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3,150,916

PROCESS FOR THE TREATMENT OF TEXTILE MATERIALS

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6 Claims. (Cl. 8—54)

It is already known to fix dyestuffs that are fixable in an alkaline medium by means of alkalies or compounds yielding alkalies, for example sodium bicarbonate, on textile materials made of fibers containing cellulose, or of silk and wool. Especially such dyestuffs have been fixed as form a homopolar chemical linkage with the textile fibers in the presence of substances reacting alkaline. The choice of the alkalies or of the alkali donators depends on the reactive groups on which said dyestuffs are based, or on the reactivity of the reactive groups with the textile fibers respectively, furthermore, on the type of the fibers to be dyed or printed, and on the dyeing process applied.

U.S. Patent No. 2,892,670 describes a process for dyeing textile materials made of cellulose, wherein the textile material is impregnated in the aqueous medium with anthraquinonoid dyestuffs, containing at least one ionogenic solubilizing group and at least an amino group, carrying as N-substituent a 1,3,5-triazine radical which is containing a halogen atom attached to a carbon atom; and wherein the so treated material is thereafter subjected to the action of an acid-binding agent and to a heat-treatment in the aqueous medium. When fixing the dyestuffs there can be used alkalies, or alkali metal bicarbonates as well as sodium trichloroacetate as acid-binding agents.

Dyestuffs that are fixable in the alkaline medium, often develop a partial inactivation in the aqueous alkaline medium; by this inactivation only weak dyeings are obtained. It was found that this inactivation which is to be attributed to hydrolytic influences, occurs in the alkaline medium and even in the weakly alkaline medium. Even when using very weak alkalies or alkali donators such as sodium bicarbonate, or when using sodium trichloroacetate as alkali donator, the stability of the printing pastes and padding liquors is only a limited one, confining the practical applicability of many dyestuffs of this series. A further disadvantage of these known processes is the sensitiveness of the unfixed padded dyeings or printings towards the action of air as well as the sensitiveness of paddings and printings with dyestuffs, having the constitution mentioned below, towards long steaming periods.

The present invention is based on the observation that such a disadvantageous influence, occurring in the form of an inactivation or reduced stability of the dyestuffs used can be avoided in the aqueous medium, by observing a weakly acid pH-value.

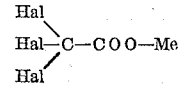
Now, we have found that it is especially advantageous to use alkali metal salts or alkaline earth metal salts of such carboxylic acids having at least 2 carbon atoms, as yield alkali or alkaline earth metal carbonates when being heated at temperatures of above 80° C. especially at temperatures from 100 to 160° C. in the presence or absence of water or steam, with separation of cleavage products that can be volatile or that are capable of being washed out, and with the simultaneous use of free carboxylic acids, as fixing agent for such water-soluble dyestuffs, so-called reactive dyestuffs, as in the presence of alkalies form a homopolar linkage with the textile fibers containing hydroxy or carbon amide groups, or as can be fixed on said fibers by the separation of water-solubilizing groups.

Because of their content of free carboxylic acids the dyestuffs containing preparations as dyebaths or printing pastes applied according to the process of the present invention show an acid reaction at room temperature and a pH value below pH 6 to about pH 4.

These acid preparations are practically of unlimited stability at room temperature or at a temperature somewhat elevated, only at high temperatures a decomposition of the salts takes place, with simultaneous setting free alkali or alkaline earth metal carbonates and with the separation of a cleavage product that is volatile or that can be washed out of the textile material.

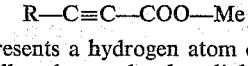
As alkali metal salts or alkaline earth metal salts of the carboxylic acids described above, there may be mentioned, for example, the following compounds:

(*a*) Alkali metal salts and alkaline earth metal salts of trihalogen derivatives of acetic acid of the general formula



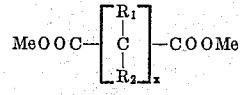
wherein Hal contains a halogen atom having an atomic weight of 35 to 126 and Me represents an alkali metal or alkaline earth metal equivalent.

(*b*) Alkali metal salts and alkaline earth metal salts of unsaturated carboxylic acids, the carbon atom of which, carrying the carboxyl group, is attached to the adjacent carbon atom by a triple linkage corresponding to the general formula



wherein R represents a hydrogen atom or a hydrocarbon radical, especially a low molecular aliphatic hydrocarbon radical, and Me represents an alkali metal or alkaline earth metal equivalent.

