Tropical Products Institute

L62

Black dyes for coir fibre 3 Practical considerations



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Summaries

SUMMARY

Black dyes for coir fibre. Part 3: Practical considerations

Earlier trials on Synacril Black A, Benzamin Black DS 167% and Suprexcel Black VY — three possible substitute dyes for Chlorazol Black E — showed the effect of variations in dyebath conditions on the amount of dye deposited on coir (Canning, A. J., Jarman, C. G. and Mykoluk, S. M. (1979) Black dyes for coir fibre. Part 2. Evaluation of selected dyes. *Report of the Tropical Products Institute*, L52). Further trials, in which both different concentrations of dye in the bath, and a standing bath were used, have now been carried out to obtain additional data on the exhaustion characteristics of the three dyes. Additionally, the effect of variations in dyebath conditions on the quality of colour, and the amount of each of the three dyes that needs to be deposited on to the fibre to produce an acceptable black, have been assessed.

With all three dyes, dyebath exhaustion was approached more slowly with increased dyebath concentration. As a consequence, when additional dye is used, dyeing time must be increased to obtain the most efficient use of dye. Since the proportion of dye that exhausts from the bath is also reduced, particularly with the higher dyebath concentrations or the shorter dyeing times, increasingly large additions of dye are required in order to intensify the visual depth of shade. With low dyebath concentrations of the two direct dyes (Benzamin Black and Suprexcel Black), allowing the liquor to cool during dyeing could reduce heating costs since the exhaustion obtained after 1.5 hours at 95°C followed by cooling was similar to that obtained after 6 hours at 95°C. However, with deeper shades the opportunity for reducing heating costs by reducing dyeing time at 95°C and then cooling is less. The inclusion of sodium carbonate in the dyebath could also result in savings on dyestuff.

When using a standing bath technique with Benzamin Black, significantly different exhaustion/time relationships were obtained with each batch of fibre. As a consequence there would be considerable practical difficulties in using this dye with this technique. On the other hand, the exhaustion of Suprexcel Black was only slightly affected by the use of replenished liquors and a standing bath technique could reduce costs. Because of its almost complete exhaustion, there would be no advantage in using a standing bath with the basic dye Synacril Black.

All the dyes gave better light-fastness properties when they were concentrated at the periphery of the fibre rather than when they penetrated more deeply: however, staining of adjacent fabrics was greater. The distribution of the dye within the fibre did not affect the visual colour.

All three dyes should produce black colours which are technically similar to those of commercial samples of black coir. The amounts which need to be deposited on the fibre to produce an acceptable black are estimated as approximately:

Benzamin Black DS 167%: 19 g per kilogram of fibre Suprexcel Black VY: 22 g per kilogram of fibre Synacril Black A: 10 g per kilogram of fibre

Fastness properties of the blacks vary with the dye and the technique used to apply it.

Information on the development of optimum dyeing conditions is appended.

RÉSUMÉ

Colorants noirs pour fibres de coco. 3 ème partie: Remarques pratiques

Des essais antérieurs sur le Synacril Black A, le Benzamin Black DS 167% et le Suprexcel Black VY — trois colorants pouvant peut-être remplacer le Chlorazol Black E — ont montré l'effet de variations dans les conditions du bain de teinture sur la quantité de colorant fixée sur les fibres de coco (Canning A. J., Jarman C. G. et Mykoluk S. M., (1979). Colorants noirs pour fibres de coco. 2 ème partie. Evaluation de colorants choisis. *Rapport de l'Institut de Produits Tropicaux*, L 52). D'autres essais dans lesquels on a utilisé des concentrations différentes de colorant dans le bain et un bain stationnaire ont été effectués pour obtenir des données supplémentaires sur les caractéristiques d'épuisement des trois colorants. De plus, l'effet de variations des conditions du bain de teinture sur la qualité de la couleur et la quantité de chacun des trois colorants qui doit être fixée sur les fibres pour produire un noir acceptable ont été évalués.

Avec les trois colorants, le bain de teinture a progressé vers l'épuisement plus lentement avec une concentration accrue du bain de teinture. Comme conséquence, lorsqu'un supplément de colorant est utilisé, le temps de teinture doit être augmenté pour obtenir l'utilisation la plus efficace du colorant. Etant donné que la proportion de colorant qui part du bain est également réduite, en particulier avec les concentrations plus élevées du bain de teinture ou les temps de teinture plus courts, des additions croissantes de colorant sont nécessaires pour intensifier la profondeur visuelle de la teinte. Avec des concentrations basses des deux colorants directs (Benzamin Black et Suprexcel Black), laisser refroidir le liquide pendant la teinture pourrait réduire les frais de chauffage étant donné que l'épuisement obtenu après 1, 5 heures à 95°C. Cependant, avec des teintes plus profondes, il est moins possible de réduire les frais de chauffage en réduisant le temps de teinture à 95°C et en refroidissant ensuite. L'inclusion de carbonate de sodium dans le bain de teinture pourrait également entrainer des économies de matière colorante.

En utilisant une technique de bain stationnaire avec le Benzamin Black, des relations épuisement/temps significativement différentes ont été obtenues pour chaque charge de fibres. Comme conséquence, il y aurait des difficultés pratiques considérables à utiliser ce colorant par cette technique. Par ailleurs, l'épuisement du Suprexcel Black n'a été que légèrement affecté par l'utilisation de liquides rechargés et une technique de bain stationnaire pourrait réduire les frais. A cause de son épuisement presque complet, il n'y aurait pas d'avantage à utiliser un bain stationnaire avec le colorant basique Synacril Black.

Tous les colorants ont donné de meilleures propriétés de stabilité à la lumière lorsqu'ils étaient concentrés à la périphérie de la fibre plutôt que lorsqu'il avaient pénétré plus profondément: cependant la coloration du tissu adjacent était plus grande. La distribution du colorant à l'intérieur de la fibre n'a pas influencé la couleur visuelle.

Les trois colorants devraient produire des couleurs qui sont techniquement analogues à celles d'échantillons commerciaux de fibres de coco noires. Les quantités qui doivent être fixées sur les fibres pour produire un noir acceptable sont estimées approximativement de la façon suivante: Benzamin Black DS 167%: 19 g par kilogramme de fibre Suprexcel Black VY: 22 g par kilogramme de fibre Synacril Black A: 10 g par kilogramme de fibre

Les propriétés de stabilité des noirs varient avec le colorant et la technique utilisée pour l'appliquer.

Les informations sur le développement des conditions optimum de teinture sont annexées.

