UNITED STATES PATENT OFFICE.

MAX FREMERY AND JOHANN URBAN, OF OBERBRUCH, GERMANY.

MANUFACTURE OF CELLULOSE.

SPECIFICATION forming part of Letters Patent No. 657,818, dated September 11, 1900.

Application filed June 20, 1899. Serial No. 721,261. (No specimens.)

To all whom it may concern:

Be it known that we, MAX FREMERY, a subject of the German Emperor, and JOHANN URBAN, a subject of the Austrian Emperor, both residing at Oberbruch, near Aachen, in the German Empire, have invented certain new and useful Improvements in the Manufacture of Cellulose; and we do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

Our invention has reference to a preliminary treatment of cellulose to facilitate and increase its solution in ammoniacal solutions of oxid of copper or in solutions of chlorid of zinc. In Patent No. 617,009, granted January 3, 1899, it has already been stated that cellulose not preliminarily treated dissolves in an ammoniacal solution of oxid of copper only to such extent that the produced solution would contain not more than about four and one-half per cent. cellulose.

In carrying out our invention we may treat cellulose in the form of any cotton free from grease or hydrate of cellulose produced, for instance, by parchmentizing cellulose by means of sulfuric acid of 59° Baumé or by precipitating solutions of chlorid of zinc or ammoniacal solution of oxid of copper containing a small percentage of dissolved cellulose, or we may treat hydrocellulose produced, for instance, by the well-known processes of Girard, (*Berichte der Deutschen Chemischen Gesellschaft* IX, 65ª.) We have discovered that by subjecting the cellulose or its derivatives to a preliminary energetic treatment with oxidizing or reducing bleaching media or chemical agents its subsequent solution in the ammoniacal solution of oxid of copper or a solution of chlorid of zinc will be effected in a much shorter time and that such cellulose can be dissolved (by employing the suitable quantity of copper and ammonia) in a larger percentage—almost double the percentage hitherto employed.

In carrying out our invention we may use various bleaching media, such as sulfurous-acid salts or chlorin in the form of hypochlorite or nascent chlorin in watery solution or chlorin water. The concentration of the bleaching agent and the duration of the action of the same will depend in each case upon the special kind or nature of the cellulose material employed to the solution. For instance, while with cotton a relatively-weak bleaching action produces the desired effect ramie fiber requires a more vigorous treatment. In the treating of wood cellulose the bleaching composition must be of a still higher power in order to obtain the desired effect; but in every case the treatment must be more energetic than is usual for other purposes.

Cotton of commerce free from grease—as, for instance, surgical wadding—dissolved in an ammoniacal solution of oxid of copper or in a solution of chlorid of zinc dissolves very slowly and only in such proportions that the solution obtained contains only up to about three or four per cent. of cellulose. The same material left for from twelve to eighteen hours in a bleaching liquid prepared from fifteen grams of chlorid of lime per liter of water will, after a preliminary washing and drying, dissolve in an ordinary ammoniacal solution of oxid of copper in the proportion of about eight per cent. and more, and that in the space of a few hours. If the bleaching is carried on still more energetically, there results a very liquid solution, which is, however, not suitable for spinning purposes.

By the employment of our process, which secures the solution of more than double the percentage of cellulose hitherto obtainable, the methods based upon the working of solutions of cellulose, such as the production of artificial silk, are rendered quite economical.

We claim as our invention—

As an improvement in the production of cellulose products from cellulose material, such as cellulose, hydrate of cellulose or hydrocellulose, subjecting the material to an energetic preliminary treatment with reducing or oxidizing bleaching media and then subjecting it to the action of an ammoniacal solution of copper, substantially as described.

In testimony whereof we affix our signatures in presence of two witnesses.

M. FREMERY.
JOH. URBAN.

Witnesses:
CLARA E. BRUNDAGE,
E. M. BRUNDAGE.