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3,056,644 DYEING COMPOSITIONS

Jack Augustus Radley, John Simon Stanley, and Norman Leslie Smith, all of Reading, England, assignors to Maybourn Products Limited, London, England, a British company

No Drawing. Filed Oct. 14, 1960, Ser. No. 62,551
Claims priority, application Great Britain Oct. 23, 1959
13 Claims. (Cl. 8—93)

This invention relates to dyeing compositions suitable for use in household washing machines.

According to one aspect of the invention, there is provided a dyeing composition in paste form comprising a substantially anhydrous mixture of a dyestuff having an affinity for a textile fibre, or of a mixture of dyestuffs having an affinity for two or more different textile fibres, for example a "union" dyestuff mixture, and a liquid non-ionic detergent.

According to another aspect of the invention, there is provided a dyeing composition in powder or granular form comprising a mixture of a dyestuff having an affinity for a textile fibre, or of a mixture of dyestuffs having an affinity for two or more different textile fibres, for example a "union" dyestuff mixture, and one or more non-ionic supplemented by one or more anionic detergents if desired, which detergent or detergents may either be solid per se or, if liquid, may be caused to assume solid characteristics.

The dyeing compositions of the invention may contain, if desired, a small proportion of an optical whitening agent for the purpose of brightening pastel shades, for example the commercial product sold by I.C.I. Limited under the trade name Fluolite M.P. or the commercial product sold by Hickson & Welch Limited under the trade name Photine C.33. The dyeing compositions may also contain agents which assist the dyeing action of the compositions, such as for example, sodium chloride or anhydrous sodium sulphate. These agents may also assist the detergent action of the composition.

The dyeing compositions when in paste form may contain, if desired, a thickening agent for the paste, for example, carboxymethyl cellulose or starch, which agent may also act as a suspending medium for soil removed from a treated textile material. The thickening agent is preferably one which is soluble both in the liquid non-ionic detergent (to impart viscosity to the paste) and in water, such as for example a polyglycol ether having a molecular weight greater than 1,000. Alternatively, the non-ionic detergent may itself act as a thickening agent.

The dyeing compositions may also contain, if desired, a small proportion of sodium hexametaphosphate for the purpose of preventing the formation of precipitates due to the hardness of the water when dyeing at high temperatures. The proportion of sodium hexametaphosphate should not be less than 0.0001% (1 part per million) by weight of the quantity of water to which the composition is to be added and is preferably not less than about 0.0005% (5 parts per million).

By employing the dyeing compositions of the invention, the unexpected discovery has been made that it is possible to carry out in a single operation in a washing machine the simultaneous deterging and dyeing of textile articles, for example curtains or clothing, made from the same or different textile fibres such as cotton, woolen, silk, viscose, acetate rayon and nylon fibres. This property of the compositions of the invention is unexpected, since many commercial detergents restrain dyestuffs from dyeing fabrics, with the result that the effect of mixing many commonly used detergents and dyestuffs is, nor-

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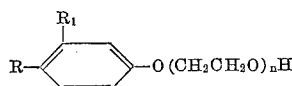
mally, to reduce the affinity of the fabric for the dyestuff, hence giving a lighter shade of dyeing. However, the compositions of the invention enable dyeing and washing to be performed at the same time, the resulting dyeing being comparable in strength with a dyeing carried out under similar conditions using the same quantity of dyestuff without a detergent being present.

It has been found that contrary to expectations the presence of water in the dyeing compositions in paste form of the invention exercises a deleterious influence on the physical properties of the paste. However, a paste of the required consistency may be obtained by the use of a substantially anhydrous liquid non-ionic detergent. This point is of importance in the filling of containers with the composition and in the dispensing of the composition into a dye-bath, and also in the storage of the composition.

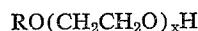
When the dyeing compositions are prepared with anionic detergents, it is necessary to alter the amount of dyestuff in the composition to obtain the same effect as a similar composition containing a non-ionic detergent. This alteration in the proportion of dyestuff in the composition is necessary to overcome the competition for active sites on the material between the detergent and the dyestuff.

