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DYEING-ASSISTANTS FOR SYNTHETIC FIBERS

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U.S. Cl. 8—172 3 Claims

ABSTRACT OF THE DISCLOSURE

In the dyeing of synthetic fibers, the use of various bis-(acetoxy- and propionoxyalkyl)acetamides, and substituted and unsubstituted N,N-dialkylbenzamides and N,N,N',N'-tetraalkylphthalamides, as dyeing assistants.

BACKGROUND OF THE INVENTION

This invention relates to the use of various N,N-lower dialkylbenzamides, N,N,N',N' - lower tetraalkylphthalamides, and bis(acetoxy- and propionoxyalkyl) acetamides as dyeing-assistants for synthetic fibers.

Many problems have been encountered in dyeing synthetic fibers such as the polyamides, e.g., "nylons," the polyesters, e.g., "Dacron," and the acrylic fibers, e.g., "Orlon," mainly because of their hydrophobic character, i.e., poor water absorbency and wettability. Furthermore, there are no reactive functional groups, such as amino and hydroxy groups, in the polymeric chains of these polymers which will assist in binding the dye molecule to the fabric.

Hydrophobic fibers, such as nylon are usually, though not always, dyed with highly colored water-insoluble organic compounds that diffuse or "dissolve" into the organic fiber to give a solid solution of the dye in the fiber.

Because these dyes are usually applied to the fiber as an aqueous dispersion, they are designated as "disperse" dyes (also included in this class are the disperse developed dyes). Among the more important representatives of this class are the aminoazo and aminoanthraquinone dyes.

Premetallized and basic (cationic) dyes are also sometimes used to dye the synthetic fibers. The premetallized dyes are usually designated as acid metallized or neutral metallized depending upon whether they are applied in an acid or neutral dyebath.

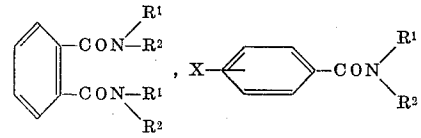
Many auxiliary products, i.e., dyeing-assistants, are used in dyeing in order to obtain commercially acceptable dye products having good water- and wash-fastness and good level shades of coloration. These dyeing-assistants comprise various wetting, penetrating, and leveling agents. The exact action of these agents in the dyeing of hydrophobic fibers is not thoroughly understood, but it is believed that they assist in the level deposition of the dye on the fiber and also assist the dye in penetrating into the fiber. In some cases, they appear to increase the solubility of the dye in the fiber.

Many of these dyeing-assistants are enumerated in Part V of the Technical Manual of the American Association of Textile Chemists and Colorists, 1966 Edition.

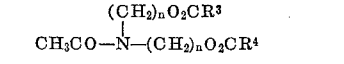
In aqueous dyeing processes the effectiveness of the dyeing-assistant is directly related to its water solubility; the greater the water solubility the greater the effectiveness. Unfortunately, the effectiveness of many commercially available dyeing-assistants is diminished because of their poor solubility in water. This limitation is partially offset by using them in an emulsified form.

SUMMARY OF THE INVENTION

This invention broadly comprises the use of the N,N-lower dialkylbenzamides, N,N,N',N'-lower tetraalkylphthalamides and bis(acetoxy- and propionoxy-lower alkyl)acetamides, having the following structural formulas:



and



wherein X may be H, —OH, or NH_2; R^1 and R^2 are alkyl groups containing up to 4 carbon atoms; $n=1$ to 3; and R^3 and R^4 are either methyl or ethyl; as dyeing-assistants for dyeing synthetic hydrophobic fibers.

We have now discovered that the water-soluble amides N,N-dimethyl-benzamide, bis(2 - acetoxyethyl)acetamide and N,N,N',N'-tetramethylphthalamide are excellent dyeing-assistants for use with the hydrophobic synthetic fibers. We have also found that some of the homologs of these compounds, although not as water-soluble as the above compounds, are also effective as dyeing-assistants when used as emulsions.

DETAILED DESCRIPTION OF THE INVENTION

In particular, the present invention comprises an improved process for dyeing hydrophobic synthetic fibers wherein water-soluble N,N-dimethylbenzamide is used as a dyeing-assistant.

