This invention relates to a process for dyeing linear polyester fiber, particularly of linear condensation polymers of terephthalic acid and a polyethylene glycol, especially ethylene glycol (commonly known as “Fiber V”), with vat dyestuffs.

Polyester fibers of the aforesaid type, such as Fiber V, have very poor affinity for dyestuffs. The best colorations thus far produced thereon have been with aqueous dispersions of water-insoluble dyestuffs having affinity for cellulose acetate. However, these dyestuffs produce only light or medium shades of poor fastness properties, especially in their resistance to light and washing.

It has also been proposed to pad dispersions of pigment dyestuffs on Fiber V, followed by heat treatment, e.g. by immersion of the padded material in molten metal for a short period of time at temperatures of the order of 400°F. While an improvement in fastness is obtained by this treatment, the depth of shade produced remains seriously limited for most dyestuffs, so that no adequate range of shades can be obtained. Vat dyestuffs, when applied in the form of an aqueous alkaline vat, fail to exhaust satisfactorily on Fiber V.

It is an object of this invention to produce colorations with vat dyestuffs on Fiber V and similar polyester fibers in a full range from light to dark shades, having excellent fastness to light and washing.

We have discovered that polyester fibers such as Fiber V can be dyed with vat dyestuffs in dark as well as medium and light shades by impregnating (e.g. padding) the fiber or textile material with an acidified vat (i.e., an acid-reacting aqueous colloidal dispersion of the leuco compound of a vat dyestuff, as produced, for example, by acidifying an aqueous alkaline vat as ordinarily applied for producing vat colorations on cellulose fibers and containing a dispersing agent which is effective in the presence of acids), and subjecting the material, thus padded or impregnated, to heat treatment at a temperature from 300 to 400°F. for a short period of time. The vat dye coagulation is then developed by oxidation in the usual manner, e.g. by passing the material through an aqueous oxidizing bath, or by exposure (especially while wet) to atmospheric oxygen, whereby shades are obtained of any desired depth, having excellent fastness to light and washing.

The best treatment of the textile material padded with an acid leuco vat dye dispersion in accordance with this invention can be carried out by radiation (e.g. by exposing the material to infrared rays), by convection (e.g. by exposure of the material to circulating hot air or other gases), or by conduction (e.g. by passage over a heated surface or immersion in a heated water-immiscible liquid). Advantageously, the material is subjected to heat treatment in a curing range employing infrared rays, circulating hot gases, or heated rollers, or by temporarily immersing the material in, or passing it through a molten metal bath maintained at the desired temperature. The padding operation and heat treatment can be conveniently carried out in a continuous manner on piece goods, warps, slubbing and the like, by progressively passing such material through padding equipment and a heating zone or apparatus.

The material can be predried after the padding operation and before the heat treatment, or drying can be effected in the course of the heat treatment. The effective heating period at the temperature range specified above is generally limited to that portion of the heating after evaporation of moisture carried by the material.

Vat dyes of the quinonoid and indigoid series can be used in the process of this invention. Acid aqueous dispersions of the corresponding leuco compounds as employed for padding the polyester fiber in accordance with the invention are prepared by vatting the dyestuff in an aqueous alkaline solution with an alkali metal hydroxide or equivalent reducing agent, incorporating therein a dispersing agent effective in acid solution, and acidifying the resulting alkaline vat to adjust the pH to the range of 2 to 5. Suitable dispersing agents are, especially, surface-active water-soluble sulfonic acids such as alkyl aryl sulfonates, e.g. higher alkyl benzene sulfonates, sulfonated condensation products of naphthalene and formaldehyde or their homologs, isopropyl- and isobutyl-naphthalene sulfonates, and aliphatic or alicyclic sulfonates such as N-ctyle-N-methyl taurine, higher alky l esters (e.g. the dioctyl ester) of sulfosuccinic acid, sulfonated rosin or its derivatives, and lignin sulfonates.

Acids employed for acidification of the alkaline vat are preferably organic acids such as acetic or formic acids, although other acids can be used to adjust the pH to the aforesaid range.

When the padded material is heat treated by immersion in, or passage through, a molten metal bath, metals or alloys suitable for use are those melting substantially below the temperature of the heat treatment, and particularly below the
boiling point of water. The latter metals and alloys can be used without difficulty from local solidification of the metal, not only in the treatment of predried materials but also with moist predried materials. A suitable material for this purpose is Wood's metal, composed of 50% bismuth, 25% lead, and 12.5% each of tin and cadmium. Other bismuth-lead alloys containing tin and/or cadmium are also known having melting points below the boiling point of water, and can be used in place of Wood's metal.

Oxidizing treatment of the material to develop the coloration thereon, following the heat treatment, is carried out according to conventional methods, e.g., by exposure while moist to atmospheric oxygen or by passage through an aqueous solution of a suitable oxidizing agent.

Preferred embodiments of the process according to the invention are illustrated in the following examples, wherein parts and percentages are by weight.

