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Dyeing Properties of Natural Dyes Extracted from Shea Butter Tree Bark (*Vitellaria paradoxa*)

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ABSTRACT

The research was designed due to the recent renewed interest in the use of natural dyes for the dyeing of textiles as a result of the problem(s) associated with dyeing using synthetic dye. Synthetic dyes are toxic, non-biodegradable and not friendly to the environment i.e. non-eco-friendly. Natural dye therefore is a perfect replacement for synthetic dyes due to their non-toxicity, biodegradable, non-hazardous and more eco-friendly. Natural dye used for the study was extracted from *Vitellaria paradoxa* Shea butter tree bark using the Soxhlet extraction technique. The extracted dye was applied on cotton fabric and evaluated based on dyeing parameters such as dye concentration, electrolyte concentration, time and temperature. Mordanting was carried out with potassium dichromate using the post-mordanting method. The percentage exhaustion–quantity of dye absorbed by cotton fibres and the fastness properties of the dyed fabric were evaluated. From the result obtained, it was observed that the percentage exhaustion increases with increase dye concentration with the highest percentage exhaustion of 95% which was obtained at 8ml dye concentration. However, increase in electrolyte concentration resulted in increase in the percentage exhaustion with the highest percentage exhaustion of 98% which was obtained at 6ml electrolyte concentration. This observation was in agreement with the effect of electrolyte concentration on percentage exhaustion. It was also observed that the percentage exhaustion increases with increase in time of dyeing. The highest percentage exhaustion of 96% which was obtained at 3h dyeing time and for temperature it was observed that that the rate of dyeing increases gradually by raising the temperature. The highest dye exhaustion of 95% was obtained at the temperature of 50°C. It may be suggested that the extracted dye in the aqueous medium is in aggregated form since they need an increase temperature to migrate into the fibre. However, the application of mordant on the dyed samples improved the dye-fibre bonding and enhanced the fastness properties with colour change rating of 3 and 4 with shade of light brown. Comparing with the mordanted samples, the mordanted samples have a better fastness properties with colour change of 5 with shade of cork. However, on the whole, it can be recommended that dye extracted from *Vitellaria paradoxa* Shea butter tree bark can be used to dye cotton fabric.

Keywords: Fastness property; Natural dyes; Mordants; Soxhlet extraction technique; *Vitellaria paradoxa*.

Introduction

Oforghor (2010) reported that the recent renewed interest in the use of natural dyes is a result of global interest in green chemistry, as it aligns well with the natural ecosystem compared to synthetic dyes. The first man-made organic dye called Mauvene was discovered by Williams Henry Parkin (1856).

Several thousands of dyes have since been prepared in the laboratory and because of generally improved properties imparted upon the dyed materials, it therefore replaced the traditional natural dye (Agarwal, 2008). Lovett, (2004) and Maria *et al.*, (2010) stated that this is as a result of global concern about the danger of the use of synthetic dyes as they are dangerous, life-threatening and carcinogenic as well as their toxicity, hazardous, non-biodegradable, non-eco-friendly, corrosive nature to the environmental. This has led to the obvious replacement of the synthetic dyes with natural ones especially the ones extracted from natural sources such as plants- roots, stem, bark, flowers, fruits, seed, nuts, leaves and even in some animal (Colchester, 2007 and Mason, 2014).

However, a dye whether natural or man-made is an organic compound used not only to just colour the surface of fibers but it must fix with the fiber and becomes part of it so that after dyeing, the fiber will not be affected during the process of washing, dry cleaning, rubbing, perspiration, light with the organic solvent. The dye should be able to give fastness to heat, bleaching and light (Bhuvan, et al.; 2004 and Eforia, 2010). The global consumption of textile is estimated at around 30 million tonnes, which is expected to grow at the rate of 3% per annum. Therefore such a huge amount of required textile materials cannot be dyed with natural dye alone. Hence, the use of eco-friendly synthetic is also essential (Samanta, 2009, Khatee and Dehghan, 2011).

