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VAT DYE COMPOSITION

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This invention relates to dye compositions and particularly to vat dye compositions in the form of dye pastes or powders, which are especially adapted for use in the preparation of aqueous solutions or dispersions for coloring textile materials and the like. The vat dye pastes or powders are particularly valuable for use in the preparation of printing pastes and padding baths. The invention also includes processes for making the vat dye compositions.

In the art of dyeing with vat dyes, two general methods are ordinarily employed. In accordance with one method, a vat comprising an alkaline solution of a reduced vat dye is prepared and the fibrous material to be dyed is worked in this solution whereby the fiber takes up the reduced compound of the dye. The material is then subjected to oxidation and other finishing treatments to convert the vat dye from the reduced to the unreduced form and further complete the fixing of the dye and finishing of the material. In accordance with the other method the dye is applied to the material to be dyed while in the unreduced form; it is then subjected to a reducing treatment whereby the dye is converted to the soluble form in the presence of the fiber and is then taken up by the fiber. It is then subjected to oxidation and/or other finishing treatments to fix the dye on the fiber, remove undesired residual material, and complete the finishing of the material.

In dyeing with vat dyes by the latter method, one of two procedures is usually followed, depending upon whether the material is to be dyed a solid color or with a pattern in one or more colors. If the material is to be dyed a solid color, the pigment padding (pad and jig) process is ordinarily employed, and if the material is to be dyed with a pattern, the printing process is generally utilized.

In the pigment padding process, the unreduced vat dye in finely-divided form is suspended in a suitable suspension medium, usually water containing added materials such as thickeners and alkaline substances. The material to be dyed is passed through this suspension, the fiber mechanically picks up the particles of insoluble dye, and the material is subsequently worked in a reducing bath whereby the dye is reduced to the soluble form and is absorbed and/or adsorbed by the fiber. The material is finally subjected to oxidation and finishing treatments to reconvert the dye to the insoluble form, fix it on the fiber, and further complete the finishing of the material.

The printing process of dyeing a textile fabric generally involves applying a so-called printing paste to the fabric in the form of a design (e. g., by means of a screen, a stencil, or an engraved roll), and then subjecting the printed fabric to further treatment to fix the dye. In general, vat dye printing pastes, in addition to the unreduced vat dye, contain substances which in themselves do not reduce the vat dye under the conditions of the printing operation but which in subsequent operations react with the dye to convert it to the soluble form (such as sodium formaldehydesulfoxylate and potassium carbonate), other substances or assistants which promote the reduction of the dye and/or absorption of the reduced compound by the fiber, and suitable gums or thickeners. The fixing treatment usually comprises subjecting the printed fabric to a so-called "steaming" or "ageing" treatment to induce reduction of the vat dye by the action of the reducing agent present on the fabric, and cause the reduced dye to be taken up by the fiber. The fabric is then subjected to oxidizing, washing, and other finishing treatments.

In dyeing with the vat dyes, particularly by the pigment padding or printing processes, the success of the operation is in a large measure dependent upon the uniformity with which the vat dye (e. g., the unreduced vat dye) is applied to the fabric. The vat dyes in unreduced form, as is well known, are insoluble in water and are not readily wetted by water. In preparing a padding bath or printing paste it is important that the vat dye be in finely-divided form, that it be uniformly distributed throughout the bath or paste, and in condition such that it may be taken up by the fiber. Further, it is important that the vat dye be capable of rapid and substantially complete reduction to the reduced form. The vat dyes, especially those which are intended for use in the pigment padding and printing processes, are generally marketed in the form of so-called dye pastes or color pastes, and dye powders. A dye paste of this kind is usually an aqueous mixture or suspension of finely-divided vat dye, which generally makes up 10 to 20 per cent of the paste. The paste usually contains a small amount of a dispersing agent such as "Leukanol" (a condensation product of formaldehyde and a naphthalene sulfonic acid) and the remainder is mainly water, a part of which is sometimes replaced by other liquids such as amino alcohols, polyhydroxy alkyl ethers or other polyhydroxy alkyl compounds, and cyclic ethers; as, for example, glycol ethers, glycol, glycerine, poly-glycerines, poly-glycols, etc. These vat dye pastes are ordinarily produced by stirring the dispersing and other agents with the filter cake of the dye resulting from filtration of a precipitate of the dye obtained in its manufacture, or by precipitation in the finely-divided form from a vat or sulfuric acid or other solution.

