

# UNITED STATES PATENT OFFICE.

EMIL HAUSSMANN, OF BERLIN, GERMANY, ASSIGNOR TO THE ACTIEN-  
GESELLSCHAFT FÜR ANILIN FABRIKATION, OF SAME PLACE.

## PROCESS OF DYEING.

SPECIFICATION forming part of Letters Patent No. 661,985, dated November 20, 1900.

Application filed July 10, 1900. Serial No. 23,068. (No specimens.)

*To all whom it may concern:*

Be it known that I, EMIL HAUSSMANN, of Berlin, in the Kingdom of Prussia, German Empire, have invented new and useful Improvements in Dyeing Processes; and I do hereby declare that the following is a full, clear, and exact description thereof, which will enable others skilled in the art to which it appertains to make and use the same.

It is well known that colors fast to milling may be produced on wool fiber by the formation of lakes. For this purpose the woolen goods are mordanted with chromium compounds and subsequently boiled in a solution of the dyestuff. The characteristic feature of the mordanting process consists in impregnating the material to be mordanted with a boiling solution of potassium or sodium bichromate with certain assistants, which serve to decompose the bichromate and simultaneously to reduce the chromic acid thus formed. Suitable additions are tartar, oxalic acid, lactic acid, or lignorosin, in combination with sulfuric acid. Sulfuric acid alone or boro-sulfuric acid or certain other additions sometimes employed set free the chromic acid without reducing it. Hence the reduction can only take place by affecting the wool fiber. I have now made the unexpected discovery that by substituting for the above-mentioned assistants neutral ammonium salts—for instance, ammonium-sulfate—a mordanting-bath of most valuable properties is obtained.

The new mordanting-bath which I propose, prepared from potassium bichromate and ammonium-sulfate, is neutral in the beginning, but becomes slowly acid on boiling, a part of the ammonium-sulfate decomposing and ammonia being given off. The latter escapes or reduces the small quantities of chromic acid, which at the same time have been set free. Further nitrogen, water, and lower oxides of chromium are formed, which latter immediately become fixed upon the wool fiber. It is evident that in the way described the mordant is but slowly, and therefore most evenly, deposited on the fiber. Moreover, if a sufficient quantity of ammonium-sulfate is added the bath may be almost completely exhausted.

My new method permits the addition of a number of mordant coloring-matters directly to the mordanting-bath without the formation of a lake occurring in the bath immediately. As the bath is neutral in the beginning, the coloring-matter only is at first absorbed by the wool. On boiling the trace of chromic acid liberated is immediately taken up by the fiber, which possesses a very great affinity for it and combines with the coloring-matter upon the fiber. The reaction continues in the same manner and is assisted by the dye-bath becoming more and more acid until both the coloring-matter and the chromic acid are withdrawn from the bath and united upon the wool.

My new mordanting process is to be regarded as a technical advantage of great importance for the following reasons:

First. The process is carried out in one single operation, the goods being brought into the boiling-bath, and no subsequent addition of any ingredients whatsoever is required.

Second. The bath is completely exhausted, so that several consecutive dyeing operations may be carried out in the same bath. Mere traces of chromium that may have been retained can be removed, if necessary, by the addition of a small quantity of lactic acid.

Third. In many cases direct shades are obtained, which in the dyeing processes hitherto employed were only obtained by a subsequent treatment with potassium bichromate. Consequently the dyer has it in his power to match directly.

Fourth. Since dyestuffs, as a rule, dye very evenly in a neutral bath, the dyeings obtained with the help of my mordanting process are distinguished by their extraordinarily even shades as compared with those produced upon a chrome-mordant. Whereas in the latter case the color-lake is immediately formed upon the surface of the woolen fiber, thereby tending to yield uneven dyeings, by my method, on the other hand, the material is first thoroughly penetrated by the dyestuff, which is then gradually fixed during the boiling of the bath. By these means even very thick and closely-woven fabrics can be readily dyed through.

Fifth. Many coloring-matters when sub-

jected to my new dyeing process yield shades which are essentially richer and much faster to milling than those produced by the processes hitherto employed.

- 5 Sixth. The preparation of the dye-bath may be essentially facilitated by keeping standard solutions containing the dyestuff and all the necessary ingredients in the proportions required. Such standard solutions  
10 could be prepared by the color-manufacturers and brought upon the market.

To further illustrate my invention, I give the following directions:

- 15 I. Dissolve in boiling water one pound of potassium bichromate, one pound of ammonium-sulfate, and one pound of anthracened in a vat of two hundred gallons. Enter with one hundred pounds of slubbing and connect with a pump. Work for two hours  
20 until the bath is exhausted. Rinse and dry.

- II. Dissolve in boiling water one pound of chrome fast yellow R, one pound of potassium-bichromate, and one pound of ammonium-sulfate in a vat of three hundred gal-  
25 lons. Enter with fifty pounds of worsted yarn at a moderate temperature, lift six times, and raise the temperature slowly to the boil and dye, gently boiling until the bath is exhausted.

III. Dissolve in boiling water 7.5 pounds of  
30 the azo-dyestuff paste obtained from diazotized picramic acid and meta-toluylene-diamine, (respectively equivalent proportions of the dyestuffs obtained from diazotized pic-  
35 ramic acid and chloro-phenylenediamine,  $\text{Cl:NH}_2:\text{NH}_2=1:2:4$  or  $1:3:5$ ), 1.5 pounds of potassium-bichromate, and 1.5 pounds of ammonium-sulfate in a piece-vat of two hundred gallons, enter with seventy-five pounds of worsted goods at a moderate temperature,  
40 and work for fifteen minutes. Raise the temperature slowly to the boil and dye, boiling until the bath is exhausted.

Having now described my invention and in what manner the same is to be performed,  
45 what I claim as new is—

The process for fixing coloring-matter on wool, said process consisting of simultaneously mordanting and dyeing woolen goods in a bath containing the dye together with  
50 chromates and neutral ammonium salts.

In witness whereof I have hereunto signed my name, this 23d day of June, 1900, in the presence of two subscribing witnesses.

EMIL HAUSSMANN.

Witnesses:

HENRY HASPER,  
WOLDEMAR HAUPT.