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Extraction, Characterization and Application of Dyes from Eucalyptus Bark and Leave Granules

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ABSTRACT
High polar solvents; 2- ethoxy - ethanol, acetone, methanol and distilled water were used to extract natural dyes from eucalyptus bark and leave granules. FTIR, NMR, Uv/Visible, GC/MS characteristics of the dyes were investigated using FTIR spectrophotometer, Bruker – Advanced 400MHz FT – NMR spectrophotometer to determine the spectral characteristics of the colorants. The extracts were applied onto mordanted and unmordanted100% cotton fabrics. Determination of wash, light and rubbing fastness tests were carried out and assessed on gray and blue scales respectively. The results indicated that there are variation in hue and shades of the pre-metallized and un-pre-metallized substrates. The spectral characteristics of the dyes showed higher absorbance from the leaves granules in distilled water and methanol. Extracts from the eucalyptus bark granules gave a deeper shade colouring brown on cotton fabrics whereas the leaves granules appeared lighter and greenish brown on the dyed substrate.The fastness ratings generally ranged from very good to excellent.

Key words: Eucalyptus, dyes, pre-metallized substrates, spectral characteristics, fastness ratings.

I. INTRODUCTION

Nature provides a wealth of plants which will yield their colour for the purpose of dyeing; many natural dyes have been used since antiquity. In the early 21st century, the market for natural dyes in the textile industry is experiencing a resurge. Western consumers have become more concerned about the health and environmental impact of synthetic dyes in manufacturing and there is a growing demand for products that use natural dyes. Completely capturing the market with natural dyed fabric is an urgent need to maintain a safe environment. Textile materials (natural and synthetic) used to be coloured for value addition, look and desire of the customers. Anciently, this purpose of colouring textile was initiated using colours of natural source, until synthetic colours/dyes were invented and commercialized. For ready availability of pure synthetic dyes of different types/classes and its cost advantages, most of textile dyers/ manufacturers shifted towards use of synthetic colourant. Natural dyes have lighter tones and are not very colorfast. But these problems can be overcome by using chemicals called mordants.(Akpuaka, 2001)

Mordants are metallic salts which produce an affinity between the fabric and the dye. Metal ions of mordants act as electron acceptors for electron donors to form coordination bonds with the dye molecule, making them insoluble in water. Besides chemical mordants natural mordants like rice stalk, Aluminum sulphate, are also gaining importance. Natural dyes can produce a wide range of beautiful shades with acceptable level of colour fastness. It is easy to design using natural colours as they complement each other well and rarely clash. Synthetic dyes, on the other hand, often look bright and garish and they require more skill in colour matching. (Eze, 2002)

Dyestuffs are fugitive and non- fugitive colour obtained from different sources. Almost all organic material will produce a colour when boiled in a dyebath, but only specific plants will yield a colour that can act as a dye. Natural dyes can be obtained from various sources like leaves and stems, Twigs and prunings, flower heads, barks ,roots ,outer skins, hulls and husks ,Heartwoods and wood shavings , Berries and Leaves seeds ,Lichens etc. (Oloyede, 1997).

Example of plants that produce natural dyes are as follows: Madder (orange to red dye), Hollyhock

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(pink dye) Hibiscus (reddish-purple dye), Dandelion (yellow dye), Indigo (navy blue dye) , Lavender (silvery gray, pale yellow dye) Marigolds (orange-yellow dye) etc. (Wikipedia ,2022).

II. MATERIALS AND METHODS

Natural dyes from granules of eucalyptus were extracted in aqueous solutions utilizing high polar solvents; ethanol, acetone, methanol and distilled water at ambient conditions.The dye extracts were characterized on Uv/visible spectrophotometer (unicam SP 800), Perkin Elmer 100 FTIR spectrophotometer. The dyes were applied onto 100% un-pre-metallized and pre-metallized cotton fabrics using Aluminum Sulphate $[Al_2(SO4)_3]$ and Sodium Chloride (NaCl) as mordants. The fastness properties of the dyes were assessed on the dyed fabrics using gray and blue scales respectively.