As noted above, vat dyes for use in the preparation of padding baths and printing paste may be supplied in the form of vat dye powders. In the case of the powders, it is important that the vat dyes contained therein not only have the properties pointed out above in connection with the vat dye pastes, but also that the powders be rapidly converted to uniform suspensions of the vat dye. Especially successful vat dye powders are described in my United States Patents Nos. 2,067,926 and 2,145,193. These vat dye powders are prepared by incorporating with the usual vat dye paste, e. g., one containing a small amount of a dispersing agent) a soluble salt of an acid alkyl ester of an oxygen-containing polybasic inorganic acid (e. g., a soluble salt of the sulfuric acid ester of a lower aliphatic alcohol) and preferably also an added substance such as, for example, dextrine, sugar, gum arabic, and the like, and then drying and grinding the resulting composition.

It is the principal object of the present invention to provide vat dye compositions which are productive of excellent dyeings and printings when employed in the usual ways. Another object of the invention is to provide vat dye pastes and powders which are adapted for use in the efficient preparation of padding baths and printing pastes containing uniformly distributed therethrough the finely-divided particles of the vat dyes. Another object of the invention is to provide vat dye pastes and powders which when used in the preparation of padding baths and printing pastes are productive of compositions characterized by the marked efficiency with which the dye is initially taken up by the fiber and the increased brilliancy and strength of the resulting dyeings and printings as compared with the dyeings and printings obtained by the use of padding baths and printing pastes prepared from ordinary vat dye pastes. A further object of the invention is to provide vat dye pastes which remain fluid on standing and which may be easily and quickly mixed with the ingredients making up the conventional padding baths and printing pastes to form homogeneous compositions containing the vat dyes in well-dispersed condition.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The vat dye compositions of the present invention consist essentially of a vat dye compound in the unreduced or other form and a soluble organic ester of a saturated aliphatic mono-carboxylic acid containing not more than 8 aliphatic carbon atoms joined carbon to carbon. The esters that have been found to be adapted for use in the compositions of the invention contain at least one sulfate, sulfonate, or phosphate group, and their ester groups are free from aliphatic radicals containing more than a total of 12 aliphatic carbon atoms per aliphatic radical and from straight carbon chains of more than 8 aliphatic carbon atoms. In speaking of soluble esters it is meant esters that are soluble in water and/or in alkaline solutions. The invention is based upon the discovery that esters of this class possess a combination of properties which make them valuable for use in compositions containing vat dyes, and especially in vat dye compositions in paste or powder form intended for use in the preparation of padding baths or printing pastes.

In preparing a vat dye paste in accordance with the present invention, a vat dye, usually in the form of a press cake (a mixture of a vat dye in finely-divided form with water as obtained in the course of manufacture of the dye) is mixed with an ester of the above class and also preferably with a dispersing agent (for example, "Leukanol" or sulfite waste liquor).

The resulting mixture is then diluted with water to the desired dye-strength; or if the paste is to be a so-called non-drying paste, that is to say, one in which the water is replaced in whole or part by a water-miscible, high-boiling alcohol such as isobutyl alcohol, glycerine, glycol, polyglycerines, polyglycols, and alkyl ethers of these compounds, the requisite amount of such alcohol is added to the mixture and water is removed therefrom by evaporation until the paste has the desired dye strength. The compositions prepared in this way comprise fluid pastes consisting of dispersions of the finely-divided vat dye. The compositions are relatively stable and do not settle on standing over a relatively long period of time. Furthermore, as compared with many available vat dye pastes, they are in a fluid or free-running state. Thus they are adapted to be efficiently and quickly incorporated with the other ingredients of padding baths and printing pastes to prepare such baths and pastes in a form such that the resulting compositions may be efficiently employed in dyeing and printing.

When preparing the vat dye compositions of the present invention in the form of vat dye flakes, scales, grains, powders, and the like, the pastes prepared as described above, but usually with little or no high-boiling water-miscible alcohol therein, may be concentrated and/or dried, as by means of a vacuum or atmospheric rotary drum drier, by spray drying, or by means of the addition to the paste of an anhydrous hydrate-forming inorganic salt. It is preferable, however, when preparing the powders to add to the paste before drying a substance such as sugar, dextrine, gum or starch, or derivatives of alginic acids and/or other suitable additions. In preparing the dry products employing a mechanical drier, the dry product scraped from the drier is in the form of flakes, scales or grains, and may be used as such, but may also be ground to produce a finely-divided powder.

