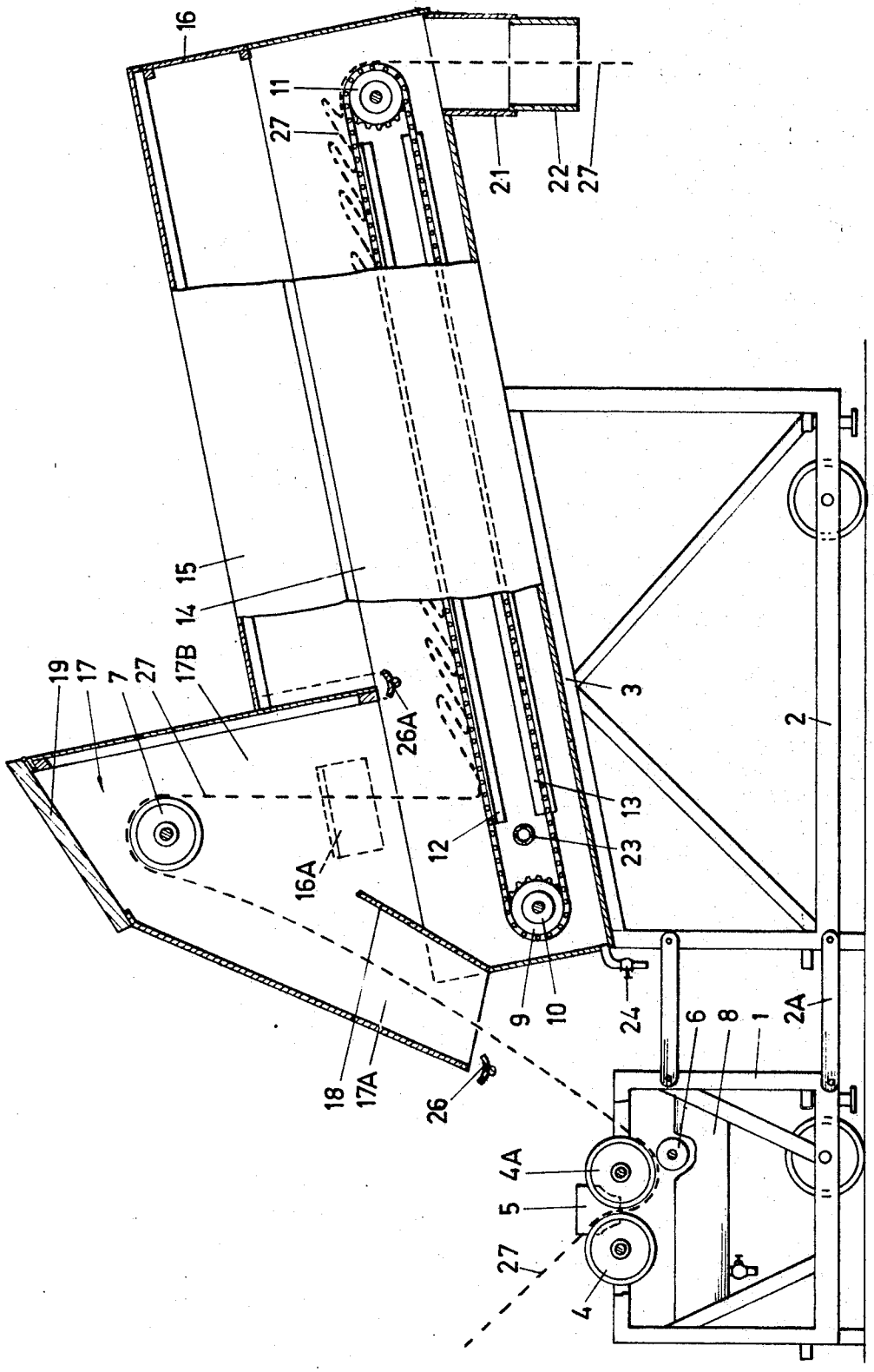
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METHOD OF DYEING WOOL AND COMPOSITION THEREFOR

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METHOD OF DYEING WOOL AND COMPOSITION THEREFOR

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ABSTRACT OF THE DISCLOSURE

Wool is dyed by treatment with a solution containing 30–35% by weight of urea, dioctylsulphosuccinate as the detergent and a dyestuff followed by steaming.

