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3,097,042

TEXTILE COLOURATION PROCESS

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7 Claims. (Cl. 8—54.2)

This invention relates to a textile colouration process and more particularly it relates to a process for the production of fast colourations on cellulose textile materials.

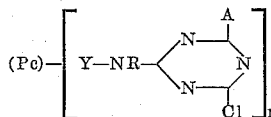
In British specification No. 797,946 there is described and claimed a process for the colouring of cellulosic textile materials which comprises impregnating the said textile materials in aqueous medium with a monoazo or polyazo dyestuff which is characterised in that it contains at least one ionogenic solubilising group and also in that it contains at least once a primary or secondary amino group carrying as N-substituent a 1:3:5-triazine radical containing at least one halogen atom attached to a carbon atom of the triazine ring, and thereafter subjecting the said textile materials to the action of an acid-binding agent in aqueous medium. Also in British specification No. 798,121 there is described and claimed a process for the colouration of cellulosic textile materials which comprises impregnating the said textile materials in aqueous medium with an anthraquinonoid dyestuff which is characterized in that it contains at least once an amino or substituted amino group carrying as N-substituent a 1:3:5-triazine radical containing at least one halogen atom attached to a carbon atom of the triazine ring, and thereafter subjecting the said textile materials to the action of an acid-binding agent in aqueous medium. Also in British specification No. 819,585 there is described and claimed a process for the colouration of cellulosic textile materials with a monoazo or polyazo dyestuff containing at least once a primary or secondary amino group carrying as N-substituent a 1:3:5-triazine radical containing at least one halogen atom attached to a carbon atom of the triazine ring, which comprises impregnating the textile material with an acid-binding agent by treatment with an aqueous medium containing up to 3% by weight of an acid-binding agent and subsequently treating the textile material with an aqueous solution of the dyestuff.

Also in British specification No. 838,336 there is described and claimed a process for the colouration of cellulosic textile materials with an anthraquinonoid dyestuff which is characterised in that it contains at least one ionogenic solubilising group and also in that it contains at least once an amino or substituted amino group containing as N-substituent a 1:3:5-triazine radical containing at least one halogen atom attached to a carbon atom of the triazine ring, which comprises impregnating the textile material with an acid-binding agent by treatment in an aqueous medium containing up to 3% by weight of the acid-binding agent and simultaneously or subsequently treating the textile material with an aqueous solution of the dyestuff.

Also in British specification No. 820,470 there is described and claimed a process for the colouration of cellulosic textile materials with a monoazo or polyazo dyestuff containing at least one ionogenic solubilising group and at least once a primary or secondary amino group carrying as N-substituent a 1:3:5-triazine radical containing at least one halogen atom attached to a carbon atom of the triazine ring, which comprises impregnating the textile material with an aqueous medium containing the dyestuff and up to 3.0% by weight of the acid-binding agent.

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Also in British specification No. 816,925 there is described and claimed a process for the colouration of cellulosic textile materials with phthalocyanine dyestuffs of the formula:



wherein (Pc) stands for a phthalocyanine radical, Y stands for a direct link or for a divalent bridging radical and the system (Pc)—Y— contains at least one solubilising group, R stands for hydrogen, alkyl, aralkyl or cycloalkyl, A stands for chlorine or O—B where B is a hydrocarbon radical or substituted hydrocarbon radical and n stands for a whole number, which comprises a treatment of the said textile materials with an aqueous solution containing up to 3% by weight of an acid-binding agent in conjunction with a treatment with an aqueous solution of the phthalocyanine dyestuff.

It is believed that the excellent fastness to wet treatments of the colourations obtained by the processes described in the above-mentioned British specifications is due to chemical reaction between the halogen atoms attached to the triazine rings present in the dyestuffs and the cellulose textile materials, so that the dyestuff is attached to the cellulose textile materials through an ether link.

It has however been occasionally found that when cellulose textile materials which have been dyed or printed with water-soluble dyestuff containing at least one dihalogeno-1:3:5-triazin-2-ylamino group, are stored in a humid atmosphere particularly in the presence of acid fumes, cleavage of the ether link occurs so that loose dyestuff is formed on the cellulose textile material and although such loose dyestuff can usually be removed by a subsequent washing treatment, staining of other portions of the cellulose textile material can result.

It has now been found that this disadvantage can be overcome by treating cellulose textile materials which have been coloured with a water-soluble dyestuff containing at least one dihalogeno-1:3:5-triazin-2-ylamino group, with a compound containing at least one primary or secondary amino group.

According to the invention there is provided an improved process for the colouration of cellulose textile materials which comprises treating the cellulose textile materials with a water-soluble dyestuff containing at least one dihalogeno-1:3:5-triazin-2-ylamino group and with an acid-binding agent and thereafter treating the cellulose textile material with an aqueous solution of a compound containing at least one primary or secondary amino group.

The water-soluble dyestuff containing at least one dihalogeno-1:3:5-triazin-2-ylamino group and the acid-binding agent may be applied simultaneously to the cellulose textile material or the dyestuff may be applied to the cellulose textile material and the so-dyed cellulose textile material subsequently treated with an acid-binding agent or the acid-binding agent may be applied to the cellulose textile material and the so-treated cellulose textile material subsequently treated with the water-soluble dyestuff containing at least one dihalogeno-1:3:5-triazin-2-ylamino group.

