

UNITED STATES PATENT OFFICE.

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BLUE FIBROUS MATERIAL AND PROCESS OF DYEING SAME.

No. 818,457.

Specification of Letters Patent.

Patented March 27, 1908.

Application filed February 7, 1905. Serial No. 244,639.

To all whom it may concern:

Be it known that I, VICTOR FUSSGÄNGER, Ph. D., chemist, a citizen of the Empire of Austria-Hungary, residing at Höchst-on-the-Main, Germany, have invented certain new and useful Improvements in the Processes of Dyeing Blue, of which the following is a specification.

I have found that by oxidizing a mixture of alkylated diamidobenzhydrols and para-oxy or para-amido diphenylamin and their derivatives new dyestuffs of a vivid blue may be obtained which are distinguished by their fastness to washing, soap, soda, alkali, and acid even in the absence of a tannin mordant.

It is known that tetramethyldiamidobenzhydrol with acids yields beautifully-blue tinted salts decolorized by an excess of acid. On tannin-mordanted cotton or silk these salts produce beautiful shades, lacking fastness, however, and being readily decomposed by soap, dilute alkalies, or acids. (Compare Nietzki, *Chem. Organ. Farbstoffe*, III edition, pages 110, 111.) The new dyestuffs obtained by this process, therefore, must not be mistaken for the known salts of the alkylated diamidobenzhydrols, as it is a question here of new condensation products.

If tetramethyldiamidobenzhydrol alone is oxidized, no dyestuff is obtained, but by oxidizing para-oxy or para-amido diphenylamin by itself a light gray is produced. If, however, the hydrol be mixed with one of these diphenylamin derivatives in a printing or padding color and oxidized on the fiber, new dyestuffs of beautiful shades and excellent fastness are obtained. In a similar manner react the derivatives of para-oxy or para-amido diphenylamin—like, for instance, para-amido-para-oxy-diphenylamin, para-diamidodiphenylamin and their homologues with tetramethyldiamidobenzhydrol and other alkylated diamidobenzhydrols. The corresponding methane derivatives, like tetramethyldiamidodiphenylmethane, when oxidized on the fiber together with para-amidodiphenylamin also yield a blue dyestuff of the same excellent properties. This is due probably to the hydrol being first formed from the methane derivative, whereupon it, reacts with para-amidodiphenylamin. Thus, for instance, para-oxydiphenylamin and tetramethyldiamidobenzhydrol yield a vivid vio-

let-blue. Para-oxydiphenylamin and tetraethyldiamidobenzhydrol yield a blue. Paraamidodiphenylamin and tetramethyldiamidobenzhydrol yield a marine blue. Paraamidodiphenylamin and tetraethyldiamidobenzhydrol yield a greenish blue. Paraamidodiphenylamin and tetramethyldiamidodiphenyl-methane yield a dark blue. Paraamidodiphenylamin and tetraethyldiamidodiphenyl-methane yield a greenish blue. Paraamidomethyldiphenylamin and tetramethyldiamidobenzhydrol yield a blue. Paraamidophenyltolylamin and tetramethyldiamidobenzhydrol yield a blue. Para-amidopara-oxydiphenylamin and tetramethyldiamidobenzhydrol yield a dark blue. Phenylpara-amidodiphenylamin and tetramethyldiamidobenzhydrol yield a blue. Para-diamidodiphenylamin and tetramethyldiamidobenzhydrol yield a dark blue. Dimethylpara-amido-para-oxyphenyltolylamin and tetramethyldiamidobenzhydrol yield a blue.

The dyestuff obtained from tetraethyldiamidobenzhydrol with para-oxydiphenylamin is especially fast to soap and soda.

The shades of the dyestuffs obtained with para-oxydiphenylamin are essentially purer and more vivid than those obtained with paraamidodiphenylamin, but to become faster to acids require a slight addition of tannin.

The present application has in common with the application on which Letters Patent were issued on August 8, 1905, No. 796,715, a partial application of paraamidodiphenylamin and its derivatives, but is distinguished from it by using alkylated diamidobenzhydrols as one of the components. Moreover, in the cited application quinonimid dyestuffs are formed on the fiber, whereas here new triphenylmethane dyestuffs may be obtained. Besides the fastness to soap and soda of the dyestuffs obtained by this process is greater and the shades are more vivid than the products of the application referred to.

The two bodies to be condensed are mixed in a printing or padding color with the requisite solvent and thickening agent, whereupon oxidizing agents are added and the whole applied to the vegetable or animal fiber. It is developed by drying or subsequent steaming. No free mineral acid is necessary, the oxidation taking place with organic salts and

chlorates. This prevents the fiber from being attacked.

