

UNITED STATES PATENT OFFICE.

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PROCESS OF DYEING SULFUR DYES.

No. 907,937.

Specification of Letters Patent.

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To all whom it may concern:

Be it known that I, ADOLF WÖSCHER, colorist, a citizen of the Empire of Germany, residing at Höchst-on-the-Main, Germany, have made certain Improvements in Dyeing with Sulfurized Dyestuffs, of which the following is a specification.

I have found that it is not essential to avoid the use of alkali sulfid in the dye bath in order to obviate the majority of the drawbacks in dyeing with sulfurized dyestuffs, provided a bisulfite be present in the bath. Thus by adopting this expedient animal fibers may be dyed with sulfurized dyestuffs without injury to the material which has been caused hitherto by the usual alkaline sulfid dye baths. The new process offers similar advantage in dyeing vegetable fibers.

The invention consists in the practical application of the observation that a dye bath prepared in the usual manner with a sulfurized dyestuff, sodium sulfid and common salt, may be treated with a large quantity of bisulfite without any of the dyestuff being precipitated. The addition neutralizes the injurious influence of the sulfid of the alkali metal to such an extent that excellent dyeings can be obtained without detrimentally affecting the fiber. By this method of working the condition of the dye bath may be compared with that of an indigo vat, only the injurious action of the alkali or alkali sulfid may be neutralized to such a point that wool can be dyed at a comparatively high - temperature without danger to the fiber. The dye baths do not become exhausted the operation being similar to that of vat dyeing. The baths may be prepared somewhat stronger than usual and in further working up, the requisite condition of the dye bath is attained by removing the dyestuffs absorbed by the goods, and by adding an alkali sulfid and bisulfite. The success of the dyeing process depends upon the adequate proportion of bisulfite being added so as to neutralize the action of the sodium sulfid. However, too great an excess of bisulfite must be avoided, as it unfavorably influences the dyeing. In most cases a suitable proportion of bisulfite has been added when a test portion of the dye bath does not turn red when a solution of phenolphthalein is added. The best temperature of the bath in dyeing wool is generally between 70-80° C., which

may be raised, however, for deep colors and lowered for light colors. Vegetable fibers are best dyed at a low temperature. By an immersion of only 1-3 minutes in concentrated baths (compare Examples VI and VII) deep dyes are obtained at higher temperature on wool and silk or half wool and half silk and at lower temperature on vegetable fibers thus permitting a continuous dyeing. The process is also suited for dyeing silk, half-silk, leather, furs, and paper, materials which could not hitherto be dyed with sulfurized dyestuffs owing to the injurious influence of the alkali-sulfid baths. Good results may be obtained with sodium, potassium or ammonium bisulfite. Instead of bisulfites, sulfites may be used in presence of suitable acids or acid salts. If desired, the dyeings may be treated subsequently with metallic salts.

An important result of the invention is that wool and vegetable fibers may be dyed simultaneously and the temperature of the dye bath may be adjusted so that the dyestuff is taken up more by the vegetable or more by the wool fiber; the higher the temperature the more the wool is dyed.

This process is applicable to various sulfurized dyestuffs, good results having been obtained, for instance, with thiogen black, sulfur black, immedial black, thiogen blue, thiogen green, immedial indone, katigen blue, katigen brown, katigen violet, thiogen dark red, immedial bordeaux, thiogen yellow and others. The fastness of the dyeings is very good.

The following examples illustrate the invention:—

Example I: The dye bath is made up per liter with 3 grams of thiogen blue B, 4, 5 grams of crystallized sodium sulfid, 4 grams of crystallized sodium bisulfite and 90 grams of crystallized Glauber's salt. The wool is dyed therein for 1 hour at 80° C.; it is then pressed, thoroughly rinsed, acidified if need be, treated in a hydro-extractor and dried.

Example II: The dye bath is made up per liter with 20 grams of liquid thiogen black B2R, 7.5 grams of crystallized sodium sulfid, 7.5 grams of crystallized sodium sulfite, 37.5 cc. of sulfuric acid of 66° Baumé specific gravity, 60 grams of Glauber's salt; the wool is dyed in the bath for 1 hour at 80° C., rinsed and dried.

Example III: A solution is prepared con-

taining per liter 10 grams of liquid thiogen black B2R, 5 grams of crystallized sodium sulfid and 10-20 grams of crystallized sodium bisulfite; leather tanned with chromium is treated in the solution for one-half hour at about 50° C. It is then washed and greased in the usual manner. In such a bath skins, for instance, sheep skins may be dyed in the usual manner by fixing them on suitable boards, care being taken that the hair alone dips into the dye bath. The temperature should be higher than 50° C. in this case.

Example IV: A dye fluid is prepared containing per liter 120 grams of liquid thiogen black M, 60 grams of crystallized sodium sulfid and 80 grams of crystallized sodium bisulfite. The ready paper sheet is passed through this fluid on a dyeing machine (for instance, Frenzel's system) then pressed and dried.

Example V: The dye bath is made up per liter with 40 grams of liquid thiogen black B2R, 20 grams of crystallized sodium sulfid, 25 grams of crystallized sodium bisulfite, 10 grams of sodium thiosulfate. Silk is dyed therein for about one-half hour at 95° C., pressed, thoroughly rinsed, finally soaped while boiling and acidified if necessary.

Example VI: The dye bath is made up

per liter with 30 grams of liquid thiogen black B2R, 20 grams of crystallized sodium sulfid, 20 grams of crystallized sodium bisulfite and 10 grams of Glauber's salt. Cotton is dyed in this bath in the cold within 1-3 minutes a deep black, whereas wool absorbs only little of the dyestuff.

Example VII: The dye bath is made up per liter with 20-60 grams of liquid thiogen black M, 20-60 grams of crystallized sodium sulfid, about 24-72 grams of crystallized sodium bisulfite, wool, silk, half wool and half silk being dyed in this bath at 50-90° C. within 1-5 minutes; the goods are then squeezed and washed.

Having now described my invention what I claim is:—

The process herein described of dyeing with sulfurized dyestuffs in an alkali sulfid bath, which consists in adding to the dye-bath sulfurous acid in the form of its salts.

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

ADOLF WÖSCHER.

Witnesses:

JEAN GRUND,
CARL GRUND.