

UNITED STATES PATENT OFFICE.

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FAST DYEING ON THE FIBER.

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A known method of producing fast dyeings on the fiber consists in impregnating the fiber with a compound adapted to be coupled and then treating the fiber with a solution of a diazo compound. In cases in which the compound adapted to be coupled has a certain affinity for the fiber it is sufficient merely to wring out the impregnated fiber and to conduct the coupling without previously drying. In producing printed colors, the material is previously prepared with a compound adapted to be coupled, such as for example 2-oxynaphthalene, and is printed with a thickened diazo-solution; or the material may be printed with a mixture of an alkali compound of 2-oxynaphthalene and a stable diazo compound, after which the material is steamed or exposed to the air and the dyeing developed by treatment with an acid.

In the known process difficulties are associated with the diazotization. By the present invention these difficulties are avoided and the process is conducted very simply by carrying out the preparation of the diazo compound on the fiber where it unites directly with the coupling component. The preparation of a diazo-solution or the use of a stable diazo compound is accordingly unnecessary.

One method of carrying out the invention consists in impregnating the fiber with a solution containing a nitrite and a compound adapted to be coupled and after wringing the damped goods treating them with a solution of a salt of a diazotizable base. The solution of this salt may contain an excess of acid and a metal salt may be added to it if desired.

In printing, the goods may be printed with a thickened mixture of both components to-

gether with a nitrite and after drying the goods the dyestuff may be developed by action of an acid or of an acid salt. The printing colours are characterized by their high stability.

Artificial threads consisting of cellulose acetate may advantageously be dyed according to the invention by treating them at a raised temperature in a solution or suspension comprising a mixture of both components and a nitrite, with the addition of a soap, Turkey-red oil or the like; after wringing, the dyestuff is formed by treatment with a warm solution of acid reaction. The production of azo dyes on the fiber may be combined with dyeing with any suitable dyes, whereby any desired mixed shades may be obtained.

The new process is also of interest in connection with art industries, such as for example painting materials by hand or by means of a stencil.

The following examples illustrate the invention without limiting it:

*Example 1.*—10 grams of cotton yarn are immersed in 200 ccm. of a solution containing per litre 10.5 grams of the anilide of 2-oxynaphthalene-3-carboxylic acid, 17.5 ccm. of a solution of sodium hydroxide of 34° Baumé specific gravity, 22 ccm. of Turkey red oil, 8 ccm. of formaldehyde of 30 per cent strength and 30 grams of sodium nitrite. The yarn is wrung and then introduced into 200 ccm. of a solution containing 0.95 grams of the hydrochloride of 2.5 dichloraniline and 7.5 grams of aluminium sulphate per liter. The dyeing obtained is reddish orange.

*Example 2.*—10 grams of cotton yarn are immersed in 200 ccm. of a solution containing per liter 3 grams of di-acetoacetic acid-ortho-tolidinid,



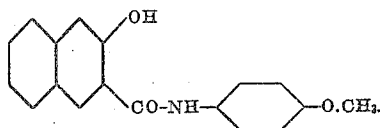
3 ccm. of Turkey red oil, 4.5 ccm. of a solution of sodium hydroxide of 34° Baumé specific gravity, 30 grams of anhydrous sodium sulphate and 25 grams of sodium nitrite. After the material has been wrung the dyeing is developed in 200 ccm. of a solution containing per litre 2.4 grams of 2-chloranilinehydrochloride and 50 grams of

sodium chloride. A clear yellow is thus obtained.

*Example 3.*—10 grams of cotton yarn are immersed in 200 ccm. of a solution containing per liter 3 grams of di-acetoacetic acid-ortho-tolidinid, 3 ccm. Turkey red oil, 4.5 ccm. sodiumhydroxide solution of 34° Baumé specific gravity, 30 grams of anhydrous so-

dium sulphate, 50 grams of sodium nitrite and 3 grams of Indigosol O, the sulfuric acid ester of the leuco compound of indigo. After the material has been wrung the dyeing is developed in 200 ccm. of a solution containing per liter 2.4 grams of 2-chloraniline hydrochloride, 50 grams of sodium chloride and 12.5 grams of aluminium sulphate. A yellowish green dyeing is obtained.