Instead of using a single non-ionic detergent in the dyeing composition, there may be employed a mixture of non-ionic and anionic detergents, particularly a mixture containing a major proportion of a non-ionic detergent and a minor proportion of an anionic detergent, for example a sulphonated alkyl benzene. Such mixtures have been found to show improved detergency over that obtained by either detergent used alone and cause comparatively little effect on the exhaustion of the dyestuff.

Of the liquid non-ionic detergents which may be employed in the dyeing compositions of the invention, it is preferred to use an alkyl aryl polyethylene glycol ether, particularly an ether having the structural formula:



wherein R is octyl or nonyl and R₁ is hydrogen, methyl, octyl or nonyl, the mean value of n being in the range of from 4 to 20, preferably from 8 to 12, e.g. the commercial product sold by I.C.I. Limited under the trade name Lissapol NX. Other preferred non-ionic detergents which may be employed are alkyl polyglycol ethers having the general formula:



wherein R is a straight chain hydrocarbon radical containing from 10 to 16 carbon atoms, preferably 12 carbon atoms, and x has a value of from 10 to 30, preferably 20. Still further preferred liquid, non-ionic detergents which may be employed in the compositions are polyethylene glycol esters of fatty and resin acids (it being recognized in the art that the chain hydrocarbon radical contains not less than 12 carbon atoms), e.g. those sold by Atlas Powder Company (U.S.A.) under the trade name Renex, and polyglycol sorbitan esters of fatty acids having H.L.B. (hydrophilic/lyophilic balance) numbers (as defined by Griffin, J. Soc. Cosmetic Chem., 1949, 1, 311) of from 8 to 20.

The non-ionic detergents exemplified above are either liquids or soft waxy solids at room temperature. In order therefore to prepare a powdered or granulated dyeing composition, it is necessary to convert these detergents into a solid form. This may be done by adding to the detergent a sufficient quantity of an inorganic salt, such

as for example sodium chloride, to produce a solid product. The free flowing properties and rapid solubility characteristics of a solid product obtained in this manner may be improved by granulation. However, for convenience in use, it is preferred to convert the detergent, when liquid, into a solid form by mixing with an absorbent solid such as for example, finely divided silica (for example the product sold in the United Kingdom by Bush, Beach & Segner Bayley Limited under the trade name Aerosil), fuller's earth, or calcium silicate. The anionic detergents employed, if liquid, may be converted into a solid form in a similar manner.

Instead of converting the liquid or waxy detergent into a solid form in the manner described above, there may be employed in the formulation of the compositions of the invention the crystalline concretions formed between the non-ionic detergent and urea, for example the product sold by the Atlas Powder Company (U.S.A.) under the trade name Renex 25, which product is the solid concretion product formed from urea and a polyoxyethylene ester of tall oil averaging 16 oxyethylene groups per molecule.

Other compounds may also be included in the composition in minor proportions, preferably those which have the effect of "suspending" soil in solution, for example carboxymethyl cellulose.

Advantageously, the composition should contain sufficient detergent to give a concentration of at least 0.02%, preferably 0.1%, by weight of detergent in the quantity of water to which the composition is to be added. The amount of dyestuff present in the composition depends, of course, on the depth of shade required for the dyed textile articles and the quantity of textile articles to be dyed. In the case of the dyeing compositions in paste form, the amount of dyestuff present advantageously does not exceed 150% by weight of the amount of detergent, since a larger amount of dyestuff would affect the physical properties of the paste. In the event of 150% by weight of dyestuff in a paste composition giving too deep a shade, a smaller amount of dyestuff may be used, the balance being made up by a dyeing assistant, e.g. anhydrous sodium sulphate or sodium chloride. The amount of thickening agent used in the dyeing composition is dependent upon the dyestuff (plus dyeing assistant if used)/detergent ratio and also upon the particular dyeing assistant, if used, typical values for the amount of thickening agent being given in the specific examples below.

