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2,945,010

PREPARING POLYESTER FIBERS OF IMPROVED DYEABILITY USING AROMATIC AMINES

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This invention relates to linear high-melting aromatic polyester fibers which can be readily dyed with a metallizable dye and to a method for producing such fibers which includes the steps of incorporating into a polyester of this type in its granular form prior to forming the fiber, a nuclearly substituted amino aromatic non-metallized compound, then forming a fiber, and then treating the fiber with an aqueous solution of a mordant salt containing a metal such as chromium, cobalt, nickel or copper. These treated fibers can be readily dyed with any of the metallizable dyes known in the art to be useful in the dyeing of other fibers than those produced from polyesters. This invention is particularly concerned with polyesters derived from terephthalic acid or 4,4'-sulfonyldibenzoic acid.

It is known that linear polyester fibers, such as those produced from polyethylene terephthalate are difficult to dye. At the present time polyester fibers are generally dyed at pressures substantially greater than atmospheric with cellulose acetate dyes in order to obtain desirable and practical shades or colors. These superatmospheric-pressure procedures require expensive equipment and consume a relatively long time in the dyeing operation. Alternatively, the dyeing of linear polyester fibers can be carried out in the presence of a dyeing assistant or swelling agent such as phenol, the cresols, benzoic acid, dichlorobenzene and the like. These procedures often result in non-uniform application of the dye to the fibers as a result of non-uniform swelling of the fibers. Furthermore, most of the dyeing assistants and swelling agents possess one or more objectionable features such as expense, toxicity, objectionable odor and the like.

It is also known in the dyeing art that compounds of metals such as nickel, chromium or cobalt react with certain dyes to produce stable, colored metal complexes known as metallized dyes. However, such dyes are not customarily employed in the dyeing of polyester fibers. The incorporation of the metal complexes in the polyester fibers during the spinning process presents special problems because of the high temperatures involved. Furthermore, strictly anhydrous conditions and other requirements are necessarily involved in the spinning process in order to minimize or avoid the decomposition of the polyester.

The general methods employed in the prior art for dyeing or mordanting fibers other than polyester fibers are not applicable to polyester fibers. For example, aqueous solutions of the metal compounds or dyes will not penetrate the polyester fibers to give the desired adherent deposit. Also, many of the commonly used mordant salts are in hydrated form and will give off water under processing conditions causing degradation of the polyester fibers. Another limiting factor in the procedures for dyeing polyester fibers is the high degree of thermal stability required. In producing the fiber, the polyester is usually melted and spun at a temperature of well above 200° C., such as at 250° C. to 300° C., and under such

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conditions many of the commonly used metallized dyes decompose to produce dark colored oxides or other decomposition products. Also, in some instances, the metallized dyes at these high temperatures actually lose their ability to function as mordants, and in other instances, the metallized dyes react with the polyesters at the temperatures used to spin the fibers to degrade the polyester.

It is quite apparent from the above discussion that the dyeing of polyester fibers presents unique and unusual problems which are not involved by use of the prior art procedures for dyeing other types of fibers.

It is an object of this invention to provide polyester fibers of improved dye affinity for metallized dyes.

It is another object of this invention to provide fibers of linear high-melting polyesters which can be readily dyed with a metallizable dye according to procedures well known in the art for dyeing other fibers normally dyeable with such dyes.

It is a further object of this invention to provide a process for preparing such fibers having these improved properties by the simple and economical procedure of (a) introducing into the polyester before spinning an aromatic amino compound of a certain class and (b) treating the spun fiber of the polyester with a mordant salt.

Other objects will become apparent elsewhere herein.

In accordance with a principal embodiment of this invention, the well known procedures for the preparation of a linear high-melting polyester fiber are modified to an extent which will permit the fibers produced to be readily dyeable with a metallizable dye by means of the following improvement in the ordinary procedures which comprises (1) incorporating in said fiber-forming polyester prior to formation of said fiber a nuclearly substituted amino aromatic non-metallized compound containing from 6 to 20 carbon atoms, (2) forming a fiber by any of the usual well known procedures, and (3) treating the fiber at an elevated temperature with an aqueous solution of a water-soluble mordant salt containing a metal such as chromium, cobalt, nickel or copper.