Wool can also be treated with a solution containing at least 20% by weight of urea and steamed prior to dyeing.

This application is a continuation-in-part of Ser. No. 476,700 filed Aug. 2, 1965, and now abandoned.

This invention relates to an improved composition, method and apparatus for the dyeing or printing of textile materials, particularly but not exclusively wool or other material consisting wholly or partly of keratin fibres.

The principal object of the above mentioned application is to provide a composition and method enabling the rate of dye fixation for such materials to be substantially increased.

Conventional dyeing methods for such materials are lengthy operations involving about 1 hour of boiling to fix the dyestuff within the fibres. Conventional printing methods require prior chlorination of the fibres and at least 30 minutes steaming to fix the dyestuff.

It is described in Australian Pat. No. 238,540 that concentrated formic acid when added to dye liquors or printing pastes accelerates the dyeing of keratin fibres because it acts as a wetting and swelling agent for keratin and as a solvent for the dye. The acid also imparts a positive charge to the fibre which attracts dye anions. However at the high concentrations of formic acid required, the corrosive and toxic fumes of the acid give rise to many difficulties, and the acid must be recovered for economic reasons. Moreover, in large scale treatments, dyeings with an uneven skittery appearance tend to be produced for reasons described later herein and the method we have discovered and described herein for correcting such uneveness cannot be used in concentrated formic acid.

We have found that the hydrogen bond breaking activity of urea in sufficiently high concentration could be used to accelerate the dyeing and printing of keratin fibres by virtue of the keratin swelling and dye dispersing action. As disclosed herein the same property can be used to accelerate the dyeing of fibres other than keratin also.

Thus according to one aspect, the invention provides a composition for the dyeing or printing of textile materials, particularly wool or other material consisting wholly or partly of keratin fibres, which composition contains a dyestuff and urea in sufficiently high concentration to accelerate the fixation of dye.

Usually for dyeing, the composition will be in the form of an aqueous liquor, i.e., a solution of the dyestuff and urea in water. For printing, the composition will usually be a paste, i.e. a thickened solution of these components.

The preferred concentration of urea in the liquor or paste is more than 20% and optimum results are obtained at concentrations of about 30% to 35%. Surface active agents, as discussed hereinafter, may be added to aid the dispersion of the dyestuff and the initial penetration of the liquor. The addition of acid is also advantageous with certain dyes since this imparts a positive charge to the fibres which attracts the dye anions.

Most classes of dye normally used to colour wool can be used in these compositions.

According to further aspect, the invention provides a method for dyeing or printing wool or other material consisting wholly or partly of keratin fibres, wherein the said material is treated with urea followed by steaming prior to the application of a dye-containing medium.

The rapid dye fixation which can be achieved with the composition and method of our invention, as described above, cannot be exploited to full advantage with exsisting dyeing techniques and machinery because the long steaming times hitherto required necessitated the use of large and expensive machines.

The present invention is thus also concerned with a practical method and apparatus which enable the potential advantages of the method and composition generally described above to be fully exploited.

According to this aspect of the present invention, there is provided a method for dyeing a textile material, particularly wool or other material consisting wholly or partly of keratin fibres, comprising the steps of impregnating the material in the form of a continuous sliver or other body of fibres with a predetermined amount of a dye liquor containing urea and a dyestuff, feeding the impregnated material into a steaming chamber, heating the material with saturated steam at atmospheric pressure before and after it is deposited on to a conveyor, and allowing the material to remain on the conveyor for a period sufficient for the desired degree of dye fixation to occur.

