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PROCESS FOR ALTERING THE ACID DYE RECEPTIVITY OF NYLON

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FIG. 1

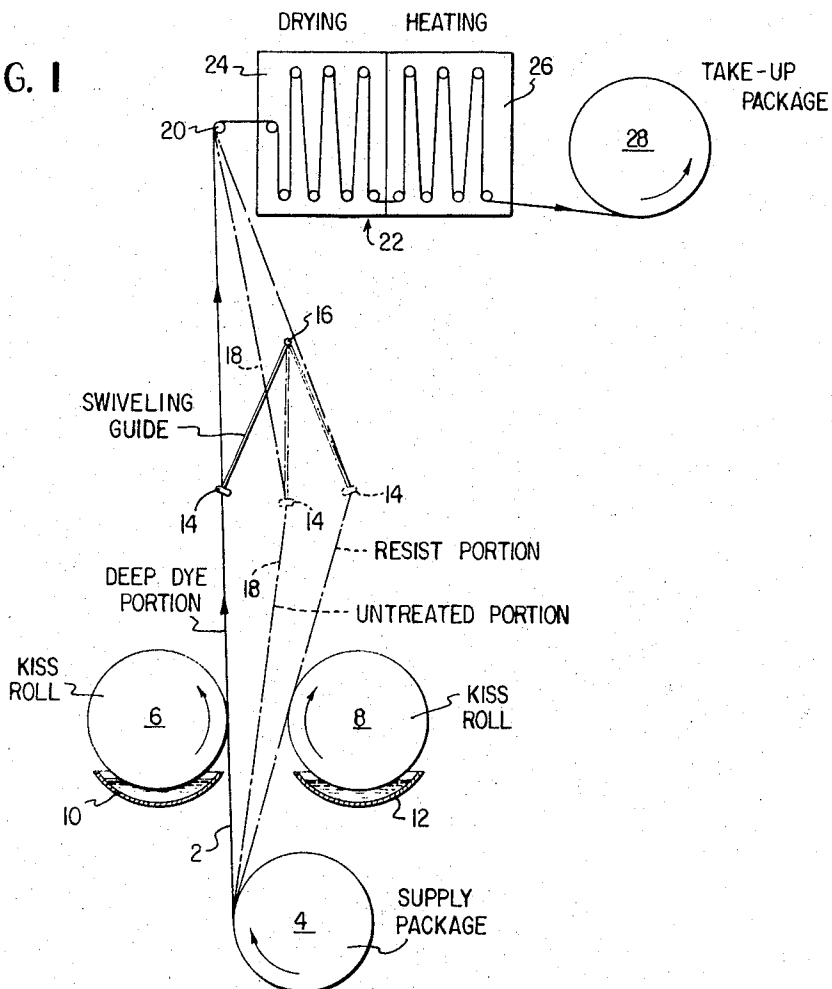
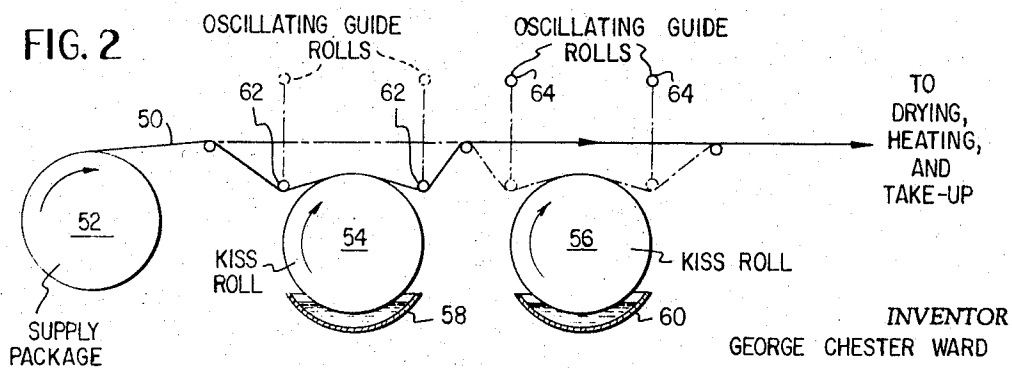


FIG. 2



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PROCESS FOR ALTERING THE ACID DYE RECEPTIVITY OF NYLON

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10 Claims

ABSTRACT OF THE DISCLOSURE

A method for increasing the affinity of nylon for acid dyes which comprises treating the nylon for which the acid dye receptivity is to be increased with an alkyl-dimethylbenzylammonium chloride prior to the dyeing operation preferably by applying the ammonium chloride compound in solution form to the nylon and drying and heating to a temperature in the range of about 50 to 200 degrees centigrade.