The water-soluble dyestuff containing at least one dihalogeno-1:3:5-triazin-2-ylamino group may be applied to the cellulose textile material by a dyeing or padding technique at a temperature of between 0° and 100° C., preferably at a temperature of between 20° and 100° C. The aqueous solution of the dyestuff used for dyeing or padding techniques may, if desired, contain customary

dyebath additives, for example migration inhibitors such as sodium sulphate, sodium alginate and water-soluble alkyl ethers of cellulose and urea.

Alternatively the water-soluble dyestuff containing at least one dihalogeno-1:3:5-triazin-2-ylamino group may be applied to the cellulose textile material by a printing technique and the aqueous printing paste containing the dyestuff may contain the conventional adjuvants for example urea, and thickening agents, for example sodium alginate. When applying such dyestuffs to the cellulose textile materials by a printing technique it is preferred to incorporate the acid-binding agent in the aqueous printing paste.

The treatment of the dyed or printed cellulose textile material with the aqueous solution of the compound containing at least one primary or secondary amino group may be carried out by immersing the said textile material in an aqueous solution of the said compound for a short time, for example between 1 and 30 minutes, preferably at an elevated temperature, for example between 50° C. and 100° C.

If desired a mixture of the said compounds can be used or the aqueous solution of the said compound can contain soap or a synthetic detergent, for example a mixture of alkyl-phenols condensed with ethylene oxide and a sulphated fatty alcohol.

If desired the coloured textile materials obtained by the process of the invention may be given a mild washing treatment, for example a treatment in an aqueous solution of soap or a detergent at a temperature of 85° C., to remove any dyestuff which has not reacted with the textile material.

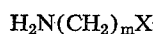
The quantity of the compound containing at least one primary or secondary amino group which is present in the aqueous solution is not critical but should not be less than 0.002 part for each 1000 parts of solution and it is preferred to use between 0.1 and 2 parts of the compound for each 1000 parts of solution.

Those compounds containing at least one primary or secondary amino group which have little or no solubility in water or are volatile in steam are preferably used in the form of their salts, for example salts with hydrochloric or acetic acids.

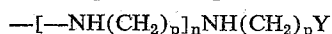
It is however preferred that the pH of the aqueous solution of the compound containing at least one primary or secondary amino group should not be less than 7.

As examples of compounds containing at least one primary or secondary amino group which may be used in the process of the invention there may be mentioned α -methylamino-acetic acid, ethylamine, diethylamine, dipropylamine, monoethanolamine, diethanolamine, dipropylamine, aniline, 2-naphthylamine-6-sulphonic acid, p-phenylene diamine, ortho-anilic acid and sulphanilic acid.

A preferred class of compounds containing at least one primary or secondary amino group are the compounds of the formula:



wherein m represents an integer from 2 to 6 and X represents an amino group or a group of the formula:



wherein Y represents an amino or hydroxy group, p represents an integer from 2 to 6 and n represents 0, 1 or 2.

As examples of this preferred class of compounds there may be mentioned ethylene diamine, 1:3-propylenediamine, N-(β -hydroxyethyl)ethylenediamine, triethylenetetramine, hexamethylenediamine and N-(β -aminoethyl)-ethylene diamine.

As examples of acid-binding agents which may be used in the process of the invention there may be mentioned sodium hydroxide, potassium hydroxide, sodium carbonate, trisodium phosphate and sodium metasilicate. If desired substances such as sodium bicarbonate or sodium

um trichloracetate can be used which on heating liberate an acid-binding agent.

The water-soluble dyestuffs used in the process of the invention may be, for example, of the monoazo, polyazo, anthraquinone, nitro or phthalocyanine series, which contain a water-solubilising group which is preferably a sulphonc acid group, and which may or may not contain coordinately bound metal atoms.

The water-soluble dyestuffs used in the process of the invention may be obtained by reacting the corresponding water-soluble dyestuff containing at least one primary or secondary amino group with a cyanuric halide such as cyanuric chloride or cyanuric bromide.

Water-soluble dyestuffs containing at least one dihalogeno-1:3:5-triazin-2-ylamino group which may be used in the process of the invention are described in British specifications Nos. 209,723, 298,494, 467,815, 772,030, 774,925, 781,930, 785,120, 785,222, 805,562, 826,405, 829,042, 828,355, 836,248, 837,035, 837,124, 837,985 and 837,990, and in Belgian specifications Nos. 556,092, 558,390, 558,801, 558,816, 558,884, 558,957, 559,782, 560,578, 560,889 and 569,115.

As examples of cellulose textile materials which may be used in the process of the invention there may be mentioned textile materials comprising cotton, linen and regenerated cellulose.

By the process of the invention colourations are produced on cellulose textile materials possessing excellent fastness to wet treatments even after the coloured textile material has been subjected to prolonged storage in a humid atmosphere.

