Pak J Sci Ind Res 2001 **44** (2) 89-92

Comparative Fastness Assessment Performance of Cellulosic Fibers Dyed Using Natural Colorants

Adeola Victor Popoola

Department of Industrial Chemistry, Federal University of Technology, P.M.B. 704, Akure, Ondo State, Nigeria

(Received 16 February 1998; accepted 17 July 2000)

Two natural dyes of plant origin, namely bixin and curcumin were extracted, purified and used as color additives in two fibrous constructions of floor carpet and cotton fabric by the 'exhaust dyeing' technique. The fastness properties of these dyes both to light and washing and their use were assessed comparatively. Curcumin showed higher average fastness ratings to light and to alkaline wash over its bixin counterpart.

Key words: Curcumin, Bixin, Dyestuffs, Fastness.

Introduction

Dyes and pigments of vegetable and mineral origins were first reported used in the Holy book when Noah was instructed to coat his ark inside and out with pitch - an organic pigment (The Holy Bible, NKJV 1985). Ever since, man has discovered ways of exploiting the natural coloring matters covering applications from painting of culturally relevant events on walls of buildings to the use as cosmetics. Other more important areas of use include the dyeing of various household articles ranging from items of clothing to leather, carpet, etc.

Although the emergence of synthetic coal tar dyes in the latter part of the 19th century led to a considerable decline in the use of these natural coloring molecules, mostly on the grounds of technical and commercial merits of the former dyes and pigments mostly of plant origin are still used today where their substitutes are still difficult to find, for instance, in food coloring and medicinal/cosmetic preparations being clinically safer than their synthetic analogs both in handling and use. Some other specialty colours mostly those of logwood and henna still continue to fulfil unique roles in hair and leather coloring. Besides most countries of the world have disposed of their use (Mclaren 1983). In the less developed nations where the cost of importing dyes is prohibitive and the technology of utilising petrochemicals is at very low ebb, a sizeable amount of natural dyes and pigments are used mostly in cottage industries. In recent years, a number of such dye yielding plants has been screened and their uses for imparting fast and substantive colours to textiles, leather etc., are being systematically investigated. Two of such dyes i.e. bixin and curcumin were developed for use in this work on two cellulosic fibrous constructions (cotton fabric and reed carpet) and the light and wash fastness properties of the dyed materials were assessed. (Popoola and Samaye 1997).

Agape@skannet.com.ng.

Materials and Methods

Extraction of dyes. Bixin and curcumin are the coloring chemicals found in the plants *Bixa orellana* and *Curcuma longa* (a family of the perennial herb, *Zingiber officinale)* respectively. *Bixa orellana* seeds were harvested, cleaned and were subsequently processed for moisture, oil and dye contents. Extraction was carried out in a soxhlet. Prior to the extraction, the seeds were dried in the oven set at 110°C for 3 h. Thereafter about 20g of the seeds were packed into a sintered glass extraction thimble contained in the soxhlet extractor and washed with *n*-hexane for 6 h to remove oil which might prevent the dyestuff from crystallising out from the extracting liquor. Subsequently, the seeds were extracted using chloroform and ethyl acetate prepared in the mixture ratio of 3:2 (vv^{-1}). Extraction process was carried out until the extractant liquid was virtually colourless indicating no further transfer of dyestuff into the mother liquor. The extracting solvent was later recovered by distilling off the solvent whilst the crude dye was separated out for drying in the oven at 105°C for 3 h for further purification prior to use.

The procedure adopted for the extraction of curcumin from ginger was similar to that described above for bixin except for the extractant used which was in this case methanol.

Dye purification. This was done by first recrystallising the crude dyes. Bixin was separated out from the crude by recrystallising in hot ethanol while curcumin was recrystallised from dry ether. The recrystallised dyes were then fractionated by column chromatography over alumina in a glass column using solvents varying in their polarities. The fractions collected were spotted on TLC (thin layer chromatography) in chloroform:acetone (90:10 vv^{-1}) for 10 min and spots detected with sulphuric/nitric acid reagents and bromocresol green. Single spots obtained on TLC for the dyes confirmed their purities.

UV visible scan. Following the purifications of the two dyes their UV visible spectra were run on UV visible spectrosol U2030 of Eagle Sci. model. They were both found to transmit yellow colour at a working maximum wavelength λ_{max} of 420 nm.

