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EFFECT OF FERRIC ALUM MORDANT ON ANNATTO-DYEING OF COTTON FABRIC

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The natural dye annatto was extracted from the plant species *Bixa orellana* and applied to pure white cotton fabric using ferric alum salt [$NH_4Fe(SO_4)_2.12H_2O$] as mordant. The quantitative determination of the amount of dye uptake in the fabric substrate was measured by extracting using stabilised DMF at 140° (dimethylformamide containing free radical inhibitor). The result obtained showed that the amount of dye uptake increased with the percentage strength of the mordant used for the pretreatment of the fabric before dyeing.

Key words: *Bixa orellana*, Ferric alum salt, Annatto-dyeing, Cotton fabric.

Introduction

Annatto is a brick-red vegetable (natural) colouring matter obtained from the ornamental plant *Bixa orellana* found mostly within the tropics. The colouring matter is contained within the pulpy substance surrounding individual seeds of the plant. Its colouring potential is attributable to the constituent carotenoid bixin which comprises between 70-80% of the total pigment mass surrounding each annatto seed. The remainder of the pigment mass consists mainly of an uncharacterised yellow pigment, the tinctorial strength of which is very much less than that of bixin together with much smaller quantities of related compounds including norbixin [1].

Annatto is a colouring matter with wide ranging applications in food colouring such as dairy products and in cosmetic industry. The use of annatto as a textile dye has not been given much attention and the present study is a systematic attempt to extend its use to textile substrate [1].

Sorption of dyes is an important step in the dyeing of textiles and this is usually studied by one of the following methods [2]. (i) finding the differences in concentrations of solution before and after the sorption, (ii) extracting the dye from the substrate with a solvent and (iii) dissolving both the dye and the substrate. The method used in this study is by extracting the non-ionic dye from the textile using stabilised dimethylformamide and then determining the amount in the resulting solution following the method of Kissa [3].

Usually, the sample is extracted several times with a small amount of a solvent and the successive extracts collected in a volumetric flask. Single step extraction for a prolonged time at the working temperature of 140° exposes the entire dye extract to the danger of decomposition. This danger is eliminated in the multiple extraction. Since the last requiring a longer time involves only a small fraction of the dye, the danger of decomposition is most minimised. In addition to the danger of dye decomposition of prolonged single extraction, partition of the dye between fibre and solvent requires that a single extraction cannot remove the dye as completely from the fibre as can repeated extractions, although the amount of the dye retained in the fibre may not always be significant.

Experimental

Materials and preparations. Annatto seeds were collected from the dried fruits of *Bixa orellana*. The seeds were ground in laboratory mortars into fine particles. The pulverised seeds were further ground into finer powder in a mechanical grinder. The dyestuff was extracted from the fine particles using continuous Soxhlet extraction method [4]. About 10gm of the particles was packed into a timble and was extracted continuously for 6 hrs. using distilled ethanol as the extractant following preliminary extraction with hexane to remove oil. At the end of the extraction, the extracting liquid was virtually colourless, indicating that the seeds have been stripped off its colourant. The solution obtained was transferred to a distillation unit to concentrate the dyestuff by distilling off the ethanol. The concentrated annatto dye was finally dried in the oven set at 105° for 12 hrs.

Dyeing of the cotton fabric. The dyed cotton fabric used was made by the conventional dyeing procedures. Fabrics of known dye content were prepared by exhaust dyeing, a known amount of scoured cotton fabric in a dyebath with a known dye concentration following pretreatments in 10 and 20% ferric alum concentrations as mordant respectively. After dyeing, the fabrics were washed free of surface held dye particles in 1% solution of non-ionic surfactant Lissapol N, rinsed with distilled water and dried. Dyeing of the unmordanted fabric was also carried out as described. The dyebath concentration was determined both before and after dyeing spectrophotometrically; the difference between the two results

representing the amount of dyes quantitatively transferred into the fabrics.

Varying concentrations of the dye solution had been earlier prepared and their absorbance studied at the wavelength of the maximum absorption of 495 nm determined for the dye and a calibration curve used for the determination of the amount of dye- uptake established.

Determination of the fastness properties of the dyed fabric. Two fastness tests were carried out on the dyed fabrics. These are fastness to light and washing. Test methods used were those of International Organisation for Standardisation (ISO) [5,6].

Fastness to light. Fastness testing to light was carried out by exposing the deep dyed fabric to 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 3-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. Fabric specimens measuring 10 x 4 cm each were placed in turn between one piece each of undyed cotton and wool fabrics measuring 5 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 weight of 2gm of the fabric used. The fabric in each case was allowed to remain in the solution at this temperature for 30 mins whilst stirring was carried out *occasionally*. The specimen was later rinsed in running cold water for about 10 mins. 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, 10 specimens were used for the test and the mean value of the results obtained taken as the fastness rating for the test.