In the following examples, which illustrate dyeing compositions in accordance with the invention, all parts are by weight.

Example 1

4.25 parts of the dyestuff C.I. Direct Yellow 12 and 5.75 parts of anhydrous glauber salts (or 2.3 parts of common salt) were mixed with 7.5 parts of the commercial product Lissapol NX and 0.66 part of polyethylene glycol 1500 and the resulting mass was ground to a smooth paste.

The paste obtained in the above manner was used to dye cotton goods in the following manner.

The paste was added to approximately 7,000 parts (9 gallons) of water contained in a suitable vessel or machine, e.g., a domestic washing machine, in which the liquid can be suitably agitated and, preferably, can be simultaneously heated to its boiling point. The liquid was agitated for a few seconds and then 454 parts of soiled cotton goods were placed in the machine and the agitation and heating continued for a further twelve minutes, after which the liquid was removed from contact with the goods which were rinsed in cold water and dried in the usual manner. The goods were strongly and levelly dyed a shade of yellow and substantially all soil had been removed.

Example 2

4.8 parts of the dyestuff C.I. Direct Blue No. 24, 5.25

parts of anhydrous glauber salts (or 2 parts common salt) and 7.5 parts of Lissapol NX were ground together to form a paste. This paste when used to dye 454 parts of soiled cotton goods as described in Example 1 yielded a deep and level dyeing of clean material.

Example 3

A paste was prepared in the manner described in Example 1, the 0.66 part of polyethylene glycol 1500 being replaced by 0.8 part of ethyl cellulose (viscosity grade 50).

Example 4

A paste was prepared in the manner described in Example 1, the 0.66 part of polyethylene glycol 1500 being replaced by 2 parts of a block copolymer of ethylene oxide and propylene oxide sold by Wyandotte Limited (U.S.A.) under the trade name Pluronic F.68.

Example 5

A paste was prepared in the manner described in Example 1, the 0.66 part of polyethylene glycol 1500 being replaced by 0.066 part of an absorbent silica sold in the United Kingdom by Bush, Beach & Segner Bayley Limited under the trade name Aerosil.

Example 6

A paste was prepared in the manner described in Example 1, the 0.66 part of polyethylene glycol 1500 being replaced by 2.66 parts of sodium alginate.

Example 7

A paste was prepared as in Example 1, the 0.66 part of polyethylene glycol 1500 being replaced by 1.066 parts of sodium carboxymethyl cellulose.

Example 8

A paste was prepared as in Example 1, the 0.66 part of polyethylene glycol 1500 being replaced by 2.13 gms. of carrageen moss extract, e.g. the commercial product sold by J. W. Cummins & Sons Limited, under the trade name Carmoss.

Example 9

2.4 parts of the dyestuff C.I. Direct Blue No. 24, 8 parts of anhydrous glauber salts, 7.5 parts of Lissapol NX and 0.4 part of an optical whitening agent sold by Hickson and Welch Limited under the trade name Photine C.33 Liquid were thoroughly mixed by milling or grinding together to form a paste. When 13.3 parts of the paste obtained in the above manner were used in the manner described in Example 1, there were obtained pastel blue shades of good brightness on soiled material.

Example 10

A paste was prepared as in Example 1, the 7.5 parts of Lissapol NX being replaced by 7.4 parts of the commercial product Renex 20.

Example 11

A paste was prepared as in Example 1, the 7.5 parts of Lissapol NX being replaced by 7.5 parts of the commercial product Lubrol W (an alkyl polyglycol ether) and the polyethylene glycol 1500 being omitted.

Example 12

A paste was prepared as in Example 9, the 7.5 parts of Lissapol NX being replaced by the commercial product Texofor F.10 (an alkyl phenol polyglycol ether).

Example 13

A paste was prepared as in Example 9, the 7.5 parts of Lissapol NX being replaced by 7.5 parts of the commercial product Renex 30.