Oforghor, (2010) and reported that extraction of colour component from source natural dye material is important step for dyeing any textile substance to maximize the colour yield. Moreover, standardization of extraction process and optimizing the variables both, for a particular source natural dye material have technical and commercial importance on colour yield and cost of extraction process as well as dyeing cost (Vankar et al.; 2007 and Ekrami et al.; 2010). The Natural dyes of different origin can be extracted using aqueous method i.e. by using water for the extraction with or without addition of salt/acid/alkaline/alcohol in the extraction bath, supercritical fluid extraction, enzyme-assisted extraction, alcoholic/organic solvent extraction by using relevant extracting equipment or soxhlet extraction method with use of alcohol and benzene mixture and finally to filtrate, evaporate and to dry using ultra filtration equipment or centrifuge rotator vacuum pump/or by extraction under reduced pressure (Mahaleet al.; 2003, Maulik et al.; 2005). Nowadays, there has been an industrial method available for extraction of colour component/purified colour substances from natural dyes for their easy applications. The collected source material is generally shadow dried in the air or sun dried within a temperature range of 37-40^{0}C for the moisture content of the source natural dye material is reduced to 10-15% with proper drying since most of the materials have moisture content of 40-80% cannot be stored without drying. Mordant (from Latin: mordere means to bite, because the mordant eats away the surface of the fiber so that the dye can seep in) are chemicals in the form of metallic salts which are generally used to create an affinity between the fibre and the pigment. The main objective of the mordant when used with adjective dyes is to open up the pores so that the colorant can penetrate the fiber, thereby helping in fixation of the dyestuff on the substrate.

Abel, (2018), Arora and Gupta, (2017), Bhatia et al,; (2017), chakra, (2014) and Oforghor *et al.* (2022) stated that the term mordant or dye fixation is a substance used to set (i.e. bind) dyes on fabrics by forming a coordination complex with the dye, which then attached to the fabric (or tissue) and these dyes are known as substantive dyes (Chengiaiah et al.; 2010, Philips, 2010 and Cheguer.; et al.; 2013,) and nowadays, natural dyes existing mostly as chemical mordant such as Alum, Copper Sulphate, Ion or chrome. Mordents are prepared in solution often with the addition of an assistant which improves the fixing of mordant to the fiber (Gajalaksmi and Abbasi, 2000, Das et al.; 2009, Grover et al.; 2011, Thompson and Thompson, 2010, Chiriu et al.; 2017). The most commonly used mordant is Alum which is used usually with cream tartar as an additive or assistance ion (Hunger, 2003, Handa et al.; 2008, Hovers et al.; 2008, Hadibarata et al.; 2013). Limitation on colors yield and poor fastness properties prompted the search for idea mordant, the chemicals which increase natural dye uptake by textile fibers. The aim of the present research study is to extract and apply natural dyers from Shea Butter tree bark *Vitellaria paradoxa* and study the dyeing and printing properties on cotton fabrics (Povette, 2001, Gupta et al.; 2004, Obenewaa, 2010, Mclark, 2011, Patel, 2011, Dora, 2013and Narbona et al.; 2021).

Experimental

Materials

The bark of Shea tree *Vitellaria paradoxa* was collected from Nasarawa State University Faculty of Agriculture, Shabu, Lafia campus. The plant materials were identified by Dr. A.O. Oforghor of Nasarawa State University.

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Cotton fabrics were purchased from Lafia modern market. Methanol (CH_3OH), Acetic Acid, Distilled water, Ethanol (C_2H_5OH), Copper, Soxhlet extractor chamber, condenser, Flat bottom flask, Water inlet and outlet, measuring cylinder, water bath, beakers, heating test tube, thermometer, heating mantles, Sample tube, glass rod, washing bottle, plastic containers, spectrophotometer, Masking tape were all of analytical grade and obtained from College of Agricultural Laboratory.