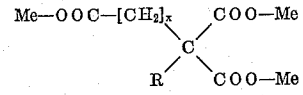
(*c*) Alkali metal salts and alkaline earth metal salts of dicarboxylic acids and the derivatives thereof of the general formula



wherein *x* stands for the integers 0, 1, 4, 5, 6, 7, or 8, the radicals R_1 and R_2 represent hydrogen atoms or a hydrocarbon radical, whereby at least one of the two radicals must represent a hydrogen atom when *x* is greater than 1, and wherein Me represents an alkali metal or alkaline earth metal equivalent.

(*d*) Alkali metal salts and alkaline earth metal salts of tricarboxylic acids and the derivatives thereof, of the

general formula



wherein X represents the integer 0 or a whole number, especially integers of 1 to 6, R represents hydrogen or a hydrocarbon radical and Me represents an alkali metal or alkaline earth metal equivalent.

(*e*) Alkali metal salts and alkaline earth metal salts of unsaturated dicarboxylic acids of the type

$$Me—OOC—C\equiv C—COOMe$$

wherein Me represents an alkali metal or alkaline earth metal equivalent.

(*f*) Alkali metal salts and alkaline earth metal salts of α, β and γ-hydroxy acids or keto acids and the derivatives thereof of the general formula

$$R—CHOH—[CH_2]_x—COO—Me$$

and

$$R—CO—[CH_2]_x—COO—Me$$

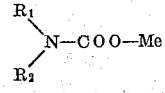
wherein x stands for the integers 0, 1 or 2; R represents a hydrocarbon radical, preferably a low molecular alkyl radical and Me represents an alkali metal or alkaline earth metal equivalent.

(*g*) Alkali metal salts and alkaline earth metal salts of keto-dicarboxylic acids and the derivatives thereof of the general formula

$$Me—OOC—[CH_2]_x—CO—[CH_2]_y—COO—Me$$

wherein x and y represent a small whole number of about 1 to 4 and Me represents an alkali metal or alkaline earth metal equivalent.

(*h*) Alkali metal salts and alkaline earth metal salts of nitrogenous carboxylic acids and the derivatives thereof of the general formula



wherein R_1 and R_2 represent hydrocarbon radicals, especially low molecular hydrocarbon radicals of the aliphatic, cycloaliphatic and aromatic series and Me represents an alkali metal or alkaline earth metal equivalent.

It is especially advantageous to use the salts mentioned sub (*a*) and (*b*).

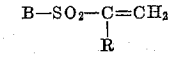
As examples of those alkali metals salts or alkaline earth metal salts of carboxylic acids yielding alkali metal or alkaline earth metal carbonates when being heated, there may be mentioned: sodium or potassium trichloroacetate, sodium and potassium tribromoacetate and the sodium and potassium salts of the following carboxylic acids: levulinic acid, propiolic acid, tetrolic acid, malonic acid, dichloromalonic acid, acetone dicarboxylic acid, cyclobutane dicarboxylic acid, acetoacetic acid, ethanetricarboxylic acid, acetylenedicarboxylic acid, β-aldehyde propionic acid, mesoxalic acid and carbamic acid. The alkali metal salts or alkaline earth metal salts mentioned above are neutral compounds, which are stable in padding liquors and printing pastes in an acid medium at temperatures up to 40° C.

As carboxylic acids that are to be used additionally there may preferably be used the same acids or a carboxylic acid which is somewhat weaker than that on which the above mentioned salts are based. Preferably lower mono or dicarboxylic acids having from 1 to about 6 carbon atoms are used. First of all, there may be used readily volatile acids, such as for example, formic acid, acetic acid, propionic acid. There may, however, also be used carboxylic acids that are difficultly volatile or non-volatile, for example, butyric acid, lactic acid, tartaric acid, citric acid, malonic acid. The additionally applied organic acids are advantageously used in quantities that are stoichiometrically smaller than alkali can be set free from the alkali donator used. Per equivalent of alkali

there may preferably be used 0.05 to 0.2 equivalent of free organic acids. In general, the volatile organic acids are already removed when drying the printed or dyed materials; difficultly volatile or non-volatile acids are neutralized during the steaming process and washed out in the following washing process.

As dyestuffs that are fixable in the alkaline medium, so-called reactive dyestuffs, there may be mentioned for example dyestuffs containing the following functional groups once or several times:

(*a*)

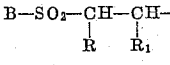


R=hydrogen, alkyl, halogen

B=dyestuff radical with solubilizing groups, for example SO_3H, COOH, which is linked with the functional group by means of a carbon atom or over a nitrogen atom.

