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The Dyeability Potential of Cellulosic Fibres Using African Yellow Wood *(Enantia chlorantha)*

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Abstract. The dye from African yellow tree, *Enantia chlorantha*, extracted by solvent extraction using acetone at a solute-solvent ratio of 1:25 was studied for its dyeability potential on cellulosic fibres. A golden yellow dye having a melting point ca 146-149 °C with 6.2% recovery was obtained. The dye was soluble in hydroxyl organic solvents. The cellulosic fibre has a greater dye uptake (26.0-23.2 mg/g) when dyed at a temperature of 80 °C than at 60 °C (22.0-21.6 mg/g). Its optimum dye-uptake, at both the temperatures, was achieved 90 min after the commencement of dyeing. However, the dyeability potential of the dye on unmordanted cellulosic fibres showed less substantivity as revealed by its low mean fastness ratings of 1.5 and 1.0 to washing and light, respectively. The tensile properties of the dyed cellulosic fibres, nevertheless, were greatly enhanced.

Keywords: Enantia dye, dye-uptake, *Enantia chlorantha,* African yellow wood

Introduction

African yellow wood, *Enantia chlorantha* (locally called, oso pupa/yaru), family Annonaceae, recognised by its bright yellow slash and conspicuous black fruits, is a fairly sized forest tree usually grown in dense shade (Dalziel, 1955). It is found in Southern Nigeria, West Cameroun, Gabon, Angola and Zaire. The wood is yellow, uniform throughout, fairly fine-grained, splitting easily and rather soft, turning brown after long exposure. It is used locally for house building and furniture work. Native caps are made from the fibrous bark in South Nigeria (Dalziel, 1955). The bark is intensely bitter to taste and has a lot of medicinal uses (Burkill, 1985). The bitterness is attributed to the presence of berberine, known to be haemostatic and febrifuge, which is present in all the species of *Enantia* (Dalziel, 1955). In comparison, *Morinda lucida* (locally called, awopa/oruwo), family Rubiaceae, having a similar texture, and outer bark blackish with deeper inner yellow, is also used as the source of a yellow dye (Adetuyi *et al.,* 2002).

An attempt has been made in this study to examine the dyeability potential of Enantia dye, closely related to Morinda yellow dye, on cellulosic fibres and its fastness properties to light, washing, perspiration and ironing.

Materials and Methods

Sample collection and treatment. The bark of the African yellow wood *E. chlorantha* was purchased from Alade Market in Idanre, Ondo State, Nigeria. The outer part of the wood bark (dark grey) was removed using pen-knife leaving only the inner yellow part which was cut into smaller pieces and oven dried at 110 °C for 6 h to reduce its moisture content considerably before extraction. The sample was finely ground and stored.

Extraction. Pulverized sample (10 g) was extracted with acetone in a Soxhlet extractor for 2 h at a solute-solvent ratio of 1:25. The solvent was removed from the extract by evaporatien (atmospheric). The colourant was transferred to an evaporating dish to which 20 ml petroleum ether (60-80 °C) was added and heated to dryness to remove the sticky nature of the extract. It was dried in an oven at 60 °C for 30 min, cooled, weighed and stored.

Determination of physicochemical properties. The following physicochemical properties were determined for the colourant.

Melting point. The melting point of the dye extract was determined using the capillary tube electrothermal method (Furniss *et al.,* 1978).

Solubility test. The solubility of the dye was tested in the following selected solvents: acetone, groundnut oil, hydrochloric acid (5%), methanol, petroleum ether, palm kernel oil (PKO), sodium hydroxide, sulphuric acid (conc), sodium hydrogen carbonate and water. The test was carried out by adding 10 mg of the colourant to each of the above solvents in a test tube, and in case the colourant was not soluble in the cold, gentle heat was applied by placing the test tube inside a heated water bath.

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Lassaigne test. The colourant was tested for the presence of nitrogen, sulphur and halogens by Lassaigne test (Furniss *et al.,* 1978).

Phenolic hydroxyl group test. This was carried out using ferric chloride-ethanol solution (1:10 w/v). A developed TLC plate was sprayed with the test solution. The presence of blue-black colour on the plate thereafter indicated the presence of phenolic hydroxyl group in the sample.

Dyeing of the cellulosic fibres. Scoured and bleached white cellulosic fibres (yarn and fabric) were used for dyeing without prior application of a mordant. The dyeing was done at two different depths, 2% and 5% for cotton yarn and fabric, respectively, carried out in an infinite dye bath in which the concentration of the colourant was maintained constant. The recipes followed were proportional to the weight of the goods (White, 1980; Giles, 1974). Dyeing was done in a single-bath dyeing machine MBMK II.