Methods

Extraction of Natural Dyes

The Shea tree barks was cut and washed to remove adhering materials, the bark was dried and pounded into powder using mortar and pestles for effective extraction**.** The natural dye was extracted tomb the plant back using soxhlet extractor. 100g of the pulverized plant bark (she tree) was packed and placed in the fitter paper and then placed in the soxlet extractor chamber in the inner part of the soxhlet extractor. The apparatus was then filled to flat bottom flask and 600ml of a chosen solvent was introduced. The dual-purpose flask is then heated gently using heating mantle. The vapour in the DPF then leaves through the by-pass tube and moves straight to the condenser where it is being condensed back to ground state. The condensed vapour (liquid) moves back to the DPF through the siphoned tube when the solvent reaches the top. The process automatically repeated itself until complete extraction is anticipated. Methanol (800ml) was used, methanol was used more than ethanol because methanol vaporized more than ethanol. Ethanol (600ml) was used as an extraction solvent to extract natural dye from the back of Shea tree. 100g of the plant powder was used at 30^0C, 40^0C, 60^0C, 80^0C and 100^0C for one hour respectively, was used for the extraction. Another extraction was done using Ethanol and Methanol also as an extraction solvent in this case, the temperature of extraction was kept constant at 76^0C but the extraction time varies at 1,2,3,4,and 5 hours respectively. These extractions was repeated using methanol, Destin water as extraction solvent. All the extracts of the Vitellaria Paradoxa tree, extracted with the same solvent was bulked together and evaporated to dryness to obtain the extracts in paste form.

Paper Chromatography

Filter paper was cut into a strip that was about 2.2cm width and 2.2cm length 2cm was measured and drawn at the bottom edge of the paper which is the starting line, a dot was marked at the 2cm. Then the dyed extract was applied on the spot line 10ml of ethanol was measured inside beaker and the paper was inserted inside for 1hr for proper result.

Retention Factor (R_f)

Retention factor is the fraction of an analyse in the mobile phase of a chromatographic system. It is used to purify substances based on certain physical, chemical or biological characteristics. It is comprised of a stationary phase and a mobile phase into which substances partition.

It was calculated as: Mobile phase = 8.3 cm

Solute = 9.5 cm

$$Rf = \frac{\text{Distance move by solute}}{\text{Distance move by solvent}}$$

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$$Rf = \frac{8.3\ \text{cm}}{9.5\ \text{cm}}$$

$= 0.87$

$= 0.9$

Figure 1. Paper Chromatography Showing Distance Move by Solute

Importance of Retention Factor

It is important because it is used in comparing the results of one chromatogram to the results of another. If the conditions in which the chromatogram are run are unchanged (same mobile and stationary phases), the retention factor for a given material should remain constant. This allows unknowns to be compared to known materials. If the retention factor of an unknown does not match that of a known material, they are not the same compound. Similar retention/factors suggest that the two samples could be the same. It also helps to predict where a particular substance will be located on the chromatogram.

Measurement of Wavelength

The method of Oforghor (2011) was adopted for measurement then the absorption spectra of the dye was measured with the aid of UNICAN sp 800B spectrophotometer in the wavelength range of 400-800nm in a glass cell of path length of 1cm, the measurements of the spectra was made at a concentration of 0.561 in ethanol as reference. The wavelength of maximum (UV-V) was found at 454nm.

Dyeing Procedure

A 10% solution was prepared from the *Vitellaria paradoxa* plant extract**.** The cotton fabric sample was dyed with the extracted dye using the exhaustion method. The fabric was introduced, and dyeing commenced at 30^0C. The temperature was raised to boil for 10 minutes. Stirring of samples was done at frequent intervals using glass rod. The temperature of dyeing at boiling point (100^0C) was maintained for 1hr after which the sample was removed and dried. The method was used for Table 2, 3, 4 and 5 by varying the Electrolyte concentration, Time of dyeing, dye concentration and application of mordents respectively. However, the temperature of the dyeing was varied. The volume required from each stock solution was calculated based on the formula.

Vol. (cm^3) = $\frac{WP}{C}$

P = percentage shade; W = weight of fabric; C = concentration of stock solution.

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Measurement of Optical Density

An instrument called colorimeter was used to measure the optical density of the extracted dye before and after dyeing. After making up the dye bath to 25ml before and after dyeing 1ml was taken and again make up to 25ml, the optical density of the dye solution will then be measured and recorded and it was repeated for each dye bath. The maximum values indicate the extent of the absorption of the dye solution. When the optical density is low after dyeing it means that there is less dye on the fabric and vice vicar versa. From the optical density before and after dyeing the percentage exhaustion was calculated thus: (Oforghor, 2011).

$$\% \text{ Exhaustion} = \frac{ODB-ODA}{ODB} \times 100$$

Where ODB and ODA represent the optical density of the dye solution before and after dyeing respectively.