Example 14

A paste was prepared as in Example 1, the 4.25 parts of the dyestuff C.I. Direct Yellow 12 being replaced by a

mixture of 1.3 parts of the dyestuff C.I. Disperse Blue 7, 0.2 parts of the dyestuff C.I. Disperse Yellow 8, 0.5 part of the dyestuff C.I. Acid Blue 5, 1.3 parts of the dyestuff C.I. Direct Blue 1 and 0.5 part of the dyestuff C.I. Direct Yellow 11.

The resulting paste dyeing composition was added to approximately 7,000 parts of water contained in a domestic washing machine in which the liquid can be suitably agitated and, preferably, can be simultaneously heated to its boiling point. The liquid was agitated for a few seconds and then 450 parts of soiled cotton, woolen, silk, viscose, acetate rayon and nylon goods were placed therein and the agitation and heating continued for 4 minutes. The heating was then continued with agitation for 10 seconds at 2 minute intervals until the goods had been immersed for a total of 20 minutes. The goods were then removed from contact with the liquid, rinsed in cold water and dried in the normal way. The goods were dyed a deep and level shade of green and all soil had been removed.

Example 15

A paste was prepared as in Example 14, but the 7.5 parts of Lissapol NX were replaced by 7.5 parts of the commercial product Renex 20.

Example 16

33 parts of the dyestuff C.I. Direct Yellow 12, 17 parts of the dyestuff C.I. Direct Yellow 11, 8 parts of the dyestuff C.I. Disperse Yellow I and 78 parts of anhydrous sodium sulphate were mixed, and 20 parts of the resulting mixture were mixed with 30 parts of the commercial product Lissapol NX, to which had been added previously 140 parts of sodium chloride. To the resultant paste 10 parts of finely divided silica, for example the commercial product sold in the United Kingdom by Bush, Beach & Segner Bayley Limited under the trade name Aerosil, were added. The resulting powdered dyeing composition was then added to approximately 45,000 parts of water contained in a domestic washing machine, in which the liquid can be suitably agitated and, preferably, can be simultaneously heated to its boiling point. The liquid was agitated for a few seconds and then 900 parts of soiled cotton, woolen, silk, viscose, acetate rayon and nylon goods were placed therein and the agitation and heating continued for 4 minutes. The heating was then continued with agitation for 10 seconds at 2 minute intervals until the goods had been immersed for a total of 30 minutes. The goods were then removed from contact with the liquid, rinsed in cold water and dried in the normal way. The goods were dyed a deep and level shade of yellow and all soil had been removed.

Example 17

A powdered dyeing composition was prepared in the manner described in Example 16, the 30 parts of Lissapol NX and finely divided silica being replaced by 60 parts of the commercial product Renex 25 (which is the urea concretion of the product obtained by forming the polyoxyethylene ester of mixed fatty and resin acids). The powdered composition was then used to dye and deterge 900 parts of soiled goods in the manner described in Example 16 and yielded a deep and level dyeing of clean material.

Example 18

2.6 parts of the dyestuff C.I. Disperse Blue 7, 0.5 part of the dyestuff C.I. Disperse Yellow 13, 1 part of the dyestuff C.I. Disperse Yellow 13, 1 part of the dyestuff C.I. Acid Blue 5, 2.6 parts of the dyestuff C.I. Direct Blue 1, 0.5 part of the dyestuff C.I. Direct Yellow 11 and 2.8 parts of sodium chloride were mixed and 20 parts of the resulting mixture were mixed with 30 parts of the commercial product Belloid MY (an ethylene oxide modified long chain aliphatic alcohol) and 140 parts of sodium chloride as in Example 16. To the resultant paste 8 parts of finely divided silica, for example the commercial prod-

uct sold in the United Kingdom by Bush, Beach & Segner Bayley Limited under the trade name Aerosil, were added. The resulting powdered composition was then used to dye 900 parts of soiled goods in the manner described in Example 16 and yielded a deep and level dyeing of clean material.