Dyestuffs containing these groups are, for example, described in U.S. Patent No. 2,657,205.

(*b*)



R=hydrogen, alkyl, halogen

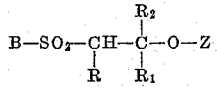
R_1=hydrogen, alkyl

X=halogen

B=dyestuff radical with solubilizing groups, for example SO_3H, COOH, which is linked with the functional group by means of a carbon atom or over a nitrogen atom.

Dyestuffs containing these groups are, for example, described in U.S. Patent No. 2,743,267.

(*c*)



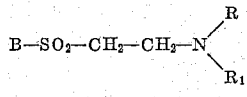
R, R_1, R_2=hydrogen or low molecular alkyl

Z=radical of a polybasic mineral acid or of a mono or polybasic organic acid.

B=dyestuff radical which may contain solubilizing groups, and which is linked with the functional group over a carbon atom or over a nitrogen atom.

Dyestuffs containing these groups are, for example, described in U.S. Patent No. 2,670,265.

(*d*)



R and R_1=aliphatic or cycloaliphatic hydrocarbon radicals, which together with the nitrogen atom can form the radical of a hydrogenated heterocyclic ring system.

B=dyestuff radical containing solubilizing groups, for example, SO_3H,—COOH—, and which is linked with the functional group over a carbon atom or over a nitrogen atom.

Dyestuffs containing these groups are, for example, described in French Patent No. 1,238,556.

(*e*) $B—SO_2F$

B=dyestuff radical containing solubilizing groups and which is linked with the functional group over a carbon atom.

Dyestuffs containing these groups are, for example, described in Belgian Patent No. 578,517.

(*f*) B—CO—R—X

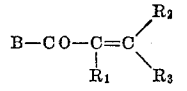
R=saturated or unsaturated hydrocarbon radical having up to 3 carbon atoms, especially the ethylene radical

X=halogen atom

B=dyestuff radical containing solubilizing groups and which is advantageously linked with the acyl radical over a nitrogen atom.

Dyestuffs containing these groups are, for example, described in Belgian Patent No. 570,437.

(g)



R_1, R_2, R_3=hydrogen atom or halogen atom, or an alkyl or hydroxyalkyl group, whereby at least one of the three radicals must represent a hydrogen atom.

B=dyestuff radical with solubilizing groups, which is linked with the functional group over a nitrogen atom.

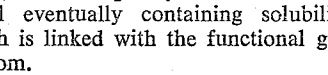
(h) $B—SO_2—NH—R—X$

R=alkylene radical with 2 to 3 carbon atoms.

X=halogen, $—O—SO_3H$, $—O—PO_3H_2$, $—O—SO_2—$alkyl, $—O—SO_2—$aryl

B=dyestuff radical eventually containing solubilizing groups and which is linked with the functional group over a carbon atom.

(i)



B=dyestuff radical which may contain solubilizing groups

B'=dyestuff radical which may contain solubilizing groups; or aryl radical which may be substituted and may contain solubilizing groups.

(j) $A—(CH_2—NH—Acyl—X)_n$

A=dyestuff radical containing solubilizing groups

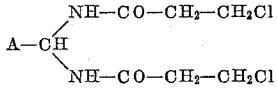
Acyl=aliphatic acyl group

X=halogen in α- or β-position of the acyl group

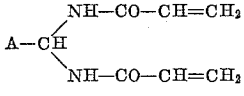
n=a whole number of about 1 to 3

Dyestuffs containing these groups are, for example, described in Belgian Patent No. 582,072.

(k)



or



A=dyestuff radical containing solubilizing groups.

It is especially advantageous to apply the process of the present invention to the dyestuffs mentioned sub *a*, *b* and *c*.

The preparation of printing pastes with the use of the fixing agents of the present invention is carried out in known manner by dissolving the dyestuffs, whereby a urea can be used simultaneously. First of all, a mixture of the dyestuff and the urea is prepared, which is dissolved in water. It is, however, also possible to dissolve said components separately and to mix the solutions. Thereafter, there are added to the dyestuff solution in any desired sequence, a neutral thickening agent, the organic acid and the neutral alkali donator. As thickening agents there may be used for example, alginates, cellulose ether, etherified carob bean flours and benzine emulsions, preferably those of the oil-in-water type.

The composition of the padding liquors corresponds to that of the printing pastes, whereby, however, it can be dispensed partially or completely with the simultaneous use of a thickening agent.