*Example 4.*—10 grams of cotton yarn are immersed in 200 ccm. of a solution containing per liter 1 gram of the para-aniside of 2-oxynaphthalene-3-carboxylic acid



3.2 ccm. of a sodium hydroxide solution of 34° Baumé specific gravity, 4 ccm. of Turkey red oil, 1.2 ccm. of formaldehyde of 30 per cent strength and 50 grams of sodium nitrite. The material is wrung and the dyeing is developed in 200 ccm. of a solution containing per liter 0.6 grams of 5-nitro-2-amino-1-methyl benzene, 7.5 ccm. of acetic acid of 30 per cent strength, 50 grams of sodium chloride and 5 grams of aluminium sulphate, the temperature being about 35° C. A Bordeaux dyeing is obtained.

*Example 5.*—20 grams of cellulose acetate artificial silk are treated for about 30 minutes at 55–60° C. in 500 ccm. of a solution containing per liter 0.4 grams of aminoazobenzene, 5 ccm. of a solution of the sodium salt of 2-oxynaphthalene of 10 per cent strength, 50 ccm. of a soap solution of 5 per cent strength and 200 ccm. of a solution of sodium nitrite of 10 per cent strength. The material is wrung and the dyeing is developed for about 30 minutes at about 50° C. in 500 ccm. of a solution containing 50 ccm. of acetic acid of 6° Baumé specific gravity per liter. A yellow red dyeing is obtained.

*Example 6.*—20 grams of cellulose acetate artificial silk are treated for about 30 minutes at 50° C. in 500 ccm. of a solution containing per liter 1 gram of 4-chloro-2-amino-1-methoxybenzene hydrochloride, 1.6 grams of the sodium salt of 2-oxynaphthalene-3-carboxylic acid, 50 ccm. of a soap solution of 5 per cent strength and 200 ccm. of sodium nitrite solution of 10 per cent strength.

After wringing the material the dyeing is developed for about 30 minutes at 50° C. in 500 ccm. of a solution containing 50 ccm. of acetic acid of 6° Baumé specific gravity per liter. A Bordeaux dyeing is thus obtained.

*Example 7.*—20 grams of cellulose acetate artificial silk are treated for about 60 minutes at 55–60° C. in 500 ccm. of a solution containing per liter 0.4 grams of 2,2'-dichlorbenzidine, 2.4 grams of the sodium salt of 2-oxynaphthalene-3-carboxylic acid, 50 ccm. of a

soap solution of 5 per cent strength and 200 ccm. of a sodium nitrite solution of 10 per cent strength. The material is wrung and the dyeing is then developed for about 30 minutes at 50° C. in 500 ccm. of a solution containing 50 ccm. of acetic acid of 6° Baumé specific gravity per liter. A blue red dyeing is obtained.

*Example 8.*—20 grams of cellulose acetate artificial silk are treated for about 60 minutes at 55–60° C. in 500 ccm. of a solution containing per liter 0.04 gram of 2,2'-dichlorbenzidine, 0.1 gram of orthotolidine, 2.4 grams of the sodium salt of 2-oxynaphthalene-3-carboxylic acid, 50 ccm. of a soap solution of 5 per cent strength and 200 ccm. of sodium nitrite solution of 10 per cent strength. After wringing the material the dyeing is developed for about 30 minutes at 50° C. in 500 ccm. of a solution containing 50 ccm. of acetic acid of 6° Baumé specific gravity per liter. A violet dyeing is obtained.

*Example 9.*—20 grams of cellulose acetate artificial silk are treated for 60 minutes at 60 to 65° C. with 500 ccm. of a solution containing per liter 0.2 grams of dianisidine, 2.4 grams of the sodium salt of 2-oxynaphthalene-3-carboxylic acid, 50 ccm. of a soap solution of 5 per cent strength and 200 ccm. of a sodium nitrite solution of 10 per cent strength. After wringing the material the dyeing is developed for about 30 minutes at 50° C. in 500 ccm. of a solution containing 50 ccm. of acetic acid of 6° Baumé specific gravity per liter. A blue dyeing is obtained.

