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# SOLID VAT DYE COMPOSITIONS

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This invention relates to a composition containing a vat dyestuff which is suitable for use in coloring fabrics. In one specific aspect, it relates to a composition suitable for use in the home by the average housewife for coloring cellulosic fibers, nylon, silk, and the like with a vat dyestuff.

Vat dyes have a reputation among ultimate retail consumers for excellent fastness to both washing and light. Commercially, vat dyes are applied to fabrics in the textile mills by treating the goods with a solution of vat dyestuff made alkaline with caustic soda and containing sodium hydrosulfite as a reducing agent. The dyestuff is converted to its leuco form and is exhausted onto the fiber. A subsequent oxidizing operation reforms the dye on the fiber. When sodium hydrosulfite is used to reduce a vat dye, the hydrosulfite is oxidized by the dye and also by air to sodium hydrogen sulfite, which in turn hydrolyzes in aqueous solution to form hydrosulfurous acid. Hydrosulfurous acid is unstable in acid solution; thus enough alkali must be present to keep the pH of the bath decidedly on the alkaline side to prevent the decomposition of the free hydrosulfurous acid and to keep the vat dye in solution. Since it is essential to the dyeing operation to provide a distinctly alkaline dye bath, it is standard commercial practice to use a strong base, i.e. caustic soda, to maintain the required degree of alkalinity. Because of the deleterious effect of caustic soda on certain fabrics, it has been proposed from time to time by workers in the art to use a weaker base to maintain the alkalinity of the dye bath. For example, Sutton, in U.S. Patent 2,662,958, teaches a method of applying vat dyestuffs to cellulose acetate using a weaker alkali than caustic soda, e.g. ammonia or sodium carbonate, in conjunction with a swelling agent for the cellulose acetate.

However, the problem of obtaining light fast and wash fast dyes which are level or uniform in shade by skilled workers in the textile mills is not akin to that of obtaining these same results in a home dyeing operation which is conducted by an inexperienced user, such as the average housewife. It is obviously desirable to supply the housewife with a unitary packaged formulation which can be used in a dyeing operation by one having no skill in the dyeing art. Such a formulation or dyeing composition, in order to be suitable, must meet the following requisites:

(1) It must, after being placed in aqueous solution, provide a dye bath of sufficient alkalinity to keep the vat dyestuff in solution and to prevent decomposition of the reducing agent,

(2) It must not provide a dye bath of such high alkalinity as to constitute a danger to the inexperienced user,

(3) It must provide a dye bath capable of producing level dyeings without using the stringent controls exercised in the textile mills during the dyeing operation, and

(4) It must be in a physical form which can be readily distributed to and handled by the ultimate consumer.

Quite surprisingly, I have discovered a novel composition that meets all four of these requirements.

It is, therefore, an object of the present invention to provide a vat dyestuff composition in powdered form which is readily adaptable for home use and which can be used without danger by an unskilled person to produce level and uniform dyeings of light and medium shades and of high fastness to light and washing.

In accordance with the invention, my novel dyeing



composition comprises the following essential ingredients in the following approximate proportions:

2–10 parts by weight vat dyestuff,
80–100 parts by weight sodium ortho-silicate,
60–70 parts by weight sodium hydrosulfite, and
1–5 parts by weight of a solid non-ionic compound containing a plurality of contiguous oxyethylene and contiguous oxypropylene groups and having a molecular weight of at least about 7500.

The composition is admixed by the user with warm water to form a dye bath in a manner hereinafter described.

Any vat dyestuff is suitable for use in my formulations. Generally speaking, the common vat dyestuffs comprise anthraquinoid, indigoid, thioindigoid, pyranoid, and carbazole types. Especially good results are obtained using anthrones, benzanthrones, violanthrones, chloroindanthrones, anthraquinone thiazoles, anthrimides, benzanthrone acridines, pyranthrones, anthraquinone imidazoles and the indophenol vat sulfur colors. I have found that about 2–10 parts by weight (e.g. grams, if the parts by weight of the other materials added are measured in grams) of dyestuff is sufficient to obtain good dyeings of light to medium shade.

The use of sodium ortho-silicate in the composition to maintain the alkalinity of the subsequently formed dye bath is particularly critical. Since this compound is less basic than caustic soda, its use substantially precludes the possibility of injury to the inexperienced user involved in handling the dyeing composition or in the accidental splashing of dye bath in the eyes or on the skin. Moreover, because it is less basic, it reduces the danger of corrosion of aluminum washing machine parts which would ordinarily occur if a dye bath containing caustic soda was placed in the machine. I have also found that, among the weaker bases, sodium ortho-silicate is peculiarly suitable as an ingredient of my dyeing composition, since it is just strong enough to maintain sufficient alkalinity in the dye bath during the dyeing operation. The electromotive potential of sodium ortho-silicate is critical in that during the dyeing operation the vat color is maintained in form of the sodium leuco salt, but as the vat color is exhausted from the bath onto the fiber, the electromotive potential drops by hydrolysis and the dye on the cloth reverts gradually to its unreduced state. Thus, the true reoxidized color is fixed on the fabric as the dyeing operation nears completion and the conventional oxidation step is thereby obviated. I have found that about 80–100 parts by weight of sodium ortho-silicate must be present in the formulation. If less than 80 parts by weight is used, the dye bath formed upon dissolving the composition in water is not sufficiently alkaline. If more than 100 parts by weight of sodium ortho-silicate is present, there exists a possibility that this extra amount might have an irritating effect on the skin of the person performing the dyeing operation.