RESUMEN

Colorantes negros para la fibra de coco. Parte 3ª: Consideraciones prácticas

Durante ensayos iniciales realizados con Synacril Black A, Benzamin Black DS 167% y Suprexcel Black VY (tres posibles colorantes alternativos al Chlorazol Black E) se manifestaron los efectos de las variaciones existentes en las condiciones del baño de tinte sobre la cantidad de colorante depositada en el coco (Canning, A. J., Jarman C. G. y Mykoluk, S. M. (1979) "Colorantes negros para la fibra de coco". Parte 2^a. "Evaluación de colorantes seleccionados". *Informe del Instituto de Productos Tropicales*, L52). En la actualidad se han llevado a cabo más ensayos ulteriores en los cuales se han usado diferentes concentraciones de colorantes en el baño y un baño estático, para obtener datos adicionales en torno a las características de agotamiento de los tres colorantes. Además de esto, se ha evaluado el efecto de las variaciones en las condiciones del baño de tinte sobre la calidad del color, y la cantidad de cada uno de los tres colorantes que ha de ser depositada en la fibra para producir un negro aceptable.

En los tres tipos de colorantes, el agotamiento del baño de tinte se produjo más lentamente con una concentración gradual del baño de aceite. Como resultado de esto, cuando se usa un colorante adicional habrá de incrementarse el período de teñido para obtener el uso más eficaz del tinte. Como la proporción del colorante salido del baño es también reducida, particularmente con las concentraciones de baño de tinte elevadas o los períodos de teñido más cortos, son requeridas adiciones cada vez mayores de colorante con el fin de intensificar el efecto visual de matiz. Con las concentraciones bajas de baño de tinte de los dos colorantes directos (Benzamin Black y Suprexcel Black), y dejando que el licor se enfríe durante el teñido, podrían reducirse los gastos, ya que el agotamiento registrado después de 1, 5 horas a 95°C sequido de enfriamiento fue parecido al registrado después de 6 horas a 95°C. Sin embargo, con los matices más intensos resulta menor la oportunidad de reducir los gastos de calentamiento mediante la reducción del período de teñido a 95°C seguido de un enfriamiento. La adición de carbonato de sodio en el baño de tinte también podría contribuir a ahorrar colorantes.

Cuando se usó el método de baño de teñido estático con Benzamin Black, se obtuvieron relaciones de agotamiento/tiempo significativamente distintas con cada grupo de fibras. Como resultado de esto, se presentarían dificultades prácticas considerables si se usara este tipo de colorante con este método técnico. Por otra parte, el agotamiento del Suprexcel Black se vio solamente ligeramente afectado por el uso de licores intensificados y la técnica del baño de teñido estático podría reducir los gastos. Debido a su agotamiento casi completo, no se obtendrían ventajas algunas mediante el uso de un baño estático con el colorante básico del Synacril Black.

Todos los colorantes presentaron mejores propiedades de resistencia a la luz cuando fueron concentrados en la periferia de la fibra, en lugar de cuando fueron introducidos más profundamente. Sin embargo, la coloración de los tejidos adyacentes fue más intensa. La distribución del colorante dentro de la fibra no afectó el color visual.

Los tres colorantes deberían producir colores negros técnicamente parecidos a las muestras comerciales del coco negro. Las cantidades precisadas que han de ser

depositadas en la fibra para obtener un negro aceptable se calculan aproximadamente en:

Benzamin Black DS 167%: 19 g por kg de fibra Suprexcel Black VY: 22 g por kg de fibra Synacril Black A: 10 g por kg de fibra

Las propiedades de resistencia a la luz de los negros varían con el colorante y el método usado para aplicarlo.

Se adjunta información sobre el desarrollo de condiciones óptimas para el teñido.

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Black dyes for coir fibre 3: Practical considerations

INTRODUCTION

Bristle fibre dyed black to simulate animal hair commands a higher price than the natural undyed fibre. Dyeing is sometimes carried out at the fibre mills but more usually in independent hackling and dyeing plants (Jarman and Jayasundera, 1975).

When the manufacture of a commonly used black dye for coir was terminated for health reasons, the Tropical Products Institute screened twenty possible substitutes and, from these, selected four which appeared sufficiently promising to warrant further investigation (Jarman and Canning, 1974). Of these, three could be used to augment the limited supplies of black dyes then available and their fastness properties and exhaustion characteristics were compared (Canning *et al.*, 1979). Of the three, Synacril Black A, a basic dye, had particular advantages. It was cheap to use and required no additional chemicals (the use of additional chemicals not only increases the cost of dyeing but causes corrosion to the galvanised iron dye vats used in Sri Lanka). However, its use could not be recommended unreservedly since it had poorer light-fastness properties than the other dyes investigated.

It was also shown (Canning *et al.*, 1979) that variations in dyebath conditions considerably affect the amount of dye exhausted on to the fibre and hence the colour. Since optimum dyeing conditions vary with changes in the relative costs of dyes, chemicals, labour and energy, it is necessary to adjust the dyebath conditions in order to take account of these.

A technique commonly used in Sri Lanka for applying dyes is the 'standing bath'. This can save costs since the used dye liquor is replenished with dye and chemicals between dyeings rather than being discarded. However, impurities in the bath could affect the exhaustion characteristics of the dyes when dyeing the second and subsequent batches of fibre.

The purpose of this report is to provide, in conjunction with that of Canning *et al.* (1979), a guide to the quantity of dye needed to produce an acceptable black from any of the three suitable dyes. To this end, data have been obtained on the effect on dyebath exhaustion both of varying the concentration of dye in the bath, and of using a standing bath. The characteristics of acceptable blacks have also been examined, and related to the optical absorption of the dyes in order that the amount of each required to produce an acceptable black could be estimated. To help dyers relate dyebath exhaustion to the amount of dye deposited on the fibre, a chart is provided in Appendix 1 (*see* Figure 7).

Particularly with direct dyes, the optimum dyeing conditions are a critical balance between technical and economic considerations. The duration of the dyeing process and the amount of sodium chloride to use are the main factors affected by these considerations and guidance on these aspects of dyeing is given in the form of a worked example in Appendix 2.

LABORATORY TRIALS

A Materials and methods

Four types of coir fibre, all obtained from either Sydenham and Corlett Ltd or Havleys Ltd, were used in these trials:

- (i) Bleached hackled bristle fibre:
- (ii) Bleached unhackled bristle fibre:
- (iii) Unbleached hackled bristle fibre; and
- (iv) Samples dyed during the preliminary trials (Jarman and Canning, 1974).

No distinction was made between hackled and unhackled fibre since hackling would not affect its chemical nature. However, fibre bleached with sulphur dioxide prior to dyeing has been shown to adsorb dyes differently from unbleached fibre (Canning et al., 1979).