The invention is illustrated but not limited by the following examples in which the parts are by weight:

Example 1

100 parts of cotton staple fibre are dyed in a solution of 1.5 parts of the dyestuff of Example 4 of British specification No. 785,222 and 150 parts of sodium chloride in 3000 parts of water for 30 minutes at 20° C. 3 parts of sodium carbonate are then added and dyeing continued for a further 1½ hours at 20° C. The cotton is then removed from the dyebath, squeezed and then treated for 15 minutes at 85° C. in a solution of 3 parts of diethanolamine and 9 parts of a mixture of alkyl-phenols condensed with ethylene oxide and a sulphated fatty alcohol in 3000 parts of water. The dyed cotton is then removed, rinsed in water and dried. The cotton is coloured a bright red shade possessing excellent fastness to wet treatments and the amount of loose dyestuff formed when the dyed cotton is stored in a humid atmosphere is very much less than a similar dyeing from which the 3 parts of diethanolamine have been omitted.

In place of the 3 parts of diethanolamine used in the above example there are used 3 parts of ethylene diamine or 3 parts of N-(β -hydroxyethyl)ethylenediamine or 3 parts of triethylenetetramine or 3 parts of hexamethylenediamine or 3 parts of N-(β -aminoethyl)ethylenediamine when similar results are obtained.

Example 2

In place of the 3 parts of diethanolamine used in Example 1 there are used 3 parts of aniline hydrochloride. A dyeing possessing similar fastness properties is obtained.

Example 3

In place of the 1.5 parts of the dyestuff of Example 4 of British specification No. 785,222 used in Example 1 there are used 1.5 parts of the dyestuffs of Example 1 of British specification No. 209,723. The cotton is coloured a bright red shade possessing excellent fastness to wet treatments and the amount of loose dyestuff formed when the dyed cotton is stored in a humid atmosphere is very much less than a similar dyeing from which the 3 parts of diethanolamine have been omitted.

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Example 4

In place of the 1.5 parts of the dyestuff of Example 4 of British specification No. 785,222 and the 3 parts of diethanolamine used in Example 1 there are used 1.5 parts of the dyestuff of Example 1 of British specification No. 209,723 and 3 parts of the sodium salt of sarcosine respectively. The cotton is coloured a bright red shade possessing excellent fastness to wet treatments and the amount of loose dyestuff formed when the dyed cotton is stored in a humid atmosphere is very much less than a similar dyeing from which the 3 parts of the sodium salt of sarcosine have been omitted.

In place of the 3 parts of the sodium salt of sarcosine used in the above example there are used 3 parts of ethylamine or 3 parts of diethanolamine or 3 parts of diethylamine or 3 parts of dipropylamine or 2 parts of ethylenediamine or 2 parts of diethanolamine or 3 parts of the sodium salt of orthanilic acid when similar results are obtained.

Example 5

In place of the 1.5 parts of the dyestuff used in Example 1 there are used 2.0 parts of the dyestuff of Example 1 of British specification No. 781,930 whereby the cotton is coloured a bright blue shade possessing excellent fastness to wet treatments and the amount of loose dyestuff formed when the dyed cotton is stored in a humid atmosphere is very much less than a similar dyeing from which the 3 parts of diethanolamine have been omitted.

Example 6

In place of the 1.5 parts of the dyestuff used in Example 1 there are used 2.0 parts of the dyestuff of Example 1 of British specification No. 785,120 whereby the cotton is coloured a bright orange shade possessing excellent fastness to wet treatments and the amount of loose dyestuff formed when the dyed cotton is stored in a humid atmosphere is very much less than similar dyeings from which the 3 parts of diethanolamine or the 3 parts of triethylenetetramine or the 3 parts of hexamethylenediamine or the 3 parts of ethylenediamine or the 3 parts of N-(β -hydroxyethyl)ethylenediamine have been omitted.

What I claim is:

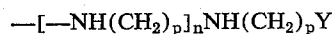
1. Process for improving the stability, during storage, of fastness to washing of cellulose textile materials which have been coloured by reaction with a water-soluble

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dyestuff containing at least one dihalogeno-1:3:5-triazin-2-ylamino group, which consists essentially in treating the so-coloured textile material with an aqueous solution of an amine of the formula:



wherein R is a radical selected from the class consisting of mono- and di-cyclic aromatic rings and the grouping $(\text{CH}_2)_m$, in which m represents one of the integers from 2 to 6; and X is a member selected from the class consisting of $-\text{NH}_2$ and



in which Y stands for a group selected from the class consisting of amino and hydroxy groups, p represents an integer from 2 to 6, and n represents one of the numbers 0, 1, and 2.

2. The process of claim 1, wherein said amine is ethylenediamine.

3. The process of claim 1 wherein said amine is N-(β -hydroxyethyl)ethylenediamine.

4. The process of claim 1, wherein said amine is triethylene tetramine.

5. The process of claim 1, wherein said amine is hexamethylenediamine.

6. The process of claim 1, wherein said amine is N-(β -aminoethyl)ethylenediamine.

7. The process of claim 1, wherein said so-coloured textile material is treated with said aqueous solution of said amine for a period of time between about 1 to 30 minutes at an elevated temperature between about 50 and 100° C.

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