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3,141,728

PROCESS FOR THE IMPROVEMENT OF THE FASTNESS TO WASHING OF DIRECT DYEINGS ON CELLULOSE-CONTAINING MATERIALS

Klaus Böckmann, Cologne-Stammheim, and Carl Taube, Leverkusen, Germany, assignors to Farbenfabriken Bayer Aktiengesellschaft, Leverkusen, Germany, a corporation of Germany

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It is known that the fastness to washing of direct dyeings on cellulose-containing materials can be improved by means of quaternary polyalkylene-polyamines and it is further known that the fastness to washing of such dyeings can also be increased by applying to the cellulose-containing materials hardenable resinous intermediate products and by then hardening these intermediate products, for example by heating to temperatures above 100° C. Finally, it is also known that hardenable resinous intermediate products have been applied to cellulose materials which had previously been dyed with direct dyestuffs and after-treated with quaternary polyalkylene-polyamines, and that the intermediate products have been subsequently hardened.

The object of the present invention is to provide a process for the improvement of the fastness to washing of direct dyeings on cellulose-containing materials by means of quaternary polyalkylene-polyamines and hardenable resinous intermediate products; this process consists in treating the direct dyeings on cellulose-containing materials with the polyalkylene-polyamines and the resinous intermediate products simultaneously and subsequently hardening the resinous intermediate products.

Surprisingly, the fastness to washing of direct dyeings on cellulose materials such as cotton and, particularly, staple fibre, is increased to a substantially greater extent by the simultaneous use of the above mentioned agents than by using them separately. Moreover, the dyeings which tend to change their shade upon treatment with quaternary polyalkylene-polyamines, show a reduced change of shade in most cases where hardenable resinous intermediate products are used at the same time.

Quaternary polyalkylene-polyamines suitable for the process according to the invention are chiefly the compounds obtainable by contacting ditertiary ethylene diamines with aliphatic dihalogen compounds which are described in U.S. Patent No. 3,009,761, furthermore quaternized polypropylene polyamines derived from the mineral acid esters of 1-amino-3-propanol which are described in German Patent No. 1,040,503 and the quaternary ammonium compounds of higher molecular weight to be used in combination with water-soluble sulfates as described in British Patent No. 761,668.

As hardenable resinous intermediate products there may be considered for example dimethylol-urea, dimethylol-ethylene-urea, dimethylol-dihydroxyethylene-urea, tetramethylol-acetylene-diurea, hexamethylol-melamine and their alkyl ethers.

The proportions of quaternary polyalkylene-polyamines and hardenable resinous intermediate products may vary within wide limits. In general, it is advantageous to use the hardenable resinous intermediate products in an excess of about the double or triple quantity by weight.

The following examples serve to illustrate the invention without, however, limiting the scope thereof.

Example 1

A staple fibre fabric dyed with 2% of the dyestuff obtainable according to Example 1 of U.S. Patent No. 2,712,006 is impregnated with the aqueous solution described below, then squeezed until its increase of weight

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amounts to 100% by weight, dried at 80-100° C. and heated to 150° C. for 5 minutes. The fabric is finally soaped as usual at 90° C. for 30 minutes, then rinsed and dried. The dyeing of the fabric thus treated has an excellent fastness to washing.

The solution employed contained per litre 50 g. of dimethylol-dihydroxyethylene-urea, 10 g. of magnesium chloride and 20 g. of the solution containing a quaternary polyalkylene-polyamine and being prepared by heating a mixture of 11.6 grams of 1,2-bis-(dimethyl-amino)-ethane and 12.7 grams of 1,4-dichloro-butane to 90-95° C. for about 40 hours, dissolving the reaction product in 50 millilitres of water and making up the solution with water to a total weight of 140 grams after the addition of 30 grams of ammonium sulfate.

If a cotton fabric dyed with the indicated dyestuff is after-treated in the manner described above, the dyeing is fast to repeated washing at 95° C.