In practicing this invention, the nuclearly substituted amino aromatic compound can be incorporated into the polyester at any time prior to its being spun into a fiber. Thus, this aromatic amine additive can be dissolved or dispersed in the polyester melt during the process for preparing the polyester itself by the usual melt polymerization technique. As another example, this aromatic amine can be introduced during the final stages of the solid-phase polymerization of the polyester wherein granules of the polyester are heated under an inert atmosphere and agitated at an elevated temperature either at atmospheric pressure or greatly reduced pressure. Alternatively, the aromatic amine can be mixed with the polyester after it has been manufactured and granulated or otherwise comminuted to form particles. For example, the aromatic amine as a liquid or powder can be mixed with the granulated polyester. The aromatic amine can be introduced into the melt spinning apparatus and mixed with the polyester in its softened condition immediately prior to its extrusion as a fiber. Most advantageously, it has been found that excellent results can be achieved by first dissolving the aromatic amine in an inert non-reactive solvent such as methyl or ethyl alcohol, benzene, toluene, hexane, kerosene, etc., and then spraying this solution onto or otherwise mixing this solution with the polyester in its granulated or otherwise pulverized form, after which the solvent is removed from the granulated polyester leaving a coating of the aromatic amine on the polyester granules. During the subsequent melt spinning operation the polyester and the aromatic amine become thoroughly mixed because the polyester granules are

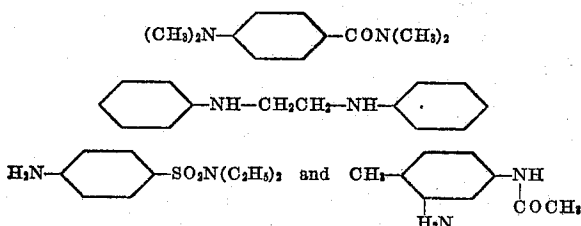
heated, fused and blended together prior to the extrusion through the spinnerette. This process appears to produce an unusually thorough blending of the aromatic amine with the polyester. Regardless of what may take place during the course of the process, the fibers which are produced have been found to be unusually receptive to interaction with an aqueous solution of the mordant salt which is then incorporated into the fibers. Of course, although this invention is primarily related to the formation of fibers, it can also be practiced in the formation of films or sheets.

Although melt spinning is the usual technique employed in forming such polyesters as are employed in accordance with this invention, it is also obvious that solution spinning or casting techniques can be similarly employed for the formation of fibers or films.

A copending application filed in the United States Patent Office, Serial No. 637,059, dated January 30, 1957 filed by the inventors herein, describes the formation of fibers having dyeability similar to those covered by the present invention, but employs the technique of introducing the mordant salt into the polyester prior to the melt spinning of the polyester whereby it is not essential to employ the amino aromatic compounds of the present invention; however, the present invention provides a highly advantageous technique which is more suitable in some circumstances, especially when the most appropriate amount of metal content for the fiber is not known at the time the fiber is spun.

By following the techniques of the present invention, it is possible to form polyester fibers which can be dyed to practical shades under ordinary conditions without the use of super-atmospheric pressures of dyeing assistants employing the metallizable dyes which are well known in the art of dyeing films, fibers, threads, yarns, fabrics, etc. These dyes are derived from synthetic and natural sources and are applied by using techniques well known and commonly employed. Many of these dyes are listed in the Technical Manual and Year Book of the American Association of Textile Chemists and Colorists such as in the 1952 edition beginning on page 168 under the heading "Alphabetical List of American-Made Dyes" including mordant dyes, mordant acid dyes, etc. Moreover, the present invention also produces polyester fibers which can be dyed with various acid wool dyes, cellulose acetate dyes and some direct cotton dyes.

The aromatic amines which can be employed include N,N'-dibutylaniline, N-ethyl-4-chloroaniline, 4,4'-methylenedianiline, 4,4'-sulfonylethanedianiline, 2,4-diaminodiphenyl, benzidine, 1,2-di(p-aminophenyl)ethane, 1,5-diaminonaphthalene, N,N,N',N'-tetramethyl-4,4'-diaminobenzophenone, etc. Other illustrative aromatic amines which can be employed include those having the following formulas:



Such aromatic compounds contain amino substituents directly connected to a benzene ring and may contain additional substituents on the nitrogen atom such as alkyl groups containing from 1 to 4 carbon atoms. Other substituents which can be connected to the benzene rings of such compounds include halogen atoms, nitro radicals, alkoxy radicals, alkyl radicals containing from 1 to 4 carbon atoms, N,N-dialkylcarboxamido radicals, N,N-dialkylsulfonamido radicals, etc. The benzene ring may be present either as one or two single rings or as a fused ring system, i.e., naphthalene. Of course, it is not intended to

include dye intermediates such as the amino derivatives of anthraquinones, etc.