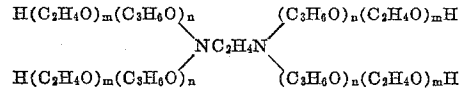
As I have already indicated, between about 60–70 parts by weight sodium hydrosulfite must be present in the formulation. If less than 60 parts is used, reduction is not complete and if more than 70 parts is used, the reduction is excessive to the point where the dye will not be absorbed by the fiber.

To insure consistently good results in the hands of an inexperienced user, my novel formulation must contain between about 1 and 5 parts by weight of a solid nonionic compound containing a plurality of contiguous oxyethylene and contiguous oxypropylene groups and having a molecular weight of about 7500–30,000. Preferred compounds of this class have the formula:

$$HO(C_2H_4O)a(C_3H_6O)b(C_2H_4O)cH$$

wherein *a*, *b* and *c* are integers having such values that

the molecular weight of the compound is at least about 7500. Compounds having the structure indicated above are solid hydrophobic condensation products of ethylene oxide and polyoxypropylene. They have a molecular weight ranging between about 7500 and 9000, and generally contain a predominance (generally from 75 to 90% by weight) of condensed ethylene oxide. Representative compounds of this type are sold commercially by the Wyandotte Chemical Company under the tradename "Pluronics." Also useful in the invention are the compounds sold under the tradename "Tetronics." These compounds are condensation products of ethylenediamine, propylene oxide and ethylene oxide and have a molecular weight as high as about 27,000–30,000. The "Tetronics" have the formula:



wherein *m* and *n* are average values sufficient to give the molecular weight indicated above. The compounds generally contain a predominance of condensed ethylene oxide. The presence of the non-ionic compound in the formulation effects a uniform and level distribution of the dyestuff on the surface of the fiber. The compound also serves as a flux in the rinsing operation to remove the silicate from the cloth.

To prepare the compositions of the invention the vat dyestuff powder, sodium ortho-silicate, sodium hydrosulfite, and solid non-ionic condensation product are dry-blended, in the above-indicated proportions, using conventional mixing techniques.

A dyeing operation, using the novel composition of the present invention, is performed as follows:

A dye bath is prepared in a washing machine or in any other suitable home receptacle by adding to about 4 to 7 quarts of warm water about 140–185 grams of the dyeing composition for each pound of goods to be dyed. The dye bath is conveniently maintained at a temperature of about 120–160° F. and the fabric to be treated is placed therein under moderate agitation for 15–30 minutes. The fabric is then removed from the bath and rinsed with water until the washings are colorless. As I have already indicated, the use of sodium ortho-silicate in my novel formulation obviates the conventional oxidizing step. If desired, however, the fabric, after removal from the dye bath, may be rinsed with a 2–10% by weight aqueous solution of sodium perborate containing a wetting agent. To insure complete fixation of the maximum possible amount of dye on the fiber, the fabric thus treated is thereafter rinsed with water.

In the examples that follow the dyestuffs used are referred to by their Colour Index (2nd edition) Name and Number, or Prototype Number in accordance with the Techincal Manual and Year Book of the American Association of Textile Chemists and Colorists, 1958 edition.

My invention is further illustrated by the following examples:

*Example I*

A dyeing composition is prepared by dry-mixing the following ingredients in the indicated quantities:

50 grams Vat Red (PR. 109), a commercially available indigo-type vat dyestuff,
1,000 grams sodium ortho-silicate,
600 grams sodium hydrosulfite, and
25 grams of a polyethylene oxide-polyoxypropylene condensate having a molecular weight of about 8000 and comprising about 80% by weight of condensed ethylene oxide.

This latter material is available commercially as "Pluronic F–68." The dyeing composition thus obtained is added to 12 gallons of water at a temperature of about 120–140° F. to form a dye bath suitable for treating 10 lbs. of goods. Ten lbs. of white cotton goods is added to the bath and is held therein using mild agitation for about 30 minutes. The cotton goods are then removed and rinsed twice with water until the final rinse is colorless. The resulting fabric has a pink color of excellent light and wash fastness.

*Example II*

The procedure of Example I is repeated using 10 lbs. of white linen. A light fast pink color is thus obtained.

*Example III*

A formulation is made having identical quantities of ingredients as that of Example I, using as a dyestuff Amanthrene Scarlet G (PR. 107), a commercially available thioindigo vat dye. Nylon goods (10 lbs.), treated in a dye bath prepared from this formulation, have a medium red color of excellent light fastness.

