Patented July 23, 1940 2,209,251

UNITED STATES PATENT OFFICE

2,209,251

DYEINGS FAST TO LIGHT

Hanns Rein, Bad Homburg vor der Hohe, Germany, assignor to I. G. Farbenindustrie Aktiengesellschaft, Frankfort-on-the-Main, Germany

No Drawing. Application March 22, 1939, Serial No. 263,402. In Germany March 24, 1938

6 Claims. (Cl. 8—120)

The present invention relates to dyeings and it especially relates to dyeings fast to light. It is an object of the present invention to produce dyeings fast to light on fibers which have been made capable of adsorbing wool dyestuffs.

As is well known, dyeings obtained by dyeing with the usual wool dyestuffs animalized materials of natural or artificial origin, especially fibrous materials from cellulose or cellulose derivatives exhibit in most cases an essentially decreased fastness to light compared with the dyeings obtained with the same dyestuffs on wool. According to the kind of fiber, dyestuff and animalizing agent, these differences in the fastness to light may be very great; thus, it occurs sometimes that dyestuffs which, as such, display a very good fastness to light, when applied to animalized fibers which have been rendered capable of adsorbing acid wool dyestuffs, for instance by introduction of nitrogen-, sulfur- or phosphorus-containing compounds, yield dyeings the fastness to light of which is too small for practical use.

Now I have found that the fastness to light of such dyeings may considerably be enhanced by incorporating with the animalized artificial or natural materials non-basic organic compounds of about 10–30 carbon atoms containing sulfuric acid radicals. Sulfonic acids of poly-halogeno-poly-hydroxy-triphenyl-methanes, for instance, have proved to be especially suitable. As artificial or natural materials there may be used above all fibers, ribbons, films and layers from regenerated cellulose, cellulose derivatives, cotton, flax, hemp, ramie and jute. The procedure may be as follows: Either the finished dyeing is subjected to the action of the products containing the sulfuric acid radicals, or the after-treatment is effected in the dyeing bath or also the animalized materials are treated before dyeing. The reaction may be performed at ordinary or elevated temperature. It is, furthermore, possible to add to the spinning solution or to the spinning or after-treating baths the sulfonation product and the animalizing agent or the product of reaction obtained from both agents.

In many cases, the present process effects, at the same time, a more uniform dyeing of wool and artificial fibers. Even by after-treating dyeings, a subsequent levelling effect is in some cases obtained.

The following examples serve to illustrate the invention but they are not intended to limit it thereto:

Example 1

Viscose fibers which have been prepared by the process of U. S. Patent 2,065,175 and which have been animalized by incorporating with the spinning solution 4% of the condensation product from trichlorinated hard paraffin and poly-ethylene-polyamine are dyed, in the presence of wool, with 2% of the following dyestuffs:

benzidine → naphthylamine-3.6-disulfonic acid
benzidine → phenol, condensed with para-toluene-sulfochloride sulfochloride
(cf. German Patent No. 261,047),
4-toluidine-2-sulfanilide→1-naphthol-4-sulfonic acid
(cf. German Patent No. 230,594),
4-acetylamino-1-aminobenzene→1-para-toluene-sulfoamino-8-naphthol-3.6-disulfonic acid
(cf. Schultz, Farbstofftabellen, 7th ed., No. 105)
aniline→2-chloro-acetylamino-5-naphthol-7-sulfonic acid
(cf. German Patent No. 557,126)
1.8-amino-naphthol-3.6-disulfonic acid→para-tolyl-1-naphthyl-amino-8-sulfonic acid
(cf. Schultz, l. c. 248),
1.4-diamino-3-phenoxy-anthraquinone-2-sulfonic acid
(cf. German Patent No. 563,201)
diethyl-benzyl-diamino-diphenyl-methane-disulfonic acid
(cf. Schultz, l. c. 806)

It results that the dyeings obtained on the animalized cellulose fiber display a considerably smaller fastness to light than the corresponding dyeings on wool. If, however, the dyeings produced on the animalized fiber or on a mixed fabric from animalized fiber and wool are treated in a rinsing bath containing per liter 0.6 g. of 3'.5'.3''.5''-tetrachloro-2'.2''-dihydroxy-triphenyl-methane-2-sulfonic acid and 0.2 cc. of acetic acid of 30 per cent strength, they become as fast to light as those produced on wool; a levelling effect is in most cases produced at the same time.