Dyestuffs used in the trials were:

Dye	Manufacturer	Colour Index (CI) Generic Name	3
Synacril Black A	ICI	CI Basic Black*	
Benzamin Black DS 167%	Bayer	CI Direct Black 17	
Suprexcel Black VY	L.B. Holliday	CI Direct Black*	

The dyes were obtained as samples directly from their manufacturers. Additional chemicals used to assist dyeing, were: Analytical Reagent grade (AR) sodium chloride; AR anhydrous sodium carbonate; and Lissapol N, an ICI non-ionic wetting and penetrating agent (this has now been replaced by Synperonic BD).

Dyeing was carried out at 95° C using a liquor to fibre ratio of 20:1 throughout. Prior to dyeing at 95°C there was an initial warm up period of half an hour during which the dyebath temperature was raised steadily from 50°C to 95°C. All dye liquors contained a wetting and penetrating agent: Lissapol N (1 gl⁻¹). Variations in the dyebath conditions are recorded under their relevant trials.

The proportion of dye adsorbed on to the fibre (dyebath exhaustion) was determined using a spectrophotometer. Measured samples of liquor, both used and unused, were diluted with a sufficient known amount of distilled water to give solutions containing concentrations of dyestuff within a range over which it had been determined that Beer's Law was obeyed, and that had absorbances[†] within the range of the instrument used when measured with a path length of 1 cm at the wavelength of maximum absorption. Absorbances of the resulting solutions, measured under the above conditions, were recorded and dyebath exhaustions were calculated as follows:

$$\mathsf{E} = 100 \left[\frac{1 - \frac{\mathsf{A}_1 \ \mathsf{D}_1}{\mathsf{A}_2 \ \mathsf{D}_2}}{\mathsf{A}_2 \ \mathsf{D}_2} \right] \text{ per cent}$$

where E = dyebath exhaustion

- A, = absorbance of diluted used liquor
- $A_2 =$ absorbance of diluted unused liquor
- D_1^2 = dilution factor of used liquor D_2 = dilution factor of unused liquor

Wavelengths of maximum absorption were 615 nm for Synacril Black A, 567 nm for Benzamin Black DS 167% and 575 nm for the Suprexcel Black VY dye.

^{*}Dye with this name not listed in Colour Index, (Society of Dyers and Colourists/American Association of Textile Chemists and Colourists, 1971), nor in its Additions and Amendments up to Number 40 (July 1981). Suprexcel Black VY is not currently available and Synacril Black A has been replaced by a close equivalent: Synacril Black AN

[†]Absorbance is: log10/Intensity of incident light \and for solutions which obey Beer's Law is proportional Intensity of transmitted light,

to concentration when the path length is constant

Light-fastness was assessed by the method described in British Standard BS 1006: 1971 *Methods for the determination of the colour fastness of textiles to light and weathering,* but the tests were terminated after fading had been produced on standard 6. Natural daylight in London was used as the fading source and results are expressed on the British Standard 1–8 scale on which 8 represents the highest degree of light-fastness.

Water-fastness was assessed by the method described in British Standard BS 2681: 1961 *Colour fastness to water*, using cotton and wool cloths obtained from the Society of Dyers and Colourists* to assess staining (the standard does not specify which cloths should be used with coir). Results are expressed using the two standard 1-5 scales, for loss of colour and for staining on fabric, on which 5 represents the least loss of colour on the former and the lightest stain on the latter.

Penetration of dye into the fibres was assessed from transverse cross-sections prepared by the grinding method (Lomas and Simmens, 1970). Sections were viewed through a microscope, and for each sample an estimate was made of the mean number of cells from the periphery of the fibre that were penetrated by dye. The mean represents several counts made on each of 25–50 fibres.

Colours of dyed coir were compared using reflectance data obtained using a Zeiss 'Elrepho' photoelectric photometer. Fibre samples examined in the photometer were made up into pads, about 0.5 cm thick to ensure that they were opaque, measuring about 5 cm² in area. In order to ensure that reflectance data on the pads were comparable, fibres were orientated in the same direction both in the pads, and in the photometer. Measurements of reflectance were made on each pad at either three or four of four wavelengths nominally described as 450 nm, 530 nm, 550 nm and 600 nm, which covered the whole of the visible spectrum. The percentage of incident light of a specified wavelength reflected was measured, with 100 per cent taken as that reflected by a magnesium oxide standard.

The relative strengths of the dyes were determined from absorption spectra measured on a Perkin Elmer Spectrophotometer. Solutions of equal strength were prepared, one for each dye, at a concentration of dye sufficiently low for light absorption to be both within the range of the spectrophotometer, and linearly proportional to dye concentration. Absorption measurements were made over a range of wavelengths for each of the three solutions. The results, in conjunction with reflectance data collected from dyed samples, were used to estimate the amount of each dye that would give equivalent, saturated black colours on 1 kg of coir fibre.

B1 Studies on dyebath exhaustion: Relationship between dyebath concentration⁺ and exhaustion

Experimental

Trials were carried out to determine differences between:

- (i) exhaustion-time relationships for different dyebath concentrations of the same dye; and
- (ii) final exhaustions with different dyebath concentrations of the same dye applied from a liquor which was allowed to cool.

The exhaustion-time relationship was determined for all three dyes. Dyeings were carried out at concentrations of 0.75 gl⁻¹, 1 gl⁻¹ and 1.5 gl⁻¹ Synacril Black in the bath on both bleached and unbleached coir; and of 1.5 gl⁻¹, 2 gl⁻¹ and 3 gl⁻¹ for both Benzamin Black and Suprexcel Black on bleached coir only. The two direct dyes were applied from liquors containing 10 gl⁻¹ sodium chloride and 1 gl⁻¹

^{*}Society of Dyers and Colourists, PO Box 244, Perkin House, 82 Grattan Road, Bradford, West Yorkshire, BD1 2JB, England

[†]Throughout this report the amount of dye used in the trials is expressed as concentration (gl^{-1}) in the dyebath. At a liquor to fibre ratio of 20:1 each 0.5 gl^{-1} represents 1 per cent on the weight of fibre

Lissapol N only. Samples were dyed separately, for increasing periods of time, and the degrees of exhaustion of the used liquors were determined.

Since there is little advantage in applying Synacril Black from a cooling liquor (Canning *et al.*, 1979), trials with cooling liquors were confined to the Benzamin and Suprexcel dyes. The dyes were applied only to unbleached coir using between 1 gl^{-1} and 2.5 gl^{-1} Benzamin Black DS 167% in the bath, and between 1.5 gl^{-1} and 3 gl^{-1} Suprexcel Black VY. Liquors used with Benzamin Black contained 5 gl^{-1} sodium chloride in addition to the Lissapol N. For Suprexcel Black, which exhausts less readily than Benzamin Black, the sodium chloride concentration was increased to 10 gl^{-1} .

