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FASTNESS PROPERTIES OF INDIGO-DYED COTTON FABRIC

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Natural indigo vat dye (CI vat blue 1) was extracted from the plant species, *Lonchocarpus cyanescence* and applied to pure white (bleached) cotton fabric intended as wearing apparel, by dyeing using alkali concentrate from cocoa pods *(Theobroma cacao)* as solubilising agent. The fastness properties of the dyed fabric to light, washing, ironing and rubbing were then assessed using standard Gray scales. The dyed fabric was found to show very good fastness to these agencies.

Key words: Fastness properties, Dyestuffs, Fabric.

Introduction

Indigo is the name of an important deep blue dye of commerce that was once known as the 'King of the dyestuffs'. It is also the common name for plants of the genus *Indigofera* from which this important dye is obtained. Indigo is one of the oldest known natural dyes, the natural product was initially classified as natural blue 1. It is also known as CI vat blue 1 (CI 73000) because this dyestuff could be applied to cotton fabrics by the vatting process now commonly used for the chemically related vat dyes. Between 700-800 species of the plant genus *Indigofera* are found widespread within the tropics and subtropical zones where they now constitute an invaluable source of raw material for the dyeing and printing industries. Although, Indigo has been long extracted and used, the process of applying the dyestuff using alkali concentrate from vegetable source as vatting agent as alternative to the caustic soda of the more successful industrial application has not been given wide ranging attention. The aim of the present study is to achieve this objective.

Materials and Methods

The indigo dye used for this work was extracted from the plant, *Lonchocarpus cyanescence*. The alkali used for the vatting process was extracted from the pods of the plant *Theobroma cacao*.

Cotton fabric was donated by O'odua Textiles, Ado-Ekiti. The fabric was pure white finished (bleached) free from both sizing agent and resin finishes.

Extraction of Indigo. Fresh indigo leaves (preferably the young foliage leaves) were collected and pounded in a mortar and the pulp molded into balls. They were allowed to dry in the sun for three days during which fermentation occured. Following the drying, 20gm of the Indigo balls was placed in a conical flask and 200ml of 18°TW caustic solution was poured over it and then corked. The content of the flask was stirred occasionally for the next 5 days. At the end of this period, the content was sieved and the vegetable debris discarded. The filterate containing the dye pigment was neutralised with 100 ml of 1M HCl to remove residual NaOH then concentrated on water bath and the residue dried in the oven at 120° for 24 hr to remove all the waters contained completely from the dyestuff.

Extraction of alkali. The alkali was extracted from cocoa pods *(Theobroma cacao)*. Fresh cocoa pods were collected from farms and were dried in the sun for 21 days and were later pulverised and ashed in a muffle furnace at a temperature of 800° [1]. The ashes were collected, cooled and transferred to a 2 litre standard flask. Water was added and thoroughly mixed to dissolve the alkali in the ash. The resulting mixture was filtered through whatman filter paper into a 250 ml beaker. The solution obtained was concentrated by heating until white needle-like alkali crystals appeared. They were dried in the oven at 105° for 3 hr.

Analysis of the alkali extract. This was done using flame photometer Unicam SP900. One gram of finely ground cocoa pod was weighed into a crucible and put in a furnace at a temperature of 800° for ashing. The ashes obtained were cooled and weighed. This was then dissolved using distilled water in a 50cm^3 standard flask and finally made up to mark. Standard solutions were prepared using Analar grades of KCl, NaCl and Ca $(NO_3)_2$. $4H_2O$. 1.9079gm of KCl, 2.5413gm of NaCl and 5.904gm of $Ca(NO_3)_2$; $4H_2O$ respectively (all sufficiently dried at 110° in the oven before use) were dissolved separately in 1 litre of distilled water. These solutions were used as standards to obtain a calibration curve.

Using the photometer, the emission intensities of these standards were obtained along side that of the test solution [1] and the metallic ions present determined.

The anions of the alkali extract were determined using the method described by Svehla [2]. The pH of the extract was also determined. The results of these determinations are summarised in Table 1.

Dyeing of cotton with indigo. Specimens of pure white cotton fabric were dyed using the indigo extracted as described earlier. 2.5 gram of the indigo was weighed into a 800cm^3 beaker and dissolved with 100 cm^3 of distilled water.

6 ml of 10% of the alkali solution obtained from Cocoa and 2.6 gram of sodium hydrosulphite ($Na_2S_2O_4$) was added one after the other to the dye bath and the solution was warmed with mild agitation on a hot plate to 50° until all the chemicals have dissolved. The bath was then made up to 500 ml of distilled water and allowed to vat for 10 min. Thereafter, the fabric was entered in a wet state and 3 sequences of dyeing/air oxidation were carried out repeatedly for 15, 10 and 5 min. keeping the fabric well agitated in the dye liquor. After the desired deep shade had been obtained, the fabric was removed, rinsed in cold water and then at the boil using 2% solution of non-ionic surfactant Lissapol N. Final washing and rinsing was done in cold running tap water before drying of the fabric [2-5].

Determination of the fastness properties of the dyed fabric. Four fastness tests were carried out on the indigo-dyed fabrics. These are fastness to light, washing, ironing and rubbing. Test methods used were those of Internatinal Organisation for Standardisation (ISO) [6,7].