Example 19

A powdered dyeing composition was prepared as in Example 18, but using 60 parts of the commercial product Renex 25 instead of the Belloid MY and finely divided silica. The resulting powdered composition was then used to dye soiled goods in the manner described in Example 16 and yielded a deep and level dyeing of clean material.

Example 20

A powdered dyeing composition was prepared as in Example 18, but there were also included in the composition 0.1 part of the optical whitening agent sold by Hickson & Welch Limited under the trade name Photine C. This composition yielded pastel blue shades of good brightness on soiled material.

Example 21

A powdered dyeing composition was prepared in the manner described in Example 20, the 0.2 part of Photine C being replaced by the optical whitening agent sold by I.C.I. Limited under the trade name Fluolite M.P. The resulting composition yielded pastel blue shades of good brightness on soiled material.

Example 22

13.61 parts of the dyestuff C.I. Direct Red 81, 0.25 part of the dyestuff C.I. Direct Yellow 44, 5.75 parts of the dyestuff C.I. Disperse Red 1, 1.5 parts of the dyestuff C.I. Disperse Red 13, 50 parts of the commercial product Renex 25 and 112 parts of sodium chloride were mixed and the resulting powdered composition was added to approximately 27,000 parts of hot water at about 80° C.

1125 parts of soiled cotton, silk, viscose, acetate rayon and nylon goods were then added to the stirred mixture, and agitation was continued for 12 minutes. The resulting dyed goods were then removed and rinsed thoroughly and dried in the normal way. The goods were dyed a deep and level shade of red and all soil had been removed.

The powdered dyeing composition described above may also be used to dye in a similar manner soiled woolen goods in which case the agitation should be carried out intermittently for the period of 12 minutes.

Example 23

A powdered dyeing composition was prepared in the manner described in Example 22 with the addition of 0.5 part of sodium hexametaphosphate to the dry mixture.

What is claimed is:

1. A method comprising concomitantly washing a fabric and dyeing the same to a predetermined shade by immersing said fabric in a water solution of an amount of dye sufficient to provide said shade and of a water-soluble non-ionic organic synthetic detergent, said dye being substantially unaffected by the presence of said synthetic detergent.

2. A method as claimed in claim 1 wherein the dye does not exceed about 150% by weight of the detergent.

3. A method as claimed in claim 1 wherein the detergent is employed in an amount to yield a concentration of no less than about 0.02% in said water.

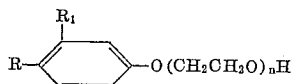
4. A method as claimed in claim 3 wherein the dye and detergent are dissolved in the water as a mixture in particulate form.

5. A method as claimed in claim 3 wherein the dye and detergent are dissolved in the water as a mixture in paste form.

6. A method as claimed in claim 3 wherein the deter-

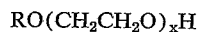
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gent is an alkyl aryl polyethylene glycol ether represented by the general formula:



wherein R is selected from the group consisting of octyl and nonyl, R₁ is selected from the group consisting of hydrogen, methyl, octyl and nonyl, and *n* is an integer in the range of from 4 to 20.

7. A method as claimed in claim 3 wherein the detergent is an alkyl polyethylene glycol ether represented by the general formula:



wherein R is a straight chain hydrocarbon radical containing from 10 to 16 carbon atoms and *x* is an integer in the range of from 10 to 30.

8. A method as claimed in claim 3 wherein the detergent is selected from the group consisting of a polyoxyethylene ester of a fatty acid having a radical containing not less than 12 carbon atoms, a polyoxyethylene ester of a resin acid having a radical containing not less than 12 carbon atoms, and mixtures thereof.

9. A method as claimed in claim 3 wherein the detergent is a polyoxyethylene ester of tall oil.

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10. A method as claimed in claim 3 wherein the detergent is combined with urea to form a solid concretion product.

11. A method as claimed in claim 3 wherein said detergent is combined with an absorbent solid.

12. A method as claimed in claim 3 further comprising dissolving a supplemental anionic detergent in the water.

13. A method as claimed in claim 3 further comprising adding a dye assistant to the water solution.

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