Extraction

100g of eucalyptus bark granuleswas poured into a beaker containing 1000ml of Ethanol, 5g of NaCl and 10g of Aluminum sulphate and left at room temperature for 24 hrs whilst stirring occasionally. The extract was filtered and preserved in a volumetric flask. The procedure above was repeated for eucalyptus leaves granules and the other chosen solvents. However, the distilled water extraction was carried out at the boil for about 1 hour using lower weight of material and higher volume water as shown in Table1

Mordanting

20g sample of bleached cotton fabric was immersed in a bath consisting of 2% o.w.f $[Al_2(SO_4)_3]$, 5% o.w.fNaCl at a liquor ratio of 20:1. The sample was treated at the boil for 1hrafter which it was rinsed, thoroughly washed and dried.

Dye application

Specific weights of Pre-metallized (mordanted) and un-premetallized cotton fabrics were immersed in a solution containing 400 ml/l of dye extract and dyed for about 15 mins. at a temperature of 45°C. 5% o.w.f of sodium chloride was then added to the dyebath and dyeing continued at the boil for 45 mins. After dyeing, the fabrics were rinsed, washed thoroughly under running tap water and dried at room temperature.

FASTNESS PROPERTIES TEST

Washing Fastness Test

The dyed fabric of size 4cm x 10cm was attached to a piece of the adjacent fabric containing (cotton and polyester), also measuring 4cm x 5cm, by sewing along one of the shorter sides with the fabric next to the face of the dyed fabric. The sewed specimen was placed in the wash bottle and the necessary amount of soap solution (5 g of premier soap and 2 g of sodium carbonate per litre) was added in a liquor ratio of 50:1 and the washing fastness is carried out in a warm water for 30 minutes. After that stipulated time, the sewed specimen was removed, rinsed twice in cold water, squeezed and dried. The change in colour of the specimen and the staining of the adjacent fabrics were assessed with grey scales.

Fastness to Rubbing

Dry rubbing

The natural dyed specimen was mounted to the holding clamp on the baseboard of the crock meter which measures 21 by 6 cm. A dry rubbing cloth was mounted flat over the end of the peg on the crock meter and fixed by means of the spring clip provided. The specimen was rubbed back and forth over a straight track 100 mm ± 8 mm long for 10 complete cycles (10 times back and forth) at one cycle per second. Staining of the rubbing cloth was assessed with grey scale.

Wet rubbing

A rubbing cloth was wet with distilled water and squeezed between blotting papers and the tests were carried out as the procedure for dry rubbing. The tested rubbing cloth was allowed to dry at room temperature. Staining of the rubbing cloth was assessed with grey scale.

Fastness to light

The sample to be tested were exposed to light frame lamp with the same characteristics from the natural light but with a much higher intensity called the (Xenon Arc Lamp) light Fastness tester. The dyed samples were measured 10cm by 5cm (Ethanol, methanol and distilled water dyed samples) from the whole fabric. The samples were fastened to the holder then placed into the cell of the Fastness tester (Xenon Arc Lamp). The specimens were exposed to light and each test sample is partly coloured during the exposure. The samples were exposed for 48 hours. The change between the exposed and covered parts of the test sample (dyed samples) were then compared with the change between the covered and exposed area

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of the standard (blue scale) which shows the change in shades and the number represent the degree of fading of the dyed samples.

Ultraviolet /Visible Spectroscopy

2mls of the liquid dye extract was measured into a 30ml flask and diluted with 8mls of each of the solvent used for the extraction to the mark. The solution was vigorously shaken to ensure uniform dispersion of the dye in each solvent, absorbance was selected as the measurement mode for the run and its recording range was set at 1 to 4. After which the range of the wavelength scans was set 550nm and a sampling interval of 1nm. 1ml of the liquid dye was pipetted into the cuvette and the sample was run and the results were recorded and saved.

