

# United States Patent Office.

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Letters Patent No. 101,175, dated March 22, 1870.

## IMPROVEMENT IN THE MANUFACTURE OF XYLOIDINE AND ITS COMPOUND.

The Schedule referred to in these Letters Patent and making part of the same

I, DANIEL SPILL, of Paradise Terrace, Hackney, in the county of Middlesex, England, have invented Improvements in the Production of Xyloidine, and of compounds containing the same, and also in the apparatus to be employed for such purposes, of which the following is a specification.

The first part of my invention relates to a method of treating cotton or other vegetable fibers or lignine, either with nitric acid or with nitric and sulphuric acids, for the purpose of converting the same into xyloidine, and of rendering the same soluble in suitable solvents.

In carrying out this part of my invention I take cotton, or other vegetable fibers or lignine, either in their normal condition, or after they have passed through any manufacturing process, or I take the refuse of the same, or the ordinary rags of commerce, either in a white, dyed, or colored condition. By preference, I first reduce them to a finely-divided state, which may conveniently be effected by any of the well-known means. I then intimately mix the same, by the employment of the mechanical means hereinafter described, either with nitric acid or with a mixture of nitric and sulphuric acids, such as are employed for making soluble gun-cotton or xyloidine, as is well understood.

Into a cylinder or vessel constructed with internal revolving arms or beating-bars, and charged with a suitable quantity of acids, as before mentioned, I introduce the fiber or fibrous materials before referred to, and immediately set the arms or beaters in motion, with the object of saturating or intimately mixing the fibers or fibrous materials with the acids quickly and uniformly.

After contact for a sufficient time, the fiber or fibrous materials are removed and the acid strained therefrom, and they are then transferred into another vessel, in which the product is pressed, so as to remove any excess of acids.

The pressed mass of converted material, which I have called xyloidine, is then opened out and transferred to a washing-vat, having a perforated false bottom and mechanical stirrers. The vat is supplied with a copious stream of water, and the stirring and washing is continued until the xyloidine is nearly or quite free from acids. The water may then be drained off through the bottom of the vat, and the xyloidine removed to a press-box to remove the excess of water, when, after such operation, the xyloidine may be opened out, and dried by any known means, when it will be found to be ready for dissolving.

The second part of my invention relates to the bleaching of xyloidine, and is as follows:

When it is desired to bleach or whiten the xyloidine, I bleach it directly after the removal of the acids,

and before removing it from the vat. This I do by any of the well-known means, preferring a solution of chlorine or a solution of chloride of lime or of soda, which I add to the xyloidine, making use of alternate stirrings and rest for a sufficient time until the xyloidine is whitened. The solution is again drained off, and the xyloidine is repeatedly washed with water, in order to remove any excess of bleaching agents or any residue from such agents, when it will be found to be ready to be submitted to pressure in order to free the same from water, and may then be opened out so as to prepare it for drying, dissolving, or other purposes.

My invention relates, thirdly, to the dyeing of xyloidine.

After draining, and before pressing the xyloidine, it may be dyed to any color or tint by any of the well-known processes for dyeing fibers, after which it may be washed and pressed, in order to remove the excess of water, and the pressed mass may then be opened out, and be thus rendered in a fit state for drying or dissolving.

The solvents which I prefer to employ for its subsequent treatment are such as are described in patents which have been granted to me, bearing date the fifteenth day of June, one thousand eight hundred and sixty-nine, and the thirtieth day of November, one thousand eight hundred and sixty-nine, and these solvents are to be used as therein described. Or, instead of the xyloidine being dyed before solution, it may be dyed during or subsequent to solution, with any colors which may be soluble in alcohol or hydrocarbon fluids, or in the solvents employed, the colors being, by preference, dissolved therein before the xyloidine is added.

My invention consists, fourthly, as follows:

In practice I prefer to dissolve one part xyloidine in from five to twelve parts of solvents, such as have been before referred to. After such solution I add such pigments as are described in my said former patents. I afterward strain the solution or paste through a fine sieve under pressure, to remove any mechanical impurities, after which operation it is in a fit condition for spreading upon surfaces or fabrics in a semi-fluid condition.

My invention consists, fifthly, as follows:

When preparing the compound for the production of solid articles, or for the covering of telegraph-wires, or for rolling into sheets, or for spreading upon fabrics or surfaces, I take the before-mentioned solution or paste, after it has been strained from impurities, and place it in a closed or air-tight chamber, provided with any suitable mechanical means for mixing or stirring the same, the spindles in connection therewith being made to pass through stuffing-boxes, the said chamber or apparatus being in connection with a condenser and a reservoir, and also, by preference, being in con-

nection with an exhausting or vacuum-producing apparatus. The air, after passing through the exhausting-apparatus, may or may not be made to re-enter the closed or air-tight chamber or apparatus, thus keeping up a continuous circulation. The latter arrangement being preferred, as that portion of the volatile solvent which would, in the former case, escape and be lost were the air allowed to escape, is retained and economised by continuous circulation. Heat is then to be applied to the mixing apparatus, say at a temperature of about 212° Fahrenheit, and the agitator or mixing arrangement is set in motion. As the operation proceeds the solvent will pass over in vapor and become condensed, and may be employed for reuse or otherwise, and the resulting mass will be found to be in a nearly dry condition and of a uniform consistency, which product may be then rolled into sheets by any well-known means, or it may be pressed or molded into forms, or otherwise fashioned.

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

1. The method herein described of treating cotton, or other vegetable fibers or lignine, with nitric acid,

or with nitric and sulphuric acids, for the purpose of converting the same into xyloidine, and rendering the same soluble in suitable solvents.

2. The process of bleaching xyloidine in the manner herein specified.

3. The herein-described process of dyeing xyloidine, either before or after the solution of the same in suitable solvents, as set forth.

4. The herein-described mode of preparing xyloidine for spreading upon surfaces or fabrics in a semi-fluid condition.

5. The herein-described process of treating xyloidine, so as to bring it to a nearly dry condition, for the production of solid articles, or for other purposes, as set forth.

In testimony whereof I have signed my name to this specification in the presence of two subscribing witnesses.

DANIEL SPILL.

Witnesses:

CHAS. MILES,  
T. W. FRIGORT.