It has been found that N,N-dimethylbenzamide has superior properties as a dyeing-assistant for use with synthetic fibers, especially the nylons. Excellent dye yields have been obtained using N,N-dimethylbenzamide and related compounds. Fabrics dyed with the assistance of these compounds exhibit strong, level shades of coloration and excellent brightness. In addition, the yarn of the dyed fabrics are fuller to the touch and have an excellent "cover" or density.

The N,N-lower dialkylbenzamides, N,N,N',N' - lower tetraalkylphthalamides and bis(acetoxy- and propionoxy-lower alkyl)acetamides described herein can be used with excellent results as assistants in dyeing synthetic, hydrophobic fibers, e.g., the nylons, in aqueous solutions.

All of the compounds preferred for this use are known and have been described in the prior art. The compound of choice in the group is N,N-dimethylbenzamide. This compound has a high degree of water-solubility which is, as previously mentioned, a desirable property for a dyeing assistant, since most, if not all, common dyeing processes employ aqueous solutions. Although other N,N-lower dialkyl benzamides can be used as dyeing assistants, they are not quite as effective as N,N-dimethylbenzamide, because as the carbon chain of the alkyl group increases in size, the water solubility decreases. Nevertheless, these compounds make excellent dyeing-assistants when used in an emulsified form.

N,N-dialkylbenzamides containing substituents such as amino and hydroxy groups in the phenyl ring have also been found to be excellent dyeing-assistants.

It has also been found that the N,N,N',N'-lower tetraalkylphthalamides, in particular the N,N,N',N'-tetramethylphthalamides, are effective assistants.

Surprisingly, we have discovered the bis(acyloxyalkyl) acetamides to be also effective as dyeing-assistants. Of these latter compounds, we prefer to use only the lower homologs of the class, in particular bis(acetoxymethyl or acetoxyethyl) acetamide and bis(propionoxymethyl or propionoxyethyl)acetamide because of their pronounced water solubility.

Nevertheless, it must be emphasized again, that even those compound described herein having poor water solubility are effective when used in an emulsified state. In such situations, an emulsifying agent or mixture is simply included in the dye bath. Commercially available emulsifiers are listed in Part V of the Technical Manual of the American Association of Textile Chemists and Colorists, 1966 Edition.

The dyeing-assistants of the present invention can be used with any commercial dyeing machine. Generally, the synthetic fabric to be dyed is first cleaned, i.e. "scoured," to remove dirt and oils that have accumulated during processing and weaving. Usually the scouring is done by immersing the fabric in a hot, aqueous, slightly alkaline solution containing a detergent and tetrasodium pyrophosphate and/or soda ash.

When an acid or acid metallized (premetallized) dye is to be used, the fabric is immersed in an aqueous bath set at about 80–100° F., containing the dyeing-assistant, e.g. N,N-dimethylbenzamide, ammonium acetate, and in cases where the dyeing-assistant is not too water-soluble, an emulsifier. Depending upon the shade of coloration desired, from about 0.5 to 10%, based on the weight of fabric (OWF), of dyeing-assistant is used. Usually about 2–3% OWF of ammonium acetate suffices.

After permitting intimate contact between the above solution and the fabric for several minutes, an appropriate dye solution is added and the dye bath is heated to about 200–220° F.

Acetic or formic acid may be added to the bath to insure complete exhaustion of the dye. N,N-dimethylbenzamide has been found effective in enhancing the dyeability of synthetic fibers, in particular nylon, with various commercial neutral and acid metallized dyes. Among the commercial metallized dyes which can be applied with the assistance of this dyeing-assistant in the manner described above are the neutral metallized "Capracyl" dyes available from the E. I. du Pont de Nemours & Co., Wilmington, Del., and the acid metallized "Chromalan" dyes available from the Allied Chemical Corporation, New York, N.Y.

The dyes predominantly used with the synthetic hydrophobic fibers are the disperse and disperse developed dyes. We have found that N,N-dimethylbenzamide and the other compounds described in the instant invention are extremely effective as dyeing-assistants with these particular dyes. In one embodiment of the present invention, the synthetic fabric is first immersed in an aqueous solution containing borax, tetrasodium pyrophosphate, and the dyeing-assistant. The fabric is removed and the disperse or disperse developed dye is added to the bath. The fabric is placed into the bath, which is then slowly heated to about 95° C. (193° F.). After keeping the fabric in contact with the hot solution for about a half hour, it is withdrawn, washed and dried. It is found that the dye is very evenly deposited throughout the fabric.

