

# UNITED STATES PATENT OFFICE.

EMIL FLICK, JR., OF OPLADEN, GERMANY.

## PROCESS OF MAKING SOLUBLE INDIGO PASTE.

SPECIFICATION forming part of Letters Patent No. 671,994, dated April 16, 1901.

Application filed January 19, 1901. Serial No. 43,837. (No specimens.)

*To all whom it may concern:*

Be it known that I, EMIL FLICK, Jr., a subject of the King of Prussia, Emperor of Germany, residing at Opladen, Rhine Province, German Empire, have invented new and useful Improvements in Processes for the Preparation of a Soluble Indigo Paste, of which the following is a full specification.

The various kinds of indigo paste at present in the market contain either a relatively small percentage of indigo-white or are very much charged with a fixed alkali, chiefly sodium hydrate, or finally require for dissolving and for preparing the indigo-vat such vast quantities of caustic alkali that the subsequent dyeing process of the fiber is greatly interfered with. It is well known that fixed alkalies attack textile fabrics very strongly, and the presence of large quantities of caustic alkalies in the vat also prevents the fixation of indigo on the fiber. For that reason it is a matter of great difficulty to dye dark shades, even if great quantities of reducing agents—hydrosulfites, for instance—are added to the vat, and the light and medium shades also show a lack in brilliancy of color. The shades obtained are rather dull and grayish blue and do not adhere fast to the fiber after all.

The object of the present invention is to avoid all those drawbacks by the preparation of an indigo paste which contains no fixed alkali at all and shows a large percentage in effective indigo-white.

The process depends on the reduction of indigotin by metals in a state of a fine division in the presence of concentrated ammonia. As metals spoken of I may employ for the reduction zinc, tin, or iron, although tin and iron do not act so energetically as zinc, and should therefore not be used unless zinc-dust be not available. Since the reduction of indigo by means of zinc-dust and ammonia proceeds very quickly and energetically, the ammonia is preferably added very gradually to the intimate mixture of indigo and zinc-dust.

The process is carried out thus: In a covered pot provided with an agitator ten kilos

of indigo pure BASF or the equivalent quantity of natural indigo is intimately mixed with about four to five kilos of zinc-dust. After the mixture has become uniform and homogeneous ten to twelve liters of twenty-five per cent. ammonia liquor are added very gradually and slowly, the agitating being continued all the while. When all the ammonia has been added, the agitator is worked for some more time—say one hour or so. The process of reduction is then finished. The pot is opened and the product of reaction quickly pressed in order to free it from the adhering liquor. If all has been carefully done and pure indigo BASF has been employed to start with, the paste obtained will contain somewhat like seventy-five per cent. indigo-white.

As previously mentioned, the zinc-dust may be replaced by tin or iron in a finely-divided state; but in that case equal weights of tin or iron and indigo should be taken, and the quantity of concentrated ammonia liquor required will have to be greatly increased to somewhat like four times the weight of the indigo employed. As a consequence the paste obtained is not so rich in indigo-white.

An indigo paste prepared in the manner described is not only suitable for the preparation of an indigo-vat, but may also be used for printing purposes in combination with the usual thickening. The indigo-vat needs no addition of fixed alkalies, whereby any injury to the textile fabrics to be dyed is avoided as a matter of course. Furthermore, owing to the absence of fixed alkalies, the indigo adheres much faster to the fiber, and there is no difficulty to produce dark shades of color by one single draw through the vat. There is therefore not only a great saving in time, but also in dyestuff. An indigo-vat prepared with a paste made according to the manner stated is also suitable for the dyeing of silk, which is altogether out of the question when fixed alkalies are present in the vat. The colors and shades obtained in dyeing silk show a great brilliancy and adhere fast to the fiber. One may also employ the indigo paste in com-

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bination with other dyestuffs. Galloeyanin BS and RS produce, with indigo paste, red shades both in dyeing and in printing.

What I claim is—

- 5 A process for the preparation of a soluble indigo paste of great strength consisting in adding to an intimate mixture of indigo and a metal in a finely-divided state very gradually a certain quantity of concentrated ammonia liquor, agitating the mass all the while
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and afterward for some time after all the ammonia has been added, and pressing the mass to free it from the adhering moisture, essentially as described.

In witness whereof I have hereunto set my hand in presence of two witnesses.

EMIL FLICK, JUNR.

Witnesses:

KARL SCHMIDT,

CHARLES L. SWINGLE.