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MANUFACTURE OF TEXTILE YARNS AND FABRICS

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This invention relates to textile yarns and fabrics to which there may be imparted a fast color and to the method of forming such textile yarns and fabrics and imparting thereto said fast color.

An object of the invention is the formation of yarns and fabrics having incorporated therein one or more components of a coloring material such that the amount of treatment after formation is reduced saving time and eliminating hazards.

Another object of the invention is the imparting of fast colors to yarns and fabrics without removing size and treating materials applied thereto as coatings. Other objects of the invention will appear from the following detailed description.

In dyeing yarns and fabrics containing filaments of organic derivatives of cellulose it has been necessary to swell the filaments in baths of swelling agents and incorporate at the surface of the filaments a dye base as a dye component; after that the material was given a rinse then diazotized and then treated with a developer or the other component of the dye in a liquid bath followed by a rinse. If there were wax or oil sizes on the filaments these had to be removed by washing prior to applying either dye component. These treatments in liquid, some of which were necessarily heated, gave rise to numerous hazards such as in yarn dyeing of entangling the filaments, stretching and breaking the filaments etc. while in fabrics such as formation of creases, matting etc. and in both yarn and fabric the necessity of relubrication and sizing.

We have found that by placing one or both components of an azo dye in the spinning solution from which the filaments are to be spun and after processing to add the other or merely diazotize there is formed a filament which may be colored evenly throughout the mass and not merely at the surface, also the only reagents that may be necessary to penetrate the filaments to form the color are of small molecular structure, even gaseous, which readily pass through sizes and lubricants eliminating the necessity of removing coatings from the yarns or filaments.

This method of dyeing accomplishes a great saving in the amount of dye used, and saves labor time. In commercial dyeing by the old method it was impossible to use all the dye in the dye bath or recover the unused dye so the practice was at the end of a run to allow the concentration of dye to decrease as low as safety would permit after which it was discarded. By the method herein described the formation of a dye bath is unnecessary and the quantity of dye used is only that amount required in actually imparting color to the yarns and fabric. There is a great saving in labor time as there are no, or there are fewer, baths to prepare and no time spent in the tedious attendance required of dye baths to see that the material is evenly treated. From 20–30% is saved in labor expenses per unit of material dyed.

In respect to the matter of hank dyeing, diazotization and developing of a yarn containing one or two components of the dye require relatively very little movement as compared with placing on the dye base, diazotizing and then developing on the hank dyeing machine and accordingly the labor of hank winding, care of the baths and rewinding from hank to packages, is saved and there will be much less damage to the filaments of the hanks. By this invention there is eliminated the further expense and skill required to produce dyed hanks entirely uniform throughout, containing no "ended" hanks which term is commonly used to describe a condition in which one side of a hank is dyed differently from another.

It is generally recognized, under the prior method of dyeing, that the best way of obtaining good dyeings and good fabric is to thoroughly cleanse the fabric free from sizes and lubricants as the molecular structure of the dye is too great to permit it to have great penetrating power. Whereas by this invention the large molecular components of the dye may be formed in the yarn it therefore is not necessary that the large molecular components penetrate the coatings and filaments but only smaller molecular structures such as the coupling component and/or the diazotizing component, need to penetrate the coatings.

By the method of this invention there is the advantage of efficient dyeing in the absence of alkalies and the reduced length of processing time in which the yarns or fabrics are in liquid baths or subject to any wetting action thus reducing the risk of stretching the fabric warpwise or otherwise reducing its wearing properties.