The present invention relates to a process for altering the acid dye receptivity or affinity of nylon. More particularly, the invention relates to a process for treating nylon textiles such as nylon yarn with an agent in order to increase the affinity of the nylon for acid dyes. Still more particularly, the invention relates to a process for producing a multi-colored or multi-shaded effect on nylon-containing materials.

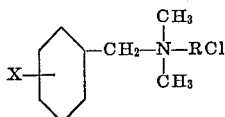
It is known that nylon, or synthetic polyamide, has several inherent, desirable properties such as strength, tenacity, abrasion resistance, high melting point and solvent and chemical resistance. Many of these properties make nylon particularly suitable for use in textile materials such as fabric, yarn, thread, cord, and the like. However, one of the desired features of any textile material, synthetic or natural, is that it be capable of being easily dyed by many different colored dyes. Nylon in some instances is not easily dyed to the desired deep shades much in demand today, especially is this true when the dye is an acid dye. The problem of dyeing nylon to a deep shade with an acid dye is particularly acute when the polyamide from which it is prepared has been polymerized in the presence of an acid type viscosity stabilizer, as for instance, an excess of dibasic acid.

Accordingly, the primary object of the present invention is to provide a process for altering the affinity of nylon for acid dyes.

Another object is to provide a process for treating nylon such as nylon yarn with an agent in order to increase the affinity of the nylon for acid dyes.

Still another object is to provide a process for producing a multi-colored effect on nylon-containing materials.

In accordance with the present invention, the affinity of nylon for acid dyes may be increased by treating at least a portion of a nylon-containing material such as nylon yarn with at least one alkyl-dimethylbenzylammonium chloride having the following structure:



where R is an alkyl ranging from C₈ to C₁₈, and where X is selected from the class consisting of hydrogen, halogen, and a lower alkyl ranging from C₁ to C₃.

The nylon-containing materials suitable for use in the practice of this invention include nylon fiber-containing fabric materials consisting wholly of nylon, such as nylon-6,6, the copolymer of hexamethylenediamine-1,6 and

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adipic acid; nylon-6, the polymer of 6-aminocaproic acid; and nylon-6,10, the copolymer of hexamethylenediamine-1,6 and sebacic acid. Other fabric or textile materials such as the various synthetic or natural fibers may be blended with the nylon in the nylon-containing materials and treated by the process of this invention. It is important to note, however, that the present invention only affects the dyeability of the nylon content of the particular nylon-containing material treated. Hence, this invention is particularly suitable for treating materials consisting wholly of nylon. The nylon-containing materials may be employed in the form of a fabric, carpet, yarn, thread, cord, or a non-woven web.

It has been found that when the nylon is treated with an alkyl-dimethylbenzylammonium chloride, and the treated nylon is then introduced into an acid dyebath, the resulting dyed nylon has a much deeper shade or color than when it is not so treated. Specifically, the alkyl-dimethylbenzylammonium chloride increases the affinity or receptivity of the nylon for the acid dye.

Exemplary alkyl-dimethylbenzylammonium chlorides which may be used in the process of the present invention are as follows:

octyldimethylbenzylammonium chloride;
decyldimethylbenzylammonium chloride;
tetradecyldimethylbenzylammonium chloride;
tetradecyldimethyl-4-chlorobenzylammonium chloride;
and
tetradecyldimethyl-3-methylbenzylammonium chloride.