It is believed apparent that there are many compounds which come within the scope of the aromatic nuclearly substituted amino compounds defined and described hereinabove. Quite obviously, these compounds are non-metallized in the sense that they do not contain chromium, cobalt, nickel, copper, or any other metal in chelated form. Moreover, it is also obvious that the essential feature of these nuclearly substituted amino aromatic compounds lies in the fact that the nitrogen atom is bonded to a carbon atom in the benzene ring. It is of little consequence whether additional substituents are present which may or may not be reactive with the polyester; however hydroxyl radicals and carboxyl radicals are disadvantageous because they tend to degrade the polyester into which the aromatic amine is to be incorporated. If the non-degrading additional substituents that are present happen to be reactive, this fact may serve to further enhance the incorporation of the aromatic amine into the polyester. Of course, any such reaction would not ordinarily destroy the existence of the incorporated aromatic ring containing a nuclearly substituted nitrogen atom. The especially advantageous compounds contemplated by this invention comprise those nuclearly substituted amino aromatic compounds employed in Examples 1, 4, 5, 6, 8 and 10 as described hereinbelow together with their closely related analogs.

The mordant salts which can be employed in treating the formed polyester fibers containing the aromatic amine are well known in the art and include the chlorides, thiocyanates, carbonates, acetates, sulfates, phosphates, monohydrogen phosphates, etc. of nickel, cobalt, chromium and copper as well as any of the well known equivalents of these salts which are commonly employed in the art of dyeing fibers, fabrics and the like with metallizable dyes. For example, the chromium salt can be present in the form of a bichromate such as ammonium bichromate.

In employing these mordant salts, they are dissolved in water which is advantageously heated to a temperature of from about 80° to about 100° C. The fibers containing the aromatic amine are then treated with the aqueous solution of the mordant salt for a sufficient period of time to allow from about 1 percent to about 10 percent (based on the weight of the fiber) of the mordant salt to be incorporated into the fiber. It is generally advantageous to carry out the treatment in the aqueous solution for about 30 to about 120 minutes. Of course, the treatment with the mordant salt can be accomplished at lower temperatures or higher temperatures and for a longer or shorter period of time. Similarly, useful results can be obtained by introducing less than 1 percent or more than 10 percent of the mordant salt into the fiber. The apparatus and processes used in mordanting fibers, threads, yarns, fabrics, etc. with salts of nickel, chromium, cobalt, copper and the like, are well known in the dyeing art and need not be further elaborated upon in this specification.

After the fiber (either as such or in the form of thread, yarn, fabric or the like) has been treated with the metal mordant salt, it can then be dyed at any suitable time by the usual methods employing any of the great variety of metallizable dyes. Such dyes are sometimes referred to as mordant or chelate dyes. The type of dye used is dependent upon the mordant present in the fiber, that is, chrome mordant type dyes are used if the fiber contains a chromium salt. These dyes are well known in the textile art and are represented by Omega Chrome Aurine GL (C.I. No. 201), Omega Chrome Brown (C.I. No. 98), Omega Chrome Red B (C.I. No. 652), and Alizarin Red S (C.I. No. 1034), etc. Suitable examples of dyes which can be employed with nickel or cobalt include those described in U.S. Patent 2,641,602, and U.S. 2,651,641, etc. Other suitable dyes are described in numerous other patents, in the publications of the American Association of Textile Chemists and Colorists as mentioned above, and in the

following copending applications: Straley and Fisher, Serial No. 458,262 filed September 24, 1954, Straley and Giles, Serial No. 466,955 filed November 4, 1954, and Straley and Wallace, Serial No. 498,112 filed March 30, 1955.

