

# UNITED STATES PATENT OFFICE.

EMIL VON PORTHEIM, OF PRAGUE, AUSTRIA-HUNGARY.

## PROCESS OF DYEING BLACK.

SPECIFICATION forming part of Letters Patent No. 586,865, dated July 20, 1897.

Application filed May 26, 1896. Serial No. 593,066. (No specimens.) Patented in Austria May 6, 1896, No. 46/1,850; in France May 9, 1896, No. 256,241; in England May 9, 1896, No. 9,912; in Italy July 3, 1896, XXXI, 41,535, LXXXI, 324, and in Hungary September 19, 1896, No. 7,502.

To all whom it may concern:

Be it known that I, EMIL VON PORTHEIM, a subject of the Emperor of Austria-Hungary, residing at Prague, in the Province of Bohemia, in the Empire of Austria-Hungary, have invented certain new and useful improvements in the Process of Producing Black Coloring-Matters on the Fiber, (for which Letters Patent have been obtained with my consent in Austria, dated May 6, 1896, No. 46/1,850; in Hungary, dated September 19, 1896, No. 7,502; in France, dated May 9, 1896, No. 256,241; in Italy, dated July 3, 1896, XXXI, 41,535, LXXXI, 324, in the name of Kinzberger & Co., and in my name in England, dated May 9, 1896, No. 9,912;) and I 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.

The object of the present invention is a process for producing black azo dyestuffs on cotton fiber impregnated with beta-naphthol sodium. These dyestuffs, produced in the manner hereinafter described, are distinguished from all similar products known till now by the easy management of the diazotizing operation, the intensity of tints, the tinctorial power, resistance to light, and stability of the diazo compound, while the said dyestuffs are not inferior, as regards the other qualities, to all similar products and therefore fill up a void in the use of the so-called "ice colors."

The invention is carried out by employing the diazo compounds of the amidochrysoïdins, such as are formed from the diazo compound of the acetyl-paraphenylenediamin by combination thereof with metaphenylenediamin or orthoparateluylenediamin or a symmetrical dimethyl-metaphenylenediamin, and by diacetylation of the obtained product. The above amidochrysoïdins may, however, be produced in any other known manner.

In carrying out the diazotizing process with regard to the amidochrysoïdin one molecule of the latter is treated with one molecule of nitrite, but similar results are also obtained by the action of two molecules of nitrite.

The stability of the diazo compounds obtained has been found to increase very much when to these compounds certain acids, such as oxalic acid, are added.

Example: 5.3 parts, by weight, of the base of amidochrysoïdin (triamidoazobenzene) are dissolved in seventy-five parts, by weight, of water and seven parts, by weight, of hydrochloric acid, (specific gravity 1.2.) Then forty-five parts, by weight, of ice are added. The cooled mixture is then diazotized with a concentrated solution of 1.75 parts, by weight, of nitrite.

The diazo liquor is mixed with one hundred and ten parts, by weight, of acetic-starch paste. Then 5.3 parts, by weight, of oxalic acid are added to it, and, finally, before printing, 2.4 parts, by weight, of acetate of sodium in form of a concentrated solution are added. The addition of oxalic acid can, however, also take place previously to the diazotizing operation—i. e., simultaneously with the addition of hydrochloric acid.

The mordanting of the cotton fiber can be performed with naphthol sodium alone or with addition of any suitable fatty acid compounds.

I claim—

1. In a process for dyeing fibrous materials black and forming the dyestuff on the fiber itself, mordanting the same first with beta-naphthol sodium and then applying thereto a diazo combination of an amidochrysoïdin base.

2. In a process for dyeing fibrous materials black and forming the dyestuff on the fiber itself diazotizing a base of an amidochrysoïdin, adding to the diazotized liquor acetic-starch paste and acetate of sodium in about the proportions set forth, and applying the compound to the fibrous material previously mordanted with beta-naphthol sodium.

3. A process for dyeing fibrous materials black and forming the dyestuff on the fiber itself, by diazotizing a base of an amidochrysoïdin, adding to the diazotized liquor acetic-starch paste, oxalic acid and acetate of sodium in about the proportions set forth, and applying the compound to the fibrous material previously mordanted with beta-naphthol sodium and a fatty acid compound.

In testimony whereof I affix my signature in presence of two witnesses.

EMIL VON PORTHEIM.

Witnesses:

CARL SCHÄNHANOL,  
ADOLPH FISCHÉ.