By the prior method of dyeing of fabrics on the winch, on the jig or in the hank form, more or less lengthy dyeing processes are required to insure penetration of the dye in between the filaments and between the crossed ends of warp and weft, this is particularly true in dyeing dark shades. It was also difficult in dyeing a roll of fabric to obtain uniformity throughout the length and width of the fabric due to currents

and change in concentration of the liquid dye bath. Also difficulty was encountered particularly in jig dyeing in obtaining the same depth of shade and uniformity of dyeing on the warp as on the weft, presumably because in the winding of the roll of fabric through the dyeing liquid, the warp is under considerable more tension than the weft. Also in highly twisted filaments if the filaments are untwisted the center was found to have a lighter dye because of lack of penetration. These difficulties are not present in dyeing according to this invention as there is uniform dispersion of the dye throughout each individual filament as well as the entire yarn and there are no lengthy wetting treatments which tend to build up different strengths of stresses in the yarn. There is found absolutely no difference between the warp and weft even in spite of the fact that the warp may be sized.

A great advantage of this invention is that uncolored yarn yet containing the dye-base may be sent out on the market and various colors imparted thereto by diazotizing and then treating in a bath of a selected coupling component. Or white yarn may be sent out on the market containing both the dye-base and the coupling component and the color later imparted to the material by merely diazotizing. In either case the dye-base is evenly dispersed throughout the yarn and the future processing, to impart the desired color or to impart the specific color, does not require the expert skill formerly necessary. For this reason each individual concern need not have a staff of highly skilled dye men.

The colors imparted to yarns and fabrics according to this method are as fast to light as the colors imparted by corresponding dyes by the former method. The colors however, are richer and more resistant to wear as the color is evenly dispersed throughout each filament and is not merely a surface color. While the strength of the filament may be weakened about 5% by the inclusion of the dye, this is less than the amount the filament is weakened by the prior methods where numerous baths, some heated or containing swelling agents, were used. The spinning solutions containing one or both components of the dye are as stable as normal spinning solutions. It passes through filters, spinnerets, etc. to all extent and purposes as does normal spinning solutions.

In accordance with our invention, then, we incorporate in the spinning solution from which filaments or films are to be formed, either by dispersion or by actually dissolving in the solvents in the spinning solution, a dye-base containing one or more primary amino groups in its molecular structure and after the subsequent formation of artificial materials, such as yarns, textile fabrics or films, we diazotize by treating with a diazotizing agent, such as the customary sodium nitrite and hydrochloric acid and then couple the diazotized dye-base with a selected developer which will impart the desired color to the textile.

A modification of our invention is to dissolve or disperse both the developer and dye-base in the spinning solution, from which filaments or films are to be formed, and after subsequent processing of the filaments or films into yarns, fabrics, etc., we diazotize the incorporated dye-base and/or developer with nitrous oxide and hydrochloric acid.

This invention is applicable in a general way to all yarns and textiles made of synthetic filaments and to foils or films. It is, however, specifically applicable to yarns and textiles made of synthetic filaments and films or foils of organic esters and ethers of cellulose. Examples of organic esters of cellulose are cellulose acetate, cellulose formate, cellulose propionate and cellulose butyrate, while examples of organic ethers of cellulose are ethyl cellulose, methyl cellulose and benzyl cellulose.

Under the term yarns are included threads, assemblies or bundles of a number of continuous filaments which may be in parallel relationship or which may be twisted together, short lengths of staple fibres, or yarn spun from such staple fibres. This invention is also applicable to single filaments or bundles of single filaments such as artificial bristles and straws, as well as foils, films or rods.

Under the term fabrics are included materials formed from yarns or filaments which have been woven, warp knitted, circular knitted, netted, knotted, braided or otherwise formed.

The spinning solutions are prepared by dissolving an organic ester or ether of cellulose or a mixture of these in such solvents as acetone, acetone and ethyl alcohol, acetone and methyl alcohol, chloroform, ethylene dichloride, ethylene dichloride and ethyl alcohol, ethylene dichloride and methyl alcohol, methyl chloride and ethyl alcohol and methyl chloride and methyl alcohol. The particular solvent or solvents employed will depend upon the organic derivative of cellulose to be dissolved. An example of a spinning solution is 1 part by weight of acetone soluble cellulose acetate dissolved in 3 parts by weight of 95/5 acetone/water solvent. Various percentages of the organic derivative of cellulose in respect to the solvent may be employed depending upon the spinning conditions and the type of film or filaments to be formed.