The impregnation may be carried out by any conventional means such as a pad mangle as used in conventional pad-dyeing processes. The amount of the dye liquor left on the padded material is about 100% by weight of the material, although lower or higher figures than this may be used if desired.

Preferably, the impregnated material is overfed to the conveyor in the steam chamber so that it is folded up on the conveyor, thus allowing a much shorter conveyor length to be used for a given steaming time.

The above defined steps may be followed by rinsing and drying in conventional machinery, such as a backwash.

As mentioned above, it is desirable to add a surface agent to the compositions of the invention, particularly for continuous pad, steam applications.

When a dye solution is applied to loose wool or sliver by a continuous process, such as padding, which leaves comparatively small amounts of the dye liquor on the wool, the liquor surrounding the individual fibres tend to break up spontaneously into droplets or beads which may be separated by distances of several fibre diameters. This effect, which is due to a combination of surface tension of the liquid and small diameter of the fibres, occurs even though the liquid thoroughly wets the fibres. The dye thus tends to be fixed on the fibres in a series of spaced bands and the mottled effect created in this manner causes the dyed fibres to have what is known as a "skittery" appearance.

This effect is overcome in existing continuous dyeing processes by using surface active agents which retard dye fixation so that the dye can diffuse along and through the wool fibres before it is fixed and even dyeing can thus be obtained. Such procedures suffer from the obvious disadvantage that long steaming times, up to an hour, are required to effect dye fixation.

In the process described herein, dye fixation is very rapid and occurs within minutes. Hence the dye must be quickly and evenly distributed over the fibre surface because the rapid fixation does not allow any useful amount of diffusion to take place along the fibre. It is therefore desirable to provide a surface active agent in the dye composition which possesses the following properties:

(1) It must not retard dye fixation because that would cancel the advantage of rapid fixation in the presence of concentrated urea.

(2) It must cause immediate and complete wetting of the wool entering the dye liquor. Besides being essential for even distribution of the liquor, it is important to displace all the air right at the liquor surface. Any entrapped air carried under the liquor by the wool will be expressed at the squeeze rollers and cause considerable foaming.

(3) It must possess strong film-forming characteristics. When the mass of wool passes through the squeeze rollers, all the fibres are pressed into intimate contact with each other and momentarily the liquor will be evenly distributed throughout the mass in the capillary channels between adjacent fibres. When the wool leaves the squeeze rollers, the fibres tend to spring apart and this will be accompanied by the spontaneous formation of beads of liquid mentioned above. Many fibre/fibre cross-over points still remain and most of the liquor will collect at these points but these are separated sufficiently to lead to uneven dyeing as described above. However, in the presence of a strong film-forming agent, the fibres tend to be held together by surface tension forces and, in any case, where fibres do become separated, a film is formed in the space between them. This assures intimate fibre/liquid contact through the mass and consequently leads to uniform dyeing.

We have found that sodium dioctylsulphosuccinate which is a common wetting agent sold under a number of trade names (e.g., "Aerosol OT") is one of the very few surface active agents which possess all three properties. Secondary alkyl sulphates and sulphonates of an approximately similar size (i.e., C_{16}–C_{18}) also appear to be suitable. Film stability can be improved by adding a small amount of a related member of the series (e.g., n-octadecylsulphosuccinamate) but in most cases the films are sufficiently stable using the dioctyl compound alone. The film-forming property can be destroyed and a skittery dyeing produced if an anti-foaming agent, solvent, or oil is added.

Thus, the preferred compositions of the present invention include a surface active agent with the properties described above. The preferred agent is sodium dioctylsulphosuccinate and the amount of agent added to the composition is preferably between 1% and 3% by weight of the composition.

The present invention also provides apparatus for carrying out the above method.

