

UNITED STATES PATENT OFFICE.

CARL DUISBERG AND PHILIPP OTT, OF ELBERFELD, GERMANY, ASSIGNORS
TO THE FARBENFABRIKEN, VORMALS FR. BAYER & CO., OF SAME PLACE.

PROCESS OF DYEING.

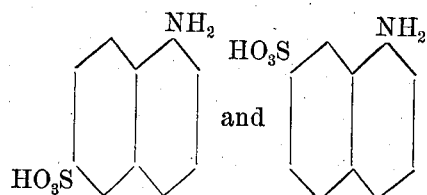
SPECIFICATION forming part of Letters Patent No. 523,809, dated July 31, 1894.

Application filed April 21, 1894. Serial No. 508,506. (Specimens.) Patented in Germany September 15, 1891, No. 65,262; in England December 28, 1891, No. 22,641; in Belgium December 31, 1891, No. 97,665; in Italy January 9, 1892, XXVI 30,988, LX, 468; in France March 21, 1892, No. 213,971, and in Austria-Hungary January 29, 1893, No. 53,601 and No. 78,592.

To all whom it may concern:

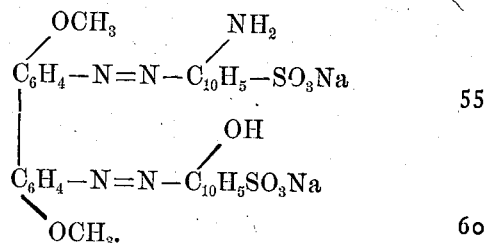
Be it known that we, CARL DUISBERG and PHILIPP OTT, chemists and doctors of philosophy, subjects of the Emperor of Germany, residing at Elberfeld, Prussia, Germany, (assignors to the FARBENFABRIKEN, VORMALS FR. BAYER & CO., Elberfeld, Germany,) have invented certain new and useful Improvements in Processes of Dyeing, (for which the aforesaid FARBENFABRIKEN has already obtained Letters Patent in Germany, No. 65,262, dated September 15, 1891; in France, No. 213,971, dated March 21, 1892; in England, No. 22,641^A, dated December 28, 1891; in Austria-Hungary, No. 53,601 and No. 78,592, dated January 29, 1893; in Italy XXVI, 30,988, LX, 468, dated January 9, 1892, and in Belgium, No. 97,665, dated December 31, 1891;) and we do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

Our invention relates to the production of azo dyes on the fiber and depends on a peculiar property of certain coloring-matters which are obtained for instance by combining one molecular proportion of tetrazotized para diamins (as for example benzidin, tolidin, called Clève's alpha naphthylamin monosulfo dianisidin, diamidodiphenylenoxid and so on) with two molecular proportions of the so acid (beta or delta) which correspond with the formula:



or with a mixture of them or with one molecular proportion of the one, and one molecular proportion of the other, of these two acids, or with one molecular proportion of the latter two acids and one molecular proportion of the known dye-stuff components. In the

latter case coloring-matters are obtained, which, for example, are illustrated by the following combination:



Such or similar dye-stuffs, when fixed on the fiber, can be further diazotized, and the resulting diazo or tetrazochlorids (which owing to their insolubility remain fixed on the fiber) can be directly combined with the known dye-stuff components (amins, phenols or sulfo or carboxylic acids thereof), and thus shades can be produced of high intensity and great fastness. Otherwise these dye-stuffs, when diazotized on fiber, can be treated with a solution of sodium carbonate in water, by means of which very fast brown shades are produced.

In carrying out our process practically we proceed as follows: About ten kilos, by weight, of unmordanted cotton are dyed by immersion for say one hour in a boiling solution prepared by dissolving 0.5 kilo, by weight, of the dye-stuff obtained from one molecular proportion of tetrazoortho diphenol-dimethyl-ether and two molecular proportions of alpha naphthylamin beta monosulfo acid (Clève's beta acid) in two hundred and fifty kilos, by weight, of hot water and adding one kilo, by weight, of common salt. Thereupon, the cotton, which is now dyed bluish-red, is washed carefully with cold water and then introduced in a solution of 0.3 kilo, by weight, of sodium nitrite in two hundred and fifty kilos, by weight, of cold water acidulated with 0.9 kilo, by weight, of hydrochloric acid (20° Baumé). By well agitating the cotton in the acid bath the diazotizing is soon finished, after which the cotton is rinsed in cold water. By placing then the thus prepared cotton in an alkali-

line solution of 0.4 kilo, by weight, of the sodium salt of beta naphthol the formation of the dye-stuff takes place on the fiber and the end of this reaction may easily be recognized
5 as the cotton gradually acquires a fast bluish-black color. When the intensity of the color no more increases, the cotton is carefully washed with water and dried.

Of course, the dye-stuff employed in the
10 foregoing example can be replaced by analogous products from other tetrazo salts or from Clève's delta acid or by similar substantive dye-stuffs derived from the said Clève's acids (with the exception however of the dye-stuff
15 from tetrazodiphenylchlorid and Clève's beta acid, which dye-stuff cannot be dyed owing to its insolubility in water), and the above employed beta naphthol may be replaced by other suitable dye-stuff components (develop-
20 ers) shades being produced which in general are from violet to blue and black. The best shades, however, are obtained by employing beta-naphthol. The process herein before defined is of a great technical value
25 as the shades produced on the fiber in the

aforesaid manner or in an analogous or similar way possess very great fastness to soaping and to the action of alkalies and acids.

When in the claim of this application we refer to "beta naphthol" we intend thereby
30 to include as well the other described equivalents therefor.

Having now described our invention and in what manner the same is to be performed, what we claim as new, and desire to secure by
35 Letters Patent, is—

The process of dyeing cotton, which consists first, in dyeing in the well known manner cotton fiber or material by means of substantive dye stuffs containing Clève's acids,
40 in subsequently diazotizing the dye stuffs fixed on the fiber, and finally combining the resulting diazo or tetrazo compounds likewise on the fiber with beta naphthol.

CARL DUISBERG.
PHILIPP OTT.

Witnesses:

THILO KROEBER,
ROBERT DEMUTH.

It is hereby certified that in Letters Patent No. 523,809, granted July 31, 1894, upon the application of Carl Duisberg and Philipp Ott, of Elberfeld, Germany, for an improvement in "Processes of Dyeing," an error appears in the printed specification requiring correction as follows: Page 1, line 30, the clause "called Clève's alpha naphthylamin mono sulfo," should be stricken out and inserted after line 32 as now numbered, same page; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed, countersigned, and sealed this 14th day of August, A. D. 1894.

[SEAL.]

JNO. M. REYNOLDS,
Assistant Secretary of the Interior.

Countersigned:

JOHN S. SEYMOUR,
Commissioner of Patents.