The amount of dyeing-assistant to be used depends upon the shade of color desired. We usually find 0.5 to 10% OWF of dyeing-assistant to be satisfactory for most purposes.

Among the disperse and disperse developed dyes effectively applied to synthetic fabrics with the aid of the dyeing assistants of this invention, are the "Acetamine," "Latyl," and "Celanthrene" dyes available from the E. I. du Pont de Nemours & Co., Wilmington, Del. The "Celanthrene" dyes are water-insoluble anthraquinone dyes, while the "Acetamine" dyes comprise the water-insoluble azo dyes.

As mentioned above, the disperse dyes are particularly useful in dyeing the synthetic hydrophobic fabrics and are widely used for this purpose. These dyes are practically water-insoluble and are usually applied as dispersions. The dyeing-assistants of the instant invention have been found to be more effective than other dye-

carriers such as benzyl alcohol, butyl benzoate, and thiodiethylene glycol.

The dyeing-assistants herein described can be used with a wide variety of synthetic hydrophobic fibers, such as the nylons, e.g. Nylon 6,6 and Nylon 6, the polyester fibers, e.g. Dacron, the acrylic fibers, e.g., Orlon, and the cellulose acetate and triacetate fibers. The optimum dyeing conditions such as temperature and pH, can be readily determined by routine experimentation.

As previously mentioned, we have found a dyeing-assistant concentration of from 0.5 to 10% OWF satisfactory.

Although we prefer to use the compounds described herein as dyeing-assistants for the neutral and acid metallized dyes and the disperse dyes, they will also assist in dyeing with the cationic, or basic dyes.

As a means of evaluating the efficacy of the organic amides herein described for use with the basic or cationic dyes, we have found the test below convenient:

A swatch of fabric dyed with a cationic dye and a swatch of the undyed fabric are immersed in a water bath containing 10% OWF of sodium sulfate decahydrate, 1% OWF of acetic acid, and 3% OWF of the dyeing-assistant being tested.

The bath is brought to the boiling point, and kept there until enough dye from the dyed swatch of fabric is transferred and sorbed on the undyed swatch so that both swatches are of the same shade.

The rapidity and effectiveness with which the compound being tested causes both swatches to have the same shade is a measure of the compound's utility as a dyeing assistant for cationic dyes.

Using this test, we have found N,N-dimethylbenzamide to be the most effective dyeing-assistant of the compounds of the instant invention for use with the cationic dyes.

The examples below are provided by way of illustrating the present invention; however, they are not to be construed as limiting the scope thereof.

EXAMPLE I

Dyeing with acid or premetallized dyes

A commercial nylon fabric is first scoured to remove oils and dirt by heating it in an aqueous bath containing a suitable synthetic detergent and soda ash at about 180–212° F. for an appropriate period of time.

A commercial dyeing machine is then charged with water at about 90° F. The nylon fabric is immersed in the bath and enough N,N-dimethylbenzamide is added to the solution to provide 1% by weight, based on the weight of the fabric (OWF). Sufficient ammonium acetate is added to yield 2–3% (OWF).

The fabric is kept in the resultant solution at about 90° F. for 10 minutes. At this point, an amount of a 1% solution of "Capracyl" Red B (C.I. Name Acid Red No. 2; Color Index No. 13020), available from the E.I. du Pont de Nemours & Co., Inc., Wilmington, Del., is slowly added to the bath solution over a period of about 10 minutes to provide from about 0.20 to 4% (OWF), depending upon the exact shade desired. The dyebath is heated at 200–220° F. for about 15 minutes.

Small amounts of acetic or formic acid may be added to the dyebath to exhaust the dye from the medium onto the fabric.

The excess dye liquid is removed from the fabric in the usual manner by either rinsing or washing and the fabric is suitably dried.

Nylon goods dyed in the above manner with N,N-dimethylbenzamide as a dyeing-assistant showed a marked superior physical appearance, as compared with fabrics dyed with the aid of commercially available dyeing-assistants.