There is added to a spinning solution, according to our invention, from 0.5 to 3% or above, based on the weight of the organic derivative of cellulose in the spinning solution, of a dye-base having at least one primary amine in its molecular structure. The dye-base may or may not be soluble in the solvent of the spinning solution. If the dye-base is insoluble in the spinning solution it is dispersed therethrough by thorough mixing. As an aid to either solubility or dispersion the dye-base may be first mixed with or dissolved in a small amount of a carrier such as a soap, modified oils and like agents.

The spinning solution is then spun into filaments or films by extruding same through suitable sized and shaped orifices into baths, either liquid or gaseous, the concentration of the constituents of which are regulated. If the spinning solution is spun into filaments of low denier they may be associated together by twisting, at the spinning cabinet to form yarns. Or if the spinning solution is spun into films or filaments of high denier they may be processed into sheets, straws, bristles and like materials.

The yarns or filaments may contain besides the dye-base and derivatives of cellulose effect materials, which may be incorporated therein by mixing same in the spinning solution or placed therein by means of swelling agents, heat and/or pressure while the yarns or filaments were in contact with such effect materials. Thus the yarns may have therein or as coating, effect materials such as pigments or delustering agents, filling materials, fire retardants, plasticizers, sizes and lubricants. Examples of pigments or delustering

agents are carbon blacks, lamp blacks, nigrosine and the oxides of metals such as tin and lead. Examples of filling materials are powdered metals such as tin, aluminum etc., or other fibres such as cotton, silk, wool, etc. Examples of fire retardants are beta chlornaphthalene, triphenyl phosphate and tricresyl phosphate. Examples of plasticizers are glycol and glycerol oleates, stearates, etc. and triethanolamine oleates or stearates. Examples of sizes are waxes, starches, gums, salts of resin acids and glycerides. Examples of lubricants are olive oil, light mineral oil, soap, etc.

At any step in the future processing of the yarn after the spinning of the yarn to its complete formation into a fabric, it may be desirable to impart color to the textile. If in the yarn stage, the yarn is diazotized by passsing the yarn as a single strand through a liquid bath of sodium nitrite and hydrochloric acid or through a chamber containing vapors of nitrous oxide and hydrochloric acid. This may be done at any winding step in the treatment of the yarn. The yarn may be diazotized in either a liquid or gaseous bath while in hank form, on bobbins or in larger packages as cheeses, warps, etc. This treatment of packages is made possible by the property of the nitrous oxide and hydrochloric acid molecules to readily penetrate into the packages and even into the filaments.

The yarn containing the diazotized dye-base is then treated in a bath containing a selected developer or coupler. The yarn will be dyed a color depending upon the particular developers used. The developer is of relatively small molecular structure as compared with the heavy colloidal structure of a complete dye. Thus large packages are penetrated by the developer and as the dye-base is uniformly distributed through the filaments the yarn is evenly dyed on both sides of hanks, the center and outside layers of wound packages and in fact throughout the entire batch in the bath.

The yarns and fabrics may be diazotized in cold liquid baths thus reducing the care necessary to prevent damage to the yarns. Both the diazotizing and developer agents may be present in the same bath, thus reducing the number of baths.

If the color is not imparted to the textile in the yarn stage, the yarn may first be formed into a fabric, say in some small plant, and the fabric then given a cold diazotizing bath and treated with a selected developer. In this manner weavers of small amounts need not have highly experienced dye men and likely damage to the fabric is very small as well as the possibility of uneven dyeing.

By a modification of our invention the processes of small amounts of organic derivatives of cellulose yarn are further protected gainst damage and uneven dyeing in that the yarn contains both the dye-base and the developer and all that remains to be done, to impart the color, is to treat with nitrous oxide and an acid. Such a treatment may be in a cold bath of sodium nitrite and hydrochloric acid. The color of the finished yarn in this case, however, is predetermined to a great extent at the time of spinning the filaments. By another modification the developer alone may be added to the spinning solution and the dye-base added later along with a diazotizer to diazotize the dye-base.