2 % Dyeing of cellulosic yarn for dye-uptake measurement. Two dyebaths, each containing 4 g/l of the dye extract, 30% sodium chloride and 3% sodium hydroxide in a liquid-to-goods ratio of 100:1, and a blank bath of the same liquid-to-goods ratio without the dye were prepared for 2% dyeing of 500 mg cotton yarn. One dyebath served as a standard solution from which various aliquots were taken for serial dilutions. Their optical density was measured on Biochron-4060 spectrophotometer at λ_{max} 390 nm of the pure dyestuff solution. The other dyebath and blank were transferred into the dye tubes mounted inside the dyeing machine and set at the dyeing temperature of 60 °C or 80 °C. On attainment of the temperature, the pre-soaked cellulosic yarn at the same temperature was introduced into the dye tube to commence dyeing . Sampling of the spent dye-liquor, about 2 ml, was done at intervals of 30 min for a period of 3 h. Prior to sampling, dyed yarn was removed from the dye-liquor. Equivalent aliquot from the blank liquor was introduced into the dye liquor to replace the dye assistants that had been removed by the dyed yarn. The dyed yarn was returned and dyeing continued till the next sampling period. The sampled solutions were diluted and their optical density measured. The dye-uptakes were determined using equations 1 and 2 (Popoola, 1983):

$$\frac{1000.C_{Df}}{W_{Df}} \qquad (1)$$

where:

W_{Df} : weight of dyed cellulosic yarn (0.5 g)

C_{Df} : the concentration of dye in yarn (% w/v), which is as given in equation (2) below

$$C_{Df} = 0.02 - C_{Ds} \qquad (2)$$

where:

0.02 (% w/v): a constant (initial concentration of dye-liquor)

C_{Ds}: the concentration of spent dye-liquor at various time intervals of dyeing, deduced from the calibration curve

After the dyeing operation, the yarn was removed from the dyebath and properly washed in a mild soap solution before being rinsed in cold distilled water and dried.

5 % Dyeing of cellulosic fabric for fastness properties test. The dyebath for 5% dyeing of the dye extract on cellulosic fabric (6 g) in the dye-liquor ratio of 30:1 contained 3 ml of the dye solution (1 g/100 ml), 30% sodium chloride and 5% sodium hydroxide. The dyeing operation was allowed to proceed for 3 h at 60 °C after which the fabric was removed from the dye bath and treated as previously described for dyed yarn. It was then fixed with hot iron.

Assessment of fastness properties. Four fastness properties, namly, washing, perspiration, hot pressing (heat treatment) and light were determined on the dyed fabric according to the International Standards Organisation (ISO) procedures (SDC, 1992). For each test, 10 specimens of the dyed article were tested and the mean value of the results obtained was taken as the fastness rating for the test.

Tensile properties of the dyed cellulosic yarn. The load-elongation at break of the dyed cellulosic yarn of various lengths was done at the constant rate of elongation of 500 per min and 500 N per tension on Instron tensile tester T5000 at Yaba College of Technology, Lagos, Nigeria.

Results and Discussion

Physicochemical properties. The results of the physicochemical properties carried out on the Enantia colourant and the colour of the dyed cellulosic fibres are summarized in Table 1. It was of low yield (6.2%) when compared with Morinda yellow dye of 16.5% yield (Adetuyi *et al.,* 2002). The solubility test results shown in Table 2 indicate that the Enantia dye was less soluble in the tested polar organic solvents than the Morinda dye. However, it was found soluble in concentrated sulphuric acid as is Morinda, which was an indication of the presence of an acidic group or electron withdrawing groups such as hydroxyl, carbonyl or nitro-groups (Mohring and Nechers, 1979).

Dyeing and dye-uptake. All the cellulosic dyed fibres, in both yarn and fabric, were uniformly and evenly coloured yellow (Table 1). Fig. 1and 2 show the calibration and the dye uptake curves, respectively, of the dyed cellulosic yarn at 60 °C and 80 °C. The dye-uptake of the yarn in the Enantia dye extract

Table 1. The physicochemical parameters obtained for colourant from African yellow wood (*Enantia chlorantha*)

Tests performed	Observations
Appearance of crystals	golden yellow
Yield	6.2%
Melting point	146-149 °C
Lassaigne's sodium fusion test	only nitrogen present
Phenolic hydroxyl group test	negative
Shade of dyed articles:	
cotton yarn	light yellow
cotton fabric	yellow

Table 2. Solubility of Enantia dye in selected solvents

Solvents	Observation
Acetone	+
Distilled water	+
Groundnut oil	-
Hydrochloric acid (5%)	-
Methanol	+
Palm kernel oil (PKO)	-
Petroleum ether	-
Sodium hydrogen carbonate (5%)	-
Sodium hydroxide (5%)	+
Sulpuric acid (conc.)	+