Samples were dyed for 1.5 hours at 95°C. The baths were then left to cool, without removing the fibre, for a period of 16 hours, after which the temperature was about 33°C. The degree of exhaustion of each bath was then determined. The trial was then repeated using identical liquors to which 2 per cent on fibre (1 gl⁻¹) of sodium carbonate had been added.

Results and discussions

The relationships between dyebath exhaustion and dyeing time (excluding the initial warm up period) are shown in Figures 1 and 2.

Relationships between dyebath exhaustion and concentration for the Benzamin and the Suprexcel dyes applied from cooling liquors are shown in Figure 3.

From Figures 1 and 2 it can be seen that the exhaustion characteristics of the three dyes differ. For example, the basic Synacril dye exhausts almost completely from the bath within an hour whereas the direct dyes are not totally exhausted even after 6 hours. Therefore, if dyeing is terminated after (say) 2 hours and the dyebath liquors discarded, considerably more of the direct Benzamin Black and Suprexcel Black dyes would be wasted than with the basic dye Synacril Black. Of the two direct dyes, Benzamin is more fully exhausted throughout the whole dyeing period.

From Figures 1, 2 and 3 it can be seen that at any particular time the proportion of dye exhausted from the bath decreases with increase in the initial dye concentration. In practice this means that if extra dye is added to produce deeper colours, a greater proportion of dye will be wasted when the used dyebath liquors are discarded; the amount wasted will depend on dyeing technique. For dyeings at 95°C without cooling (*see* Figures 1 and 2), in general this effect appears to be most marked for higher initial concentrations and accentuated with shorter dyeing times. With cooled liquors, however, (*see* Figure 3) this effect was least marked at higher initial concentrations.

Figure 3 shows the effect of allowing the dye liquor to cool after a relatively short period at 95°C (1.5 hours) before removing the fibre. Although few trials were made, an observation of practical value is that sodium carbonate added to the dye liquor increases the proportion of dye exhausted on to the fibre, and so could be useful in reducing costs since dyestuff (which is relatively expensive compared to sodium carbonate) could be saved. Without sodium carbonate, exhaustions from the cooled liquor were generally lower than when dyeing was carried out at 95°C for 6 hours. However, savings in fuel costs could be of importance and in further work this aspect should be investigated more fully.

Coir is a coarse lignocellulosic fibre with a relatively low surface area to mass ratio, and it has impurities intermingled with the cellulose. Because of this it would be expected that surface saturation could occur, especially at high dyebath concentrations, and that exhaustion would be highly dependent on penetration. It can be seen from the results that maximum dyebath exhaustion is approached relatively slowly for high initial dyebath concentrations, and it is possible that a slow rate of penetration into the fibres is responsible for this.

Figure 1

Exhaustion of Synacril Black A at 95° C on to coir from a neutral liquor at a liquor to fibre ratio of 20:1 with 1gl-¹ Lissapol N in the bath



Concentration of dye:

 $1 \blacktriangle = 0.75 \text{gl}^{-1}$ $2 \spadesuit = 1 \text{gl}^{-1}$ $3 \blacksquare = 1.5 \text{gl}^{-1}$

Figure 2

Exhaustion of different concentrations of Benzamin Black DS 167% and Suprexcel Black VY on to bleached coir from liquors at a liquor to fibre ratio of 20:1 and containing 10gl-1 sodium chloride and 1gl-1 Lissapol N



Figure 3

Exhaustion of different concentrations of Benzamin Black DS 167% and Suprexel Black VY on to unbleached coir after 1.5 hours at 95°C followed by 16 hours cooling



Conclusions

Percentage dyebath exhaustion, which will govern the depth of black produced, varies considerably with dyeing technique. It is difficult, therefore, to decide the optimum concentration of dye for the production of an acceptable black. However, these trials, along with those reported by Canning *et al.* (1979), will help dyers choose the method which best suits their particular circumstances.

B2 Studies on dyebath exhaustion: Trials with a standing bath

Experimental

Trials were carried out using mainly Benzamin Black DS 167%, but limited trials on Suprexcel Black VY were included. Synacril Black A was not included because there is no advantage in using a standing bath with this dye: it exhausts so completely and requires so few auxiliaries that no worthwhile savings could be made (Canning *et al.*, 1979).

Three separate trials were carried out:

- (i) comparison of the exhaustions of both Benzamin Black DS 167% and Suprexcel Black VY on bleached coir from liquors containing no sodium carbonate;
- (ii) comparison of the exhaustions on bleached and unbleached fibre using Benzamin Black DS 167% with sodium carbonate in the liquor; and
- (iii) as for (ii) but without sodium carbonate in the liquor.

The effect of sodium carbonate on the exhaustion of Benzamin Black DS 167% was assessed by comparing the results from trials (ii) and (iii). Care was taken to ensure that these last two trials were comparable by making certain that the heating periods involved identical conditions of time and temperature.

Liquors containing 2 gl^{-1} dyestuff (equivalent to 4 per cent on weight of fibre at liquor ratio 20:1), 10 gl^{-1} sodium chloride, and 1 gl^{-1} Lissapol N were used throughout the trials. For trials with sodium carbonate, 1 gl^{-1} (2 per cent on weight of fibre) of sodium carbonate was added to the liquors.

Each liquor was used to dye, consecutively, three identical samples of coir. Each sample was dyed in a flask under reflux for 3 hours at the boil (to keep the liquor agitated after the initial 0.5 hour heating) then removed from the hot liquor. Liquors were cooled and replenished with dye, chemicals, and water between samples of fibre. Vat 1, Vat 2, and Vat 3 are the three terms used to respectively identify the three consecutive dyeings from one liquor.

- i.e. Vat 1: original liquor
 - Vat 2: once used liquor
 - Vat 3: twice used liquor

Amounts of dye and chemicals needed to replenish the baths were calculated on the assumptions that sodium chloride, sodium carbonate and Lissapol N were not consumed in dyeing, and that heating under reflux prevented loss of water through evaporation.

The quantity of dye in the residual liquor was calculated from its concentration as determined with the spectrophotometer. The volume of each cooled used liquor was measured and an appropriate amount of dye was then added to replace that removed from the bath by the fibre and its adhering liquor. The liquor was then restored to its original volume by adding a solution containing the appropriate concentrations of chemicals, but no dye. This addition replaced chemicals and water lost with liquor adhering to the removed fibre. The replenished liquor was boiled briefly, to dissolve the dye, then cooled again before use.