Tetradecyldimethylbenzylammonium chloride is particularly suitable for use as the treating agent. Preferably, however, a mixture of the alkyl-dimethylbenzylammonium chlorides are used, such mixtures commonly being known as benzalkonium chlorides. Especially preferred for use herein, are the benzalkonium chlorides commercially known as BTC-824 and BTC-776, which contain the following alkyl mixtures (weight basis):

Alkyl	BTC-824, percent	BTC-776, percent
C ₁₂ -----	5	50
C ₁₄ -----	60	30
C ₁₆ -----	30	17
C ₁₈ -----	5	3

The alkyl-dimethylbenzylammonium chloride is applied to the nylon preferably as a pretreatment step prior to the acid dyebath, however, if desired, the chloride may be added to the acid dyebath so as to apply concurrently alkyl-dimethylbenzylammonium chloride and acid dye to the nylon. It should be noted that when the alkyl-dimethylbenzylammonium chloride is applied to the nylon concurrently with the acid dye, the resulting dyed nylon will have a substantially uniform deeper shade than what is generally obtained by using the dye alone. However, when using the preferred alkyl-dimethylbenzylammonium chloride pretreatment, only a portion of the nylon may be treated with the alkyl-dimethylbenzylammonium chloride and a portion not so treated. In this manner, the resulting dyed nylon will have a dual-colored or dual-shaded effect which is highly desirable in fabrics used today, particularly carpets. The pretreated portion will have a deep shade and the non-treated portion a lighter shade.

Normally, when the nylon is pretreated with the alkyl-dimethylbenzylammonium chloride, or mixtures thereof, the alkyl-dimethylbenzylammonium chloride is dissolved in water to form an aqueous treating solution. The alkyl-dimethylbenzylammonium chloride, or mixtures thereof, are present in the aqueous solution at a concentration in the range of from about 0.5 to 20% by weight, and preferably from about 1 to 6%, for example 1.5%.

When the alkyl-dimethylbenzylammonium chloride and acid dye are applied concurrently to the nylon, the alkyl-

dimethylbenzylammonium chloride, or mixtures thereof, are present in the acid dyebath solution at a concentration in the range of from about 0.3 to 10.0% by weight, and preferably from about 0.3 to 1.0%, for example 0.5%.

The nylon may be conveniently treated with the alkyl-dimethylbenzylammonium chloride at a temperature in the range of from about 20 to 60° C., and preferably from about 25 to 35° C., for a period of time in the range of from about 5 to 60 minutes, and preferably from about 10 to 20 minutes. However, if desired, higher and lower temperatures and times may be used.

The alkyl-dimethylbenzylammonium chloride may be applied to the nylon in any convenient manner such as by immersion, padding, spraying, brushing or coating. If the multi-colored effect is to be imparted to the nylon then the alkyl-dimethylbenzylammonium chloride may be applied to portions of the nylon, as discussed herein-after. The entire yarn skein may then be immersed in an acid dyebath to yield a dual-colored, dyed product. The design or coloring effect on the nylon material may be altered as desired by those skilled in the art.

When the nylon, or a portion thereof, is pretreated with the alkyl-dimethylbenzylammonium chloride prior to the acid dyebath treatment it is desirable after applying the alkyl-dimethylbenzylammonium chloride to the nylon to first remove any excess alkyl-dimethylbenzylammonium chloride solution from the nylon, for example, by squeezing between rollers, and then to dry and heat at least the treated portions, and preferably all of the nylon (treated and untreated) at a temperature in the range of from about 50 to 200° C. Preferably, the nylon is dried and heated at a temperature in the range of from about 100 to 200° C., and more preferably from about 100 to 120° C., for a period of time in the range of from about 10 seconds to 20 minutes, and preferably from about 1 to 3 minutes. Any convenient drying and heating means may be used, such as placing the nylon in an oven or running the nylon over heated rolls. In addition, an infra-red heater or subjecting the nylon to a steam treatment at five pounds pressure for a few minutes, e.g., 5 minutes, may also be used to dry and heat the nylon. The alkyl-dimethylbenzylammonium chloride treated, squeezed, dried and heated nylon may then be introduced into the acid dyebath treatment zone.