Thus, according to a further aspect of the present invention, there is provided apparatus for dyeing textile material, in particular wool or other material consisting wholly or partly of keratin fibres, with a dye liquor containing urea and a dyestuff, said apparatus comprising a chamber containing a conveyor to receive and hold a continuous sliver or other body of fibres, previously impregnated with the dye liquor, for a period sufficient to allow the desired degree of dye fixation to occur, and means for steaming the impregnated fibre body before falling on to and whilst on the conveyor.

Preferably, the apparatus also includes a pad mangle for impregnating the continuous sliver or other body of fibres with the dye liquor.

The chamber is provided with entrance and exit flues or like passages by which the fibre body enters and leaves the chamber. Preferably, the entrance flue is arranged so that the fibre body travels vertically through at least a portion thereof before being deposited on the conveyor and the fibre body is steamed while passing through the

said flue. It is preferable that the openings of both inlet and outlet flues point downwards so that steam is retained in the chamber by virtue of its lower density than air. However, in some applications, such as the steaming of loose wool, it is convenient for the opening of the inlet flue to be upwards.

It is also preferred that the apparatus is arranged so that the throughput of the pad mangle, or other impregnating means, is in excess of the conveyor speed so that the padded fibre body is overfed to the conveyor. It is further preferred that the conveyor is arranged to slope upwardly away from the point at which the fibre body is fed onto the conveyor so that the exit flue is at sufficient height to allow the fibre body to fall directly from the conveyor into suitable containers or else directly into apparatus for subsequent processing such as backwash.

The above and other aspects of the present invention will be explained in detail and enlarged upon in the following description which relates to a preferred embodiment of the apparatus of the invention and in which reference will be made to the accompanying drawing which shows a partly cut away elevation of an apparatus in accordance with the preferred form of the invention.

The apparatus shown in the drawing is a partly sectioned side elevation of an apparatus intended for the dyeing of wool or other textile slivers and consists of two main frames generally indicated as **1** and **2**, coupled together by removable sections **2A**, and an inclined subframe **3** which supports the conveyor as will be described hereinafter. On frame **1**, a conventional pad mangle is mounted. The pad mangle consists essentially of two rubber-covered rollers **4**, **4A** which, together with side plates **5**, constitute a trough for the dye liquor. In use, the pressure at the nip between the rollers **4**, **4A** is adjusted to control the amount of dye liquor left on a sliver after it passes through the dye liquor and the nip.

Transfer rollers **6** and **7**, synchronized with rollers **4**, **4A** serve to carry the impregnated sliver away from the pad mangle, and up into the flue **17A** and allow it to drop on to the conveyor belt **9** (as described hereinafter). Roller **6** is optional and can occupy any convenient position to allow latitude in positioning the mangle. The sliver may pass directly around roller **4A** and into the flue **17A**.

The conveyor belt **9** is made up of stainless steel rods bent at each end to form a chain link which couples each rod to the next one. It is supported and driven by two pairs of suitably toothed chain wheels **10** and **11** which are mounted on axles at either end of the subframe **3**. The subframe **3** and the conveyor belt **9** are inclined upwardly in the direction of travel to raise the outlet end to a convenient height, approximately 4 or 5 ft., for the reasons described above. Chain wheel pair **11** is powered to drive the conveyor. The conveyor belt is further guided and supported by Teflon rubbing strips carried on rails **12**, **13** beneath the upper and lower spans respectively of the belt **9**. The conveyor belt **9** is enclosed in an insulated rectangular chamber **14** which is also mounted on the subframe **3**. A large portion of the upper part of chamber **14** is fitted with a hinged lid **15** of triangular cross-section. The lid **15** contains an inspection window **16** in the gable section at the top end. The remaining part of chamber **14** is fitted with a vertical flue **17**, enclosing the roller **7**. The flue **17** is divided into two parts, **17A**, **17B** by the baffle **18**. The lower section **17A** opens downwards and provides the entry for the impregnated sliver. A hinged lid **19** at the top of flue **17** provides access for threading up the sliver or leader tape before the start of a run. Inspection windows **16A** are located on the vertical sides of flue **17**. The upper end of chamber **14** terminates on the underside in a downwardly-opening vertical flue **21** which may be fitted with an extension **22** of adjustable length.