Example 1

10 parts of Indanthrene Brilliant Pink R Paste (Colour Index No. 1210) were vatted with 2.5 parts of caustic soda and 2.75 parts of sodium hydroxysulfite in about 200 parts of water, having dissolved therein 3 parts of a higher alkyl benzene sodium sulfonate dispersing agent. 15.3 parts of aqueous 28% acetic acid were added to acidify the vat. The mixture was then diluted with water to a volume equal to that of 250 parts of water. Fiber V piece goods were padded with the resulting leuco vat dye suspension at 160°F and dried. The material was then heated in a curing range at 350°F for 5 minutes, and then scoured in an aqueous detergent solution, oxidation of the dyestuff to develop its coloration being effected by atmospheric oxygen. A brilliant red shade was produced having excellent fastness to light and washing.

Example 2

10 parts of Indanthrene Brilliant Pink R Paste, employed in Example 1, were vatted with 1.7 parts of caustic soda and 2 parts of sodium hydroxysulfite in about 50 parts of water containing 1 part of a sulfonated formaldehyde-naphthalene condensation product as a dispersing agent. 11.4 parts of 28% acetic acid, diluted with 200 parts of water, were added to acidify the vat, and sufficient water was added to adjust the volume of the resulting dispersion to that of 150 parts of water. Fiber V stubbing was padded with the resulting aqueous leuco vat dyestuff dispersion at 180°F, the material retaining about 100% of its weight of the padding liquor. The material was then passed, while wet, through a Wood's metal bath maintained at 350°F, the duration of the immersion in the metal bath being 2 minutes. As the material emerged from the bath, steam escaped, the fiber being left practically dry. The vat dye coloration was developed by passage through a 1% aqueous solution of potassium persulfate. A brilliant dark red shade was obtained having excellent fastness to light and washing, the coloration being much brighter than that produced with the same dyestuff on cotton or wool.

Example 3

10 parts of Indanthrene Brilliant Violet RR Paste (Colour Index No. 1104) were vatted with 2.5 parts of caustic soda and 2.5 parts of sodium hydroxysulfite in about 200 parts of water containing 5 parts of the sulfonated formaldehydene-naphthalene condensation product of the preceding example. The alkaline vat was acidified by adding 16.6 parts of 28% acetic acid, and the resulting liquor was diluted with water to increase its volume to that of 350 parts of water. Fiber V piece goods was padded with resulting liquor at 180°F, and then dried. The padded material was heat treated in a curing range for a period of 5 minutes at 350°F, and then scoured in an aqueous detergent solution, oxidation being effected by atmospheric oxygen. A brilliant violet coloration was produced having excellent fastness properties, as obtained in the preceding examples.

Example 4

5 parts of indigo were padded with 2 parts of sulfonated caslor oil, and vatted with 5 parts of caustic soda and 7.5 parts of sodium hydroxysulfite in the presence of 1 part of sulfonated naphthalene-formaldehyde condensation product in about 50 parts of water, and the resulting acid vat was diluted with sufficient water to render its volume equal to that of 150 parts of water. Fiber V piece goods was padded with the resulting acid vat, and then passed, while wet, through a Wood's metal bath maintained at 350°F, duration of immersion in this bath being 2 minutes. Upon oxidation in an aqueous oxidizing bath as hereinafter described, a deep navy blue shade of unusual brilliance was obtained, having excellent fastness properties and a much brighter color than shades produced with indigo on wool or cotton.

Example 5

4 parts of Indanthrene Brown R (Colour Index No. 1191) were vatted with 2.5 parts of caustic soda and 2.5 parts of sodium hydroxysulfite in about 50 parts of water containing 1 part of sulfonated naphthalene-formaldehyde condensation product. The solution was acidified by adding 15.5 parts of 28% acetic acid diluted with about 50 parts of water, and the volume of the resulting acidified vat was adjusted by dilution with water to a volume equivalent to that of 150 parts of water. Fiber V material was padded with the resulting acid bath and passed, while wet, through molten Wood's metal maintained at 350°F, the duration of immersion in the metal bath being 2 minutes. Upon subsequent oxidation in an aqueous persulfate bath, the material was dyed a deep brown shade of excellent fastness to light and wet processing.

Example 6

10 parts of Indanthrene Brilliant Green B Double Paste (Colour Index No. 1101) were vatted with 2.5 parts of caustic soda and 2.5 parts of sodium hydroxysulfite in about 300 parts of water containing dissolved therein 5 parts of sulfonated formaldehyde-naphthalene condensation product. The solution was acidified by adding 16.6 parts of 28% acetic acid, and diluted with water sufficient to render the volume equal to that of 250 parts of water. Fiber V material was padded with the resulting acid vat, and after drying, was passed through a curing range in which it was heated at 350°F, for 5 minutes. After scouring with an aqueous detergent bath, a full bright green shade was produced having excellent fastness to washing and light processing.