Example 2

Cuprammonium fiber is animalized by incorporating with the spinning solution 3% of the condensation product named in Example 1 and treated in the acid spinning bath with low content of acid, with 2 g. per liter of 4.3'.5'.-3''.5''-penta-chloro-2'.2''-dihydroxy-triphenyl-methane-2-sulfonic acid. Instead of adding the sulfonic acid to the acid spinning bath, the fibrous material may also be treated, after washing, with a solution of 2 g. per liter of the above sulfonic acid, a small amount of acetic acid being added.

The material thus treated is then dyed with 1% of the dyestuff para-amino-benzoyl-ethyl-alpha-naphthylamine→1-benzoylamino-8-naphthol-4.6-disulfonic acid (cf. German Patent No. 269,213); a dyeing is thus obtained which displays a considerably better fastness to light than the dyeing obtained on a fiber not subjected to the said pretreatment. The same result is obtained if the

starting material is a mixed yarn consisting of equal parts of wool and the above-mentioned fiber.

Example 3

Fibers which have been animalized by means of the product obtained by the action of carbon disulfide upon poly-ethylene-imine are dyed with the dyestuff mentioned in Example 2. The dyed material is then after-treated in a rinsing bath containing 0.6 g. per liter of the tetrachloro-dihydroxy-triphenyl-methane-sulfonic acid named in Example 1 and acetic acid. The dyeing thus obtained is considerably faster to light than without the said treatment.

Example 4

Cellulose fibers are pre-treated with the condensation product from ethylene-imine and octodecyl-bromide and then dyed with one of the wool dyestuffs mentioned in the preceding examples. The dyeing thus obtained is treated in a rinsing bath, acidified with acetic acid, and containing per liter 1 g. of the bis-(1.2-dichlorobenzene-4-sulfonyl-3'-aminobenzene-1'-sulfonyl-3''-aminobenzene-1''-sulfonyl)-diamino-stilbene-disulfonic acid named in German Patent No. 544,976. A considerable improvement of the fastness to light is thus obtained.

Example 5

Viscose artificial silk is animalized by adding to the viscose spinning solution, shortly before the spinning process, the product obtained by the action of 3'.5'-3''.5''-tetrachloro-2'.2''-dihydroxy-triphenyl-methane-2-sulfonic acid upon the condensation product from trichlorinated hard paraffin and polyethylene-polyamine; the material thus obtained is then dyed, in the manner usually applied in wool dyeing, in a neutral bath with the dyestuff No. 864 (Schultz, l. c.) or with the dyestuff No. 785 (Schultz, l. c.). The fastness to light of the dyeings thus obtained is practically the same as that of the wool, which has been dyed simultaneously, the tinctorial strength being, surprizingly, in better conformity with that of the wool than is the strength of a dyeing produced on an animalized fiber obtained as usual without the addition of the above product.

The following table indicates a certain number of further examples which illustrate the process of the present invention. The following dyestuffs have been used:

(1) Para-amino-benzoyl-ethyl-alpha-naphthylamine→ benzoyl-K-acid (cf. German Patent No. 269,213) (A)

(2) The dyestuff obtained by condensation of para-chloro-benzaldehyde with ethyl-benzyl-meta-toluidine, trisulfonation, oxidation and melting with para-phenetidine (cf. German Patents Nos. 293,352 and 287,003) (B)

Benzidine → naphthylamine-3.6-disulfonic acid
Benzidine → phenol, condensed with para-toluene-sulfochloride

(cf. German Patent No. 261,047) (C)

(4) The dyestuff prepared from anisaldehyde, ethylbenzyl-aniline-sulfonic acid and para-phenetidine according to the process of Example 5 of German Patent No. 293,352. (D)

(5) 4-amino-3-methyl-hexahydro-diphenyl-azo-sulfotolyl-H-acid (cf. German Patent No. 533,617) (E)

(6) H-acid→ 1.8-naphthylamino-sulfonic acid (F)

(7) 4-toluidine-2-sulfanilide→ 1.4-naphthol-sulfonic-acid (cf. German Patent No. 230,594) (G)

(8) Sodium 1.4-diamino-2-phenoxy-anthraquinone-3-sulfonate (cf. German Patent No. 563201) (H)

(9) Primulin-sulfonic acid→phenyl-gamma-acid (I)

(10) Aniline→ 2-chloro-acetylamino-5-naphthol-7-sulfonic acid (cf. German Patent No. 557,126) (K)

(11) A mixture of 3.3'-toluidine-6.6'-disulfonic acid→ 2 mols of aceto-acetic acid anilide and dyestuff B (L)

(12) Aniline (2 mols)→N-(2'-hydroxy-1'-naphthoic-acid-6'-sulfonyl)-1-amino-8-naphthol-4.6-disulfonic acid (cf. German Patent No. 274,082) (M)