For trials (i) and (ii), determinations of dyebath exhaustion were made throughout the dyeing period using small, measured, cooled, and suitably diluted samples taken from the liquor; for trial (iii), only the final exhaustion of each vat after dyeing for 3 hours at the boil was determined.

Results and discussions

Dyebath exhaustions for each of the three vats throughout the 3-hour dyeing period at the boil are shown in Figures 4 and 5. Figure 4 indicates the exhaustions of both the Benzamin dye and the Suprexcel dye, on to bleached coir in the absence of sodium carbonate. Figure 5 shows the exhaustions of only the Benzamin dye from liquors containing sodium carbonate on both bleached (*see* Figure 5(a)), and unbleached (*see* Figure 5(b)), coir. Final exhaustions for each of the three vats of the Benzamin dye applied without sodium carbonate to both bleached and unbleached fibre are given in Table 1.

Table 1

Exhaustion after 180 minutes boiling of Benzamin Black DS 167% at 2 gl⁻¹ in a standing bath containing 1 gl⁻¹ Lissapol N and 10 gl⁻¹ sodium chloride

	Bleached coir %	Unbleached coir %	
Vat 1	86.5	79.5	-
Vat 2	85.0	74.0	
Vat 3	77.5	70.5	

Figure 4

Exhaustion of Benzamin Black DS 167% at 2gl-1 and Suprexcel Black VY at 2gl-1 on to bleached coir in a standing bath containing 1gl-1 Lissapol N and 10gl-1 sodium chloride



Vat 3

The results in Figure 4 for bleached coir show that differences in the exhaustion from the three vats occur. With the Suprexcel black dye the differences are slight. Therefore, if a 3-hour process is suitable, when using dyes such as Suprexcel, up to three batches of fibre, possibly more, could be dyed from the same liquor without adversely affecting the colour. For example, with a 2 gl⁻¹ concentration of dye in the bath only about half the dye in the vat exhausts on to the fibre after 3 hours. Thus three batches of fibre dyed from one *standing bath* would require only about two-thirds of the dye needed to prepare three *fresh dyebaths*. Amounts of other chemicals needed would also be reduced.

The exhaustion of the Benzamin dye was affected similarly in all experiments (compare Figures 4, 5(a) and 5(b)). In each case the quantity of dye exhausted decreased with each subsequent vat except for a short period for Vat 2.

The results also confirm the findings (Canning *et al.*, 1979) that dyebath exhaustion was greater for bleached fibre than for unbleached (*see* Table 1) but that the effect is less marked (compare Figures 5(a) and 5(b)) when sodium carbonate is included in the liquor.

From a comparison of Table 1 with Figure 5 it can be seen that addition of sodium carbonate reduces final dyebath exhaustion for both bleached and unbleached coir. The results in Figure 4 differ from those shown in Table 1 but it is probable that these differences arose from inadequate control of the heating period.

Figure 5

Exhaustion of Benzamin Black DS 167% at 2gl-1 in a standing bath containing 1gl-1 Lissapol N, 10gl-1 sodium chloride and 1gl-1 sodium carbonate



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For dyes such as Benzamin Black, the use of a standing bath will not be greatly advantageous. Since the degree of exhaustion of this dye is relatively high only small savings of dyestuff can be made, and the necessity of increasing the concentration of dye and sodium chloride in order that colours can be reproduced from each successive vat would make the use of a standing bath a complicated procedure.

Conclusions

- 1 The exhaustion of dye from replenished liquors is not necessarily characteristic of that from fresh liquors.
- 2 For the Suprexcel dye, a standing bath can save costs. With a concentration of 2 gl⁻¹ of the dye re-use of the old liquor would save about half the cost of dye needed for a second batch of fibre, and would consume smaller amounts of auxiliary chemicals.
- 3 The use of a standing bath for dyeing coir with the Benzamin dye is of doubtful value, and might prove more costly than the use of fresh liquor for each batch of fibre.

C1 Studies on the quality of colour: Effect of dyeing technique

Experimental

Several samples of unbleached coir were dyed using 1.5 gl^{-1} or 2 gl^{-1} Suprexcel Black DS 167% and 1 gl^{-1} of sodium carbonate in the bath. Dyeing times and temperatures and the concentration of sodium chloride in the liquor were varied. The degree of dyebath exhaustion was determined for each used liquor and the concentration of dye deposited on each sample of fibre was calculated. Six samples, each containing approximately 21 g* of adsorbed dye per kilogram of fibre, were selected for evaluation. The dyeing conditions used with the six samples are summarised in Table 2.

Fibre from each of these samples was tested for light-fastness and water-fastness using British Standard methods (see p. 7), and sectioned by the grinding method (see p. 7) in order to assess penetration of dye into the fibres. Additionally their colours were compared by means of reflectance data measured with the photometer (see p. 7).

Table 2

Sample number	Concentration of dye gl ⁻¹	Concentration of sodium chloride gl ⁻¹	Time at 95° C minutes	Cooling time hours	Dye adsorbed on fibre g(kg) ⁻¹
1	1.5	30	123	<u></u>	19.6
2	1.5	30	183		20.8
3	2.0	10	180		20.8
4	1.5	10	360	<u>125</u> 7	22.4
5	2.0	5	90	16	20.6
6	1.5	10	90	16	22.2

Dyebath conditions for samples dyed with Suprexcel Black VY and quantity of dye adsorbed

Results and discussions

Assessments of fastness to water and light, and of penetration are given in Table 3; measurements of reflectance are given in Table 4.

Small differences were found between the fastness properties of the six samples.

Penetration, which was relatively shallow compared with fibre diameter (*see* Table 3), improved with longer dyeing times at 95°C (compare sample 4 with other results in Table 3). There was also evidence that reduced concentration of sodium chloride

^{*}The 2 per cent shade (i.e. 2 g of dye in the bath for each 100 g of fibre) of Suprexcel Black VY acceptable to 2 of 4 respondents in the preliminary trials (Canning and Jarman, 1974) was estimated to contain 21 g deposited dye per kilogram of fibre. Since a liquor to fibre ratio of 20:1 was used in the preliminary trials, 2 per cent shades were dyed using a concentration of 1 g^{1-1} dyestuff in the liquor.

with increased concentration of dye assisted penetration at 95°C (compare samples 2 and 3, Table 3); but this trend was not evident with relatively short dyeing times followed by cooling (compare samples 5 and 6, Table 3).

