

UNITED STATES PATENT OFFICE.

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PRODUCTION OF BROWN TO VIOLET TO BLACK SHADES.

1,011,084.

Specification of Letters Patent.

Patented Dec. 5, 1911.

No Drawing.

Application filed November 30, 1909. Serial No. 530,657.

To all whom it may concern:

Be it known that I, HENRI SCHMID, chemist, citizen of the Swiss Republic, residing at Mülhausen, in Alsace, Germany, have invented new and useful Improvements in the Production of Brown to Violet to Black Shades, of which the following is a specification.

In the specification of Letters Patent No. 863,761, is described the production of brown shades by oxidizing on the fiber, or fabric, by means of a chlorate in the presence of vanadium compound, para-phenylene-diamin, or a homologue, or isomer, thereof, in the absence of sufficient mineral acid to combine with the whole thereof. Further, in the specification of the application for Letters Patent Serial No. 530,658 it is stated that by employing, instead of para-phenylene-diamin itself, a mixture thereof with an amino-hydroxy compound, various shades of brown can be produced. I have now discovered that if, in these processes, the para-phenylene-diamin, or homologue, or isomer, thereof, and the amino-hydroxy compound be employed so that the whole thereof, or at any rate the greater part thereof, is in the form of the free base, the oxidation can be carried out by means of chlorate and potassium ferrocyanid. The difficulty hitherto experienced has been due to the fact that if the para-phenylene-diamin be not used in the form of a free base, but in the form of a salt thereof, for instance in the form of its hydrochlorid, the addition of potassium ferrocyanid causes the diamin to be precipitated and, consequently, either no result is obtained, or the result obtained is unsatisfactory. The shades produced according to my present invention can be made to vary, and are generally deeper and have a more violet tinge than those produced by means of the vanadium salt and a chlorate. On soaping the material, however, the shades tend to become browner.

The process of this invention is an improvement on the vanadium-chlorate process, because the material, after being padded, is less sensitive and the color has less tendency to spread. The shades can be reserved and white, or colored, discharge effects produced in a manner similar to that described in the aforesaid specification of Letters Patent No. 863,761. The quantity

of potassium ferrocyanid employed can vary, for instance between 10 and 30 grams per liter of the bath. With a concentrated padding bath, containing, for instance, from 25 to 40 grams of para-phenylene-diamin per liter, deep brown-black can be obtained, and this can also be reserved in a similar manner. The said brown-black withstands light treatment with soap, but on treatment in a somewhat stronger alkaline bath it tends to become dark brown. The black obtained according to this process is better than anilin black, because the former does not tend to become green, and because the material is not weakened during the production of the said black. If meta-phenylene-diamin be employed, instead of the para compound, similar results are obtained. Meta-toluylene-diamin gives rise to an olive-black.

The homologues of the phenylene-diamins are for the purposes of this invention equivalent to the phenylene-diamins themselves.

The following are examples of the preparation of baths in accordance with my invention, which, however, is not limited to these examples.

Example 1—Padding bath for brown.—Dissolve twelve, to fifteen, grams of para-phenylene-diamin, twenty grams of sodium chlorate, twenty grams of ammonium chlorid, and from ten, to twenty, grams of potassium ferrocyanid in water, and make up to one liter.

Example 2—Padding bath for black.—Dissolve thirty grams of para-phenylene-diamin, thirty, to forty, grams of ammonium chlorid, thirty, to forty, grams of potassium ferrocyanid, and from thirty, to thirty-five, grams of sodium chlorate in water, and make up to one liter.

The material is padded with either of the aforesaid baths and dried, then steamed for from five, to ten, minutes, and washed and, if necessary, treated with soap solution. In order to obtain discharge effects, the material can, after drying and before being steamed, be printed with a reserve paste containing sodium acetate, potassium sulfite, and Rongalite C, with gum thickening.

Now what I claim is:—

1. The process of producing brown to violet to black shades on the fiber by apply-

ing to the material a mixture containing a phenylene-diamin, a chlorate and a ferrocyanid, at least one-half of the phenylene-diamin being in the free state, and then causing the oxidation of the phenylene-diamin substantially as described.

2. The process of producing brown to violet to black shades on the fiber by applying to the material a mixture containing a phenylene-diamin and an amino-hydroxy compound, a chlorate and a ferro-cyanid, at least one-half of the amino groups being in the free state, and then causing the oxidation of the phenylene-diamin and of the amino-hydroxy compound substantially as described.

3. The process of producing brown to violet to black shades on the fiber by applying to the material a mixture containing a phenylene-diamin, a chlorate and a ferrocyanid, at least one-half of the phenylene-diamin being in the free state, then drying the material and subjecting it to the action of steam.

4. The process of producing brown to violet to black shades on the fiber by applying to the material a mixture containing phenylene-diamin and an amino-hydroxy compound, a chlorate and a ferrocyanid, at least one-half of the amino groups being in the free state, then drying the material and subjecting it to the action of steam.

5. The process of producing brown to violet to black shades with discharge effects on the fiber by applying to the material a mixture containing para-phenylene-diamin, a chlorate and a ferrocyanid, at least one-half of the para-phenylene-diamin being in the free state, then drying the material, printing it with a paste containing a reduc-

ing agent and subjecting it to the action of steam.

6. The process of producing brown to violet to black shades with discharge effects on the fiber by applying to the material a mixture containing para-phenylene-diamin and an amino-hydroxy compound, a chlorate and a ferrocyanid, at least one-half of the amino groups being in the free state then drying the material, printing it with a paste containing a reducing agent and subjecting it to the action of steam.

7. The process of producing brown to violet to black shades with discharge effects on the fiber by applying to the material a mixture containing para-phenylene-diamin, a chlorate and a ferrocyanid, at least one-half of the para-phenylene-diamin being in the free state, then drying the material, printing it with a paste containing a reducing agent and a coloring matter, and subjecting it to the action of steam.

8. The process of producing brown to violet to black shades with discharge effects on the fiber by applying to the material a mixture containing para-phenylene-diamin and an amino-hydroxy compound, a chlorate and a ferrocyanid, at least one-half of the amino groups being in the free state, then drying the material, printing it with a paste containing a reducing agent and a coloring matter, and subjecting it to the action of steam.

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

HENRI SCHMID.

Witnesses:

GEORGE GIFFORD;
ARNOLD ZUBER.