+ soluble; - insoluble

increased porgressively with time at the two dyeing temperatures. After 90 min of dyeing, a sudden fall in the dye uptake was noticed in the cellulosic yarn dyed at 80 °C, while that at 60 °C was fairly constant (Fig. 2). However, the dye uptake of the dyed yarn at 80 °C was higher than that at 60 °C. This observation was contrary to the expected behaviour of a direct colourant on cellulosic fibres (Giles, 1974). The deviation may be attributed to the purity of the Enantia colourant and the fibre morphology. It has been reported that changes in fibre morphology, as a result of high dyeing temperature with time, affects the dye-uptake of fibre (Burdett, 1975), as heat treatment of the fibre or yarn affects the amorphous content of the fibre, which is the region accessible to dyestuffs. The fibre at 80 °C presented a more accessible region for dye absorption than at 60 °C and these regions were more for the first 90 min of dyeing commencement. Thereafter, the colourant bleeds out of these sites. Thus, increasing dyeing time at this particular temperature probably affects the mobility of the chain segments of the fibre molecules, thereby increasing its crystallinity and decreased its dye-uptake.

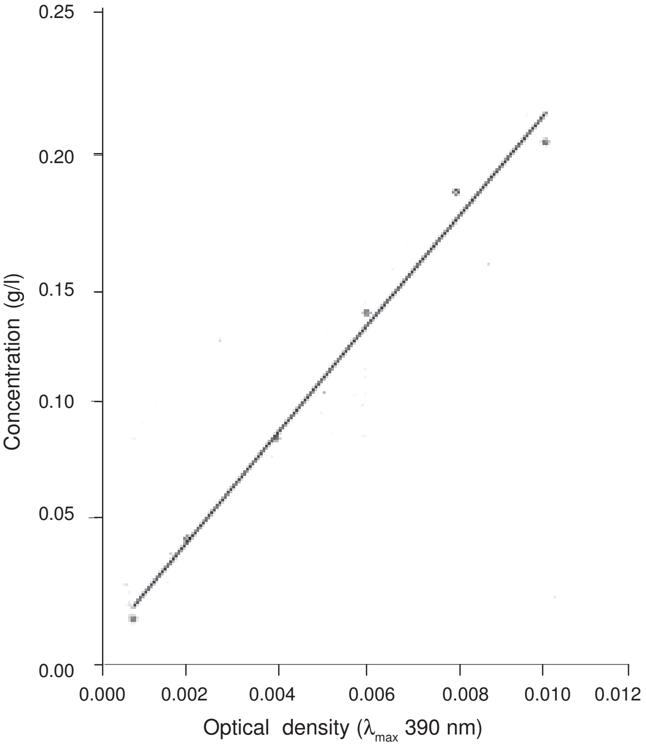

Fig. 1. Optical density versus concentration (g/l) calibration curve for Enantia dye in alkaline medium.

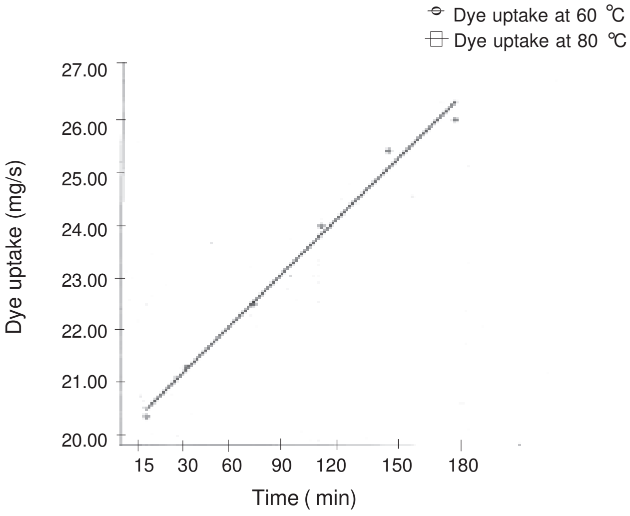

Fig. 2. Dye uptake of cotton yarn in Enantia dye at various time intervals (min) at 60 °C and 80 °C.

Fastness properties of the dyed cellulosic fabric. The results obtained for the fastness ratings of the dyed fabric are given in Table 3. The dyed fabrics have poor mean fastness ratings of 1.0 and 1.6 on a Grey scale of 8 and 5 to light and washing, respectively. This shows that the dye structure itself was not stable (fugitive) to high energy radiation, such as ultraviolet. It also suggested a less extensive conjugation or a straight chain for the dye molecule (Kemp, 1991). Its low fastness rating to washing suggested that the dye was less substantive to cotton fabric and the substantivity can be improved with

Table 3. Fastness ratings of *Enantia chlorantha* dyed cellulosic fabric

Specimen no.	Washing	Perspiration	Hot pressing	Light
1	2	3	4	1
2	1	3+	4	1
3	1+	3	4	1+
4	2	3	4	1
5	1	3+	4+	1+
6	1+	3	4	1
7	2+	3	4+	1
8	1	2	4	1
9	2	3	4	1
10	1	3	4	1
Mean fastness rating	1.6	3.0	4.1	1.0

Grey scale rating: 1-2 low fastness; 3-4 moderate fastness ; 5-6 high fastness

the use of a suitable mordant, either natural or synthetic. However, the dyed fabric had good fastness ratings of 3.0 and 4.0 based on a scale of 5 in respect of perspiration and hot pressing, respectively. This is an indication of the stability of the dye factor to alkaline/acidic liquid treatment (perspiration solution) and dry heat that the material may be exposed to during usage.