In general, we find that with fabrics dyed with the assistance of N,N-dimethylbenzamide, the yarn is fuller to the touch and has a better surface "cover" or density.

Commercially acceptable results can be obtained using 0.5 to 10% (OWF) of the dyeing assistant.

EXAMPLE II

Example I is repeated, with substantially the same results, using the following dyes:

ACID (METALLIZED) DYES

"Chromolan" [1]	C.I. Name	Color Index No.
Blue NGG	Acid Blue 158	14880
Green BL	Acid Green 12	13425
Orange R	Acid Orange 76	18870
Yellow N	Acid Yellow 54	19010

[1] Allied Chemical Corp., New York, N.Y.

EXAMPLE III

When nylon fabrics are dyed according to the procedures of Examples I and II, replacing the N,N-dimethylbenzamide with from 0.5 to 10% (OWF) of the dyeing-assistants below, strong, level shades of dyeing with good brightness are obtained:

bis(acetoxymethyl)acetamide
bis(2-acetoxyethyl)acetamide
bis(propionoxymethyl)acetamide
bis(2-propionoxyethyl)acetamide
N-methyl-N-ethylbenzamide
N,N-dimethyl-4-aminobenzamide
N,N-dimethyl-2-hydroxybenzamide
N,N,N',N'-tetramethylphthalamide
N,N,N',N'-tetraethylphthalamide

EXAMPLE IV

Nylon fabrics are dyed according to the procedure of Examples I and II using from 0.5 to 10% (OWF) of the dyeing-assistant below. Because of their poor water solubility, however, they are used as emulsions (see Part V of the Technical Manual of the American Association of Textile Colorists, 1966 for a list of commercial emulsifiers).

N,N-diethylbenzamide
N,N-di-n-propylbenzamide
N,N-di-n-butylbenzamide
N,N-diethyl-2-hydroxybenzamide
N,N-di-n-propyl-4-aminobenzamide
N,N,N',N'-tetra-n-butylphthalamide

The dyed fabrics have strong, solid, level shades and are commercially acceptable.

EXAMPLE V

Examples I to IV are repeated, with substantially the same results, using instead of nylon fabric a commercial fabric derived from one of the following synthetic fibers:

cellulose acetate
cellulose triacetate
the acrylic fibers "Orlon" and "Creslan"
the polyester fibers "Dacron" and "Kodel."

EXAMPLE VI

Use of disperse, disperse developed, and nylon dyes

To a 2 liter beaker is added 1420 ml. of water (20 times the weight of the carpet stock to be used). To the beaker is then added 2.13 g. (3% OWF) of borax and 0.71 g. (1% OWF) of sodium tetrapyrophosphate decahydrate. The pH of the resultant solution is about 9.0–9.5. To the aqueous solution is then added 0.71 g. (1% OWF) of N,N-dimethylbenzamide.

An 18 in. long, 7 in. wide white nylon carpet stock, weighing 71.0 g. is immersed in the above solution, and the bath is heated to 80° F. (26.5° C.). After 10 minutes of immersion, the nylon carpet sample is withdrawn from the solution. The following dye solutions are then added with stirring to the solution: 2.84 ml. of a 1% stock solution (4% OWF) of "Acetamine" Yellow CG (C.I. Name Disperse Yellow 3; Color Index No. 11855); and 8.52 ml. of a 1/10% stock solution (12% OWF) of "Celanthrene" Blue CR. All of these dyes are obtainable from the E. I. du Pont de Nemours & Co., Wilmington, Del. The nylon carpet stock is replaced in the bath and kept there at 80° F. for 15 minutes. The bath temperature is raised 3° F. per minute (1.8° C. per minute), with constant stirring, until the temperature is 205° F. (96° C.). The carpet stock is kept in the stirred bath for 30 minutes at this temperature and is then removed and immersed in water at 80° F. It is removed from the wash water and air-dried to provide a dyed carpet stock having a good solid level color shade and a good fabric "cover" or density.

Commercially acceptable results are obtained when either 0.5% (OWF) or 10% (OWF) of the dyeing assistant is used.

