

UNITED STATES PATENT OFFICE.

MAX BECKE AND ALBERT BEIL, OF HÖCHST-ON-THE-MAIN, GERMANY, ASSIGNORS TO THE FARBWERKE, VORM. MEISTER, LUCIUS & BRÜNING, OF SAME PLACE.

PROCESS OF DYEING WITH SULFUR DYES.

SPECIFICATION forming part of Letters Patent No. 680,472, dated August 13, 1901.

Application filed May 4, 1900. Serial No. 15,532. (No specimens.)

To all whom it may concern:

Be it known that we, MAX BECKE, chemist, a citizen of the Empire of Austria-Hungary, and ALBERT BEIL, chemist, a citizen of the Empire of Germany, both residing at Höchst-on-the-Main, Germany, have invented a new and useful Improvement in the Method of Dyeing with Sulfur Dyestuffs in the Vat, of which the following is a specification.

10 Sulfur dyestuffs obtained by heating various organic substances with sulfur, with or without sulfids of alkali metals, are now of great and increasing importance in the dye industry. The process now generally adopted

15 in dyeing with dyestuffs of this class consists in preparing the bath with the sulfur dye, common salt, sodium carbonate, and a sulfid of an alkali metal, the dyeing taking place at nearly boiling-point, the material being

20 kept as much as possible below the surface of the bath. This mode of working, however, has great drawbacks and requires scrupulous attention, for if the goods be exposed to the air even for a comparatively

25 short time unevenness will ensue by the dyestuff separating out on the parts exposed to the air, and thus spots are produced which cannot be removed. The reason why sulfur

30 colors produce uneven and spotted dyes is, according to our observation, to be found in the following: Sodium sulfid, which the dyestuff either contains when manufactured or which is added to the bath on dissolving the

35 dyestuff, has a reducing influence besides a dissolving action on the dyestuff. Besides the dissolved dyestuff the bath contains also the dyestuff reduced to the leuco compound. The dyestuff not reduced and the dyestuff

40 reduced appear to possess different affinities for the fiber. Therefore they dye the fiber in a different degree of shade, and as the leuco compound readily oxidizes in contact with the oxygen of the air at ordinary temperature an opportunity is given by the present

45 mode of dyeing for producing unevenness and spots by the dyestuff separating out on the material near the boiling-point when exposed to air, which on further working in the dye-bath cannot be removed, or

only incompletely, there being no excess of 50 reducing agents in the bath. Relying on these observations, we have found that very good results may be obtained in dyeing with sulfur dyes by transforming them completely

55 into the leuco compounds, dyeing with them in this form, and finally transforming the leuco compounds into the dyestuff by oxidation by the air or other oxidizing agents—in fact, by dyeing the dyestuffs according to the

60 indigo-vat dyeing process. The hydrosulfite-vat is best suited for reduction, while the copperas-vat or zinc-vat gives worse results, to be accounted for by the formation of compounds

65 soluble with difficulty of the metallic salts and the leuco compounds of sulfur dyes. The raw melts are best not employed for reduction, but the dye-acids obtained by oxidizing or acidifying the dissolved raw melts. The

70 preparation of the vat and the dyeing therein are in general the same as with indigo, only the additional excess of lime is generally not

75 required, as when dissolving indigo-white. The leuco compounds possess great affinity for the fiber. They dye rapidly and evenly, while cold rinsing will hardly, if at all, re-

80 move them from the fiber. The “turning green” of the shade begins immediately in an exceedingly even manner on the goods being taken out of the bath and rinsed. The

85 formation of the dyestuff being completed by exposure to the air, the shades may be transformed into thorough fast tints by subsequent treatment with metallic salts or other oxidizing agents, as is done after dyeing in a bath

90 containing sulfids of alkali metals. As with the indigo-vat, the same material may be introduced several times, the colors becoming stronger and deeper by giving two or more ends. The remarkable fact here is that the

95 shades treated with oxidizing agents, such as sulfate of copper, after the first immersion yield on a second and subsequent immersion disproportionately stronger and more powerful colors than when merely exposed to the air.

The method described is of particular importance, as the reduction of the sulfur dyes may be combined with the vat-dyeing of indigo, it thus being possible to obtain in one

vat with the recognized fast-sulfur dyes and indigo shades just as fast, yet essentially cheaper, than those obtained with indigo alone and also faster shades than those of indigo, which are topped with other colors. For dyeing in the vat the most suitable are the dyestuffs known as "immedial-blue C," "immedial-black G and V extra," (Cassella,) "Katijen-blue," (Bayer,) "Noir Vidal," (St. Denis,) "Clayton fast-black D," and other sulfur dyestuffs.

Example: Four kilos of the dyestuff obtained from para-oxy-para-amido-diphenyl-amin-o-sulfonic acid are treated with sixty liters of hydrosulfite solution of 13° Baumé specific gravity at 50° to 60° centigrade till the color of the solution becomes brown yellow. The leuco compound thus obtained is then poured into the vat containing about two thousand liters of cold water. The mode of working this vat depends upon the nature

of the material to be dyed and is carried out according as loose material, yarns, stuffs, cops, &c., are employed in a similar manner as with indigo.

Having now described our invention, what we claim is—

The herein-described process of dyeing with sulfur dyestuffs, which consists in transforming the dyestuffs by reduction into leuco compounds, dyeing with them and developing the dyestuff by oxidation, substantially as set forth.

In testimony that we claim the foregoing as our invention we have signed our names in presence of two subscribing witnesses.

MAX BECKE.
ALBERT BEIL.

Witnesses:

HEINRICH HAHN,
ALFRED BRISBOIS.