The esters employed in the vat dye compositions of the present invention are such as are derivable from any saturated aliphatic monocarboxylic acid containing less than 9 carbon atoms (such as, for example, acetic acid, propionic acid, normal and isobutyric acids, normal and isocaproic acids, normal and isovaleric acids, normal and isoheptylic acids, caprylic acid, their isomeric acids, etc.), and an organic hydroxy compound, especially an alcohol, containing not more than 8 aliphatic carbon atoms in a straight carbon chain and a total of not more than 12 aliphatic carbon atoms. The aliphatic monocarboxylic acids from which the esters are derivable may contain substituents; such as, for example, halogens, hydroxyl, amino, and aromatic radicals.

The said esters may be derivatives of open-chain aliphatic monohydric or polyhydric alcohols, or of mono- or polyhydric hydroaromatic alcohols, cycloaliphatic alcohols, heterocyclic alcohols, aralkyl alcohols, mixed aliphatic-hydroaromatic alcohols, or of mono- and polyhydric alcohol ethers. As examples of such alcohols there may be mentioned ethyl, propyl, allyl, butyl, amyl, hexyl, heptyl, octyl, and the like alcohols; cyclohexanol, methyl cyclohexanol, cyclopentanol, tetrahydrofurfuryl alcohol, benzyl alcohol, phenyl ethyl alcohol, phenyl, methyl carbinol, the various terpene alcohols; ethylene, propylene, di-ethylene, and triethylene glycols, and the like; and the methyl and ethyl ethers of ethylene, di-ethylene, or other glycols. As typical alcohols there may be also mentioned the following alcohols which are obtainable as by-products in the production of methanol by the catalytic hydrogenation of mixtures of carbon monoxide and carbon dioxide: 4-methyl-1-pentanol; 2-methyl-1-pentanol; 2,4-dimethyl-1-pentanol; 3-methyl-2-pentanol; 2,4-dimethyl-3-pentanol (di-iso-propyl-carbinol); 2-methyl-3-pentanol; 4-methyl-1-hexanol; 2,4-dimethyl-1-hexanol; 2,5-dimethyl-3-hexanol; 2-ethyl-1-butanol; and others of like character.

The above parent alcohols may be primary or secondary but all of them are characterized by containing no straight carbon chain of more than 8 aliphatic carbon atoms. The esters derived from alcohols wherein the direct chain between the esterified alcohol function and the nearest branching point in the ester group is not longer than 4, or at most 6 carbon atoms, are particularly valuable for use in the vat dye compositions.

Where in the appended claims an ester of an alcohol and an aliphatic monocarboxylic acid is referred to it will be understood that an ester derivable from an alcohol selected from any of the above described classes and an aliphatic monocarboxylic acid is intended, unless otherwise indicated.

It will be understood that the alcohols from which the esters are derivable may contain substituents which do not alter the essential physical or chemical characteristics of the esters. As indicated above, polyhydric alcohols may be employed and in this case either one or both of the hydroxyl groups may be esterified with the carboxylic acid group of the acid employed. When only one of the hydroxyl groups is esterified, one or more free hydroxyl groups remain, which are not objectionable and in some cases appear to have an advantageous effect upon the properties of the esters. The amino group may also be mentioned as an example of a substituent which may be present in the alcohol residue of the esters, valuable esters adapted for use in the vat dye compositions being derivable from amino alcohols.

It has been found that the best results are obtained with esters of the above class which contain sulfonate and/or sulfate groups and which display high solubility in aqueous solutions and/or alkaline printing pastes. Accordingly, the preferred esters are those which are derived from lower aliphatic alcohols (i. e., containing 2 to 6 carbon atoms), cycloalkyl alcohols, and aromatic alcohols. It has been found that esters which are derivatives of isobutyl alcohol are of markedly high value. For similar reasons, the esters derived from the shorter chain-length aliphatic monocarboxylic acids (i. e., containing 2 to 4 carbon atoms) are of particular value. Acetic acid is preferred as the aliphatic monocarboxylic acid. While preferred esters contain one or more sulfonate and/or sulfate groups, it has been found that those esters which contain at least one sulfonate group are especially valuable. Further, although the solubilizing group of the sulfonated and/or sulfated esters may be in the ester radical, the acyl radical, or both, it is preferred to employ such esters in which the solubilizing group, preferably a sulfonate group, is in the acyl radical and the ester radical is free from solubilizing groups.