Instead of the vat dyes employed in the examples, which are representative of those of the indigoil, thiodigold, anthraquinone, and high-
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er polycyclic quinone vat series, other vat dye-
uffs of this and related classes can be applied in
a similar manner. The vat dyeuffs are ad-
antageously added in aqueous alkaline solution
by treatment with an alkaline metal hydroxystate
or with other reducing agents heretofore em-
ployed for preparing alkaline vats. A dispersing
agent which is active in acid solution, e. g. of
the type employed in the examples, or other
surface-active sulfonic acids, is added to the alkaline
vat to preserve dispersion of the free vat dye-
stuff leuco compound which is formed upon
acidification. Acidification is accomplished by
adding an acid, as described above, so as to ad-
just the pH to 2 to 5.
The resulting acid vat is advantageously padded
on the material, especially at tempera-
tures from 100 to 200° F., the amount of padding
liquor retained by the goods being of the same
order as the weight of the fabric. As indicated
above, the padded material can be dried before
heat treatment or in the course of heat

treatment. The heat treatment of the invention in-
volves heating the material at 300 to 400° F., and
maintaining it at this temperature for 25
seconds to 5 minutes, especially in a curing range
or a molten metal bath.

Development of the coloration by oxidation
can be effected by atmospheric oxygen, e. g., in
the course of an aqueous scouring operation fol-
lowing the aforesaid heating treatment, or by
passing the material through an oxidizing bath
such as aqueous hydrogen peroxide, alkalai metal
per sulphates, bichromates and similar compounds.
The colorations produced are generally brighter
than those produced with the same dye on cot-
ton or wool, and can be made of any desired depth
of shade. The fastness of the resulting colora-
tion to light and washing is excellent, even in
the case of deep shades.
The polyester fibers which are dyed with vat
dyesuffs in accordance with this invention are
linear condensation superpolymers of tereph-
thalic acid (or an ester-forming functional de-
rotive thereof) with a polymethylene glycol
(preferably, ethylene glycol) having up to 10
methylenegroups in a chain interconnecting a
pair of terminal hydroxy groups. The fiber is
produced by forming the polyester into filament
and stretching, whereby axial molecular orienta-
tion occurs, as indicated by production of well
defined X-ray fiber diagrams. The resulting fiber
is strong, flexible, and of high melting point and
low solubility in water and most organic solvents.

Variations and modifications, which will be
obvious to those skilled in the art, can be made
in the procedure hereinafter described without
departing from the scope or spirit of the inven-
tion.

We claim:
1. A process for dyeing synthetic linear polye-
ester fiber material, which comprises impregnat-
ing said material with an acid-reacting aqueous
dispersion of the leuco compound of a vat dye,
and heating the impregnated material at a tem-
perature of 300 to 400° F. for a period ranging
from 25 seconds to 5 minutes, prior to develop-
ment of the coloration by oxidation of the leuco
compound on the fiber.
2. A process for dyeing synthetic linear poly-
esther fiber material, which comprises padding
said material with an aqueous dispersion of the
leuco compound of a vat dye having a pH of 2 to
5, at a temperature of 100 to 200° F., and heat-
ing the impregnated fiber at a temperature from
500 to 400° F. for a period ranging from 25
seCONDS to 5 minutes, prior to development of
the coloration by oxidation of the leuco compound
on the fiber.
3. A process as defined in claim 2, wherein
the padded material is dried, prior to the heat treat-
ment, at temperatures from 300 to 400° F.
4. A process as defined in claim 2, wherein
the heat treatment is effected by immersing the ma-
terial in a molten metal bath maintained at a
temperature from 300 to 400° F., the duration of
the immersion ranging from 25 seconds to 5
minutes.
5. A process as defined in claim 2, wherein
the aqueous dispersion of the vat dye leuco com-
pound is prepared by acidifying an aqueous alka-
line vat of the vat dyeuff, containing an organic
sulfonate dispersing agent, with an organic acid
in sufficient amount to yield a pH of 2 to 5.
6. A process as defined in claim 2, wherein
the material is progressively padded with the aqueous
acidic leuco vat dye dispersion, and progressively
exposed to a heating medium, the duration of said
exposure being such as to maintain the tem-
perature of the material at 300 to 400° F. for 25
seconds to 5 minutes.
7. A process for dyeing textile fiber of a poly-
esther of terephthalic acid and ethylene glycol with
a vat dyeuff, which comprises padding said
textile fiber with an aqueous dispersion of a vat
dye leuco compound, obtained by acidifying an
aqueous alkaline vat of the vat dye containing
an organic sulfonate dispersing agent to a pH
of 2 to 5, exposing the padded material to a heat-
ing medium so as to maintain the temperature
of the material at 300 to 400° F. for a period of 25
seconds to 5 minutes, and exposing the heat
treated material to an oxidizing agent to con-
vert the vat dye leuco compound to the corre-
sponding vat dye on the fiber.

HANS LUTTERINGHAUS,
HENRY R. MAUTNER,
ALEX A. ARCUS.

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