III. RESULTS AND DISCUSSION

Table 1 Showing results for dye extracted from eucalyptus plant (bark and leave granules)

Plant (Eucalyptus)	**Solvent**	**Weight of granules (g)**	**Volume of solvent (mls)**	**Volume of extract (mls)**	**Time** (Hours)
Bark	Ethanol	100	1000	540	24
Bark	Methanol	100	1000	440	24
Bark	Acetone	100	1000	580	24
Bark	Distilled water	100	800	520	12
Leave	Ethanol	100	1000	510	24
Leave	Acetone	100	1000	560	
Leave	Methanol	100	1000	420	24
Leave	Distilled water	100	8000	510	12

Table 2: Showing washing fastness results of bark and leave granules dyes extracted from different solvents on both mordanted and un-mordanted fabrics

Solvent	**Mordanted fabric**				**Non-mordanted fabric**			
	Bark		Leave		Bark		Leave	
	CSH	STN	CSH	STN	CSH	STN	CSH	STN
Ethanol	5	4-5	4-5	4	4-5	3-4	4	3
Methanol	4-5	4	4	4	4	3	3-4	3
Acetone	5	4-5	4-5	4-5	4-5	4	4	4
Distilled water	4-5	4-5	4	3-4	4	3	3-4	3

KEY:
CSH------ Change in Shade
STN------- Staining of white

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Table 3: Showing Results for Light Fastness Test of Bark and Leave Granules Dyes Extracted from Different Solventson both mordanted and un-mordanted fabrics

Solvent	**Mordanted**		**Non-mordanted**	
	Bark	Leave	Bark	Leave
Ethanol	7	6	6	5
Methanol	6	6	5	4
Acetone	7	6	6	5
Distilled water	7	6	5	4

Table 4: Showing Results for Rubbing Fastness Test.

Dye	**Granules**	**Un-mordanted dyed sample**		**Mordanted dyed sample**	
		wet	**dry**	**wet**	**dry**
Ethanol extract	Bark	3	4	4-5	4-5
	Leave	4	4-5	4	4
Methanol extract	Bark	3-4	4-5	4 -5	4-5
	Leave	4	4-5	4-5	4-5
Acetone extract	Bark	4-5			
	Laeve				
Distilled water extract	Bark	4-5	4-5	4	4
	Leaf	4-5	4-5	4-5	4-5

Table 5: Showings the Absorbency (A) of the Extracted dye using a wavelength of 550nm

Dye	**Granules**	**Absorbance (A)**	**Peak**	**Position (nm)**
Ethanol extract	Bark	1.912	1	550nm
	Leave	6.984	2	
Methanol extract	Bark	3.856	3	550nm
	Leave	4.776	4	
Acetone extract	Bark	4.327	5	550nm
	Leave	6.854	6	
Distilled water extract	Bark	4.728	7	550nm
	Leaf	6.736	8	

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The results presented in Table 1 indicate that the dyes extracted gave good yield (v/v) with higher volume from ethanol and distilled water perhaps due to their higher polarity. Extracts from eucalyptus bark gave rich brown coloration while those from the leaves appeared greenish brown. Dyes extracted from acetone and ethanol showed deeper shades than those of the other solvents. The greater dept of shade arising from acetone and ethanol and water extracts could be attributed to their higher polarity compared to methanol. The dyed pre-metallized fabrics appeared deeper in shade than their un-pre-metalized counterparts. Reasons for this is thought to be that the mordants created more reactive sites in the cotton cellulose molecule which gave rise to more dye attraction and uptake consequently resulting to more dye molecules being attached to the cellulose polymer thereby giving deeper shades.

The fastness ratings of the dyes as presented in Tables 2, 3 and 4 suggest that dyes extracted from eucalyptus bark granules applied onto mordanted cotton fabrics showed better wash, light and rubbing fastness properties than those extracted from the leaves. Still on the results shown in Tables 2,3 and 4 dye extracts from ethanol, acetone and distilled water and applied onto pre-metallized cotton fabrics gave very good to excellent fastness ratings compared to those of ethanol on un-pre-metallized cotton substrate. This could perhaps be due to the high polarity of the solvents as well as greater affinity of the modified cellulose fabrics for the dyes.Investigated spectral characteristics of the dyes as presented in Table 5 reveals that the visible absorption measurement gave maximum absorbency peaks at wavelength of 550nm. Distilled water extract from eucalyptus bark granules recorded absorbency peak at 4.728, Methanol 3.856 ethanol 1.912. The absorbency peaks for leave granules however, were higher as shown in Table 5.