In view of the foregoing disclosure, it will be readily apparent that a large number of esters are available for use in the compositions of the invention. The following esters (described in the form of the free acids of the inorganic acid residues contained therein) are given as examples of typical esters of the class hereinabove disclosed: isobutyl sulfoacetate; n-amyl-beta-sulfo-beta-furylpropionate; n-amyl sulfoacetate; isobutyl sulfatobutyrate; isobutyl phosphatobutyrate; C-phenyl-isobutyl sulfopropionate; normal-hexyl sulfoacetate; cyclohexyl-sulfoacetate; normal-amyl sulfatobutyrate; 4-methyl-1-pentyl sulfoacetate; isobutyl sulfobutyrate; 2-methyl-1-pentyl sulfobutyrate; 2,4-dimethyl-1-pentyl sulfoacetate; tri-isopropylcarbinol sulfoacetate; glyceryl-di-(sulfoacetate); 4-methyl-1-hexyl sulfoacetate; monoethyl-ether of triethylene glycol sulfoacetate; triethylene glycol sulfoacetate; triethylene glycol sulfatoacetate; mono-acetylglycerine sulfoacetate; methyl-cyclohexyl sulfoacetate; tetrahydrofurfuryl sulfobutyrate; tetra-hydrofurfuryl-aminoethyl sulfoacetate; phenyl-methyl-carbinol sulfoacetate; secondary octyl sulfoacetate; octyl sulfoacetate; the sulfuric acid esters or phosphoric acid esters of the glyceryl esters of acetic acid; the glyceryl esters of glycollic or lactic acid which are sulfated or phosphated in the glyceryl radicals; the sulfoethyl ester of beta-hydroxyethylamidopropionic acid (the sulfoethyl ester of N-hydroxyethyl succinamic acid); sulfatopropyleneglycol acetate; sulfatoethyleneglycol butyrate; and the monosulfate of glycerine monosulfoacetate.

The esters employed in the present invention may be prepared in any well-known or suitable manner. For example, the sulfated esters may be prepared by reacting the ester of the corresponding unsaturated aliphatic acid with concentrated sulfuric acid or the ester of the corresponding hydroxy-aliphatic acid with chlorsulfonic acid, and neutralizing the resulting product with the desired inorganic or organic base. The sulfonated esters may be prepared by reacting the ester of the corresponding halogenated aliphatic acid with sodium sulfite.

The esters are preferably employed in the form of their neutral soluble salts. The alkali-metal salts, and the salts obtained by neutralizing the acid esters with organic derivatives of ammonia, are especially important. As examples of suitable organic derivatives of ammonia, the following are mentioned: the alkylolamines (e. g., mono-, di-, or triethanolamine); the alkylol diamines (e. g., 1,2-di-(2′-ethanolamino)-ethane and 1,3-di-(2′-ethanolamino)-2-propanol); the quaternary alkylolamines, the di-quaternary alkylolamines (e. g., tetra-hydroxyethylamine ammonium hydroxide, hexa-hydroxy-ethyl-ethylene diammonium hydroxide); and the quaternary aralkyl-alkyl ammonium hydroxides or the aralkyl-hydroxyalkyl-ammonium hydroxides (e. g., tri-methyl-benzyl ammonium hydroxide, tri-(hydroxy-ethyl)-benzylammonium hyroxide, etc.).

The valuable effects resulting from the inclusion of the above described esters in vat dye compositions in accordance with the present invention are noticed both in vat dye pastes and in powders from which dyeing compositions (baths and printing pastes) are prepared and also in the dyeings and printings obtained with the ultimate compositions.

The value of vat dye compositions is primarily dependent upon the excellence of the dyeings and printings obtained therefrom. The physical form of vat dye pastes and powders prior to their use in dyeing, and also prior to their incorporation in the ultimate dyeing compositions, has an important effect on the coloring properties of these compositions. In the vat dye pastes, the esters described above act to increase the fluidity of the pastes and also apparently to prevent flocculation of the dye particles. In the vat dye powders, the esters make possible the rapid disintegration of the dye particles in dye baths and aqueous printing compositions, a result which is believed to be due to the fact that the esters are so combined or commingled with the vat dye particles as to form therewith a composition resembling a solid dispersed suspension. The particles of the vat dyes, therefore, are prevented from combining to form larger particles which would be difficult to disperse in a padding bath or printing paste.

When the vat dye pastes and powders are incorporated with the other usual ingredients of vats, padding baths, or printing pastes, several advantageous results are obtained. First, as previously noted, the compositions may be mixed readily with such ingredients to form homogeneous dyeing compositions. Further, it appears that the esters act to increase the solubility of leuco dyes and increase the strength and brilliancy of the dyeings and printings obtained. This is because, while leuco vat dyes are regarded as being soluble in alkaline solutions, and because of this solubility may be taken up by the fiber to be dyed, they are not freely soluble and their increased solubility caused by the esters accelerates the rate of their absorption or adsorption by the fiber.