In the modified process there is added to the spinning solution a dye-base containing at least one primary amino group in its molecular structure and a developer. After spinning and at any step in the processing up to finished fabrics, the material is diazotized with nitrous oxide which has high penetrating properties. In this process, as well as in the formerly described process, the sizes and/or lubricants on the filaments need not be removed as the diazotizing agent, also the developer, pass through such sizes and finishes readily. It is preferable, however, to avoid the use of, or to allow for, gelatins, which are themselves diazotizable and produce colored materials, thus throwing the goods or fabrics off color. The gelatins usually diazotize and couple to form yellowish dyes which produce complications in attempts at their removal.

It is found that the following dyes and like amino compounds may be used as the dye-base:

A.
1. p-Nitraniline
2. p-Nitro-o-anisidine
3. m-Nitro-p-toluidine
4. m-Nitro-o-anisidine
5. p-Chloraniline
6. α-Naphthylamine
7. Benzidine
8. Dianisidine
9. p-Aminobenzene-azo-naphthylamine
10. Amino-azo-benzene

With the above amino compounds or dye-bases, there may be used one or more of the following developers or couplers, depending upon the color desired.

B.
1. α-Naphthylamine
2. Dimethylaniline
3. m-Phenylene diamine
4. α-Naphthol
5. β-Naphthol
6. β Oxynaphthoic acid
7. p-Xylidine
8. β-Naphthylamine
9. Acetoacetic ester
10. Mono-di-or trihydric-phenol
11. Hydroxy-toluenes
12. Cresol
13. Resorcine

As examples of the colors obtained by various selections of developers to be used with selected dye-bases the following chart is given. The numerals following the letter "A" refer to the dye-base opposite that number above and the numerals following the letter "B" refer to the developer opposite that number above.

A8+B6=blue black
A8+B3=scarlet
A8+B6 or B10=neutral black

A9+B6=black
A9+B3=brownish black
A9+B2=deep purplish maroon
A9+B13=maroon
A9+B4=reddish blue
A9+B5=deep purple

A10+B10=greenish yellow
A10+B13=orange

It is thus seen that by spinning the yarn containing a dye-base such as p-aminobenzene-azo-naphthylamine the selected color can later be imparted thereto by the simple and nondangerous expedient of diazotizing and treatment with a developer which will couple with the dye-base to give the desired color. No swelling agents, alkalies or long heated baths are necessary.

The dye-base may be used to the extent of 0.5 to 5% of the weight of the yarn. The developer if added to the spinning solution may be employed in like amounts. If the developer is to be added to spun yarn in a bath the bath contains the customary concentration employed by former processes.

By this method dyes may be used which have no affinity for organic derivatives of cellulose. Any azo dye may be employed, of which one component is compatible with the derivative of cellulose, when it is present in the spinning solution in very small amounts.

It is to be understood that the foregoing detailed description is given merely by way of illustration and many variations may be made therein, without departing from the spirit of our invention.

Having described our invention, what we desire to secure by Letters Patent is:

1. The method of forming textiles comprising filaments of cellulose acetate which comprises adding to the spinning solution, from which the filaments are formed, a dye-base containing a diazotizable amino group and a developer, forming the filaments and then diazotizing.

2. The method of forming textiles comprising filaments of cellulose acetate which comprises adding to the spinning solution from which the filaments are formed, a dye-base containing a diazotizable amino group and a developer, forming the filaments, coating the filaments with a permeable finish and then diazotizing.

3. The method of forming textiles comprising filaments of cellulose acetate which comprises adding to the spinning solution, from which the filaments are formed, a dye-base containing a diazotizable amino group and a developer, forming the filaments, coating the filaments with a size and then diazotizing in a vapor bath.

4. The method of forming textiles comprising filaments of cellulose acetate which comprises adding to the spinning solution, from which the filaments are formed, a dye-base containing a diazotizable amino group and a developer, forming the filaments, coating the filaments with a size and then diazotizing in a cold liquid bath.

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