Table 3

Properties of colours produced from approximately 21 g Suprexcel Black VY deposited on each kilogram of unbleached coir

Sample	Light-	Water-fastness	i		Penetration
number	fastness	Change in colour	Stain on cotton	Stain on wool	cells deep at periphery of fibre)
1	4-5	5	1	1-2	1-2
2	4-5	5	1	1-2	1-2
3	4	5	1	1-2	2
4	3-4	4BR	1-2	2	2+
5	4	5	1	1-2	1+
6	4	5	1	1-2	1-2

Note: See Table 2 for differences in dyeing conditions

Improved penetration seemed to be associated with improved water-fastness (with respect to staining), loss of water-fastness (with respect to change in colour), and with loss of light-fastness, (compare results for sample 4 with those for other samples, Table 3). However, many of the differences in fastness properties of the six samples were inconclusive, since differences of half a grade in fastness assessments are within the limits of experimental error.

Table 4

Reflectance of coir with approximately 21 g Suprexcel Black VY deposited on each kilogram of fibre

Sample	Percentage of	Percentage of incident light reflected					
number	450 nm	530 nm	550 nm	600 nm			
1	2.60	2.65	2.55	2.75			
2	2.70	2.70	2.60	2.80			
3	2.80	2.90	2.85	3.05			
4	2.80	2.80	2.85	3.05			
5	2.85	2.90	2.80	3.00			
6	2.80	2.80	2.70	2.90			

Each of the six samples reflected visible light similarly (see Table 4). The amount of light reflected from each of four wavelengths through the visible spectrum was of a similar low order for all samples, with red light (600 nm) being reflected slightly more strongly than other colours. Differences between reflectance figures obtained with the six samples could not be related to dye penetration, nor to the amount of deposited dye (compare samples 3 and 5, 1 and 6, 3 and 4 using Tables 2, 3 and 4).

It would appear that the technique used to deposit a predetermined amount of dye on the fibre does not significantly affect the visual colour obtained. Failure to relate differences in the reflectance characteristics of the six samples to dyeing technique indicates that the very small differences measured originate from other sources.

The depth to which dye penetrates into the fibre does, however, appear to be affected by dyeing technique. With fixed amounts of deposited dye, increased penetration implies that the dye has spread thinly through a greater proportion of the fibre substance. This would be expected to lead to improved water-fastness since dyes are retained more strongly at a low concentration in the substrate. However, the dyed material would be less able to resist change of colour when dye is removed from, or destroyed at, the surface of the fibre. This is because exposure of the natural colour of the surface of the fibre occurs with a relatively small loss of dye. Sample 4 shows these characteristics: fastness to water with respect to staining was slightly higher than for other samples. Thus, this sample was not only well penetrated, but also contained a relatively low concentration of dye at the surface.

Distribution of dye through the dyed portion of the fibre is uneven; dye concentration in the substrate decreases with depth of penetration. For sample 3, which was also well penetrated, the use of excessive dye in the bath would have maintained a high concentration of dye in the surface of the fibre. Similarly the use of cooling liquors, short dyeing times and, probably, the use of relatively high concentrations of sodium chloride*, could tend to concentrate the dye at the surface of the fibre. Thus, it is not surprising that samples 1 to 3, and 5 and 6, had better light-fastness than sample 4, and also poorer water-fastness with respect to staining.

Conclusion

The quality of colour produced from the deposition of a given quantity of an individual dye can be affected by dyeing technique. Techniques which favour penetration of dye are likely to give lower fastness ratings for loss of colour through light or water than techniques which favour surface deposition of dye; but fastness ratings with respect to staining could be higher. In these particular trials visual colour was unaffected.

C2 Studies on the quality of colour: Relative strength of alternative dyes

Introduction

Samples assessed by coir trade representatives in the preliminary trials (Jarman and Canning, 1974) were examined in the photometer in order to establish both the nature of light reflected from blacks which were acceptable, and the nature of differences between the blacks which could be detected by some respondents.

Experimental

Samples examined were:

Two commercial samples, identified as A and B for the purpose of this report; a sample dyed with 2 gl⁻¹ Suprexcel Black in the bath and a sample dyed with 1.5 gl⁻¹ Benzamin Black in the bath, each of which was acceptable to at least three of four respondents; and samples dyed with 1 gl⁻¹ Benzamin Black in the bath, and 1 gl⁻¹ Suprexcel Black in the bath, both of which were acceptable to two of the four respondents.

Shades of Synacril Black were not included in this examination. Since only one shade of this dye was produced in the preliminary trials, comparisons were not possible.

The optical absorbances of the three dyes in solutions of equal concentration (25 mgl⁻¹) were measured and the results were used to estimate the relative strengths of the dyes.

Results and discussions

(i) *Quality of acceptable blacks*. The percentage of incident light reflected from commercial samples A and B, and the samples from preliminary trials are given in Table 5.

From Table 5 it can be seen that all samples (including the commerical samples) reflected similarly both in intensity (between 2.4 and 3.1 per cent for any wavelength) and in composition. However, slight variations are apparent, and it cannot be ruled out that these will cause some observers to notice slight differences in colour. For example, Jarman and Canning (1974) reported that some observers found a purple tint to samples dyed with Benzamin Black, and since from Table 5 it can be seen that the Benzamin dyed samples reflect fractionally more red and blue than they do green,

^{*}Increased concentration of sodium chloride makes direct dyes more substantive towards cellulose. The migration of dye is possibly also inhibited by the resultant stronger bonding to the fibre.

Table 5

	Percentage reflection	of incident	light from	black coir	acceptable to	coir merchants
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Sample		Percentage reflected light		
Dyestuff	Concentration in dyebath gl ⁻¹	450 nm (Blue)	550 nm (Green)	600 nm (Red)
Benzamin Black DS 167%	1.0	2.70	2.60	2.70
Benzamin Black DS 167%	1.5	2.50	2.40	2.50
Suprexcel Black VY	1.0	2.90	2.95	3.10
Suprexcel Black VY	2.0	2.90	2.95	3.05
Commercial sample A	unknown	2.50	2.50	2.60
Commerical sample B	unknown	2.90	2.90	3.00

this observation appears to be supported. Since, however, Benzamin dyed samples were found to be acceptable to several observers it would appear that personal preference is important in choice of an acceptable black. The results also show that there was only a marginal reduction in reflectance (which was not perceived by most observers) from samples dyed with considerably larger amounts of the same dye. This indicates that 2.5–3 per cent reflectance is normal for black coir and so in practice it is wasteful to attempt to improve on such blacks by increasing the dyebath concentration. It also indicates that the small reflectances measured by the photometer may be due to surface characteristics of the coir. It can, therefore, be concluded that the sample dyed with 1 gl⁻¹ Benzamin in the bath represents an acceptable black colour.