Any of the acid dyes conventionally used for dyeing nylon are suitable for use in the present invention. The term "acid dye" normally is a dye which is applied from a dye liquor containing sulfuric, formic or acetic acid, or ammonium sulfate. Sometimes the dye is applied from a neutral dyebath and, in a few instances, from a slightly alkaline dyebath. Sodium sulfate, Glauber's salt, is often added when dyeing with sulfuric, formic or acetic acid. The pH value of the various dyebaths varies from about pH 2 to pH 8. Many of the acid dyes possess good solubility in water, which is of importance for direct print styles on polyamide fibers. Chemically, the acid dyes usually comprise azo, anthraquinone, triphenylmethane, azine, xanthene, ketonimine, nitro and nitroso compounds. Exemplary acid dyes are: Orange II (C.I. Acid Orange 7), Black 10B (C.I. Acid Black 1), Capracyl Yellow GW C.I. acid yellow 124, Capracyl Red B C.I. acid red 182, Capracyl Blue G C.I. acid blue 165, Capracyl Violet BD, Nylanthrene Navy LFWG, Capracyl Red BB C.I. acid red 178, Fast Light Red BA C.I. acid red 37, Vialon Blue FFG C.I. acid blue 209.

The acid dye may be applied to the nylon by any of the conventional techniques well known in the art, for example, immersion, padding, spraying, brushing or coating. Preferably, the nylon is immersed or dipped into a dyebath which contains the acid dye either alone or in admixture with an alkyl-dimethylbenzylammonium chloride. The acid dyebath may be maintained at a temperature in the range of from about 70 to 180° C., and preferably from about 90 to 160° C. The nylon is normally

present in the dyebath for a period of time in the range of from about 5 to 120 minutes, and preferably from about 15 to 30 minutes. Generally, after the nylon has been dyed, the nylon is squeezed to remove excess dye, rinsed, and then dried in the conventional manner.

As previously mentioned, the preferred mode of the present invention is to treat the nylon to yield a multi-colored effect thereon. Hence, only a portion of the nylon is pretreated with the alkyl-dimethylbenzylammonium chloride and the entire amount dyed to yield a dual-colored effect. If desired, additional shades may be imparted to the nylon by using other agents which alter the dyeability of nylon such as condensation products of aryl, usually carbocyclic, sulfonic acids and an aldol. The aldol is an aldehydic or ketonic carbonyl compound, e.g., formaldehyde, acetaldehyde, benzaldehyde, benzoin, and acetone. These condensation products normally retard or decrease the affinity of the nylon to the acid dye. A preferred condensation product is a formaldehyde naphthalenesulfonic acid condensation product, commonly known as Erional NW. For example, a first portion of a nylon yarn may be treated with an alkyl-dimethylbenzylammonium chloride, a second portion of the nylon yarn may be treated with Erional NW, and a third portion of the nylon yarn not treated with any agent. The excess agents may be squeezed from the respective treated nylon portions and then the entire skein of nylon yarn preferably subjected to a heat treatment to dry and heat the skein. The skein of nylon yarn is then introduced into an acid dyebath.

The resulting nylon product has a tri-colored effect. The portion treated with the alkyl-dimethylbenzylammonium chloride is dyed a darker shade than is normally obtained with the particular dye, the untreated portion is dyed the usual dye shade, and the portion treated with the Erional NW is dyed a lighter shade than is normally obtained with the particular dye, the untreated portion is dyed the usual dye shade, and the portion treated with the Erional NW is dyed a lighter shade than is normally obtained with the dye. The respective agents may be applied to the nylon in any desired pattern and by any means known to those skilled in the art.

For a better and more complete understanding of the present invention, its objects and advantages, reference should be had to the following detailed description and to the accompanying drawings in which:

FIGURE 1 is a schematic diagram illustrating a method for obtaining a multi-colored effect on the nylon; and

FIGURE 2 is a schematic diagram illustrating an alternative method for obtaining a multi-colored effect on the nylon.

Referring to FIGURE 1 of the drawings, a skein of nylon yarn 2, e.g., nylon 66 yarn, is supplied from any conventional type supply package or roll 4. The nylon yarn 2 is conveyed between a pair of conventional kiss rolls 6 and 8, which are rotating in opposite directions as illustrated in FIGURE 1 of the drawings.