The vat dye compositions of the present invention are particularly valuable as compared with ordinary vat dye compositions when they are applied to synthetic fibers composed of regenerated cellulose, or to unbleached natural fibers, such as cotton and raw silk. It is known that these latter fibers do not, as a rule, readily absorb the reduced vat dyes because of their coating of natural waxes which act as resist agents.

As before stated, the soluble esters of this invention may be used per se or in admixture with other substances ordinarily used in the art of dyeing or printing textiles. These substances may be in the nature of the alkyl partial inorganic esters specifically disclosed in my patents above referred to, other substances used in dyeing and printing, such as the alkylolamines, polyhydric alcohols, and ethers, e. g., the ethers of diethylene glycol, glucosic compounds, dextrines, gums, starches, etc., or compounds in the nature of printing catalysts, such as heavy metal compounds or the heavy metal salts of alkyl partial esters, such as isobutyl-nickel-sulfate, or the heavy metal salts of the soluble carboxylic esters of the present invention, themselves.

Inasmuch as the quality and dispersibility of the resulting dye composition are dependent to a considerable extent upon the original degree of dispersion of the dye in the suspension or in the dry form, the vat dye is preferably treated in the highly dispersed form. A suitable dispersing agent is preferably incorporated into the dispersion or suspension so as to aid in preventing precipitation and agglutination of the dye particles during storage or during the drying and to increase the fluidity and render easier the incorporation of the soluble salts of the carboxylic acid esters.

Among the dispersing agents which may be employed for this purpose there may be mentioned, for example, the sulfonic acids of benzene, hydyroxybenzenes, naphthalene, hydroxynaphthalenes, their nuclear alkyl, nuclear aralkyl, and hydrogenated derivatives, as well as aldehyde condensation products thereof (as for example, 2,6-naphthalene-disulfonic acid, 1,7-naphthalene-disulfonic acid, 2,8-naphthalene-disulfonic acid, isopropyl-naphthalene-sulfonic acids, dibutyl-naphthalene-sulfonic acids, amyl-naphthalene-sulfonic acids, butyl-benzyl-naphthalene-sulfonic acids, cyclo-hexyl-naphthalene-sulfonic acids, methylene-di(β-naphthalene-sulfonic acid), methylene-di(di-isopropyl-naphthalene-sulfonic acid), in the form of the free acids or salts (e. g., alkali metal salts), etc.; sulfite cellulose waste liquors and their mildly oxidized products and evaporated residues; sulfonated resins; abietene, abietine, and abietane sulfonic acids; soaps; sulfonated higher fatty acids, fats and oils; and the like, as well as mixtures of two or more of such substances.

Further, additional substances having diluent antifoaming, assisting, wetting, catalytic, enolyzing, or other suitable action in the subsequent use of the compositions may also be incorporated into the compositions of the present invention, preferably while the latter are in the form of aqueous suspensions or in the form of non-drying pastes containing high boiling alcohols or alcohol ethers.

In preparing the vat dye pastes and powders in accordance with a preferred manner of proceeding, the mixture of vat dye in aqueous or high boiling solvent dispersion is agitated with the appropriate amount of the carboxylic acid ester salt together with the other substances, if added, until a homogeneous paste is formed, or the mixture of the aqueous suspension of the dispersed vat dye and a carboxylic acid ester salt together with the other substances, if added, is evaporated with agitation until a thick paste is formed, with or without the aid of vacuum and preferably at a temperature not exceeding 100° C. The resulting paste constitutes a valuable vat dye paste. If desired, it may be brought to substantial dryness to form a dry vat dye composition. Thus, the paste may be evaporated to dryness on an atmospheric or vacuum rotary drum drier, and the dry product scraped off in the form of flakes, scales, or grains which may be used as such; or the paste, with or without preliminary evaporation, may be admixed with a suitable hydrate-forming soluble salt in an anhydrous condition, such as anhydrous trisodium phosphate, and the admixture stirred to produce a dry product. If desired, the dry product may be disintegrated in any suitable manner into a powder.

Mixtures of the carboxylic acid ester salts or mixtures of them with alkyl inorganic ester salts, disclosed in my patents referred to above may also be employed in accordance with the present invention. Further, the esters used in the compositions of the invention may be used together with or in place of the esters disclosed in my applications Serial Nos. 253,346 and 253,347, filed of even date herewith.