Dyeing of the products. Both cotton fabric (scoured and bleached, supplied courtesy of Nichemtex Textile Industry, Lagos, Nigeria) and reed fibers obtained from the plant *Thaumatococcus danielli* commonly used for carpet construction were dyed by 'exhaust dyeing' technique. Dyeing was done without mordants. Two separate dyebaths were prepared, each containing the same amount of 1 g of the pure dyestuff dispersed in 1000 cm^3 of 96% ethanol contained in a 2l culture flask equipped with a thermometer and a stirrer. One bath was used for the dyeing of the cotton fabric while the other was used for the reed fiber. Both the fabric and the reed, 22 each, was introduced into the dyebath at the dyeing temperature Td of 80°C and dyeing was carried out continuously for 16 h to allow for saturation of both products with the dyestuffs. A high, uniform stirring speed was maintained throughout the period of dyeing to ensure that dyestuffs were in a dispersed state. After dyeing, the specimens were removed and washed in water and dried in a vacuum oven.

Determination of fastness properties. Fastness tests to light were carried out on the two dyed fibers to provide a comparative basis for assessing the stability of the two dyes to UV radiation. The fastness to alkaline wash could only be carried out on the cotton fibers because the ISO (International Standard Organisation) method employed (Schlaeppi 1974) could not be used on the reed fibers.

Fastness to light. Fibers (both cotton and reed) dyed according to the procedure already described were exposed to artificial source of light to induce fading using Shirley Institute's light fastness tester SDL 237. Method used was that described by ISO (Schlaeppi 1974). After testings, samples were assessed on the standard 8-point Gray Scale. Ten specimens were used for the test and the mean value of the results obtained was taken as the fastness rating for the stability of the dyed fiber to light.

Fastness rating to alkaline wash. Fastness to washing test No. 3 specifically designed for cellulosic fabrics was used (Popoola 1996). Fabric specimens measuring 10cm x 4cm each were placed in turn between one piece each of undyed cotton and wool fabrics measuring 5cm x 4cm and stitched around leaving a portion of the specimen uncovered. A beaker containing 100ml solution of 0.5 g of a non-ionic surfactant, Matexil DN (ICI) was heated to the boil. The fabric in this case was allowed to remain in the solution at this temperature for 30 min, with occasional stirring. The specimen was later rinsed in running cold water, the stitch line removed along two sides and the specimens hanged out to dry. After drying, the change in color of the dyed specimens was assessed both in terms of alteration of shade of the uncovered portion of the dyed fabric and the degree of staining of the undyed portion against the standard 5-point Gray scale. As in the case of fastness to light, ten specimens were assessed and the mean value for the 10 specimens was taken as the stability of the dyed fabric to washing.

Results and Discussion

One of the cardinal requirements for a dye or pigment to be used as color additive mostly in textile fibers is that of fastness (or stability) to a number of agencies which tend to militate against their desired properties both in storage and use thus limiting their serviceability. Principally, these agents include UV radiation, water, gas fumes, dry heat (in case of sublimable colorants) etc. In this study, the stabilities of both dyes to UV radiation and alkaline wash were tested on the

Table 1
Fastness rating of cellulosic fibrous constructions to light, dyed with bixin and curcumin dyes

Serial No	Bixin		Curcumin	
	Cotton	Reed	Cotton	Reed
1	4	4	5	4
2	3	3	4	3
3	3	2	5	3
4	4	3	5	3
5	4	3	4	4
6	4	4	4	3
7	4	4	5	4
8	5	4	4	4
9	4	4	4	4
10	3	4	4	4
Mean value	3.8	3.2	4.4	4.0

dyed fibers. Fastness rating to light on a 8-point scale are given in Table 1 while the fastness to washing of cotton fabric only on a scale of 5 is given in Table 2.

From the results presented above, the following trends are clearly discernible.

(a) For the fastness rating to light, the average mean fastness (on an 8-point scale) is comparatively higher for cotton dyed material than the reed fibers for each dye class.

(b) The mean fastness to light rating for curcumin dye is higher than that of bixin though used on the same cellulosic material.