Determination of Wash Fastness of Dyed Samples

2g of joy soap was dissolved in 100ml of warm water and stirred until it become a homogenous solution. The dyed fabric sample was washed and squeezed gently for 5min. Rinse and dried then gray scale was used for assessing the fastness (Oforghor *et al.*, 2011).

Assessment of change in color

This assessment was based on the magnitude of visual contrast between the original (untreated) 100% white cotton fabric and the tested sample. It was assessed using the Grey scale for color change and staining, The Grey scale of accessing change in color consisted of five (8) pairs of pieces of cotton fabric (Oforghor *et al.* 2011).

Dyeing Procedure with Mordant

The one (1) mordant application methods was used using the same dyeing procedure Post mordanting: 4gm of copper sulphate was used for mordanting.

Screen Printing Procedures

STEP 1: The design is created.

To start, the printer takes the design I want to create on the finished product, and prints it out onto a transparent acetate film. This was used to create the stencil.

STEP 2: The screen is prepared.

Next, the printer chooses a screen to suit the complexity of the design, and the texture of the fabric being printed. The mesh screen is then coated with a layer of light-reactive emulsion, which will harden when developed under bright light.

STEP 3: The emulsion is exposed.

The acetate sheet featuring the design is then laid onto the emulsion-coated screen, and the whole thing is exposed to a very bright light. The light hardens the emulsion, so the parts of the screen which are covered by the design remain in liquid form.

STEP 4: The emulsion was washed off creating the stencil.

After the screen has been exposed for a set time, the areas of the screen not covered by the design turned hard. Any unhardened emulsion is then carefully rinsed away. This leaves a clear imprint of the design on the screen for the ink to pass through. The screen is then dried, and the printer makes the necessary touch-ups or corrections to make the imprint as accurate as possible to the original design. The stencil is now ready to be used.

STEP 5: The item was prepared for print.

The screen is then placed on the printing press. The facing cap being printed was laid down flat onto the printing board, underneath the screen.

STEP 6: The ink was pressed through the screen onto the item.

The screen was lowered down onto the printing board. Ink was mixed with the dye thoroughly, and added to the top end of the screen, and a squeegee is used to pull the ink along the full length of the screen. This presses the ink through the open areas of the stenciling printing the design on the facing cap underneath.

STEP 7: The facing cap was dried, checked and finished.

The printed product then passes through a dryer, which 'cures' the ink and creates a smooth, colourfast finishing. The final product was checked and washed thoroughly to remove any residue, before being passed on for use.

Results

Table 1. Effect of Concentration on Dye Exhaustion *Vitellaria paradoxa*

Concentration	ODB	ODA	% Exhaustion
2ml	0.891	0.662	25
4ml	0.891	0.245	73
6ml	0.891	0.248	72
8ml	0.891	0.045	95
10ml	0.891	0.063	93
12ml	0.891	0.067	92

Table 2. Effect of Electrolyte on Dye Exhaustion *Vitellaria paradoxa*

Electrolyte	ODB	ODA	% Exhaustion
0ml	0.891	0.057	94
2ml	0.891	0.128	86
4ml	0.891	0.031	97
6ml	0.891	0.020	98
8ml	0.891	0.526	41
1oml	0.891	0.274	69

Table 3. Effect of Time on dye Exhaustion

Time	ODB	ODA	% Exhaustion
0h	0.891	0.267	70
1h	0.891	0.091	90
2h	0.891	0.201	77
3h	0.891	0.034	96
4h	0.891	0.077	91
5h	0.891	0.185	79

Table 4. Effect of Temperature on dye Exhaustion

Temperature	ODB	ODA	% Exhaustion
30^0C	0.891	0.175	80
40^0C	0.891	0.053	94
50^0C	0.891	0.054	95
60^0C	0.891	0.192	78
70^0C	0.891	0.125	86
80^0C	0.891	0.248	72

Table 5. Effect of mordanting on dye Exhaustion

Mordant	Method of mordanting	Colour	Colour change
Copper sulphate	Post-mordant	Coffee brown	5

Table 6. Effect of washing fastness on dyed sample

Sample	Colour of the dyed sample	Colour change
Temperature	Light brown	2
Concentration	Brown	3
Electrolyte	Carton brown	4
Time	Coffin brown	5

Key: 1=Very Poor; 2=Poor; 3=Fair; 4=Good; 5=Excellent.