As the kiss rolls 6 and 8 rotate they pass through baths 10 and 12, respectively. Preferably, the kiss rolls have an absorbent material or pad affixed thereto so that when they pass through the baths, a portion of the bath solution is absorbent thereon. Thus, when the kiss rolls contact portions of the nylon yarn as it passes therebetween they impart their respective solutions to the portions of the yarn that are contacted with the rolls. Bath 10 contains an aqueous solution of alkyl-dimethylbenzylammonium chloride such as a BTC-824 chloride mixture, the concentration of BTC-824 being 3% by weight of the aqueous solution. The bath 12 contains an aqueous solution of Erional NW, e.g., an Erional NW concentration in the range of from about 1 to 6% by weight such as 3%. Each of the baths may be maintained at a temperature of about 30° C.

The yarn 2 is caused to contact the kiss rolls by a swivelling guide 14, which is attached to a conventional

swivelling means 16. The swivelling guide is in continuous contact with the yarn 2 such that as the guide 14 travels from left to right the yarn is also caused to travel from left to right. In this manner, by controlling the rate of swing or swivel of the guide 14, a first portion of the yarn 2 is contacted with the kiss roll 6, which imparts BTC-824 solution thereto, a second portion is not contacted with either kiss roll (as indicated by the dotted line 18), and a third portion is contacted with kiss roll 8, which imparts Erional NW thereto.

The treated and untreated nylon yarn 2 is then conveyed via a conventional guide roll 20 into an oven or heating chamber 22. While it is not shown in the FIGURE 1, it is desirable to pass the yarn 2 between a pair of squeeze rolls to remove excess solution therefrom prior to introducing the yarn into the heating chamber 22. The heating chamber 22 comprises a drying section 24 and a heating section 26. The nylon yarn passes through the drying section 24, which may be maintained at a temperature of 100° C., into the heating section 26, which may be maintained at a temperature of 120° C. The dried and heated yarn is then withdrawn from the heating chamber 22 and collected on a conventional take-up package or roll 28.

The treated, dried and heated nylon yarn may be subsequently introduced into a conventional acid dye-bath not shown in FIGURE 1. After the treated yarn has been dyed in the acid dye bath the portions of the yarn which were treated with the BTC-824 solution will have a deep dye shade, the untreated portions of the nylon yarn will have the normal dye shade, and the portions which were treated with the Erional NW will have a lighter shade than is normally obtained with the acid dye.

Referring to FIGURE 2 of the drawings, an alternative method for obtaining the multi-colored effect is shown. Nylon yarn 50 is withdrawn from a supply package 52 and is conveyed past kiss rolls 54 and 56, which rotate in the same direction (as illustrated in FIGURE 2) and which pass through baths 58 and 60, respectively. The kiss rolls and baths operate in the same manner and under the same conditions as previously discussed with reference to FIGURE 1, with the bath 58 containing an aqueous solution of BTC-824 and the bath 60 containing an aqueous solution of Erional NW.

Pairs of conventional oscillating guide rolls 62 and 64 are positioned and controlled so that they cause portions of the yarn 50 to contact the rolls 54 and 56 whereby the respective solutions are imparted to the yarn. Specifically, the oscillation of the guide roll pairs 62 and 64 are controlled such that a first portion of the yarn 50 contacts roll 54, a second portion does not contact either roll 54 or 56, and a third portion contacts roll 56. In this manner a tri-colored effect is imparted to the nylon yarn when the yarn is subsequently dyed in an acid dye-bath. As indicated in the FIGURE 2, the yarn 50, after passage over the kiss rolls, is conveyed to drying, heating and take-up operation (not shown) prior to being dyed.

If desired, disperse dyes such as Eastman Blue BNN C.I. disperse blue 3 and Amacel Yellow G C.I. disperse yellow 3 may be used in admixture with the acid dyes to treat the nylon-containing materials. It has been found that treating the nylon with an alkyldimethylbenzylammonium chloride does not seriously affect the disperse dye affinity of the nylon. Hence, cross dye effects may be obtained by combining a disperse dye with an acid dye.