The choice of the alkali donator depends on the other components used. Generally, there are used the alkali metal salts of the carboxylic acids applied, since hereby the formation of insoluble precipitates is avoided to a large extent. Furthermore, when using alkali metal salts, the steaming periods generally are shorter than the steaming periods are, when alkaline earth metal salts are used. As alkali metal salts there preferably are used by economical reasons sodium salts or potassium salts, but also all the other alkali metal salts can be used. As alkaline earth

metal salts there may advantageously be used magnesium salts.

As fibrous material there may be used natural or synthetic fibers containing hydroxy or carbon amide groups, for example, fibers of natural or regenerated cellulose, such as cotton, linen, viscose rayon, cuprammonium rayon, staple fiber, furthermore wool, natural silk and polyamide fibers as well as mixtures containing said fibers.

The fixation of the dried prints or pads is performed either by neutral steaming, whereby according to the dyestuff or alkali donator used, steaming periods within the range of about 2 to about 30 minutes at temperatures of about 100 to 120° C. are required, or by heating the material at temperatures in the range of 100 to 200° C., preferably 140 to 160° C. The fixation periods in the latter case are within about 15 seconds to 8 minutes.

By applying the combination of the salts of the carboxylic acids mentioned above and the free organic acids according to the process of the present invention, the stability of the dyestuff containing preparations as padding liquors and printing pastes is improved considerably and a reduced sensitiveness of the unfixed prints and dyeings to air is obtained as compared with the processes in which the hitherto known alkali donators are used. Furthermore, with the salts of the carboxylic acids used in the presence of free organic acids there are obtained considerably better dyestuff yields than those obtained with processes in which inorganic fixing agents are applied to fibers, especially to fibers of regenerated cellulose. A further technical advantage is the reduced sensitiveness of the prints to long steaming periods, so that, for example, the dyeings are better reproducible, especially when applying dyestuff mixtures.

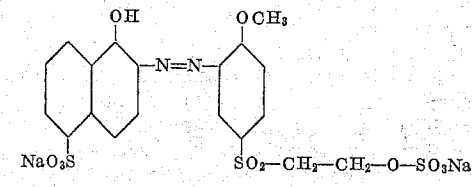
When using dyestuffs with 2 functional groups of the type described above and applying the salts of the carboxylic acids together with free organic acids to fibers of regenerated cellulose, there are obtained softer prints than those obtained according to the hitherto known processes. In the process of the present invention it is possible with the use of said salts of the carboxylic acids together with free organic acids to add the alkali donator to the printing paste or to the dyeing liquor before the mixture of the other components is cooled completely to room temperature. In practice this method means a considerable simplification and time saving. Without simultaneously using free organic acids and still earlier when using inorganic alkali donators, for example, sodium bicarbonate, such a working method produces an untimely decomposition of the dyestuff, thus weaker dyeings are obtained.

When dyeing in a long liquor and using the claimed salts of carboxylic acids in the presence of free organic acids, the dyeing process can be carried out at temperatures up to 100° C., whereby the textile material is better penetrated with the dyestuff than in a dyeing process carried out at low temperatures. Even such sensitive dyestuffs can be used as are decomposed in a neutral or weakly alkaline solution at elevated temperatures.

The following examples serve to illustrate the invention but they are not intended to limit it thereto, the parts being by weight unless otherwise stated.

Example 1

40 parts of the dyestuff of the following constitution

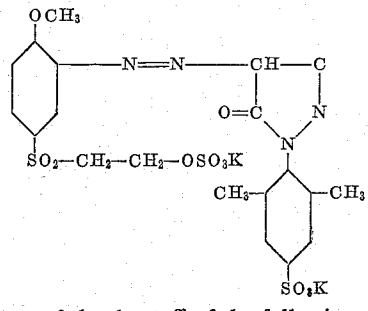


were mixed with 75 parts of urea and the mixture was dissolved in 300 parts of hot water. To the solution were added 400 parts of a neutral alginate thickening of 4% strength, 6 parts of acetic acid of 15% strength and 80

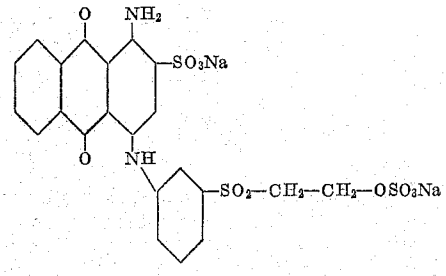
parts of an aqueous solution of trichloroacetic acid of 50% strength adjusted to a pH of 7 by means of sodium carbonate or sodium hydroxide solution. Finally, the printing paste was made up to 1000 parts with water or a neutral alginate thickening. A cotton fabric was printed with the printing paste in known manner, the printed fabric was dried, then steamed neutrally for 8 to 10 minutes, rinsed, soaped and rinsed once more. By this working method the same results as regards colouring strength and fastness to washing are obtained as in the printing processes carried out with the usual alkalies (alkali metal bicarbonates, alkali metal carbonates). When using acetic acid and sodium trichloroacetate, the stability of the printing paste is more than 6 weeks whereas the stability is only 2 to 3 days when using alkali metal carbonates.