(ii) Absorbance of the three dyes. Figure 6 shows the absorbance throughout the visible spectrum (390–700 nm) obtained with solutions of each dye at 25 mgl⁻¹ concentration and a path length of 1 cm. It can be seen that the Synacril dye absorbs more strongly than the Benzamin dye over almost the entire spectrum and that the Benzamin dye absorbs more strongly than the Suprexcel dye. However, it is also evident from Figure 6 that the dyes absorb differently from one another and that it is not possible to reproduce the absorption characteristics of one dye simply by adjusting the concentrations of the others. Nevertheless, since the Benzamin dye is known to produce an acceptable black colour on coir, data from Figure 6 can be used to predict the approximate amount of the other two dyes needed to produce similarly acceptable black colours by calculating the concentrations of them which would be necessary to produce equal total absorption over the visible spectrum (390-700 nm). Since for solutions which obey Beer's Law absorbance is proportional to concentration, this was done by determining the ratio of areas under the graphs between these limits using the weight of paper enclosed by the graphs as representing area. The concentrations of Suprexcel to Benzamin to Synacril that should give equal areas under the curves, i.e. the same total absorbance of visible light stand in the ratio of 1.18:1:0.54.

The sample from the preliminary trial dyed using 1 gl⁻¹ of the Benzamin dye was shown to give an acceptable black colour. It is estimated that, under the conditions used in the preliminary trial, dyebath exhaustion would have been about 95 per cent which means that some 19 g of Benzamin dye was adsorbed on to each kilogram of fibre. Using the above ratios, therefore, 22.4 g of Suprexcel dye or 10.28 g of Synacril dye deposited on each kilogram of fibre should give acceptable black colours. Dyers should, therefore, use dyeing conditions which would lead to these amounts of dye being adsorbed on the fibre, and particularly bear in mind that additional dye will usually be needed to take into account incomplete exhaustion.

In practice, some observers find that fibre that has adsorbed about 10 g of Synacril dye per kilogram of fibre has a slight greyish-green hue and is not acceptable to them. The difference in colour between samples containing concentrations of the Synacril and Benzamin dyes that should give similar total absorption of visible light is not unexpected, in view of the differences between the spectra. However, use of more Synacril dye should compensate for the colour difference arising from the relatively weak absorption at some wavelengths, and in practice about 13 g of Synacril dye per kilogram of fibre has been found to produce an acceptable black colour.





Similarly, small adjustments to the predicted amount of Suprexcel dye needed may be necessary to compensate for slight differences between the absorption spectra of this and the Benzamin dye.

Appendices

APPENDIX 1

Amount of dye deposited at given dyebath exhaustion

Percentage dyebath exhaustions do not give a ready indication of the quantity of dye deposited on the fibre. Figure 7 shows the quantity of dye deposited at any specified percentage exhaustion of dyes applied at 1, 2, 3, 4, 5 and 6 per cent shades. The conversions shown on this chart, which apply to all dyes, will not be affected by the dyeing techniques used.

Figure 7

Quantity of dye deposited on fibre at exhaustions of between 0 and 100 per cent from liquors containing up to 6 per cent on the weight of fibre of dyestuff



Exhaustion of dye from liquor (percentage)

APPENDIX 2

Estimation of the optimum concentration of sodium chloride for use with direct dyes

Introduction

The optimum conditions for dyeing depend not only on technical considerations, but also on the circumstances of the dyer. This appendix is designed to illustrate, by way of a hypothetical example, how both economic and technical considerations affect the development of an optimum dyeing method.

It is assumed that dyebath concentrations equivalent to 3 and 4 per cent on fibre (i.e. 1.5 gl^{-1} and 2 gl^{-1} at a liquor ratio of 20:1) are near the optimum for Benzamin and Suprexcel dyes respectively. These values were chosen since data on their exhaustion were available (*see* Figure 8)*.

It is also assumed that a liquor ratio of 20:1, with 1 gl⁻¹ of Lissapol N in the liquor, unbleached coir and dyeing time of 6 hours or less are appropriate. The exhaustion-time curves shown in Figure 8 are valid only for 3 and 4 per cent shades of the respective dyes, and only when the dyes are applied as stated.

The following costs were assumed:

Benzamin Black DS 167%	6-	£4.00 per kilogram dye powder
Suprexcel Black VY	_	£3.60 per kilogram dye powder
Sodium chloride (salt)	_	1.8 pence per kilogram, the approximate UK ex-
		factory price when purchased in 50 tonne lots; 5
		pence per kilogram, the estimated wholesale price
		of 50 kg lots; or 15 pence per kilogram, the approxi-
		mate UK retail price for a refined salt
Time	_	1.2 pence per hour per kilogram of fibre

Prices for dyes and salt were realistic in the UK in 1975–1976: Suprexcel was last available in 1972 at a price of £1.63 per kilogram. The cost of 'time', which involves mainly labour costs and fuel costs, will vary considerably with the scale of operation (dyeing a 1 tonne lot of material will take a similar time to the dyeing of a 1 kg lot, and will not require a proportional increase in manpower). The cost of chemicals, inclusive of Lissapol N, and water per kilogram of fibre will not be affected by the scale of operation unless discounts are obtained.

To estimate the optimum concentration of salt it is necessary to determine first the appropriate dyeing time. However, since time and salt concentration cannot be considered independently of each other, an approximate idea of the appropriate amount of salt needed will help in the initial stages of developing the optimum dyeing technique. With dyes which exhaust reasonably well, the optimum concentration of salt is usually between 5 gl⁻¹ and 20 gl⁻¹. With deep colours, such as black, it is more likely to lie in the range of 10-20 gl⁻¹. It is assumed that the higher range will apply to the two dyes considered here.

Estimation of dyeing time

(i) With 3 per cent Benzamin Black DS 167%. It is first assumed that a minimum of 1 hour at 95° C is needed for the dye to penetrate an adequate depth into the fibre. From curves 3 and 5 on Figure 8(a) the relevant range of dyebath exhaustions is between 52 per cent, after 1 hour at 10 gl⁻¹ salt, and 86 per cent, after 6 hours at 20 gl⁻¹ salt. There must be estimated the time at which the added cost of increased dyeing time is equal to the cost of the resultant extra dye recovered from the bath. Beyond this time, increases in exhaustion become more costly to achieve than savings on the dyestuff.

With dye used at the rate of 3 per cent on fibre, the cost of dye would then be 12 pence per kilogram of fibre.