The amount of the soluble carboxylic acid esters employed in the preparation of the compositions of the present invention may be varied over a considerable range, depending in part upon the concentration of vat dye in the composition, the use to which the composition is to be put, and whether or not other diluents and other materials having a catalytic, assisting, wetting, enolyzing, dispersing, or other action, are included in the composition.

The invention may be employed in connection with vat dyes of all types including those derived from anthraquinones, indophenols, various indigoid, thioindigoid and indirubin compounds, etc.; as for example, indanthrones, pyranthrones, flavanthrones, dibenzanthrones, isodibenzanthrones, perylene quinones, anthranthrones, dibenzpyrene quinones, anthrimidocarbazoles, naphthacridones, indigo, thioindigo, indirubin, etc., including derivatives thereof, such as their halogen, nitro, sulfur, and/or alkoxy derivatives.

It should be noted that while the vat dyes in the compositions of the invention are preferably in the unreduced form, they may also be in the reduced or leuco form. For example, leuco ester salts of vat dyes and leuco compounds of vat dyes prepared by careful acidification of an alkaline vat, or by other methods, may be used, if desired.

Although it is generally preferable and desirable, in order to take full advantage of their properties, to employ the esters in vat dye pastes or powders which are used in the preparation of padding baths or printing pastes, the esters may also be mixed with the other ingredients used in padding baths or printing pastes at the time these compositions are prepared. In proceeding in this way, the dye may be introduced into the mixture in the form of a press cake or an ordinary dye or other form of paste, the ester or esters being added at any desired or suitable point in the preparation of the compositions.

In the forgoing description, the use of the vat dye compositions in the dyeing and/or printing of textile materials has been stressed since this is the most important application of the compositions. The compositions, however, especially when in the form of powders, may be used in the preparation of dispersions of vat dyes for use in the pigment form in the dyeing of paper, the tinting of fibers and other materials, and the like. The ready dispersibility of the compositions makes them especially valuable for such purposes.

The invention will be further described in connection with the following specific examples (in which the parts are by weight), which are given to illustrate the invention. It will be realized, however, that the invention is not limited thereto but that changes may be made in the materials treated and their proportions, manipulative steps, and other conditions without departing from the scope of the appended patent claims.

Example 1

1800 parts of National Vat Brown G (di-naphthyl-thioindigo) in the form of a press cake containing about 16 per cent of dye solids and the rest principally water, are mixed with 36 parts of "Tamol NNO" (which is the sodium salt of a naphthalene sulfonic acid condensed with formaldehyde), 80 parts of the ethyl ether of ethylene glycol and 100 parts of glycerine, and 187 parts of sodium isobutyl sulfoacetate are then added and the whole is mixed thoroughly. The resulting dilute paste is then evaporated at a temperature of about 100° C. to form 1800 parts of a vat dye paste in which the vat dye particles are thoroughly dispersed and which remains in fluid condition on standing. The paste is especially adapted for incorporation with the usual additional ingredients for the preparation of padding baths and printing pastes which give dyeings and printings of increased strength and brilliancy as compared with the dyeings and printings obtained with the same compositions but omitting the sodium isobutyl sulfoacetate.

Example 2

1462 parts of National Vat Orange R (Color Index No. 1217), in the form of an aqueous paste containing about 13 per cent of dye solids are substituted for the Vat Brown G press cake in Example 1. A similar vat dye paste is obtained which has the valuable properties pointed out in connection with the paste of Example 1.

Example 3

3600 parts of National Vat Pink FF (6,6′-dichlor-4,4′-dimethyl-thioindigo) in the form of a press cake containing about 25 per cent of dye solids and the rest principally water are mixed thoroughly with 480 parts of sulfite buck (a sulfite waste liquor product) dissolved in 800 parts water. To the resulting mixture 8.15 parts of caustic soda, 3180 parts of dynamite glycerine, and 540 parts of sodium isobutyl sulfoacetate are added and the resulting mixture is agitated to form a dilute paste which is then evaporated as described in Example 1 to form 6000 parts of a vat dye paste. Similarly to the paste described in Example 1, the vat dye paste of this example is especially adapted for use in the preparation of padding baths and printing pastes which give unusually valuable dyeings and printings.