*Example IV*

A formulation is made having identical quantities of ingredients as that of Example I, using as a dyestuff Vat Brown 5 (PR. 121), a commercially available thioindigo vat dyestuff. Silk goods (10 lbs.) treated in a dye bath prepared from this formulation, have a brown color of excellent light and wash fastness.

*Example V*

A dyeing composition is prepared by dry-mixing the following ingredients in the indicated quantities:

75 grams Vat Green 1 [CI (1st ed.)-1101], a commercially available dimethoxyviolanthrone vat dyestuff,
900 grams sodium ortho-silicate,
700 grams sodium hydrosulfite, and
50 grams of a polyethylene oxide-polyoxypropylene condensate having a molecular weight of about 8000 and comprising about 80% by weight of ethylene oxide ("Pluronic F–68").

A dye bath is prepared as described in Example I and 10 lbs. of rayon goods is added thereto. The goods are kept in the dye bath under mild agitation for about 20 minutes. They are then removed, rinsed three times with water and thereafter allowed to dry. The resulting fabric has a medium green color of excellent light and wash fastness.

*Example VI*

The procedure of Example V is repeated, with the exception that the dyestuff used in the formulation is Vat Blue 6 (CI–1113), a chloro-indanthrone vat dyestuff. The resulting fabric has a blue color of excellent light and wash fastness.

*Example VII*

The procedure of Example V is repeated, with the exception that the dyestuff used in the formulation is Vat Yellow 2 (PR–9), an anthraquinone thiazole vat dyestuff. The resulting fabric has a medium yellow color of good light and wash fastness.

*Example VIII*

The procedure of Example V is repeated, with the exception that the dyestuff used is Vat Brown 1 (PR–118), an anthrimide-type vat dyestuff. The resulting fabric has a medium brown color of excellent light and wash fastness.

*Example IX*

The procedure of Example V is repeated, with the exception that the formulation described therein is modified by substituting for the dyestuff 25 grams of Vat Green 3 (PR–293), a benzanthrone acridine vat dyestuff. The resulting fabric has a light green color of excellent light and wash fastness.

*Example X*

The procedure of Example IX is repeated, with the exception that the dyestuff used is Vat Orange 9 (CI–1096), a pyranthrone vat dyestuff. The resulting fabric has a light golden orange color of excellent light and wash fastness.

*Example XI*

The procedure of Example IX is repeated, with the exception that the dyestuff used is Vat Brown 3 (CI–1151), an anthramide-type vat dyestuff. The resulting fabric has a light brown color of excellent light and wash fastness.

*Example XII*

The procedure of Example IX is repeated, with the exception that the formulation is modified by substituting for the dyestuff 80 grams of Vat Blue 43 (CI–969), an indophenol vat sulfur color. The resulting fabric has a medium blue color of good all-round light fastness properties.

*Example XIII*

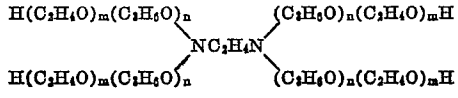
The procedure of Example XII is repeated, with the exception that the dyestuff used is Vat Brilliant Orange GR (PR–287), an anthraquinone imidazole and the non-ionic compound used is "Tetronic 704," a condensation product of ethylenediamine, propylene oxide and ethylene oxide having a molecular weight of about 27,000. The resulting fabric has a medium orange color of excellent light and wash fastness.

I claim:

1. A solid, storage stable dyeing composition for home use consisting essentially of 2–10 parts by weight of vat dyestuff powder, 80–100 parts by weight of sodium ortho-silicate, 60–70 parts by weight of sodium hydrosulfite and 1–5 parts by weight of solid non-ionic material selected from the group consisting of

$$HO(C_2H_4O)a(C_3H_6O)b(C_2H_4O)cH$$

and



wherein *a*, *b*, *c*, *m*, and *n* and integers having such values that the molecular weight of said material is about 7500–30,000, said composition being capable of providing, when admixed with water, a dye bath wherein the color becomes reoxidized on the fiber as the dyeing operation nears completion.



2. A solid, storage stable dyeing composition for home use consisting essentially of 2–10 parts by weight of vat dyestuff powder, 80–100 parts by weight of sodium ortho-silicate, 60–70 parts by weight of sodium hydrosulfite and 1–5 parts by weight of solid nonionic material having the formula:

$$HO(C_2H_4O)a(C_3H_6O)b(C_2H_4O)cH$$

wherein *a*, *b* and *c* are integers having such values that the molecular weight of said material is at least 8000, said composition being capable of providing, when admixed with water, a dye bath wherein the color becomes reoxidized on the fiber as the dyeing operation nears completion.

3. A composition according to claim 1 wherein said dyestuff is an anthraquinoid type vat dyestuff.

4. A composition according to claim 1 wherein said dyestuff is an indigoid type vat dyestuff.

5. A composition according to claim 1 wherein said dyestuff is a thioindigoid type vat dyestuff.

6. A composition according to claim 1 wherein said dyestuff is a pyranoid type vat dyestuff.

7. A composition according to claim 1 wherein said dyestuff is a carbazole type vat dyestuff.

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