Example 2

30 parts of the dyestuff of the following constitution



and 10 parts of the dyestuff of the following constitution

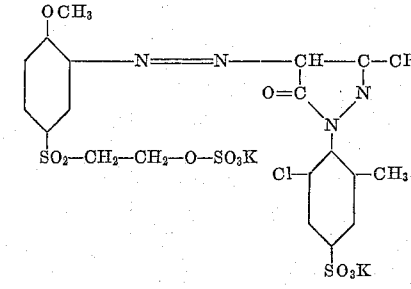


were mixed with 30 parts of urea and the mixture was dissolved in 300 parts of hot water. To the solution were added in usual manner a neutral thickening, 8 parts of acetic acid of 15% strength and 40 parts of sodium trichloroacetate and the whole was then made up to 1000 parts. After printing on staple fiber, drying the printed material, neutral steaming for 8 minutes at 101° C. and carrying out the usual rinsing and washing process, there was obtained a green print having a good fastness to washing.

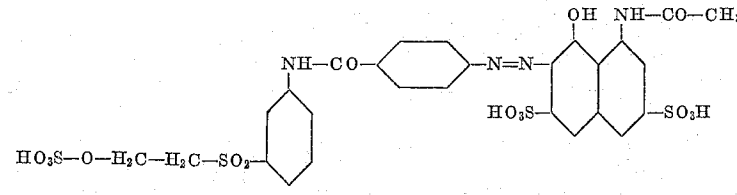
When the same printing process was carried out with a printing paste that had been allowed to stand for 6 weeks a print of practically the same strength and the same tint was obtained as that obtained with the freshly prepared printing paste, whereas, when allowing printing pastes having the same composition but in which sodium bicarbonate is used as alkali donator, already after a few days, there were obtained prints that are changed in colour and tinted more weakly, and with printing pastes prepared with sodium trichloroacetate but without free acetic acid there were obtained prints changed in colour after having allowed the printing paste to stand for 2 weeks.

Example 3

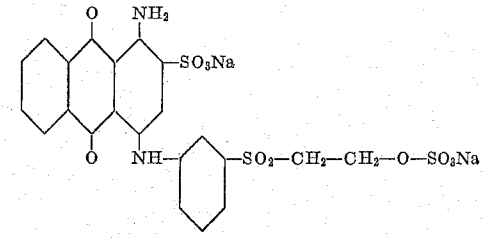
6 parts of the dyestuff of the following constitution



(as yellow component) and 3 parts of the dyestuff of the following constitution



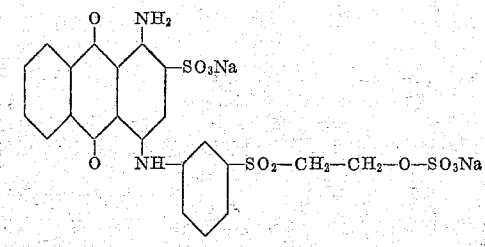
(as red component) and 3 parts of the dyestuff of the following constitution



(as blue component) were mixed with 30 parts of urea and the whole was dissolved in 300 parts of hot water. To the solution were added a neutral thickening, 3 parts of tartaric acid and 30 parts of sodium trichloroacetate and the whole was then made up to 1000 parts. A cotton fabric was printed with the printing paste in known manner. Single parts of the printed fabric were then steamed for 5, 10, 15 and 25 mintues. Subsequently, the fabric was rinsed, soaped and dried in usual manner. Independent of the steaming period, the prints showed a completely uniform tint, whereas prints with printing pastes prepared with sodium bicarbonate, show a slowly weakening of the red component, so that a change of the tint occurs.