The invention is additionally illustrated by the following example wherein a skein of nylon 66 yarn is partially dipped in a 1.5% aqueous solution of BTC-824, removed, and the excess BTC-824 squeezed out. Another portion of the skein is dipped in a 3% aqueous solution of Erional NW, removed, and the excess squeezed out. Still another portion of the skein is left undipped. The entire skein is then dried and heated for two minutes at 120° C. The entire skein is then dyed in an acid dye bath

maintained at 90° C., with a 2% aqueous solution of Capracyl Brown BRN for 1 hour, rinsed and dried.

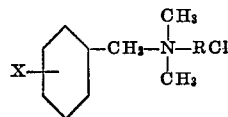
The resulting skein has three shades, as follows: The portion treated with the BTC-824 is dyed a dark brown, the untreated portion is dyed a light reddish brown, and the portion treated with the Erional NW is dyed a very light reddish tan. This process will work equally well on nylon 6.

The principle, preferred construction, and mode of operation of the invention have been explained and what is now considered to be its best embodiment has been described in the foregoing specification. However, it should be understood that the invention which is intended to be protected herein may be practiced otherwise than as specifically illustrated and as described without departing from the scope of the appended claims.

What is claimed is:

1. A process for increasing the affinity of nylon for acid dyes, which comprises

(a) pretreating at least a portion of a nylon-containing material with at least one alkyldimethylbenzylammonium chloride having the following structure:



where R is an alkyl ranging from C₈ to C₁₈, and where X is selected from the class consisting of hydrogen, halogen, and a lower alkyl ranging from C₁ to C₃,

(b) drying and heating at least the pretreated portions of the nylon-containing material at a temperature in the range of from about 50 to 200° C., and
(c) thereafter dyeing the nylon-containing material with an acid dye.

2. The process of claim 1 wherein all of the nylon-containing material, pretreated and untreated, is dried and heated prior to the acid dye treatment.

3. The process of claim 1 wherein only a portion of the nylon-containing material is pretreated with an aqueous solution of the alkyldimethylbenzylammonium chloride, said aqueous solution containing a concentration of alkyldimethylbenzylammonium chloride in the range of from about 0.5 to 20.0% by weight.

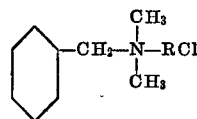
4. The process of claim 1 wherein only a portion of the nylon-containing material is pretreated with an aqueous solution of the alkyldimethylbenzylammonium chloride at a temperature in the range of from about 20 to 60° C., for a period of time in the range of from about 5 to 60 minutes.

5. The process of claim 1 wherein an aqueous solution containing a mixture of alkyldimethylbenzylammonium chlorides is used to pretreat the nylon-containing material.

6. The process of claim 1 wherein the alkyldimethylbenzylammonium chloride is an aqueous solution of a mixture of said chlorides having different alkyl groups, said mixture comprising 5% by weight C₁₂ alkyl chlorides, 60% by weight C₁₄ alkyl chlorides, 30% by weight C₁₆ alkyl chlorides, and 5% by weight C₁₈ alkyl chlorides.

7. A process for producing multi-colored effects on nylon-containing material, which comprises

(a) treating a first portion of the nylon-containing material with at least one alkyldimethylbenzylammonium chloride having the following structure:



where R is an alkyl ranging from C₈ to C₁₈.

- (b) treating a second portion of the nylon-containing material with a formaldehyde naphthalenesulfonic acid condensation product,
- (c) drying and heating the treated nylon-containing material at a temperature in the range of from about 100° C. to 200° C. for a time in the range of from about 10 seconds to 20 minutes, and
- (d) thereafter dyeing the nylon-containing material in an acid dyebath to yield multi-colored nylon.
8. The process of claim 7 wherein a tri-colored effect is obtained on the nylon-containing material by treating one portion thereof with said alkyldimethylbenzylammonium chloride, treating a second portion thereof with said formaldehyde naphthalenesulfonic acid condensation product, and preserving a third portion of the nylon-containing material untreated with said alkyldimethylbenzylammonium chloride and said condensation product, and thereafter dyeing the nylon-containing material.
9. The process of claim 7 wherein excess alkyldimethylbenzylammonium chloride and excess condensation product are mechanically removed from said nylon-containing material prior to drying and heating.

10. The process of claim 7 wherein the acid dyebath contains a mixture of acid dye and disperse dye.

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