*Example 10.*—20 grams of cellulose acetate artificial silk are treated for about 60 minutes at 60 to 65° C. in 500 ccm. of a solution containing per liter 1.2 grams of 1-aminonaphthalene, 3 grams of the sodium salt of 2-oxynaphthalene-3-carboxylic acid, 50 ccm. of a soap solution of 5 per cent strength and 500 ccm. of a sodium nitrite solution of 10 per cent strength. After wringing the material the dyeing is developed for about 30 minutes at 55 to 60° C. in a solution containing 50 ccm. of acetic acid of 6° Baumé specific gravity per liter. A black dyeing is obtained.

After dyeing and washing, the artificial silk is soaped for about 15 minutes at 40–50° C.

*Example 11.*—In 40 ccm. of water are dissolved 6 grams of di-acetoacetic acid-ortho tolidinid, 6 ccm. of Turkey red oil and 1 ccm. of sodium hydroxide solution of 34° Baumé specific gravity. There are then added to the solution 5 grams of 2-chloraniline, 2 grams of sodium nitrite and 40 grams of neutral starch tragacanth thickening (6:100). The unprepared goods are printed with this printing colour, dried and then developed at boiling temperature in a solution containing 20–30 grams of aluminium sulphate or sodium bisulphate per liter. The

goods are then washed, soaped and again washed. A clear yellow is obtained.

If there is added to the above printing colour Indigosol (the ester of the leuco compound from indigo as obtainable in commerce) a green shade is obtained.

*Example 12.*—In 40 ccm. of water are dissolved 2 grams of the para-anisidide of 2-oxy-naphthalene-3-carboxylic acid, 8 ccm. of Turkey red oil and 6.4 ccm. of sodium hydroxide solution of 34° Baumé specific gravity. There are then added to the solution 1.2 grams of 1-methyl-2-amino-5-nitrobenzene, 1.2 grams of sodium nitrite and 40 grams of neutral starch tragacanth thickening (6:100). The further treatment is as in the preceding example. A Bordeaux dyeing is obtained.

*Example 13.*—A printing colour is prepared as follows: 3 grams of the beta-naphthalide of 2-oxy-naphthalene-3-carboxylic acid, 6 ccm. Turkey red oil, 9 ccm. sodium hydroxide solution of 34° Baumé specific gravity, 20 ccm. of water, 1.4 grams of the hydrochloride of para-phenylenediamine-azo-1-methoxy-4-methyl-2-aminobenzene, 0.8 grams of sodium nitrite and 40 grams of a neutral starch tragacanth thickening (6:100). The material printed with the above colour is treated as in Example 11. A dark gray is obtained.

What I claim is,—

1. In the process for producing fast dyeings on the fiber involving the diazotization, by treatment with a soluble nitrite and a

reagent having an acid reaction, of a diazotizable amine on the fiber in the presence of a compound adapted to be coupled, the steps which consist in impregnating the fiber with a mixture comprising the compound adapted to be coupled and the soluble nitrite, and, thereafter, treating the so-impregnated fiber with a second mixture comprising the reagent having an acid reaction.

2. In the process of producing fast dyeings on cellulose acetate fiber involving the diazotization, at a temperature of about from 30° C. to about 75° C., by treatment with a soluble nitrite and a reagent having an acid reaction, of a diazotizable amine on said fiber, in the presence of a compound adapted to be coupled, the steps which consist in impregnating cellulose acetate fiber in a bath comprising the compound adapted to be coupled and the soluble nitrite, removing the fiber from said bath, wringing the fiber, and developing the dyeing on the so-impregnated and wrung fiber by treatment of the same in a second bath comprising a reagent having an acid reaction.

3. A process for producing fast dyeings on the fiber, which comprises impregnating the fiber with a solution containing a compound adapted to be coupled and a nitrite, and treating the moist fiber, after wringing, with a solution containing a salt of a diazotizable base.

In testimony whereof I affix my signature.

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