IV. CONCLUSION

Natural dyes were extracted from eucalyptus bark and leave granules in aqueous solutions of high polar solvents. The dye extracts were of high yield and gave wide range of hues and shades notably appearing reddish brown for eucalyptus bark and lemon green to greenish brown for the leaves. All the dye extracts were visibly soluble in water and were applied with ease from aqueous solutions, though under different conditions. The dyes exhausted well on both pre-metallized and un-pre-metallized cottonfabrics with deeper shadesfor the former; giving varied depth of shades. It is thought that the mordant created more reactive sites in the cellulose molecule which gave rise to more dye attraction and uptake resulting to more dye more molecules being attached to the cellulose polymer leadingto deeper shades.Fastness tests carried out on the dyed cotton fabrics indicated that the dyes generally have very good to excellent fastness properties to wash, light and rubbing. It can thus be deduced that eucalyptus plant which abounds locally is one natural plant from which high quality natural dyes can be obtained and could find useful application onto cellulosic fabrics especially cotton which is hugely cultivated in large quantity in Africa. This discovery could go a long way in providing employment for Nigerian youths and as well reduce the impact of importation of synthetics dyes consequently saving foreign exchange.

REFERENCES

[1]. Akpuaka, U.O and Inyang, N.E.U. (2001). Local Plant and Their Application in P.H Titration Dyeing of Fabrics and Paper. Nigeria Journal of Science Education, 5 (1): 25 – 33.

[2]. Eze, S.O; Okereafo, A (2002). Industrial and analytic potentials of dye extracts from Telfairaaccidentalis (Fluted pumpkin). J. Chem. Soc. Nig. 27,143-146.

[3]. Fryxell, P. A. (2003). Morphology of the base of seed hairs of Gossypium I. gross morphology, Bot. Gas., 124, 196–199.

[4]. Gibson, J. R. (2001). Temperature effects on growth, development, and fiber properties, in Cotton Physiology, J. R. Mauney and J. M. Steward, eds, The Cotton Foundation, Memphis, TN, pp. 47–56.

[5]. Gautam, R. (2006). "The water footprint of cotton consumption: An assessment of the impact of worldwide consumption of cotton products on the water resources in the cotton producing countries". Ecological Economics. 60: 186–203.

[6]. Hsieh, Y.-L. (2001). Variations of mature cotton fiber tensile properties – association with seed positions and fiber lengths, Textile Research Journal 71(12): 1079–1086.

[7]. Maxwell, (2003). Textiles of Southeast Asia: tradition, trade and transformation (revised ed.). Tuttle Publishing. p. 410. ISBN 978-0-7946-0104-1.

[8]. Ochonogor, E.C (2000). Improvised Indicators for Chemical Analysis in Mag

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Akala (Ed.) 41st Annual Conference Procedures of Science Teachers Association of Nigeria Ibadan: Heine Mann Education Book P/C. Pp. 419 – 421.
[9]. Oloyede, O. I. (1997). Extracting Acid Bases and Indicators from Locally Available Materials: Implication for the Integrated Science Teachers. A Paper Presented At The 40th Annual Conference of Science Teachers Association of Nigeria (STAN) Held At Kano.
[10]. Rebenfeld, L. (2007). Mechanical properties of native fibrous materials, Proc. 1st Cleveland Symp. Macromol. A. G. Walton, ed., Elsevier, Amsterdam, 177–201.
[11]. Rupert B. (2011) ,Human factors in cotton culture; a study in the social geography of the American South (U of North Carolina Press, 1929) online free
[12]. Seagull, R. W. (1996). Changes in microtubule organization and wall microfibril orientation during in vitro cotton fiber development: an immunofluorescent study, Can. J. Bot., 64, 1373–1381.
[13]. Williford, J. R. (2008). "100 Years of Cotton Production, Harvesting, and Ginning Systems". Transactions of the ASABE. 51 (4): 1187–98. doi:10.13031/2013.25234. Archived from the original on 25 April 2015. Retrieved 28 August 2015