

UNITED STATES PATENT OFFICE.

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PROCESS OF DYEING AND PRODUCT THEREOF.

964,816.

Specification of Letters Patent.

Patented July 19, 1910.

No Drawing.

Application filed June 17, 1909. Serial No. 502,812.

To all whom it may concern:

Be it known that I, ROBERT E. SCHMIDT, doctor of philosophy, chemist, citizen of the German Empire, residing at Elberfeld, Germany, have invented new and useful Improvements in Processes of Dyeing and Products Thereof, of which the following is a specification.

I have made the surprising discovery that the acidylized aminoanthraquinones can be used as valuable vat dyes. They yield with alkaline reducing agents vats which dye the textile fiber from yellow to red to blue shades of excellent fastness.

In order to illustrate my new process more fully the following example is given, the parts being by weight:—Stir up 40 kilos of diacetyl-1.5-diaminoanthraquinone (10 per cent. paste) with 36 liters of warm water, add to this mixture 40 liters of a hydrosulfite solution 20° Bé. and 6 liters caustic soda lye 30° Bé. After a short time the color is completely dissolved. Prepare a dyebath which should contain 2000 liters water, $\frac{1}{2}$ liter caustic soda lye 30° Bé. and 2 liters hydrosulfite solution and 80 parts of common salt and add to this cold bath the dye solution prepared as above indicated, through a sieve. Stir the bath well, let stand during some minutes, enter 100 kilos well wetted out cotton and work for 1 hour in the cold bath. After dyeing, squeeze out well, rinse, and acidulate the goods in a bath containing 2 ccm. sulfuric acid of 66° Bé. per liter, rinse and finally soap. A very fast clear orange-yellow shade is thus obtained.

Wool is dyed in an analogous way: Stir up 10 kilos diacetyl-1.5-diaminoanthraquinone (10 per cent.) with 8–10 kilos caustic soda lye 30° Bé. and 30 liters of a hydrosulfite solution 17° Bé. When all the color is thoroughly dissolved, add this solution to the dyebath which consists of 1200–1500 liters of water of 30° Bé., to which $\frac{1}{2}$ liter caustic soda lye 30° Bé. and 2 liters of a hydrosulfite solution 17° Bé. and 20–40 kilos Glauber's salt has previously been added. Enter 50 kilos wool, work for $\frac{1}{2}$ to 1 hour at 30° C., the goods being continually immersed in the liquor. After dyeing, squeeze out well, expose, rinse, acidulate, wash and dry.

A very fast orange-yellow shade is thus obtained.

Silk and charged silk are dyed in an analogous way. In order to use the acidylized aminoanthraquinones for printing I proceed as follows, the parts being by weight:—100 parts of diacetyl-1.5-diaminoanthraquinone (10 per cent. paste) are mixed with 74 parts of tragacanth thickening (65:1000), 1.1 parts of tin crystals, 7 parts of copperas powder, 8 parts of tartar powder are added and the paste is well stirred. Print, run through soda lye (18° Bé.) at 85°, acidulate, rinse and soap at 60° C. A very fast clear orange-yellow shade is thus obtained.

Other reducing agents *e. g.* hydrosulfites may be used. Other acidylized aminoanthraquinones may be used with the same success *e. g.* formyl-1-aminoanthraquinone (dyes orange), diformyl-1.4-diaminoanthraquinone (dyes orange-brown), diformyl-1.5-diaminoanthraquinone (dyes orange), acetyl-1-aminoanthraquinone (dyes greenish-yellow), diacetyl-1.4-diaminoanthraquinone (dyes yellow-red), diacetyl-1.8-diaminoanthraquinone (dyes orange), cinnamyl-1-aminoanthraquinone (dyes yellow), acetyl-1-amino-4-chloroanthraquinone (dyes greenish-yellow), acetyl-1-aminoanthraquinone-4-mercaptan-ethylether (dyes orange-red), isovaleryl-1.5-diaminoanthraquinone (dyes yellow), dicinnamyl-1.5-diaminoanthraquinone (dyes blue).

I claim:

1. The process of treating textile fiber consisting in reacting on an acidylaminoanthraquinone with alkaline reducing agents to form a solution, applying the leuco compounds thus produced to the fiber and then oxidizing these leuco compounds, thus producing from yellow to red to blue shades on the fiber.

2. The process of treating textile fiber consisting in reacting on an acidylaminoanthraquinone with alkaline hydrosulfites to form a solution, applying the leuco compounds thus produced to the fiber and then oxidizing these leuco compounds, thus producing from yellow to red to blue shades on the fiber.

3. The process of treating textile fiber consisting in reacting on diacetyl-1.5-diaminoanthraquinone with hydrosulfite and

caustic soda lye to form a solution, applying the leuco compounds thus produced to the fiber and then oxidizing these leuco compounds, thus producing yellow shades on the fiber.

4. As a new article of manufacture textile fiber impregnated with the oxidation products of the leuco compounds of an acidylaminoanthraquinone, which treated fiber upon being touched with a watery solution containing 0.4% of anhydrous hydrosulfite of sodium and 0.8% of caustic soda changes its color of yellow to red to blue into a brownish shade which on exposure to air assumes again its original color.

5. As a new article of manufacture yellow textile fiber impregnated with the oxidation products of the leuco compounds of diacetyl-

1.5-diaminoanthraquinone, which treated fiber remains unchanged upon being boiled with a 10% muriatic acid and upon being touched with a cold solution of an alkali-hypochlorite and which upon being touched with a watery solution containing 0.4% of anhydrous hydrosulfite of sodium and 0.8% of caustic soda changes its yellow color into a brownish red shade, which on exposure to air assumes again its original appearance.

In testimony whereof I have hereunto set my hand in the presence of two subscribing witnesses.

ROBERT E. SCHMIDT. [L. s.]

Witnesses:

OTTO KÖNIC,
CHAS. J. WRIGHT.