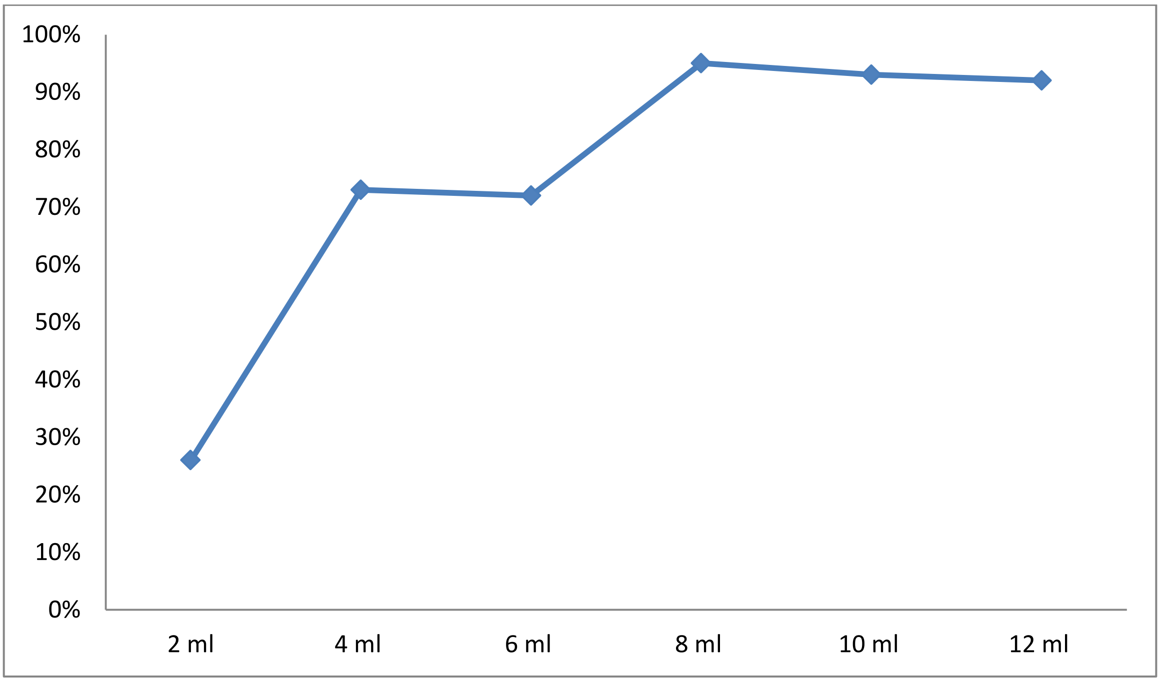

Figure 1. Effect concentration on dye exhaustion

% Exhaustion of extracted dye concentration of *Vitallaria paradoxa* bark at various concentrations using methanol and acetic acid as electrolytes.

