

UNITED STATES PATENT OFFICE.

MAX SCHÜMANN, OF LUDWIGSHAFEN, GERMANY, ASSIGNOR TO THE BADISCHE ANILIN & SODA FABRIK, OF LUDWIGSHAFEN, GERMANY, A CORPORATION.

BROWN SULFUR DYE AND PROCESS OF MAKING SAME.

SPECIFICATION forming part of Letters Patent No. 687,072, dated November 19, 1901.

Application filed August 13, 1901. Serial No. 72,158. (No specimens.)

To all whom it may concern:

Be it known that I, MAX SCHÜMANN, doctor of philosophy and chemist, a citizen of the free Hansa town of Hamburg, residing at Ludwigshafen-on-the-Rhine, in the Kingdom of Bavaria, Germany, have invented new and useful Improvements in the Manufacture of Brown Sulfur Dyestuff and Processes of Producing the Same, of which the following is a specification.

I have discovered that if 2.4.1 dinitro-acetanilid (which can be obtained by the nitration of acetanilid) is heated with sulfur and sodium sulfid brown coloring-matters are obtained which dye cotton from the alkaline sulfid bath. The brown shades thus obtained possess a considerable fastness against washing, acids, and light, so that the subsequent treatment with chrome compounds or oxidizing agents, such as is usual with sulfur dyes, is unnecessary in the case of the new coloring-matters obtained according to my invention. The shade obtained varies to a certain degree, according to the quantity of alkaline sulfid used in the process for their production, red-brown to pure-brown shades being obtained. If the dyed cotton be treated with copper sulfate, a pure brown results, and upon treating with chromates the shade becomes slightly yellower; but the fastness to washing, which before the treatment is excellent, is not improved. My new coloring-matter dissolves in water with a red-brown to brown color. On the addition of concentrated hydrochloric acid or acetic acid (containing about thirty per cent. of $C_2H_4O_2$) to this solution a red-brown to black-brown precipitate is formed; but caustic-soda lye causes no appreciable change. In concentrated sulfuric acid it dissolves with an orange-brown to brown color and in alcohol with a dirty-yellow to greenish-yellow color.

The following example will serve to further illustrate the nature of my invention and the manner in which the same may be carried into practical effect; but the invention is not confined to the example. The parts are by weight.

Example: Mix together two hundred and

fifty (250) parts of crystallized sodium sulfid with one hundred (100) parts of sulfur in a pot provided with a stirrer and heated in an oil-bath. When the sulfur is dissolved, add to the melt, which should be at a temperature of, say, about one hundred and thirty degrees (130°) centigrade, sixty-five (65) parts of dinitro-acetanilid and then continue heating, so that the temperature of the oil-bath rises slowly to two hundred and twenty degrees (220°) centigrade. The melt becomes stiff. When it is no longer possible to stir it readily, remove it from the pot and spread it out in a thin layer upon iron plates and then in this condition bake it in an air-bath for several hours at a temperature of about two hundred degrees (200°) centigrade. The resulting product can be directly employed in dyeing. It is readily soluble in water containing sodium sulfid, yielding a red-brown solution.

If in the above example three hundred and twenty-five (325) parts of sodium sulfid be used instead of two hundred and fifty (250) parts and the process be otherwise carried out as described, the coloring-matter obtained yields purer brown shades.

Now what I claim is—

1. The process of producing brown coloring-matter containing sulfur by heating dinitro-acetanilid with sulfur and alkaline sulfid, substantially as described.

2. The brown coloring-matter containing sulfur obtainable as hereinbefore described, which dissolves in water with a brown color, which solution, on the addition of hydrochloric acid or acetic acid, yields a brown precipitate, whose solution in concentrated sulfuric acid is brown, and in alcohol yellow, and which dyes cotton brown shades which become slightly yellower on treatment with chromates, substantially as described.

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

MAX SCHÜMANN.

Witnesses:

- WALTER VOIGTLAENDER-FETZNER,
JOHN L. HEINKE.