A steam pipe **23** is mounted between the upper and lower portions of the conveyor belt and runs across the chamber **14** near chain wheels **10**. The pipe **23** is provided with two parallel rows of holes along its length so that steam supplied to the pipe in operation is ejected sideways and downwards and fills the whole of chamber **14** and flue **17**.

The reason for the downward openings of both inlet (**17**) and outlet (**21**) flues is to contain the steam in the chamber and flue by taking advantage of its lower density compared with air. This allows a temperature as near as possible to 100° C. in saturated steam at atmospheric pressure to be maintained without elaborate steam sealing means or without the escape and wastage of large quantities of steam. For efficient operation in this regard, the highest part of the opening of inlet flue **17A** should be slightly higher, preferably 4 to 6 inches, than the opening of outlet flue **21**. The steam level is thus maintained a few inches inside the outlet flue and the small amount of escaping steam all leaves via the inlet flue, countercurrent to the direction of sliver travel. The optional extension **22** to outlet flue **21** provides a means of adjusting the balance between inlet and outlet flues. The level of the opening of inlet flue **17A** is near the lowest level reached by the sliver when it drops onto the conveyor **9**, the steam pipe **23** is below this level, and the baffle **18** extends well up into flue **17**, all in order to minimize the possibility of cold air pockets forming in the lower parts of the steaming chamber **14**, which by cooling the sliver would slow down the rate of dyeing.

The roller **7** must be located some distance, of the order of 30 inches or more, above the conveyor **9** for the following reasons. The length of travel of the sliver must be sufficient for it to reach operating temperature (approx. 100° C.) before it is folded up. The sliver folds up more uniformly when dropped from some height. The line of travel between roller **6** and **7** in the steam zone should not deviate too far from vertical otherwise the sliver may tend to sag and break. With the preferred dye liquor composition, the sliver tends to foam when first contacted with the steam and the length of free travel should be sufficient to allow partial collapse of the foam so that the folded-up sliver is not too bulky.

The whole of flue **17**, lid **15**, chamber **14** is insulated with a 2" thickness of glass fibre or synthetic foam. An outlet **24** is provided at the lowest point of the chamber for the drainage of condensed water. A steam heated gutter **26** is provided at the inlet opening to prevent water dripping onto the sliver as it enters the flue and two sloping gutters **26A** are located under the abutment of the hinged lid **18** and flue **17**. All metal fittings and linings inside the flues and chamber are constructed from stainless steel to prevent rust staining of the textile material. The slope of all surfaces of the lids and flue above the sliver is sufficient for condensed water to run to the side or to a gutter rather than to drop off onto the textile material. Additional means of heating, such as closed steam coils, are not required because it is preferred to heat the sliver, impregnated with the preferred dye liquor composition, by means of saturated steam at approx. 100° C. However, it is not necessary to arrange elaborate means of ensuring steam saturation because the small degree of superheating normally encountered in expanding the steam from a line pressure above atmospheric (for example, a rise of 1 to 3° C. above 100° C. at atmospheric pressure) can be well tolerated when a dye liquor of the preferred composition is used.

In operation of the above described apparatus, one or more wool slivers (shown as dashed line **27**) are fed side by side from a suitable source (not shown) through the dye bath between the rollers **4**, **4A** and through the nip of these rollers. The sliver then passes around either roller **4A** or roller **6**, into the flue **17** via the opening in portion **17A** and thence over the roller **7**, after which it drops vertically on to the lower end of the conveyor belt **9**.