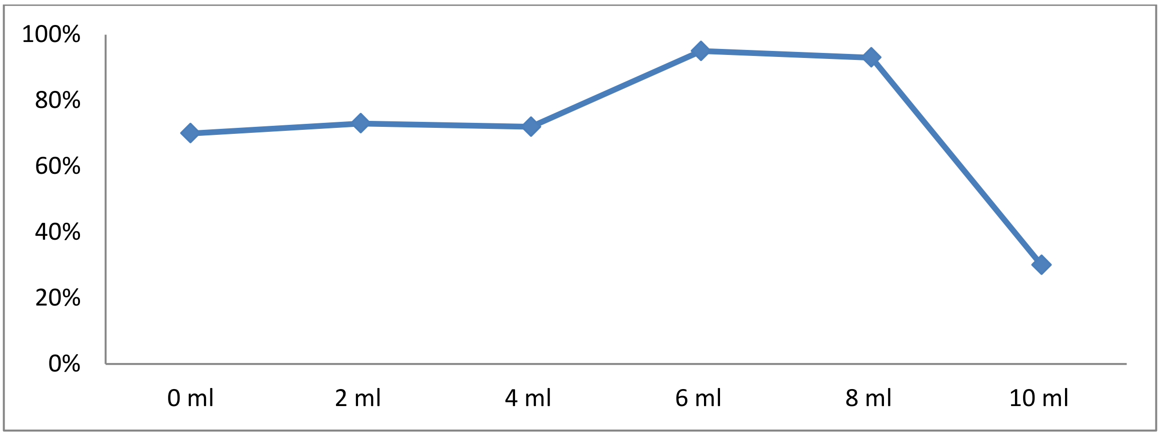

Figure 2. Effect of electrolytes on dye exhaustion

% Exhaustion of extracted dye concentration of *Vitallaria paradoxa* bark methanol and acetic acid as electrolytes.

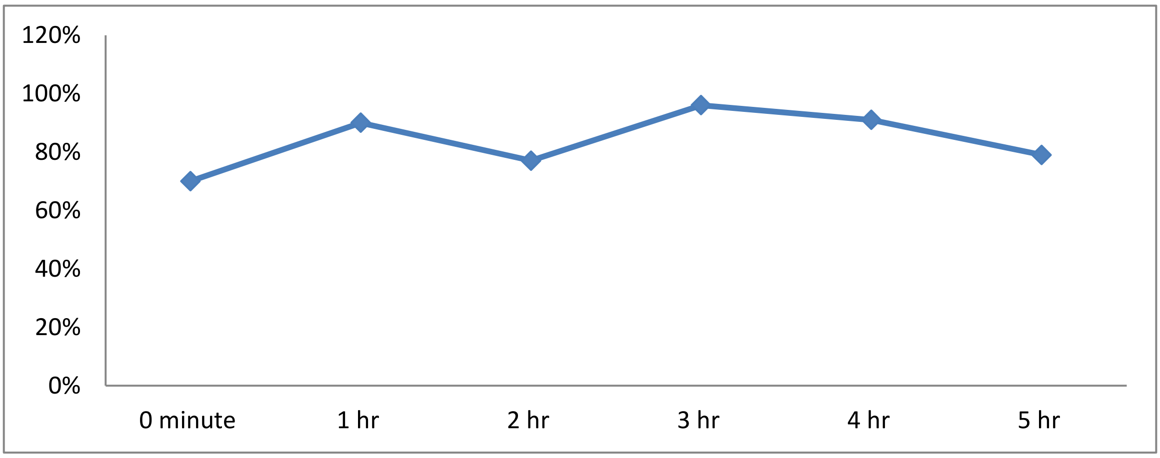

Figure 3. Effects of time on dye exhaustion

% Exhaustion of extracted dye concentration of *Vitallaria paradoxa* bark at various time using methanol and acetic acid as electrolytes.