Tensile properties of the dyed cellulosic yarn. The tensile properties of the dyed cellulosic yarn at the dyeing temperatures of 60 °C and 80 °C are presented in a load-elongation curve shown in Fig. 3. The dyed yarn was less extensive than the undyed yarn (control). The dyed cellulosic yarn at 80 °C was stronger than that dyed at 60 °C, corroborating maximum dye-uptake at this temperature (80 °C), which contributed to the yarn strength and the subsequent fabric to be produced from them.

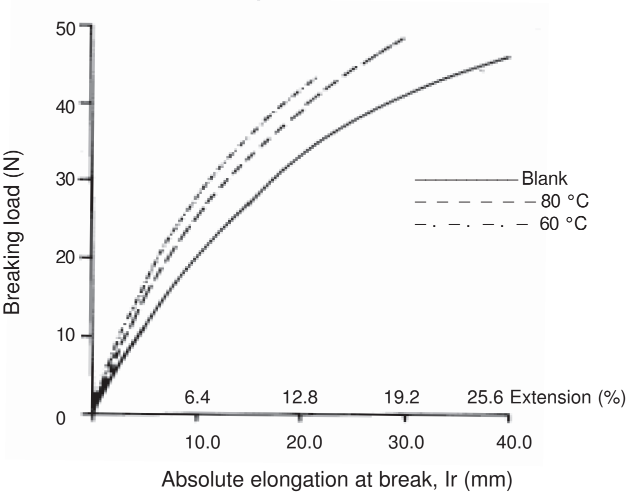

Fig. 3. Load-elongation curves for 15-cm Enantia dyed cotton yarn at 60 °C and 80 °C.

In conclusion, based on the performance of the colourant in this study, and with a little modification to the dyeing processes to increase its substantivity as earlier suggested, the Enantia yellow colourant may be best used for cottage cellulosic textile industry (because of low yield) at 80 °C for a dyeing period of 90 min.

References

Adetuyi, A. O., Popoola, A. V., Lajide, L. 2002. Isolation, purification and uv-visible spectroscopic study of the colourant from *Morinda lucida* plant. *J. Appl. Sci.* **5**: 3174-3185.

Burdett, B. C. 1975. Influence of fibre structure on dye uptake. In: *The Theory of Colouration of Textiles*, C. L. Bird and W. S. Boston (eds.), pp. 111-115, Dyers Comp. Publ., England.

Burkill, H. M. 1985. *Revision of Dalziel's Useful Plants of West Tropical Africa,* vol. **I:** pp. 111-112, 2nd edition, White Friars Ltd., England.

Dalziel, J. M. 1955. *The Useful Plants of West Tropical Africa*, pp. 4-6, Watmughts Ltd., Bradford, England.

Furniss, B. S., Hannaford, A. J., Rogers, V., Smith, P. W. G., Tatchell, A. J. 1978. *Vogel's Textbook of Practical Organic Chemistry,* pp. 100-222, 4th edition, Longman Publishers, London, England.

Giles, C. H. 1974. *A Laboratory Course in Dyeing,* pp. 65, 126-127, 3rd edition, The Society of Dyers and Colourists Publishers, Bradford, England.

Kemp, W. 1991. *Organic Spectroscopy,* pp. 245-248, 3rd edition, Macmilan Publishers, Ltd., London, UK.

Mohring, J. R., Nechers, D. C. 1979. *Laboratory Experiments in Organic Chemistry*, pp. 469-498, 3rd editoin, D. van Nostrand Comp., New York, USA.

Popoola, A. V. 1983. The Effects of Heat and Liquid Treatments on Structure and Properties of Polyethylene Terephthalate. *Ph. D. Thesis,* pp.1- 92, UMIST, London, UK.

SDC. 1992. *Standard Methods for the Determination of Colour Fastness of Textiles and Leather*, pp. B03 (1-7), C03 (1-3), E04 (1-3), P01 (1-2), 3rd edition, The Society of Dyers and Colourists, SDC Publishers, Bradford, England.

White Jr., M. 1980. Dyeing with direct dyes. *American Colour and Chemical Corp., Charlotte Dyeing Primer (Part 1)* **12:** 88-89.