Example 2

A staple fibre fabric is dyed with 1% of the dyestuff obtainable by coupling 1 mol of tetrazotized 4,4'-diamino-3,3'-dihydroxyphenyl with 2 mols of 2-amino-5-hydroxynaphthalene-1,7-disulfonic acid and subsequently converting the disazo compound formed into the copper complex. The fabric is finally impregnated with the aqueous solution described below, then squeezed until its increase of weight amounts to 100% by weight, dried at 80-100° C. and heated to 150° C. for 5 minutes. The fabric is finally soaped, rinsed and dried in conventional manner. The dyeing thus treated has an excellent fastness to washing.

The aqueous solution employed contained per litre 50 g. of dimethylol-dihydroxyethylene-urea, 10 g. of magnesium chloride, 5 g. of ammonium chloride and 30 g. of the solution containing a quaternary polyalkylene-polyamine and being prepared according to Example 1 of German Patent No. 1,040,503 in the following manner: 26 g. 1-amino-3-chloropropane hydrochloride were added to 40 g. of 40% aqueous sodium hydroxide solution and stirred at room temperature for 24 hours. Then the solution from which sodium chloride had separated was heated for two hours to 35-40° C., for 2 hours to 60-65° C. and for 2 hours on the boiling water bath. Thereupon in the course of about 4 hours 55 g. of dimethyl sulfate were dropped into the solution which was always kept weakly alkaline by the addition of sodium hydroxide solution. Finally the neutral solution was made up with water to 185 g. and filtered.

The dyeing is also very fast to washing if after-treated in the manner described above with a solution containing per litre 50 g. of dimethylol-dihydroxyethylene-urea, 10 g. of magnesium chloride and 40 g. of the solution containing a quaternary polyalkylene-polyamine and being prepared according to Example 3 of German patent specification No. 960,178 in the following manner: 90 parts by weight of polyethylene-imine (50%) were dissolved in 200 parts by weight of water and peralkylated at 20° C. with 300 parts by weight of dimethyl sulfate under neutralization with calcium hydroxide. Thereupon mineral acid was added and the reaction mixture was heated for some hours to 90-95° C. After neutralizing the reaction product with ammonia the precipitated calcium sulfate was filtered off and the filtrate filled up to 1500 parts by weight.

Example 3

A staple fibre fabric dyed with 2% of the dyestuff of Example 1 of U.S. Patent No. 2,917,504 is impregnated with the aqueous solution described below, then squeezed until its increase of weight amounts to 100%

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by weight, dried at 80–100° C. and heated to 140° C. for 10 minutes. The fabric is finally soaped, rinsed and dried in conventional manner. The dyeing of the fabric thus treated is very fast to washing.

The aqueous solution employed contained per litre 100 g. of hexamethylol-melamine, 10 g. of zinc chloride and 20 g. of the solution containing a quaternary polyalkylene-polyamine and being prepared according to Example 1 above.

Example 4

Mercerized cotton dyed with 1.5% of the dyestuff of Example 3 of U.S. Patent No. 2,777,839 is impregnated with the aqueous solution described below, then squeezed until its increase of weight amounts to 100% by weight, dried at 80–100° C. and heated to 160° C. for 3–4 minutes. The cotton is finally soaped, rinsed and dried in conventional manner. The dyeing of the cotton has an excellent fastness to washing.

The aqueous solution employed contains per litre 75 g. of dimethylol-dihydroxyethylene-urea, 10 g. of magnesium chloride and 15 g. of the solution containing a quaternary polyalkylene-polyamine and being prepared according to Example 1 above.

We claim:

1. Process for improving the fastness to washing of direct dyeings an cellulose-containing materials which comprises treating the direct dyeings on cellulose-con-

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taining materials with a quaternary polyalkylene-polyamine and a resinous intermediate product simultaneously and subsequently hardening the resinous intermediate product.

2. Process according to claim 1 where the polyalkylene-polyamine is a member of the group consisting of compounds obtainable by contacting ditertiary ethylene diamines with aliphatic dihalogen compounds, quaternized polypropylene polyamines derived from the sulfuric acid ester of 1-amino-3-propanol and quaternary ammonium compounds of higher molecular weight in combination with water soluble sulfates.

3. Process according to claim 1 wherein the resinous intermediate product is a member of the group consisting of dimethylol-urea, dimethylol-ethylene-urea, dimethylol - dihydroxyethylene - urea, tetramethylol - acetylene diurea, hexamethylol-melamine and their alkyl ethers.

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