The rate of feed through the rollers **4**, **4A** and the speed of the conveyor **9** are adjusted so that there is a considerable overfeeding of the sliver on to the conveyor, ratios of up to 40:1 being usable but 15 to 30:1 being envisaged for normal operation. The sliver is thus piled up on the conveyor in a series of pleats and is slowly carried up the chamber **14** by the conveyor. On reaching the top of the conveyor **9**, the sliver falls through the flue **21** and the extension **22** into a container or directly into a washing apparatus such as a conventional backwashing plant (not shown).

In general, the time taken for a given part of the sliver to pass from the dye bath to the end of the apparatus can be varied up to 10 minutes, but is usually 2 to 5 min. Because of the small volume of dye contained in the dye bath and the rapidity with which the resulting shade can be observed, the changing or correction of colour or shade of the emerging sliver can be effected quickly. This is in contrast with conventional continuous dyeing processes in which periods of up to 1 hour may elapse before dyed material and an indication of the shade can be obtained.

The apparatus and the method of this invention can be used for continuously fixing the dye on melange or Vigoureux printed sliver. Melange or Vigoureux printing involves passing a sliver between a felt-covered roller, which is furnished with dye liquor from a trough underneath, and a fluted roller. The sliver is impregnated with bands of liquor only where the flutes press it into the felt. In conventional methods the sliver is then collected in or on containers, transferred to a steam box or autoclave, steamed batchwise for an hour or more and then rinsed in a backwashing machine. Each fibre contains several alternating coloured and white bands and requires very little blending later to give a more even "melange" effect than can be obtained by mixing dyed and undyed slivers. In applying the present invention to such a process, the dye liquor is made up with urea as described herein. The sliver is printed in the conventional way and then, instead of batching-up, is conveyed directly into a steaming chamber such as is shown in the drawing and finally through a backwashing machine. The steaming chamber shown in FIG. 1 can be uncoupled from the pad mangle by removing the coupling sections **2A** and transferred to the outlet end of a melange printing machine which already has a transfer roller in an appropriate position to fulfill the function of roller **6**.

The method and apparatus described herein is particularly suitable for uniformly dyeing of wool top previously given a shrinkproofing treatment which is normally difficult to dye evenly in conventional batch equipment because of the very rapid absorption of dye by such wool, coupled with the generally poor circulation of liquor in such equipment. Rapid absorption is an advantage in the method and apparatus described here. Certain reactive dyes, in particular, can be used on such wool to produce level very bright, fast shades after only a few minutes' steaming whereas conventional methods of application require prolonged boiling to achieve a high level of fastness, with consequent dulling of shade.

The method of the invention and the operation of the above described apparatus is illustrated but not limited by the following examples in which parts are expressed by weight.

EXAMPLE 1

A wool sliver was padded continuously, to leave on the wool 100% of a solution containing 35 parts of urea, 2 parts ammonia (0.880), 1 part potassium chromate, 1 part C.I. Mordant Blue 9, (Color Index No. 14,855) 1.5 parts of sodium dioctylsulphosuccinate and 59.5 parts of water. The sliver was then passed through a steam chamber at 100° C. with a contact time of 5 min. which fixed 94% of the dye. Fixed dye is defined as the dye

not removed by extraction with 50% ethanol containing 0.5% ammonia. This solvent removes more dye than warm water and detergent.

EXAMPLE 2

A sample of white wool sliver was padded to leave on the wool 100% of a solution containing 35 parts of urea, 2 parts acetic acid, 2 parts of sodium secondary heptadecyl sulphate, 2 parts of C.I. Acid Green 73, and 59 parts of water and steamed for 1 min. 95% of the dye applied to the sliver was fixed.

EXAMPLE 3

Eighteen wool tops (each 2½ oz./5 yd.) were padded continuously at the rate of 4½ yd./min., to leave on the wool 110% of a solution containing 35 parts of urea, 1.5 parts acetic acid, 1 part of C.I. Acid Orange, 85, 1.5 parts of sodium dioctylsulphosuccinate, and 61 parts of water. The sliver was then passed through the steam chamber of the apparatus described herein with reference to FIG. 2 at 100° C. with a contact time of 5 minutes. Over 95% of the dye applied to the sliver was fixed. The slivers were then backwashed and dried in the normal way.