Example 4

40 parts of the dyestuff of the following constitution

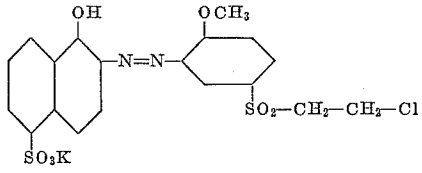


were mixed with 30 parts of urea and the mixture was dissolved in 300 parts of hot water. After addition of 50 parts of an alginate thickening of 4% strength the solution was diluted with 535 parts of cold water and 6 parts of acetic acid of 15% strength and 40 parts of sodium trichloroacetate were added. The material to be dyed (cotton or staple fiber) was then padded with the solution, dried uniformly, steamed for 5 to 10 minutes, or thermofixed for 5 minutes at 150° C. and rinsed and soaped in usual manner. By this working method results are obtained which are in many respects better than those

obtained in a pad-dyeing process with the usual alkalies (alkali metal carbonates, alkali metal bicarbonates). For example, when using sodium trichloroacetate the dyed goods are not or scarcely sensitive to airing, whereas an airing for 1 to 2 days of the dyed goods prepared with sodium bicarbonate causes an obvious weakened tint owing to the weaker fixation. Furthermore, the finished dyeings on staple fiber prepared with sodium trichloroacetate and free organic acids have an essentially more brilliant tint and a stronger coloration than those prepared with alkali metal carbonate.

Example 5

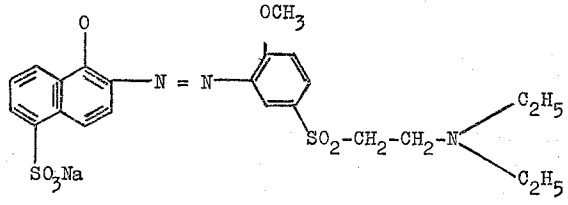
30 parts of the dyestuff of the following constitution



were mixed with 30 parts of urea and the mixture was dissolved in 300 parts of hot water. To the solution were added in usual manner a neutral thickening, 3 parts of a solution of lactic acid of 50% strength, 40 parts of sodium trichloroacetate and the printing paste was made up to 1000 parts. Subsequently, a fabric of staple fiber was printed in usual manner, steamed and washed. A beautiful, full shaded red tint was obtained.

Example 6

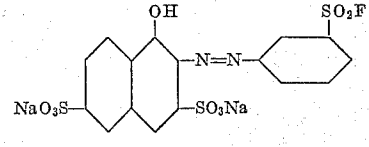
30 parts of the dyestuff of the following constitution



were mixed with 30 parts of urea and the mixture was dissolved in 200 parts of hot water. To the solution were added in usual manner a thickening agent, 3 parts of butyric acid, 400 parts of an aqueous solution of sodium acetoacetate of 10% strength, and the printing paste was made up to 1000 parts with water. Subsequently, a cotton fabric was printed, steamed and finished. A full shaded red tint was obtained.

Example 7

30 parts of the dyestuff of the following constitution

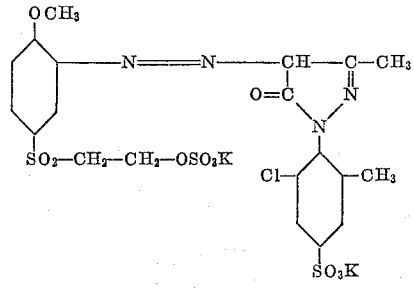


were dissolved together with 30 parts of urea in 300 parts of water. After addition of a thickening agent, 3 parts of citric acid and 40 parts of sodium trichloroacetate,

the printing paste was made up to 1000 parts with water. Subsequently, a fabric of viscose rayon was printed, steamed and washed in usual manner. A beautiful orange tint was obtained.

Example 8

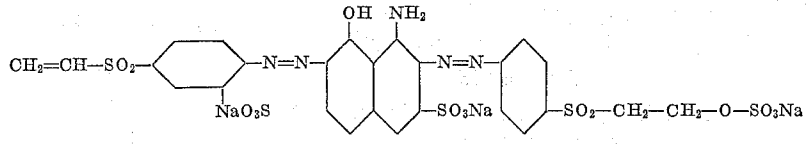
30 parts of the dyestuff of the following constitution



were mixed with 30 parts of urea and the mixture was dissolved in 300 parts of hot water. To the solution were added in usual manner a thickening agent, 8 parts of acetic acid of 15% strength and 40 parts of sodium tribromoacetate and the whole was made up to 1000 parts. Subsequently, a cotton fabric was printed, steamed and washed in the usual manner. A beautiful yellow tint was obtained. The stability of the printing paste is about 1 week with sodium bicarbonate, and more than 8 weeks with acetic acid and sodium tribromoacetate.