Figure 8

Exhaustion of direct dyes over a period of 6 hours at 95°C with different concentrations of sodium chloride in the dye liquor





Concentration of sodium chloride:

1 = 0gl-1 2 = 5gl-1 3 = 10gl-1 4 = 15gl-1 5 = 20gl-1 6 = 25gl-1 7 = 30gl-1

Figure 9 Cost of Benzamin Black DS 167% deposited on to fibre with different concentrations of sodium chloride



Concentration of sodium chloride:

 $1 = 0gl^{-1} 2 = 5gl^{-1} 3 = 10gl^{-1} 4 = 15gl^{-1} 5 = 20gl^{-1} 6 = 25gl^{-1} 7 = 30gl^{-1}$

Figure 8(a) can now be adjusted to show the rate at which this 12 pence worth of dye is deposited on to the fibre: the exhaustion axis is converted to pence, with 100 per cent exhaustion equivalent to 12 pence. The cost of time at the rate of 1.2 pence per hour is shown as the dotted line (0) in Figure 9.

At some time each exhaustion curve has the same slope as the cost of time. These times for curves 2, 3 and 4 in Figure 9 are marked with a dot, and are those at which the cost of dye exhausted on to the fibre and the cost of time expired are increasing at the same rate. Thus, for these circumstances, the economic dyeing time will be two hours within about five minutes – provided that the optimum concentration of salt lies within the range of $5-15 \text{ gl}^{-1}$ and that these conditions will produce the quality of colour required.

(ii) With 4 per cent Suprexcel dye. For Suprexcel dye (Figure 8(b)) a different result is obtained. Using £3.60 per kilogram as the cost of the dye, and 4 per cent dye, the cost of dye used in the dyebath will be 14.4 pence per kilogram of fibre. 'Time' costs are unchanged and the economic time will still be about 2 hours for curve 3 (10 gl⁻¹ salt), but for curves 2 and 4 it would be about 1.5 and 2.5 hours respectively.

Determination of salt concentration

Having established an approximate optimum dyeing time, the salt concentration to apply the dyes can be determined. It is first necessary to establish on a cost basis the relationship between dye exhaustion and salt concentration for a dyeing time of 2 hours. This is shown in Figure 10 using intersections of the lines representing a 2-hour dyeing time and the respective exhaustion curves on Figure 9 to provide the



Weight of fibre

Note: All costs are based on 1 kg of fibre dyed at a liquor to fibre ratio of 20:1

necessary data after converting the exhaustion axis to represent the cost of dye exhausted from the dyebath^{*}. For the purposes of comparison further curves for a dyeing time of 6 hours are included.

Lines representing three costs of salt in the dyebath have been added to Figure 10. These lines are used to determine the optimum salt concentration in the same way that the line for the cost of time was used earlier.

From Figure 10 it will be seen that the four curves for dye exhaustion vary in shape; optimum salt concentrations for each are in Table 6. (For S6, 4 per cent Suprexcel dye, the optimum for salt costing 1.8 pence per kilogram could not be determined).

^{*}See Figure 9 for relationships between the cost of exhausted Benzamin dye and salt concentration after 2 hours dyeing. For Suprexcel dye an axis representing 14.4 pence at 100 per cent exhaustion was used.

Table 6

Dyestuff	Price	Dyeing	Optimum concentra	ition of sodium chlo	oride (gl ⁻¹ at LR 20:
	of time a dyestuff 95°C hours	95°C hours	@ 1.8p kg-1(0 ₁)	@ 5p kg-1(0 ₂)	@15p kg-1(0 ₃)
3 per cent Benzamin	£4.00 kg-1	2	28.75	16.25	6
Black DS 167%		6	18	12	6
4 per cent Suprexcel	£3.60 kg-1	2	30	21.5	8.5
Black VY		6	30	23.75	11.75

Optimum sodium chloride concentration for applying 3 per cent Benzamin Black DS 167% and 4 per cent Suprexcel Black VY

The figures in Table 6 exemplify optimum salt concentrations changing with:

(i) salt costs:

(ii) dye; and

(iii) dyeing time.

Other considerations in the development of the optimum process

Balancing the optimum amount of dye and salt. From Figure 10 it can be seen that with increase in the price of salt, the optimum salt concentration will become less. However, if the salt concentration is reduced the quantity of dye exhausted onto the fibre will also reduce. This will reduce the visual depth of colour (unless there is excess dye in the bath) and the fibre may not be acceptable. Therefore, it would be necessary to deposit more dye on the fibre by, for example, increasing the quantity of dye in the dyebath. Since exhaustion curves for different percentages of dyestuff are unlikely to follow those shown in Figures 8(a) and 8(b), additional curves would be required to estimate accurately the optimum dyeing conditions; for small changes in the percentages depth of shade it is probably satisfactory to estimate the quantities of dye and salt from Figure 10.

For example, from curve B6 in Figure 10 if the price of salt changed from 5 to 15 pence per kilogram, the optimum concentration of salt for a 3 per cent shade of Benzamin dye applied for 6 hours at 95° C would reduce from 12 gl⁻¹ to 6 gl⁻¹. However, the percentage exhaustion of the dyebath would fall also: from 85 to 76 per cent. At 3 per cent shade this fall in exhaustion of 9 per cent would represent 2.7 g per kilogram less dye on the fibre.

Assuming that 85 per cent exhaustion of a 3 per cent shade gave the correct depth of visual colour, then an additional 2.7 g per kilogram of dye would be required. Since only 76 per cent of the extra dye used will exhaust at the lower optimum salt concentration (6 gl⁻¹), the quantity of extra dye needed in the bath to make the colour good would be $(2.7/76 \times 100)$ or 3.55 g per kilogram of fibre.

However, adding extra dye to the dyebath increases the cost and this would in turn increase the quantity of salt which could be used to economic advantage. Thus, a dyer would preferably use a smaller amount of extra dye than indicated by this estimation, together with a little extra salt to restore an optimum balance.

The effect of dyeing time. An adjustment to dyeing time will alter the visual depth of colour produced. From Figure 8 it can be seen that, with shorter dyeing times, less dye exhausts on to the fibre and thus more dye, or more salt, would be needed in the dyebath if dyeing time is reduced. Small adjustments of dyeing time can, therefore, be helpful in the development of an optimum technique.

The effect of the 'warm up' period. The warm up period is an extremely important part of the dyeing process since it is at this stage that even or uneven colours are produced. In order to obtain even colours, this part of the process can be varied but alterations will change the quantity of dye exhausted during warm up. This will slightly change the shapes of the exhaustion curves with time and for salt concentration since the maximum possible exhaustion of the dyebath will not change. Since the shapes of these curves determine the optimum conditions, variations in the warm up period will alter optimum conditions.

Conclusion

The considerations which affect the determination of the optimum salt concentration and optimum dyeing times are complex and it is doubtful whether the dyer could determine these for each dyeing. However, the information presented in these reports should help coir dyers to use estimation methods to arrive at a useful compromise. It should be emphasised that the use of bleached coir, sodium carbonate or alternative wetting agents will also affect the course of dye exhaustion. These three aspects have not been mentioned in this appendix.

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