The fiber-forming polyesters with which this invention is particularly concerned include any of the known highly polymeric linear polyesters employed in the preparation of synthetic organic fibers by the melt spinning method. Thus, this invention applies to those fibers which are known in the art and which can be employed in the preparation of yarns, threads, woven fabrics, unwoven fabrics, various textile materials, ropes, etc., and which are characterized by melting points of at least 150° C. and generally 200° C. or higher and numerous other chemical and physical characteristics well known to those acquainted with the art of manufacturing synthetic organic fibers and products produced therefrom. Examples of linear polyesters which are useful in the preparation of synthetic organic fibers include the polyesters derived by the condensation of one or more of the following acids or esters thereof with one or more glycols:

- (1) Terephthalic acid with ethylene glycol,
- (2) 4,4'-sulfonyldibenzoic acid with pentamethylene glycol,
- (3) Terephthalic acid with 1,4-cyclohexanedimethanol,
- (4) 4,4'-ethylenedioxydibenzoic acid with tetramethylene glycol,
- (5) 4,4'-ethylenedioxydibenzoic acid with 2,2-dimethyl-1,3-propanediol,
- (6) Terephthalic acid and glutaric acid with 1,4-cyclohexanedimethanol,
- (7) 4,4'-diphenic acid with ethylene glycol,
- (8) Dimethylmalonic acid with 2,2-dimethyl-1,3-propanediol,
- (9) Terephthalic acid with tetramethylene glycol and neopentyl glycol,
- (10) 4,4'-sulfonyldibenzoic acid and sebacic acid with hexamethylene glycol,
- (11) Trans-1,4-dicarboxycyclohexane with 1,4-cyclohexanedimethanol, etc.

The fiber-forming polyesters of this invention are most advantageously formed from one or more of a combination of a bifunctional dicarboxy constituents selected from the group comprising 4,4'-diphenic acid, terephthalic acid, 4,4'-benzophenonedicarboxylic acid, 1,2-di(p-carboxyphenyl)ethane, 1,2-di(p-carboxyphenoxy)ethane and 4,4'-sulfonyldibenzoic acid condensed with a glycol selected from the group comprising glycols containing from 2 to 12 carbon atoms including 2,2-dimethyl-1,3-propanediol, other gem-dialkyl aliphatic glycols containing from 6 to 12 carbon atoms, 1,4-cyclohexanedimethanol, trans-quinitol, ethylene glycol, pentamethylene glycol, etc. In addition to the bifunctional dicarboxy compounds and glycols mentioned above, various aliphatic dicarboxylic acids or esters and other glycols such as the ether glycols can be introduced as modifiers into the preparation of the fiber-forming polyesters in accordance with methods known in the art. As already mentioned, the techniques for preparing a linear highly polymeric high-melting fiber-forming polyester (including copolyesters) are well known.

Even though all of the above examples illustrate high-melting, fiber-forming polyesters which can be spun to form useful fibers, it is apparent that there are others having similar utility and those skilled in the art will readily recognize the obvious equivalents of these fiber-forming polyesters. It is also to be noted that while most of these fiber-forming polyesters are derived from aromatic dicarboxylic acids, there are a few which can be derived from aliphatic dicarboxylic acids such as dimethylmalonic acid or trans-1,4-dicarboxycyclohexane and that these few exceptions to the usual rule that aromatic polyesters are generally the most satisfactory represent, of course, obvious equivalents to the aromatic

polyesters with which this invention is primarily concerned. The polyesters used in practicing this invention are prepared by the condensation of a dicarboxylic acid or ester thereof with a glycol. Batchwise or continuous

- 5 processes for preparing these polyesters by either melt-polymerization techniques or solid-phase polymerization techniques are known in the art and need no further elaboration in this specification. Generally, after such polyesters in their highly polymeric form have been produced, they are granulated. Some times the granulation is performed prior to the ultimate solid-phase polymerization. In any event, the fiber-forming polyesters are generally introduced into the melt-spinning apparatus in the form of granules. For that reason, as well as for other reasons, the most advantageous technique described above is that wherein the aromatic amine is incorporated into the polyester by dissolving it in a volatile nonreactive organic liquid to form a solution, mixing this solution with the said polyester in granulated form and evaporating the volatile liquid.

This invention can be further illustrated by the following examples of preferred embodiments although it will be understood that these examples are included merely for purposes of illustration and are not intended to limit the scope of the invention unless otherwise specifically indicated.

Example 1

Polyethylene terephthalate was granulated to a particle size of 60 mesh. One hundred grams of the polymer was stirred with a solution of 4 g. N,N'-diphenylethylenediamine in 200 cc. of methyl alcohol. The methyl alcohol was evaporated while the mixture was being stirred. This treatment deposited the diamine on the surface of the polyester granules. Fibers were spun by extruding the melted polyester through a multihole spinneret. The fibers were then drafted and heat set in the usual way. The fibers were collected in the form of a skein and treated as follows:

Twenty grams of fibers, in the form of a skein, was heated for 1 hour at 95-100° C. in 300 cc. of water containing 1.5 g. of nickel thiocyanate. After washing and drying, the fibers dyed well with nickel chelate dyes.