Example 9

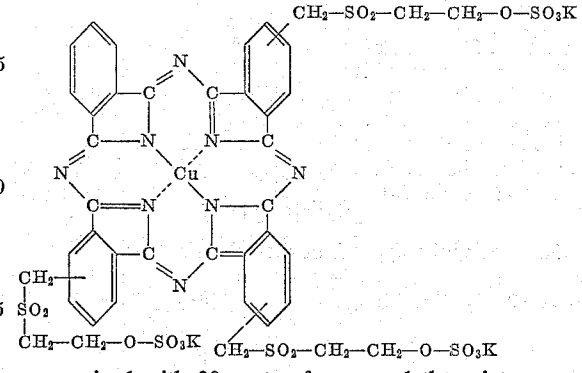
80 parts of the dyestuff of the following constitution



were mixed with 30 parts of urea and the mixture was dissolved in 300 parts of hot water. Subsequently, there were added a neutral alginate thickening, 6 parts of acetic acid of 15% strength and 30 parts of sodium trichloroacetate and the paste was made up to 1000 parts with water. Subsequently, a fabric of staple fiber was printed, steamed and washed in usual manner. The prints prepared with the use of acetic acid and sodium trichloroacetate exhibit a softer handle than those prepared with sodium bicarbonate.

Example 10

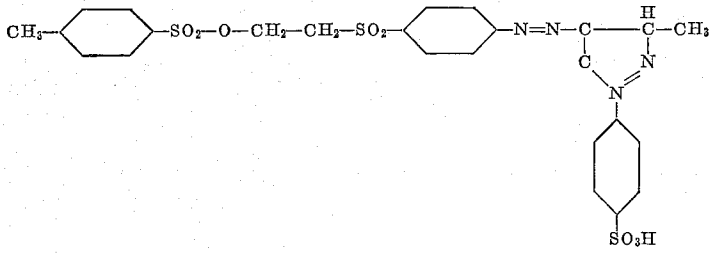
30 parts of the dyestuff of the following constitution



were mixed with 30 parts of urea and the mixture was dissolved in 300 parts of hot water. To the solution were added a thickening agent, 3 parts of a solution of lactic acid of 50% strength and 40 parts of sodium trichloroacetate and the paste was made up to 1000 parts with water. When printing a fabric of cotton or staple fiber and subsequently steaming and washing it, a beautiful turquoise blue tint was obtained.

Example 11

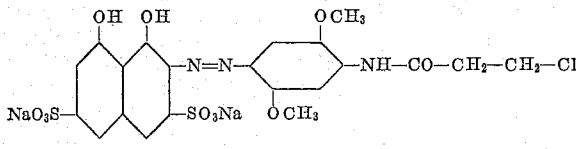
30 parts of the dyestuff of the following constitution



and 100 parts of urea were dissolved in 300 parts of hot water. To the solution were added a thickening agent, 3 parts of a solution of lactic acid of 50% strength, and 40 parts of sodium tribromoacetate and the paste was made up to 1000 parts with water. A cotton fabric was printed with this paste, steamed and washed. A yellow print was obtained.

Example 12

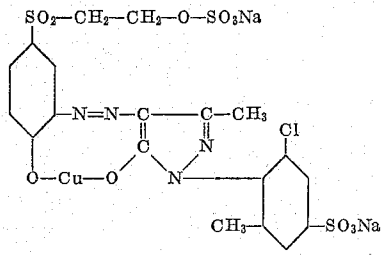
30 parts of the dyestuff of the following constitution



and 30 parts of urea were dissolved in 300 parts of hot water. By addition of a thickening agent, 3 parts of butyric acid and 20 parts of sodium acetoacetate and water the paste was made up to 1000 parts. When printing a fabric of staple fiber with this paste and steaming and washing it, a reddish blue print was obtained.

Example 13

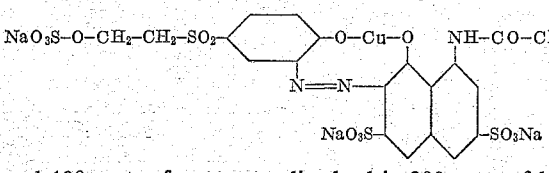
40 parts of the dyestuff of the following constitution



were mixed with 100 parts of urea and the mixture was dissolved in 200 parts of hot water. To the solution were added 500 parts of a benzine-in-water emulsion containing 4% of a polyglycol stearic acid condensation product as emulgator, 40% of an alginate thickening (of 4% strength), 30% of heavy petrol and 26% of water, there were furthermore added 3 parts of butyric acid and 20 parts of sodium acetoacetate. The paste was made up to 1000 parts with water. A cotton fabric was printed with this paste, steamed for 5 to 10 minutes and rinsed and soaped in the usual manner. A deep brownish yellow print was obtained.