Example 4

400 parts of indigo paste (containing 20 per cent pure indigo as shown by indigotin titration and resulting from the precipitation of indigo from an alkaline solution of indoxyl by aeration) are stirred with 2 parts "Leukanol" (a condensation product of naphthalene sodium sulfonate with formaldehyde) until a thin fluid paste is obtained. 13 parts cane sugar and 5 parts 2-methyl-1-pentanol sodium sulfobutyrate are then added, the mixture being stirred until solution of the sulfo alkyl ester is complete, and the resulting mixture is then evaporated with stirring until a thick paste is formed. The thick paste is then evaporated to substantial dryness either by means of a rotary double drum drier or else placed in drying pans in vacuum. The dry product is ground and screened. An indigo powder is obtained containing 80 per cent indigo which, when added to water or the dye liquor, disperses readily. When employed for the preparation of an indigo vat with the aid of the usual reducing agents (e. g., sodium hydrosulfite and caustic soda) the powder disperses instantly and reduces very rapidly producing a clear vat which dyes cotton yarns an even blue shade of high tinctorial value.

Example 5

To 200 parts National Carbanthrene Violet 2R Paste (Color Index No. 1104), containing 11 per cent dye solids in the form of an aqueous suspension, are added 2 parts "Leukanol," 5 secondary hexyl sodium sulfobutyrate, and 71 parts cane sugar or "Cerelose."

The mixture is stirred until all the soluble material is dissolved and is then evaporated to dryness on a double drum drier, whereby a flaked material is obtained which is ground to a powder and screened through a 60 mesh screen. The resulting product is a violet colored powder instantly dispersible when added to water and is especially suitable for the dyeing of piece goods by the pad-jig dyeing process. When the powder is made into a printing paste containing the usual ingredients used for the printing of vat dyes, the prints obtained therewith yield very brilliant violet shades, fully penetrated and level.

Example 6

2765 parts of Carbanthrene Flavine GC (Color Index No. 1095) press cake containing 13.8 per cent total solids, of which approximately 11 per cent represent actual dyestuff, are stirred in a beaker while 95 parts "Tamol NNO" are added until a fluid paste is obtained. Thereafter, 10 parts trisodium citrate, 320 parts sodium isobutyl sulfoacetate dissolved in 320 parts water, and 1600 parts dynamite glycerine are added in succession and the 5111 parts then obtained are subjected to evaporation until 3200 parts of a color paste are obtained. This paste is then passed through a micropulverizer, finally screened through a 200 mesh screen, and is ready for use as a printing color. Unusually strong and well penetrated yellow prints are obtained therewith.

Example 7

To 4410 parts Brilliant Indigo KMR (6,6'-dibrom-indigo) paste containing 20 per cent actual dye solids, 1800 parts dynamite glycerine are added and the mixture is then evaporated in a glycerine bath at 100° C. to a total of 2923 parts of concentrated color paste. A color paste especially suitable for printing is then obtained therefrom as follows:

To 2260 parts of the above concentrated color paste, 200 parts water, 140 parts isobutyl sodium sulfoacetate dissolved in 172 parts water, and 28 parts diacetone alcohol are added, giving a total of 2800 parts of an Indigo KMR color paste which when thoroughly mixed is screened and is then ready for use. When made up into a vat dye printing paste, very deep and fully penetrated navy blue shades are obtained therewith.

In the above examples, the esters used may be replaced by equivalent amounts of any one or a mixture of the class herein disclosed; for example, any one or a mixture of the esters specifically mentioned above.

The process of making the vat dye compositions of the invention, either in paste, powder, or flake form, is further applicable to any vat dye. Thus any vat dye of the previous examples may be replaced by any of the following:

	Color Index No.
Brilliant Indigo 4G	1189
National Vat Scarlet G	1228
National Vat Violet R	1222
National Vat Green G	1199
Carbanthrene Yellow G	1118
Carbanthrene Golden Orange 2RT	1097
Carbanthrene Golden Orange G	1096
Carbanthrene Blue Green FFB	1173
Carbanthrene Violet 2R	1104
Carbanthrene Dark Blue DR	1099
Carbanthrene Flavin GC	1095
Carbanthrene Blue GCD	1113
National Vat Orange R	1217

The process is of special value for the compounding of blacks of the anthraquinone or indigo-thioindigoid series.

The esters of this invention may also be added directly to printing pastes whereby unusual results are obtained, since an optimum amount may be added thereto without limitation.