Example 2

Another skein of the fibers described in Example 1 weighing 20 g. was treated in the same way except that cobalt acetate was used. The fibers dyed well with cobalt chelate dyes.

Example 3

Another skein (20 g.) of the fibers described in Example 1 was boiled for 1 hour in a solution containing 1.5 g. ammonium bichromate and 2 g. formic acid dissolved in 200 cc. of water. The fibers were tinted a light green by the chromium. They were readily dyed to heavy shades with chrome mordant dyes.

Example 4

A copolyester was made having the composition 0.83 mole 4,4'-sulfonyldibenzoic acid +0.17 mole succinic acid +1.0 mole pentamethylene glycol. It was granulated to a particle size of 60 mesh and treated with an ethyl alcoholic solution of 4,4'-sulfonyldianiline to give a deposit of 3% diamine, based on the polymer. Fibers were spun by the melt process. A skein of the fibers (100 g.) was boiled for 1 hour in 1200 cc. of water containing 5 g. of sodium bichromate and 6 g. of lactic acid. The fibers dyed well with chrome mordant dyes.

Example 5

Polyethylene terephthalate was made by the usual melt polymerization method (see U. S. 2,465,319 patented March 22, 1949). At the end of the reaction, 4% by weight of 1,2-di(p-aminophenyl) ethane was added to the melt. After the amine had been thoroughly mixed, the melt was cooled and granulated. Fibers spun from the polymer dyed well with chrome mordant dyes after

they had been treated with bichromate as described above. The fibers dyed well with nickel chelate dyes after they had been treated with a nickel salt such as thiocyanate.

Example 6

A polyester made from 1,2-di(p-carboxyphenoxy) ethane and ethylene glycol was granulated to 60 mesh. Four percent by weight of N-ethyl-4-chloroaniline was deposited on the granules by evaporation of an alcohol solution. Fibers were spun and drafted by the usual methods. A skein of the fibers (20 g.) was boiled in 200 cc. of water containing 1.2 g. of sodium bichromate and 1.0 g. of tartaric acid. The fibers dyed well with chrome mordant dyes.

Example 7

Another skein of the fibers (20 g.) described in Example 6 was heated at 90-95° C. for 45 minutes in 200 cc. of water containing 2 g. of nickel thiocyanate. The fibers dyed well with nickel chelate dyes.

Example 8

Five percent N,N,N',N'-tetramethyl-4,4'-diaminobenzophenone was incorporated in polyethylene terephthalate as described in Example 5. Fibers were spun and drafted by the usual methods. Treatment of the fibers with nickel, copper, chromium, and cobalt salts gave products that dyed well with acid wool dyes and mordant dyes of the types described hereinabove, e.g. Acid Anthracene Red 3B (C.I. 487), Acid Anthracene Yellow GR (C.I. 187), Acid Blue B (C.I. 707), Acid Fast Violet BG (C.I. 695), Acid Orange ROS (C.I. 161), Xylene Light Yellow 2G (C.I. 639), Neolan Black ALU (Pr. 143), Neolan Blue GG (Pr. 144), Neolan Orange GRE (Pr. 463), Neolan Pink BA (Pr. 326).

Example 9

A polyester was prepared from terephthalic acid and 1,4-cyclohexanedimethanol as described in the copending application of Kibler, Bell and Smith, Serial No. 554, 639 filed December 22, 1955. This polyester was granulated to 60-mesh and 100 grams of the granulated polyester was sprayed with a solution of 6 grams of N,N'-diphenylethylenediamine dissolved in 300 cc. of ethyl alcohol. The ethyl alcohol was then evaporated and the dry granules were then melt-spun in the usual manner. A skein of fibers (20 g.) was heated in 300 cc. of water containing 2.2 grams of nickel acetate. The treated fibers dyed well with nickel chelate dyes. Similar results were obtained employing a polyester prepared from trans 1,4-cyclohexanedicarboxylic acid and 1,4-cyclohexanedimethanol as described in the above-mentioned Kibler et al. application.