The process may be modified with para-oxydiphenylamin by applying the different components successively and by oxidizing them together or by separately preparing the leuco compound from hydrol and the diphenylamin derivative. It is then applied to the fiber and oxidized by steaming with chlorates or other oxidizing agents, such as potassium bichromate.

Example I.

20 grams of tetramethyldiamidobenzhydrol of ninety per cent. are dissolved on the water bath with 10 grams of para-oxydiphenylamin, 100 cubic centimeters of acetic acid of fifty per cent., 20 cubic centimeters of lactic acid of fifty per cent. stirred into 500 grams of acid-starch thickening, adding 10 grams of chlorate of sodium, 20 cubic centimeters of water, 10 cubic centimeters of aluminium chlorid of twenty per cent., 5 cubic centimeters of cero-chlorid of twenty per cent., 195 cubic centimeters of water. 1 kilogram.

Example II.

10 grams of commercial tetramethyldiamidobenzhydrol and 10 grams of para-oxydiphenylamin are heated with 100 cubic centimeters of acetic acid of 8° Baumé specific gravity and 20 cubic centimeters of lactic acid of fifty per cent. and stirred into 500 grams of acetate-of-starch thickening, adding 10 grams of chlorate of sodium, 20 cubic centimeters of water, 10 cubic centimeters of aluminium chlorid of 20° Baumé specific gravity, 10 cubic centimeters of cero-chlorid of twenty per cent. and 170 cubic centimeters of water, being added. 1 kilogram.

The printed fabric is dried and steamed for a short time in the Mather-Platt apparatus or for one hour in a vessel without pressure, then washed and soaped.

Example III.

20 grams of tetramethyldiamidobenzhydrol of about ninety per cent., 10 grams of para-oxydiphenylamin, 100 cubic centimeters of acetic acid of 8° Baumé specific gravity, 50 cubic centimeters of lactic acid of fifty per cent., 500 grams of acid-starch thickening, 10 grams of tartaric acid, 5 cubic centimeters of cero-chlorid of 43° Baumé specific gravity, 185 cubic centimeters of water, 10 grams of chlorate of sodium, 20 cubic centimeters of water, 20 grams of tannin, 20 cubic centimeters of water. 1 kilogram.

The fabric is printed, steamed in the Mather-Platt or in a closed vessel, passed through an antimony-bath, washed, soaped, and dried.

Example IV.

20 grams of the leuco base from tetramethyldiamidobenzhydrol and para-oxy or para-amido diphenylamin are dissolved in 100 cubic centimeters of acetic acid of 8° Baumé and stirred into 500 grams of acid-starch thickening and then made up with 185 cubic centimeters of water to 1 kilogram.

The fabric is printed, dried, passed through a bath of five grams of potassium bichromate and three grams of sulfuric acid to the liter at about 60° centigrade, washed, and soaped.

Example V.

A { 18 grams of commercial tetramethyldiamidobenzhydrol and 11 grams of para-amidodiphenylamin are dissolved in 100 cubic centimeters of acetic acid of fifty per cent. and 20 cubic centimeters of lactic acid of fifty per cent. and stirred into 100 grams of tragacanth water, sixty grams to the liter, 200 cubic centimeters of water, 70
B { 14 grams of chlorate of sodium, 20 cubic centimeters of water, 15 cubic centimeters of aluminium chlorid of 20° Baumé specific gravity, 10 cubic centimeters of cero-chlorid of twenty per cent., 400 cubic centimeters of water. Before use, pour color B into A and make up to 1 liter. 75

The fabric is padded, dried, and the blue developed by steaming for a short time or on the drying-cylinder or in the oxidation-chamber.

The process may be applied to vegetable or animal fibers, but with woolly material the reducing power of the fiber has to be considered, which requires greater quantities of chlorate or chlorinated or acidified woolstuffs.

Resist effects may be obtained in the usual manner by reducing agents, such as tin salt, potassium sulfite, hydrosulfite, and the like.

Having now described my invention, what I claim is—

1. The process herein described for the manufacture of fast-blue dyestuffs on the fiber, which consists in saturating the fiber with a mixture of alkylated diamidobenzhydrols and diphenylamin derivatives and in oxidizing same together by means of an oxidizing agent.

2. The process of dyeing, which consists in preparing leuco compounds from a mixture of alkylated diamidobenzhydrols and diphenylamin derivatives, saturating the fibers with said compounds and oxidizing them to a dye by oxidizing agents.

3. As a new product, a fibrous material of blue shade, obtained by oxidizing a mixture of alkylated diamidobenzhydrols and diphenylamin derivatives within the fiber, being fast to water, soap, soda, dilute alkalies and acids and reduced by hydrosulfite.

In testimony that I claim the foregoing as my invention I have signed my name in presence of two subscribing witnesses.

VICTOR FUSSGÄNGER.

Witnesses:

ALFRED BRISBOIS,
BERNHARD LYDECKER.