Example 8

A standard printing thickener is prepared as follows:

	Parts
Corn starch	100
British gum	300
Gum tragacanth 5% solution	100
Water	500

are thoroughly mixed together and boiled to a smooth paste. To 520 parts of the above paste are added 170 parts K_2CO_3 and 60 parts H_2O, and heat applied until a homogeneous paste is obtained. After cooling of this paste to about 150° F. there are added 140 parts sodium formaldehyde sulfoxylate, 50 parts glycerine and 60 parts water, and the paste stirred until all ingredients are thoroughly mixed and dissolved. A blue vat dye printing paste is then prepared by adding to 200 parts Carbanthrene Blue GCD (Color Index No. 1113) 680 parts of the above printing thickener, and 120 parts n-amyl sodium sulfoacetate, stirring until the ingredients are thoroughly mixed and dissolved. Cotton and rayon fabrics are printed therewith by means of a rotary intaglio printing machine; the material is then dried and aged for 4 minutes in an air-free ager at 102° C. (saturated steam) after which the prints are rinsed in cold running water, soaped at the boil, rinsed, and dried. Thus a very brilliant heavy shade of blue is obtained which shows superiority in brilliance, penetration, levelling and fastness to crocking versus prints obtained with the same printing paste but when the n-amyl sodium sulfoacetate has been left out.

In the above example the n-amyl sodium sulfoacetate may be replaced by any one of the esters disclosed above.

I claim:

1. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to a vat dye compound and a small amount of a dispersing agent, a soluble organic ester of an aliphatic monocarboxylic acid containing 2 to 4 carbon atoms and an aliphatic alcohol containing 2 to 6 carbon atoms, said ester containing at least one solubilizing radical selected from the group consisting of the sulfonate, sulfate, and phosphate radicals.

2. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to a vat dye compound and a small amount of a dispersing agent, a soluble alkali metal salt of an organic ester of an aliphatic monocarboxylic acid containing 2 to 4 carbon atoms and an aliphatic alcohol containing 2 to 6 carbon atoms, said ester containing in the acyl radical thereof at least one solubilizing radical selected from the group consisting of the sulfonate, sulfate, and phosphate radicals.

3. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to an unreduced vat dye and a small amount of a dispersing agent, a soluble organic ester of an aliphatic monocarboxylic acid containing 2 to 4 carbon atoms and an aliphatic alcohol containing 2 to 6 carbon atoms, said ester containing a sulfonate radical in the acyl radical thereof.

4. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to an unreduced vat dye and a small amount of a dispersing agent, an alkali metal salt of an ester of an aliphatic alcohol and an aliphatic monocarboxylic acid containing 2 to 4 carbon atoms, the alcohol residue of said ester containing 2 to 6 carbon atoms, said ester containing a sulfonate radical in the acyl radical thereof.

5. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to a vat dye compound and a small amount of a dispersing agent, a soluble salt of an ester of an aliphatic alcohol and sulfoacetic acid, the alcohol residue of said ester containing 2 to 6 carbon atoms.

6. A vat dye composition for use in the preparation of printing pastes and padding baths comprising an unreduced vat dye, a dispersing agent, and an alkali metal salt of an ester of an aliphatic alcohol and sulfoacetic acid, the alcohol residue of said ester containing 2 to 6 carbon atoms.

7. A vat dye paste for use in the preparation of printing pastes and padding baths comprising an unreduced vat dye, a dispersing agent, and sodium isobutyl sulfoacetate.

8. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to a vat dye compound and a small amount of a dispersing agent, a neutral soluble salt of isobutyl sulfoacetate.

9. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to an unreduced vat dye and a small amount of a dispersing agent, an alkali metal salt of isobutyl sulfoacetate.

10. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to a vat dye compound and a small amount of a dispersing agent, a neutral soluble salt of isobutyl sulfobutyrate.

11. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to an unreduced vat dye and a small amount of a dispersing agent, an alkali metal salt of isobutyl sulfobutyrate.

12. A vat dye paste for use in the preparation of printing pastes and padding baths comprising an unreduced vat dye, a dispersing agent, and sodium isobutyl sulfobutyrate.

13. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to a vat dye compound and a small amount of a dispersing agent, a neutral soluble salt of an ester of an aliphatic monocarboxylic acid containing 2 to 4 carbon atoms with an alcohol containing 6 carbon atoms.

14. A vat dye composition for use in connection with the coloring of textile fibers by printing and pigment padding processes comprising, in addition to an unreduced vat dye and a small amount of a dispersing agent, an alkali metal salt of cyclohexyl sulfoacetate.

15. A vat dye paste for use in the preparation of printing pastes and padding baths comprising an unreduced vat dye, a dispersing agent, and sodium cyclohexyl sulfoacetate.

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