Fastness to light. Fastness testing to light was carried out by exposing the deep dyed fabricto an artificial fading lamp source using the Shirley light fastness tester model SDL 237. Samples for testing were mounted in cells. After pre-set test cycle, samples were assessed using the standard 8-point Gray scale. Ten specimens were used for the test and the mean value of the results obtained taken as the fastness rating for the test.

Fastness to washing. Test method 3 specifically designed for cellulosic fabric was used. Fabric specimens measuring 10 cm x 4 cm each were placed in turn between one piece each of undyed cotton and wool fabrics measuring 5 cm x 4 cm and stitched around leaving a portion of the specimen uncovered. A beaker containing 100 ml. solution of 0.5 gm Lissapol N, was heated to the boil (giving a liquor ratio of 50: 1 on the 2 gm weight of the fabric). The fabric in each case was allowed to remain in the solution at this temperature for 30 min. whilst stirring was carried out occasionally. The specimen was later rinsed in running cold water for about 10 min. the stitch line removed along 2 sides and the specimen hanged out to dry. After drying, the change in colour of the uncovered portion of the fabric was assessed on the 5 point Gray scale. As in the case of fastness to light, ten specimens were used for the test and the mean value of the results obtained taken as the fastness rating for the test.

Fastness to ironing. Essentially, this fastness test assesses the resistance of the dye to transfer by sublimation. In this study the fastness to ironing in the dry state was assessed as follows:

A dyed specimen measuring 10 x 4 cm was positioned on a dry bleached white cotton cloth measuring 14 x 4 cm and an iron set at cotton rating and having a pressure gauge of 30 gm cm^{-2} was placed on top of the fabrics for 15 sec. After this, the colour change in terms of degree of staining of the white fabric was assessed.

Fastness to rubbing . This test was carried out to assess the resistance of the dyed fabric to transfer by rubbing using Shirley's crockmeter SDL 238A. This was done by rubbing a dyed specimen of the fabric measuring 10 cm by 5 cm in contact with similar but undyed white (control) fabric back and forth ten times in 10 sec. The degree of staining of the undyed specimen was thereafter rated against the standard Grey scales.

Results and Discussion

The result of the analysis of the alkali extract used as solubilising agent in this study is shown in Table 1. The alkali is found to consist principally of mixtures of caustic soda and potash and carbonates. These salts particularly the caustic-soda/potash together with sodium hydrosulphite served as the effective vatting agents for the indigo dye. The vatting which is essentially a reduction process is a necessary condition for the conversion of the insoluble indigo into the soluble 'leuco' sodium salt of the pigment. The reduced, but soluble sodium salt once formed is absorbed by the fabric and is subsequently converted to the insoluble, oxidised pigmented form in the fabric upon exposure to air or by treatment with appropriate oxidising agent.

Vat dyes are known to be most stable in alkaline medium as this will ensure the dyebath's retention of the reduced state during the period of the dyeing. Accordingly, the pH of 11.50- 11.85 found for this alkali extract helps to achieve this condition which is necessary for satisfactory dye uptake.

The results of the fastness tests to the various agencies light, washing, ironing and rubbing of the indigo-dyed cotton fabric are shown collectively in Table 2. From the Table, it can be seen that the indigo dyed fabric showed very good mean fastness to light rating of 5.20 considering the 8-point scale on which this property has been assessed.

Similarly, the mean fastness ratings to other agencies i.e. fastness to washing of 3.45, dry ironing of 3.75 and rubbing of 3.90 for the indigo dyed cotton fabric are considered very good on a 5-point scale on which they were assessed. The explanation for the satisfactory fastness rating rests on the fact that, Vat dye such as indigo, unlike other classes of dyes which are held on to textile fibres by chemical bonds, operate upon a different dyeig principle [8]. The dye is mechanically trapped within the

TABLE 1. PHYSICAL DATA OF ALKALI EXTRACT FROM *T. CACAO.*

Crystal form	White needle-like crystals.
pH	11.50-11.85
Cations found	Na^+ , K^+
Anions found	OH^- , CO_3^{2-}

TABLE 2. FASTNESS RATINGS OF INDIGO DYED COTTON FABRIC INTENDED AS WEARING APPAREL.

Fastness test specimens	Light	Washing	Dry ironing	Rubbing
1	6	4	4	3
2	5	4	4	2
3	4	4	4	4
4	6	3+	3	4
5	5	4	3+	3
6	5	4	4	4
7	6	3	4	5
8	6	4	3	4
9	5	4	4	3
10	4	3	4	3+
Mean rating	5.20	3.65	2.75	3.90

fibre structure as insoluble pigments resulting in dyeings having outstanding resistance to most agents encountered in use. Since the entrapped pigments are water insoluble, they cannot be easily washed off in water. The mechanical trapping of the colouring pigments within the textile material is a result of lack of appropriate functional groups in the dye which are capable of forming chemical bonds with the -OH (hydroxyl) groups present in the fibre [9].

Apart from the solubilising effect of the caustic solution on the dye, it also has a swelling effect on the fabric especially as the fabric was held loose during the period of dyeing. This effect is responsible for an improved dye-uptake in the fabric. This is achieved by causing the individual fibre to swell thus promoting further entrapment of the colouring pigment within the fibres interstices (matrix) and consequently, high fastness ratings to the various agencies observed.

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