Example 14

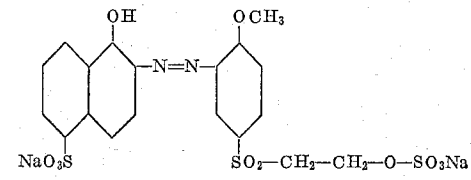
30 parts of the dyestuff of the following constitution



and 100 parts of urea were dissolved in 200 parts of hot water. After addition of 500 parts of a benzine-in-water emulsion described in Example 13, 3 parts of tartaric acid and 40 parts of sodium tribromoacetate, the paste was made up to 1000 parts with water. A fabric of staple fiber was printed with the paste thus prepared, steamed and washed. A full shaded red violet print was obtained.

Example 15

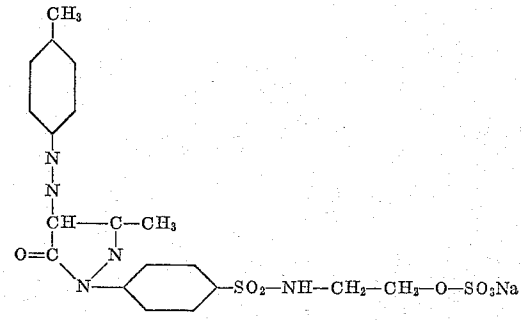
30 parts of the dyestuff of the following constitution



and 30 parts of urea were dissolved in 300 parts of hot water. After addition of a neutral alginate thickening, 5 parts of malonic acid and 40 parts of sodium malonate, the paste was made up to 1000 parts with water. A fabric of cotton or staple fiber was printed with the paste thus prepared, steamed for 10 minutes, and washed. A full shaded red print with a good fastness to washing was obtained.

Example 16

30 parts of the dyestuff of the following constitution



and 100 parts of urea were dissolved in 300 parts of hot water. To the solution were added a thickening, 3 parts of a solution of lactic acid of 50% strength and 40 parts of sodium trichloroacetate and the mixture thus obtained was then made up to 1000 parts with water. A cotton fabric was printed with the paste thus prepared, heated for 10 minutes and washed subsequently. A brilliant yellow print was obtained.

We claim:

1. In a process for coloring cellulose material and textile material containing carbon amide groups with a water-soluble reactive dyestuff which is fixable in an alkaline medium, the improvement comprising applying thereto an aqueous preparation containing (*a*) the aforesaid dyestuff, (*b*) as fixing agent, a salt selected from the group consisting of alkali metal salts and alkaline earth metal salts of a carboxylic acid having at least two carbon atoms, which salt liberates carbonate when heated to a temperature above 80° C., and (*c*) a free carboxylic acid in an amount that the preparation has a pH value below pH 6, and heating the treated material to a temperature above 100° C.

2. In a process for printing cellulose material and textile material containing carbon amide groups with a water-soluble reactive dyestuff which is fixable in an alkaline medium, the improvement of applying thereto an aqueous printing paste containing (*a*) the aforesaid dye-

stuff, (*b*) as fixing agent, a salt selected from the group consisting of alkali metal salts and alkaline earth metal salts of a carboxylic acid having at least two carbon atoms, which salt liberates carbonate when heated to a temperature above 80° C., (*c*) a free carboxylic acid in an amount that the printing paste has a pH value of below pH 6, and (*d*) a thickening agent, and fixing the printings by heating the materials to a temperature above 100° C.

3. The improvement as claimed in claim 1, wherein the fixation is performed by steaming the materials at a temperature of 100 to 120° C. during 2 to 30 minutes.

4. The improvement as claimed in claim 1, wherein the fixation is performed by dry heating the materials to a temperature in the range of 100 to 200° C. during 15 seconds to 8 minutes.

5. The improvement as claimed in claim 1, wherein the free carboxylic acid is applied in an amount from 0.05 to 0.2 equivalent of free organic acid per equivalent of alkali formed from the aforesaid alkali metal and alkaline earth metal salt.

6. The improvement as claimed in claim 1, wherein an alkali metal salt of the trichloroacetic acid is used as fixing agent.

References Cited in the file of this patent

UNITED STATES PATENTS

2,892,670 Alsberg et al. ---------- June 30, 1959