EXAMPLE VII

Comparable results are obtained when Example VI is repeated with the following dyes:

Disperse dyes

	C. I. Name
"Latyl" [1]	
Yellow 4RL	Disperse Yellow 23 (Color Index No. 26070).
"Celanthrene" [1]	
Fast Blue 2G	Disperse Blue 7 (Color Index No. 62500).
Red 3BN	Disperse Red 15 (Color Index No. 60710).
"Acetamine" [1]	
Scarlet B	Disperse Red 1 (Color Index No. 11110).
Orange GR	Disperse Orange 3 (Color Index No. 11005).

Disperse developed dyes

	C. I. Name
"Acetamine" [1]	
Diazo Black RB	Disperse Black 1 (Color Index No. 11365).
Diazo Navy RD	Disperse Black 6 (Color Index No. 37235).

[1] E. I. du Pont de Nemours & Co., Wilmington, Del.

EXAMPLE VIII

When nylon fabrics are dyed according to the procedures of Examples VI and VII, replacing the N,N-dimethylbenzamide with from 0.5 to 10% (OWF) of the dyeing assistants below, strong, solid, level shades of dyeing with good brightness are obtained:

bis(acetoxymethyl)acetamide
bis(2-acetoxyethyl)acetamide
bis(propionoxymethyl)acetamide
bis(2-propionoxyethyl)acetamide
N-methyl-N-ethylbenzamide
N,N-dimethyl-4-aminobenzamide
N,N-dimethyl-2-hydroxybenzamide
N,N,N',N'-tetramethylphthalamide
N,N,N',N'-tetraethylphthalamide

EXAMPLE IX

Nylon fabrics are dyed according to the procedure of Examples VI and VII using from 0.5 to 10% (OWF) of the dyeing assistants below. Because of their poor water solubility, as compared with the dyeing assistants of Example III, they are maintained in an emulsified form during dyeing by incorporating a suitable commercial emulsifier (see Part V of the Technical Manual of the

American Association of Textile Colorists, 1966) in the dye bath.

N,N-diethylbenzamide
N,N-di-n-propylbenzamide
N,N-di-n-butylbenzamide
N,N-diethyl-2-hydroxybenzamide
N,N-di-n-propyl-4-aminobenzamide
N,N,N',N'-tetra-n-butylphthalamide

The dyed fabrics have strong, solid, level shades and are commercially acceptable.

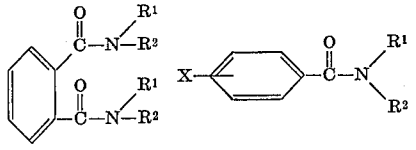
EXAMPLE X

Examples VI, VIII, and IX are repeated, with substantially the same results, with the disperse and disperse developed dyes of Example VII, using instead of nylon fabric, a commercial fabric derived from one of the following synthetic fibers:

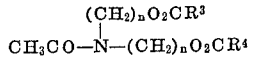
cellulose acetate
cellulose triacetate
the acrylic fibers "Orlon" and "Creslan"
the polyester fibers "Dacron" and "Kodel"

What is claimed is:

1. In the process of dyeing synthetic fibers using an aqueous system comprising a dye and a dyeing-assistant, the said dye being selected from the group consisting of acid metallized, neutral metallized, dispersed and disperse developed dyes, the improvement which comprises incorporating in the dyebath as a dyeing assistant up to 10% by weight, based on the weight of the fibers, of a compound selected from the group consisting of:



and



wherein X is H, —OH or —NH_2; R^1 and R^2 are alkyl groups containing up to 4 carbon atoms; n=1 to 3; and R^3 and R^4 are either methyl or ethyl.

2. In the process of dyeing synthetic fibers using an aqueous system comprising a dye and a dyeing-assistant, the said dye being selected from the group consisting of acid metallized, neutral metallized, dispersed and disperse developed dyes, the improvement which comprises incorporating in the dye bath as a dyeing-assistant up to 10% by weight, based on the weight of the fibers, of N,N-dimethylbenzamide.

3. In the process of dyeing nylon fibers using an aqueous system comprising a dye and a dyeing-assistant at temperatures between 80 and 210° F., the said dye being selected from the group consisting of acid metallized, neutral metallized, dispersed and disperse developed dyes, the improvement which comprises incorporating in the dye bath as a dyeing-assistant up to 10% by weight, based on the weight of the fibers, of N,N-dimethylbenzamide.

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