EXAMPLE 4

Eighteen ends of wool sliver (each 3 oz./5 yd.) were padded continuously at the rate of 10 yd./min. to leave on the wool 120% of a solution containing 35 parts of urea, 2.5 parts of a 60% solution of acetic acid, 2 parts of a 75% solution of sodium dioctylsulphosuccinate, 1.05 parts of the metal complex dye C.I. Acid Yellow 114, 0.875 part of C.I. Acid Yellow 79, 0.175 part of C.I. Acid Green 40, and 58.4 parts of water The sliver was then passed through the steam chamber of the preferred form of the apparatus described herein with reference to FIG. 1 with the steam temperature at 102° C. and with a contact time of 5 min. About 93% of the total amount of dye was fixed and the unfixed portion was removed by backwashing followed by drying in the normal way. The dyeing was perfectly even with a washfastness equal to that of the same dyes applied by conventional methods.

EXAMPLE 5

Four batches of wool sliver of 250 lb. each were each divided into eighteen ends (each 3 oz./5 yd.) which were padded continuously at the rate of 6 yd./min. to leave on the wool 130% of a solution containing 35 parts of urea, 2 parts of a 60% solution of acetic acid, 2 parts of a 75% solution of sodium dioctylsulphosuccinate, 3 parts of the dye known by the trade name of Levalan Dark Brown TL (C.I. Acid Brown 331), and 58 parts of water. The sliver was then passed through the steam chamber of the preferred form of the apparatus as in Example 4 but with a contact time of 10 min. giving a dye fixation of about 95%. Removal of the unfixed dye in the backwash was facilitated by operating the bowls at 50° C. with countercurrent water flow and adding concentrated commercial ammonia solution to the second bowl at the rate of 1 part per 100 parts of wool travelling through. The sliver was then dried in the normal way.

The resultant dark brown shade was the same in each of the four batches, uniform within each batch and had the same washfastness as the dye applied by normal batch dyeing methods.

EXAMPLE 6

Wool top was given a continuous antifelting treatment involving sequential passage through dilute aqueous solutions of acidic chlorine, mildly alkaline reducing agent and an epichlorhydrin modified polyamide resin, followed by drying. Eighteen slivers (each 3 oz./5 yd.) of wool previously treated in this way were paded continuously at the rate of 15 yd./min. to leave on the wool 130% of a solution containing 35 parts of urea, 1.5 parts of a 75% solution of sodium dioctylsulphosuccinate, 0.0036 part of the dye known by the trade name of Lanasol Red B (C.I. Reactive Red 65), 0.0036 part of Lanasol Scarlet 2R (C.I. Reactive Red 78) and 63.5 parts of water. The sliver was then steamed as in Example 4 but with a contact time of 2 minutes followed by backwashing and drying as in Example 5. There was complete fixation of dye and the resulting bright pastel pink shade was even and completely washfast. Shades as pale as this are difficult to apply even to treated wool by conventional methods and such methods tend to yellow the wool and so produce a dull shade.

We claim.

1. A method of dyeing wool comprising the steps of pading a continuous sliver or other continuous body of wool fibres to leave on the fibres a predetermined amount of a dye liquor comprising a dyestuff, 30–35% by weight of urea and 1–3% by weight of sodium dioctylsulphosuccinate, and steaming the padded fibres to fix the dyestuff on the fibres.

2. The method of claim 1 wherein the fibres are steamed at 100–102° C. for 1 to 10 minutes.

3. A composition for the pad dyeing of wool comprising a dyestuff, 30–35% by weight of urea and 1–3% by weight of sodium dioctylsulphosuccinate.

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