sulfate	3	Fair fastness
Wool	Aluminium sulfate	4/5	Between good & excellent fastness

In this study, cotton fabrics recorded wash fastness ratings of 4 for ferrous sulfate, 3 for copper sulfate, and 4– 5 for aluminium sulfate. Wool fabrics recorded the same ratings for the mordants used. Ramprasath et al. (2017) similarly observed higher wash fastness values on wool compared to cotton when dyed with Hibiscus sabdariffa, attributing the difference to stronger binding of anthocyanin molecules with the amino and carboxyl groups present in protein fibres. Ali and Khan (2024) reported that wool fabrics dyed with Hibiscus sabdariffa extract achieved wash fastness ratings ranging from 3 to 4, with copper mordant providing deeper shades but lower fastness compared to aluminium mordant, which is consistent with the present findings. Likewise, Haji et al. (2020) examined anthocyanin dyes on wool and found that pre-mordanting with aluminium salts significantly improved wash fastness to 4/5, while iron and copper mordants gave only moderate ratings. These comparisons confirm that aluminium mordant generally enhances dye-fibre bonding and stability, while copper tends to yield weaker wash fastness despite producing intense colours. Egbujor et al., (2019) reported that different shades of purple were obtained from the dye extracted from H. sabdariffalinn (zobo) at different concentration when dyeing the cotton fabric. The cotton fabric was mordanted using potassium dichromate which was immediately used for dyeing but was unable to penetrate the dyestuff from Hisbiscussabdariffalinn due to the inability of the mordant to form coordination complex with the dye extracted from H. sabdariffalinn (zobo). The unmordanted cotton for H. sabdariffa was not resistant (fastness) to the cotton fabric. The dyed





cotton fabric was able to penetrate the mordanted using aluminium potassium sulphate but the colour changed to violet after washing with toilet soap.

SUMMARY

This study focused on extracting, characterizing, and applying natural dye from Hibiscus sabdariffa on cotton and wool fabrics. The Soxhlet extraction method was used with methanol as the solvent at a 1:4 ratio of sample to solvent. From 900 g of dried hibiscus calyces, 4.5 g of powdered dye was obtained, giving a yield of 0.5 percent. The low yield compared to some earlier studies was likely due to differences in solvent type, ratio, and the drying method used.

UV-Visible spectroscopy showed an absorption maximum at 520 nm, which is typical for anthocyanin-rich extracts. FTIR analysis revealed peaks linked to hydroxyl, carbonyl, and aromatic functional groups, confirming the presence of phenolics, flavonoids, anthocyanins, and carbohydrate-based compounds.

The dye was applied to cotton and wool fabrics using three mordants: aluminium sulfate, ferrous sulfate, and copper sulfate. Wool generally absorbed more dye than cotton, with percentage exhaustion ranging from 35.90 to 39.11 percent for wool and 22.00 to 26.40 percent for cotton. Aluminium sulfate gave the highest exhaustion for both fabrics.

Wash fastness tests showed that both fabrics dyed with aluminium sulfate had the best resistance to colour loss, while copper sulfate gave the lowest ratings. Cotton fabrics recorded wash fastness ratings between 3 and 4–5, while wool fabrics ranged from 3 to 4. This aligns with earlier research showing stronger dye-fibre bonding in protein fibres like wool.

CONCLUSION

Natural dyes are notable for their bright colours to fabrics. They can be used in textiles, pharmaceutical, food, cosmetics, plastics, photographic and paper industries. Hibiscus sabdariffa is a viable source of natural dye for cotton and wool fabrics. The process of extraction and dyeing is environmentally friendly. Use of mordants can also be considered for improving the fastness of dyed clothes. Although the yield was relatively low in this study, the extract produced strong colours with acceptable fastness properties, especially when used with aluminium sulfate mordant. Wool showed better dye uptake and wash fastness than cotton, confirming its higher affinity for anthocyanin-based dyes. This research supports the potential of Hibiscus sabdariffa as a sustainable alternative to synthetic dyes, with added benefits from its bioactive compounds. Further research will help to explore the undiscovered important uses of dye extracted from Hisbiscus sabdariffalinn (zobo), and other plant.

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