These observations can be interpreted in terms of both the relative substantivities of the two dyes to the substrates as well as the stabilities of the dyestuffs themselves to ultra violet rays. Since a higher stability (fastness) value is recorded in respect of cotton dyed materials in both cases, a preliminary inference can be drawn that the dyestuffs are both more substantive to and exhibit more affinity to cotton fabric than the reed plant. This can be either as a result of stronger force of attraction between the dyes and the cotton fabric and / or higher uptake of the dyes in cotton. It is noted, however, that since both substrates are cellulosic, similar forces of attraction are expected between the two substrates and the dyes. The more acceptable explanation therefore rests on a differential level of dye uptake by the fibers. In a previous

Table 2

Fastness rating of cotton fabric to washing dyed using bixin and curcumin dyes

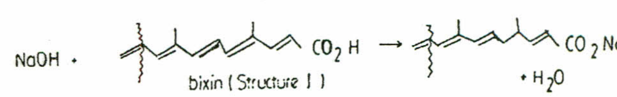
Serial No	Bixin		Curcumin	
	Deg. of staining	Alteration	Deg. of staining	Alteration
1	2	2	3	3
2	2	2	3	3
3	3	2	3	2
4	3	2	3	3
5	2	3	3	4
6	2	3	3	3
7	3	2	3	2
8	3	3	2	3
9	2	2	3	3
10	2	2	2	3
Mean value	2.4	2.0	2.8	2.9

study carried out using similar dye obtained from the plant *Pterocarpus erinaceous* it was shown that the cotton fabric had a crystallinity value of ca. 42.30% while that of the reed fiber was ca. 56.40. Since the reed fiber is more crystalline than the cotton counterpart, it permits lower level of dye absorption than the less crystalline cotton. Indeed, higher level of dye uptake was obtained for cotton fiber either with or without mordant than for the reed fiber using dyestuff from African rosewood plant *(P. erinaceous)*. Another significant factor which controls the fastness ratings of the two dyes on the substrates is the relative structural stabilities of the dyes to ultra violet rays. Generally, dyestuffs with open chain structures though conjugated are less stable to light energy than those having conjugated benzenoid or fused macrocyclic structures. The structures of the two dyes as shown from previous works (Godwin and Weedon 1965) indicated bixin as an extended open chain conjugated system (Structure I) whilst curcumin is a more stable structure having two benzenoid rings at its two terminals which exert considerable resonance stability on the dye structure in addition to that obtained from the conjugated system (Structure II).

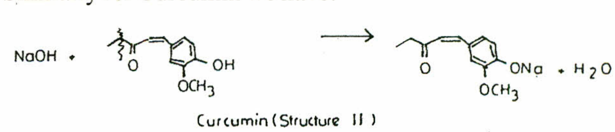
Thus the relative higher stability of curcumin to light energy is the result of its increased stability to ultra violet radiation.

The fastness ratings of the two dyestuffs to alkaline wash both in terms of degree of staining and alteration of shade are just about average considering the fact that this property was assessed on a scale of 5. Like fastness to light, however, slightly higher values were obtained for curcumin which shows being more stable to alkaline wash than bixin. A probable explanation for this lies in the chemistry of the reaction of the dyes with the washing liquor. Bixin has been shown to react well with alkali as 5% caustic soda (NaOH) converts it to a soluble sodium salt which though can be reversed by treatment with mineral acid, (HCl, H_2SO_4 etc) (Popoola 1996). Similarly curcumin is readily converted to its soluble salt in the presence of alkali according to the respective equations 1 and 2 below.

The loss in shade resulting from the alkaline washing of the dyed fabric can be attributed to the removal of the dyestuffs from the dyed fabric as a result of their conversion to soluble



Similarly for Curcumin we have:



products/salts of the dyes and consequently a lowering of the fastness rating to washing observed.

Acknowledgement

The fund for this project was provided by UNESCO-Regional office for Science and Technology in Africa, Nairobi, Kenya under its UNESCO-UNISPAR (University-Industry Partnership Programme) scheme for which the author is deeply grateful.

References

Godwin G, Weedon W 1965 *Chemistry and Biochemistry of Plant Pigments*, Acad. Press pp 245-253.

Holy Bible (The NKJV 1985) *Genesis*, Chap. 6 Vs. 14. Thomas Nelson Pub. p 6.

McLaren K 1983 *The Color Science of Dyes and Pigments.* Adam Higher Publ (Lond.) pp 23.

Popoola A V 1996 Development of indigenous pigments for industrial uses, CAPL, Lagos. pp 17.

Popoola A V, Samaye E O 1977 Dye production for cottage mat weaving and batik printing industries, UNESCO-UNISPAR Project report, pp 5-6.

Schlaeppi F 1974 Color fastness measurements. *Text Chem and Col* **6** 143.