(13) A mixture of para-toluidine-ortho-sulfanilide→ 4'-sulfophenyl-3-methyl-5-pyrazolone (cf. German Patent No. 226,239) and para-toluidine-ortho-sulfanilide→ 1.4-naphthol-sulfonic acid (cf. German Patent No. 230,594) and the dyestuffs Nos. 1199 and 1200 (Schultz, l. c.). (N)

(14) A mixture of 1-amino-4-(4'-acetylamino-anilido)-anthraquinone-2-sulfonic acid (cf. German Patent No. 280,646) and ortho-amino-tolylsulfone $\xrightarrow{\text{acid}}$ 2.8.6-amino-naphthol-sulfonic acid and sulfonated 3-hydroxy-2-quinophthalone (cf. French Patent No. 775,935) (O)

	Animalizing agent	Dyestuff	Treating agent	Treating manner	Treating temperature
					Degrees
1	Reaction product from trichlorohard-paraffine and polyethylene-polyamine.	2% of dyestuff A	3'5'3''5''-tetrachloro-2'2''-dihydroxy-triphenyl-methane 2-sulfonic acid.	Treatment after dyeing	20
2	do	2% of dyestuff B	do	do	20
3	do	2% of dyestuff B	do	Treatment before dyeing	20
4	do	1% of dyestuff A	do	do	20
5	do	2% of dyestuff C	do	Treatment after dyeing	20
6	do	2% of dyestuff D	do	do	20
7	do	2% of dyestuff Nr. 51 (Schultz, l. c.).	do	do	20
8	do	2% of dyestuff E	do	do	20
9	do	2% of dyestuff F	do	do	20
10	do	2% of dyestuff G	do	do	20
11	do	2% of dyestuff H	do	do	20
12	do	2% of dyestuff J	do	do	20
13	do	2% of dyestuff K	do	do	20
14	do	0.9% of dyestuff L	do	Treatment immediately after spinning.	20
15	do	2% of dyestuff D	See Nr. 1	Treatment after dyeing	60
16	Pentachlorohard-paraffine and polyethylene-polyamine.	2% of dyestuff D	do	do	100
17	Pentachlorohard-paraffine and ethylenediamine.	2% of dyestuff D	do	do	100

	Animalizing agent	Dyestuff	Treating agent	Treating manner	Treating temperature
					Degrees
8	Polyethyleneimine	2% of dyestuff D	See Nr. 1	Treatment after dyeing	100
9	Trichlorohard-paraffine and polyethylene-polyamine treated with dimethylsulfate.	2% of dyestuff D	do	do	100
10	Carbon disulfide and polyethyleneimine.	2% of dyestuff D	do	do	100
11	Octodecylbromide and ethyleneimine.	2% of dyestuff D	do	do	100
12	Reaction product from trichlorohard-paraffine and polyethylene-polyamine treated with 3' 5' 3'' 5''-Tetrachloro-2' 2''-dihydroxy-triphenyl-methane-2-sulfonic acid.	1% of dyestuff A	do	Treatment before spinning	
23	do	2% of dyestuff M	do	do	
24	Mixed yarn consisting of 50% wool and 50% material as per Nos. 1-15.	1.5% of dyestuff N	do	Treatment after dyeing	20
25	do	0.75% of dyestuff O	do	do	20

I claim:

1. The process of improving the fastness to light of dyeings obtained on animalized fibrous textile materials by means of wool dyestuffs which comprises incorporating in these materials a polyhalogen-polyhydroxy-triphenylmethane-sulfuric acid.

2. An animalized fibrous textile material which contains a polyhalogen-polyhydroxy-triphenyl-methane-sulfuric acid.

3. The process of improving the fastness to light of dyeings obtained on animalized fibrous textile materials by means of wool dyestuffs which comprises incorporating in these materials 3', 5', 3'', 5''-tetrachloro-2', 2''-dihydroxy-triphenyl-methane-2-sulfonic acid.

4. The process of improving the fastness to light of dyeings obtained on animalized fibrous textile materials by means of wool dyestuffs which comprises incorporating in these materials 4, 3', 5', 3'', 5''-penta-chloro-2', 2''-dihydroxy-triphenyl-methane-2-sulfonic acid.

5. An animalized fibrous textile material which contains 3', 5', 3'', 5''-tetrachloro-2', 2''-dihydroxy-triphenyl-methane-2-sulfonic acid.

6. An animalized fibrous textile material which contains 4, 3', 5', 3'', 5''-penta-chloro-2', 2''-dihydroxy-triphenyl-methane-2-sulfonic acid.

HANNS REIN.