## STATES PATENT OFFICE.

## HERMANN MULLER, OF PARIS, FRANCE.

## PROCESS OF INDIGO DYEING.

No. 808,398.

Specification of Letters Patent.

Patented Dec. 26, 1905.

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To all whom it may concern:

Be it known that I, HERMANN MULLER, a subject of the Grand Duke of Baden, residing | hundred and fifty cubic centimeters water, at 2 Passage Violet, Paris, in the Republic of 5 France, have invented certain new and useful Improvements in Indigo Dyeing, of which the following is a full, clear, and exact description.

This invention is designed to facilitate and 10 simplify the dyeing with indigo of textile fabrics either in the piece or yarn, to reduce the time necessary for the preparation of the bath, and to effect considerable economy in the process of dyeing. The invention may be applied 15 to the dyeing of cotton, wool, silk, linen, or unions of these fibers.

Hitherto in the process of indigo dyeing the preparation of the bath occupied about from five to six days, and in the dyeing of cotton 20 or other cloth it was considered necessary to dip the cloth or material four or five times into the vat to obtain a dark color. By my present process I am able to prepare the bath in from three to four hours and use it imme-25 diately after preparation and to dye to an equal

shade in a single operation. The old vats are dispensed with and I employ a Jigger or Foulard machine or other suitable form of dyeing-machine with rollers or cylinders.

My invention consists, essentially, in the preparation of a dye-bath from indigo (natural or artificial) with the addition or combination of one or more of the following materials: stannous oxid, (SnO.) stannic oxid, 35 (SnO<sub>2</sub>,) hydrated stannous oxid, Sn<sub>2</sub>O (OH)<sub>2</sub>, hydrated stannic oxid, (H2SnO3,) acetic acid, caustic soda in solution, chromate of potash, and hydrochloric acid.

According as the material to be dyed is 4° cotton, wool, or silk, it is necessary to proceed differently and to use dye-baths of different composition.

Good results in the dyeing of cotton are obtained from a dye-bath prepared as follows: 45 fifty grams powdered indigo made into a paste and boiled with one hundred and fifty cubic centimeters stannic oxid (SnO<sub>2</sub>) dissolves in acetic acid, forming an acetate of tin 20° to 25° Baumé. When the acetic acid is nearly 5° evaporated, into this is poured slowly and mixed four hundred and fifty cubic centime-

ters caustic soda in solution, 40° Baumé, and boiled until the indigo is dissolved. Add three boil again, and strain the solution through a 55 metal sieve. The solution will be of a yellow color, and the materials to be dyed are passed through it (the bath may be either cold or hot) for about ten to fifteen seconds, then wellsqueezed out and slightly aired, and subse- 60 quently passed into a bath for one-half to one minute of two grams bichromate of potash, one liter water, twenty-five cubic centimeters hydrochloric acid. The material on removal is washed, soaped, rinsed, and dried.

Good results in the dyeing of wool and silk materials are obtained from a dye-bath prepared as follows: fifty parts powdered indigo made into a paste and heated with two hundred parts acetic acid, ninety per cent. Add two 70 hundred parts stannic oxid, (SnO2.) The whole is boiled and just sufficient caustic soda in solution added to dissolve and reduce the indigo, then boiled, and brought up to one thousand parts by addition of water thereto. The ma- 75 terials are passed for about fifteen to twenty seconds into this hot bath, then slightly aired, and passed through a cold bath of a fixing solution preferably containing two grams bichromate of potash, one liter water, twenty- 80 five cubic centimeters hydrochloric acid.

Instead of using stannic oxid (SnO<sub>2</sub>) to form an acetate of tin for the preparation of the dye-bath one can use equally as well other oxids of tin-such as stannous oxid, (SnO,) hy- 85 drated stannic oxid, (H2SnO3,) or hydrated stannous oxid, (Sn<sub>2</sub>O(OH)<sub>2</sub>.) Also the solution of caustic soda can be replaced by a solution of caustic potash and the bichromate of potash by any other soluble chromate.

What I claim as my invention, and desire to secure by Letters Patent, is—

1. The process of dyeing fabrics and analogous materials which consists in feeding the material through a dyeing-machine having a 95 dye-receptacle, subjecting it to a dye-bath composed of indigo, an oxid of tin, an acid and an alkaline solution, then airing said material, and finally treating it with a fixing solution.

2. The process of dyeing fabrics and analogous materials which consists in feeding the

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material through a dyeing-machine having a dye-receptacle, subjecting it to a dye-bath composed of indigo, an oxid of tin, acetic acid, and an alkaline solution, then airing said material, and finally treating it with a solution of bichromate of potassium and hydrochloric acid.

In witness whereof I subscribe my signature in presence of two witnesses.

HERMANN MULLER.

Witnesses:
Hanson C. Coxe,
John Barker.