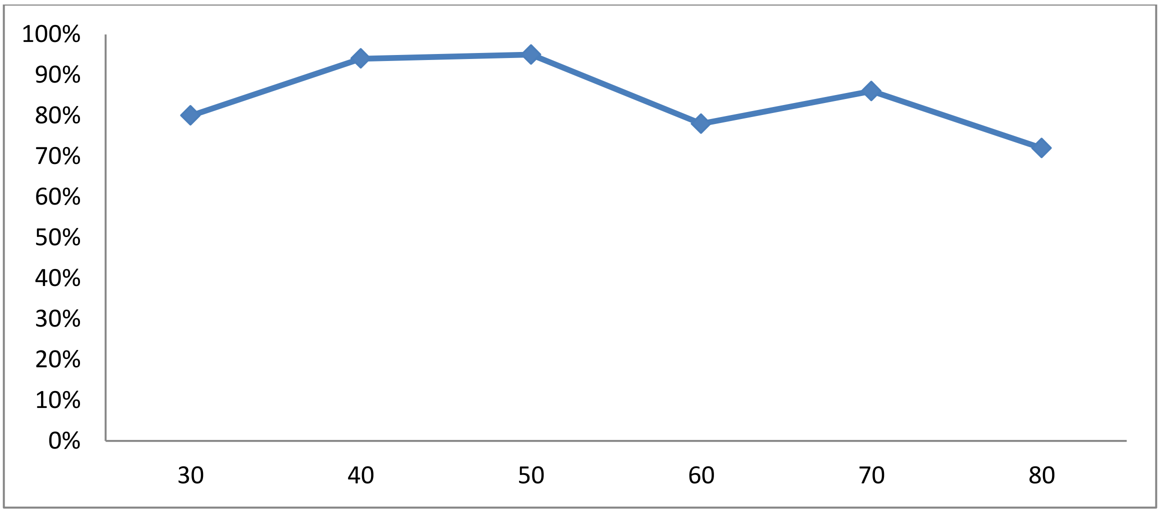

Figure 4. Effect of temperature on dye exhaustion

% Exhaustion of extracted dye concentration of *Vitellaria paradoxa* bark at various Temperature using methanol and acetic acid as electrolytes.

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Discussions

Table1 shows that the optical density after dying decreases with the increase of the percentage exhaustion with maximum at 90%. Dye exhaustion increase as the dye concentration increases. Oforghor et al. (2020) reported that this may explain that the presence of more dye hinders absorption as the dyed to the fabric. It was observed that bright and shade was produced when cotton fabric was dyed with 90% dye percentage exhaustion. The dye behavior of absorption on cotton fabric depends on the characteristics of dye towards the fabric. However, in Table 2, the effects of electrolyte (acetic acid) concentration on colour strength, it was observed that colour strength increases as electrolyte concentration increases but a further increase of the electrolyte did not lead to increase in percentage of dye exhaustion because the fabric has already absorb the quality it can, hence after 85% the increase was pronounced (Oforghor, 2011).

Meanwhile, in Table 3, the effects of dyeing time on colour strength, it was observed that colour strength increases as dyeing time increases but a further increase of the dyeing time after 3h did not lead to increase in percentage of dye exhaustion because the fabric has already absorb the quality it can, hence after 96% the increase remains the same (Oforghor, 2011).

Table 4 shows the effect of temperature on percentage dye exhaustion, it was observed that the absorption of dye by cotton fabric increased with the increase of dying temperature. It seems that dye exists in solution as an aggregate of various when the temperature of the solution increase the thermal energy coming from inside impeded aggregation, and therefore, increase in the amount of the non-aggregates dye properties in solution. The effect of temperature on percentage dye exhaustion was almost the same between 80-90%. However, maximum percentage dye exhaustion was obtained at 50^0C with dyeing levelness.

Washing Fastness Properties of Dye Extracts from *Vitellaria paradoxa* on Cotton Fabric

The result of wash fastness properties of dye extract from *Vitellaria Paradoxa* on cotton fabric were presented in Table 5. The result showed that when cotton was dyed without mordant, a fastness grade 3 was experienced which indicates poor to fair fastness. When the mordant was applied (copper sulphate) the fastness grade increased to the range of 5 on cotton fabric. This indicates an excellent rating of fastness. This shows that more of the dye molecules are fixed on the fabrics by post-mordanting and so can only be slightly washed off.

Conclusion

Natural colorants were successfully extracted from the bark of *Vitellaria paradoxa* using different solvents. Dye extracts from the bark of *Vitellaria paradoxa* was extracted using ethanol as solvent. Methanol and ethanol was the best solvents for the extraction of colourants from Shea tree bark. The dye extracts were applied on cotton fabric with and without mordants. The results obtained in this study suggest that the natural dye extracted from both plants possess intrinsic affinity for natural cotton fabrics. The observed potential affinity of the dye extract for the textile subtracts used for the study may be due to the presence of biomordants such as tannins. The result of the study show dye extracts imparted non-spectral colours (brown) on the textile subtracts. However, the addition of mordant's improved the fastness performance of the dye extracts on the fabrics. Copper sulphate was proffered as a potential mordant because of it ability to produce better light and washing fastness on cotton fabric

Recommendation

The benefits of studying plant dyes and their application on cotton fabrics will improve the socio-economic and aesthetic value in the economy with respect to the result of the study, the following is recommended for further consideration.

Appendix

Figure 5. Dyed and Un-dyed fabric

Declarations

Source of Funding

This study did not receive any grant from funding agencies in the public or not-for-profit sectors.

Conflict of Interests

The authors declare that there is no conflict of interest regarding the publication of this paper.

Consent for Publication

The authors declare that they consented to the publication of this study.

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