Example 10

A copolyester was made having the composition 0.5 mole 4,4'-sulfonyldibenzoic acid +0.5 mole azelaic acid +1.0 mole pentamethylene glycol. One hundred grams of the polyester and 5 g. of 4,4'-methylenedianiline were dissolved in 600 cc. of tetrachloroethane and films were coated from the solution. After drying, the films were heated for 1 hour at 95-100° C. in 2% water solutions of the following salts: (a) copper chloride, (b) cobalt chloride, (c) nickel thiocyanate, and (d) ammonium bichromate. The films were rinsed, dried, stretched lengthwise and widthwise and heat-set. All of the films dyed to some extent with acid wool dyes, the deepest color being obtained on sample (a). Samples (b) and (c) dyed to deep shades with the nickel and cobalt chelate dyes described in U.S. Patents 2,641,602 and 2,651,641. Sample (d) was tinted to a pale green, showing that the ammonium bichromate had been reduced to the trivalent state within the film structure. The film dyed well with chrome mordant type dyes.

Although the invention has been described in considerable detail with reference to certain preferred embodiments thereof, it will be understood that variations and modifications can be effected without departing from

the spirit and scope of the invention as described hereinabove and as defined in the appended claims.

We claim:

1. A process for preparing polyester fibers which can be readily dyed with metallizable dyes comprising the following steps, (1) forming a mixture of (A) a linear, highly polymeric, condensation polyester of (a) at least one bifunctional dicarboxylic acid comprising at least 50 mole percent of an acid selected from the group consisting of terephthalic acid, 4,4'-sulfonyldibenzoic acid, 4,4'-benzophenonedicarboxylic acid, 4,4'-ethylenedioxydibenzoic acid, 4,4'-ethylenedibenzoic acid, 4,4'-diphenic acid and trans-1,4-cyclohexanedicarboxylic acid and (b) at least one bifunctional glycol containing from 2 to 12 carbon atoms, said polyester being fiber-forming and having a melting point of at least 150° C., with (B) a non-metallized aromatic compound having from 6 to 20 carbon atoms and from 1 to 2 carbocyclic rings, which aromatic compound is entirely composed of carbon atoms, hydrogen atoms, at least one amino group directly connected to a benzene ring and only those other substituents selected from the group consisting of (a) halide atoms, (b) nitro radicals, (c) alkoxy radicals, (d) carbonyl radicals, (e) hydrocarbon radicals, (f) sulfonyl radicals each of which substituents is directly connected to a benzene ring, and (g) hydrogen atoms, said mixture containing from about 2 to about 10% by weight of said compound (B), (2) forming a fiber of said mixture of (A) and (B) and (3) bringing said fiber into intimate contact with an aqueous solution of a water-soluble mordant salt of a metal selected from the group consisting of chromium, cobalt, nickel and copper, said contact being at a temperature of from about 80° C. up to the boiling point of said aqueous solution whereby there is formed a fiber which can be readily dyed with a metallizable dye, said fiber containing from about 1% to about 10% based on the weight of the fiber of said mordant salt.
2. A process as defined by claim 1 wherein (A) is composed of polyethylene terephthalate.
3. A process as defined by claim 1 wherein (A) is composed of poly-1,4-cyclohexanedimethylene terephthalate.
4. A process as defined by claim 1 wherein said mixture of (A) and (B) is performed by dissolving said compound (B) in a volatile non-reactive organic liquid to form a solution, mixing this solution with the said fiber-forming polyester (A) in granulated form and evaporating the volatile liquid.
5. A process as defined by claim 1 wherein (A) is composed of polyethylene terephthalate, (B) is N,N'-diphenylethylenediamine and said mordant salt is nickel thiocyanate.
6. A process as defined by claim 1 wherein (A) is composed of 4,4'-sulfonyldibenzoic acid polyester, (B) is 4,4'-methylenedianiline and said mordant salt is nickel thiocyanate.
7. A process as defined by claim 1 wherein (A) is composed of terephthalic acid polyester with 1,4-cyclohexanedimethanol, (B) is 4,4'-methylenedianiline and said mordant salt is nickel acetate.
8. A process as defined by claim 1 wherein (A) is composed of 4,4'-sulfonyldibenzoic acid polyester, (B) is 4,4'-sulfonyldianiline and said mordant salt is sodium dichromate.
9. A process as defined by claim 1 wherein (A) is composed of polyethylene terephthalate, (B) is 1,2-di(p-aminophenyl) ethane and said mordant salt is sodium dichromate.

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