Extraction and determination of amount of dye-uptake. 0.1 Gram dyed sample was weighed accurately in each case into a small conical flask and was covered with about 5 cm^3 of the extractant. The flask was dipped partially in an oil bath set at 140° ± 0.1° being shaken continuously. Three different extractions of between 2-30 mins durations were carried out. This ensured virtually complete stripping of the dye from the fabrics as the dyed fabrics appeared colourless after the extractions. The extracts were collected together in a 100 cm^3 volumetric flask, the contents of the volumetric flask were rinsed with solvent from wash bottle into the volumetric flask. The extract was allowed to equilibrate at room temperature and made up to mark using the extracting solvent. The concentration of the dye was then measured using Griffith's Spectro-20 meter.

Results and Discussion

Extraction of dye from fibre is essentially the reverse of the dyeing operation. Stripping of dyes from fibres (desorption) involves the transfer of dye from the fibre's matrix into the solution phase. It is known that the rate of dyeing is temperature controlled, increasing sharply with increasing dyeing temperature. The factors that affect the dyeing of cotton are equally important during the stripping of dyes from it. In other words, the rate of desorption or extraction of the dye from the cotton fabric is found to increase with increase in solvent temperature. Beside the thermal method of enhancing the dye desorption from a fibre, other methods based on the use of plasticisers to lower the effective glass transition temperature of the polymer have been discussed.

The result of the amount of dye extract from the untreated and premordanted annatto-dyed cotton fabric are shown in Table 1. The effectiveness of the stabilised DMF as an extractant for this dye at 140° can be seen from the percentages of the dye removed from the fabric (shown in parenthesis) during the 3-stage extraction process. In two of the three samples, i.e. in the mordanted specimens, 99% of the amount of dye in the fabric was quantitatively removed by the extractant during the 2-mins. duration of the first extraction whilst the specimens were virtually completely stripped off the dye at the second extraction i.e. within 5 mins. of the extraction time. In case of the untreated fabric, the specimen was completely stripped off its dyestuff within the first 2 mins. of the extraction indicating that the mordant is also playing a vital role in fixing the dyestuff in addition to the increase in dye uptake which it caused. Thus in all cases, further extraction was carried out to ensure complete stripping of the dye.

The role of ferric alum mordant. The role of the mordant in the dye uptake was assessed by comparing the amount of dye taken up by the mordanted fabric with the fabric dyed without prior treatment in the mordant. The increase in dye uptake arising from mordanting of the cotton using 10 and 20% solutions of ferric alum is shown in Table 2. The percentage increase in each case has also been calculated. It is seen that there is an increase in the amount of dye uptake by an average of 9.12% for the fabric pretreated in 10% ferric alum mordant solution to an average of 21.82% for the 20% mordant treated fabric.

TABLE 1. EFFECT OF ALUM MORDANT ON THE DYE UPTAKE OF ANNATTO-DYED COTTON FABRIC (mg/gm OF FABRIC).

	Cumulative timing (mins)	Untreated	10% Alum	20% Alum
1st Extract	2	0.475 (100)	0.515 (99)	0.574 (98.6)
2nd Extract	5	0.475 (100)	0.520 (100)	0.580 (99.7)
3rd Extract	10	0.475 (100)	0.520 (100)	0.582 (100)

Mordants such as ferric alum or synthetic ones such as Ketanol O and Tanninol BM on treatment with natural fibres such as cotton, jute etc., are capable of increasing both the level of dye uptake and fixation of the dye. This is usually achieved by forming complexes with the dyestuff, open up the fibre's structure to enhance both the uptake and fixation of the dye.

Fastness properties of the dyed fabric. The results of the fastness tests to the two agencies i.e. light and washing of the annatto-dyed cotton fabric are shown in Table 3. From the results, it can be seen that the fabric showed poor fastness to light rating of 3 on a scale of 8 but appreciably better wash fastness of 3.6 rating on a scale of 5. It is to be recommended therefore that if the pigment were to be used on textiles, interior furnishings rather than wearing or outer apparels are to be dyed using the dyestuff.

TABLE 2. PERCENTAGE INCREASE IN DYE UPTAKE OF ANNATTO-DYED COTTON AS A RESULT OF MORDANTING USING A FERRIC ALUM SALT. DYE UPTAKE IN CONTROL (UNMORDANTED) FABRIC 0.475 mg/gm OF FABRIC.

		Increase in mg/gm	% Increase
10% Alum	1st Extract	0.040	8.42
	2nd Extract	0.045	9.47 Average = 9.12
	3rd Extract	0.045	9.47
20% Alum	1st Extract	0.099	20.84
	2nd Extract	0.105	22.10 Average=21.82
	3rd Extract	0.107	22.52

TABLE 3. FASTNESS RATINGS OF ANNATTO-DYED COTTON FABRIC (FOR INTERIOR FURNISHING).

Specimens	Light	Washing
1	4	3-4
2	3	4
3	3	3
4	3	4
5	2	3
6	3	3-4
7	4	3
8	2	4
9	3	4
10	3	4
Mean ratings	3.0	3.6

Conclusion

The treatment of cotton fabric with 10 and 20% solutions of $(NH_4Fe(SO_4)_2.\ 12H_2O)$ ferric alum respectively led to an average of 9.12 and 21.82% in the amount of equilibrium dye-uptake in the fabric. This increase in dye uptake is attributed to the opening up of the fibre's structure by the ferric alum mordant to facilitate both dye penetration and fixation.

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