

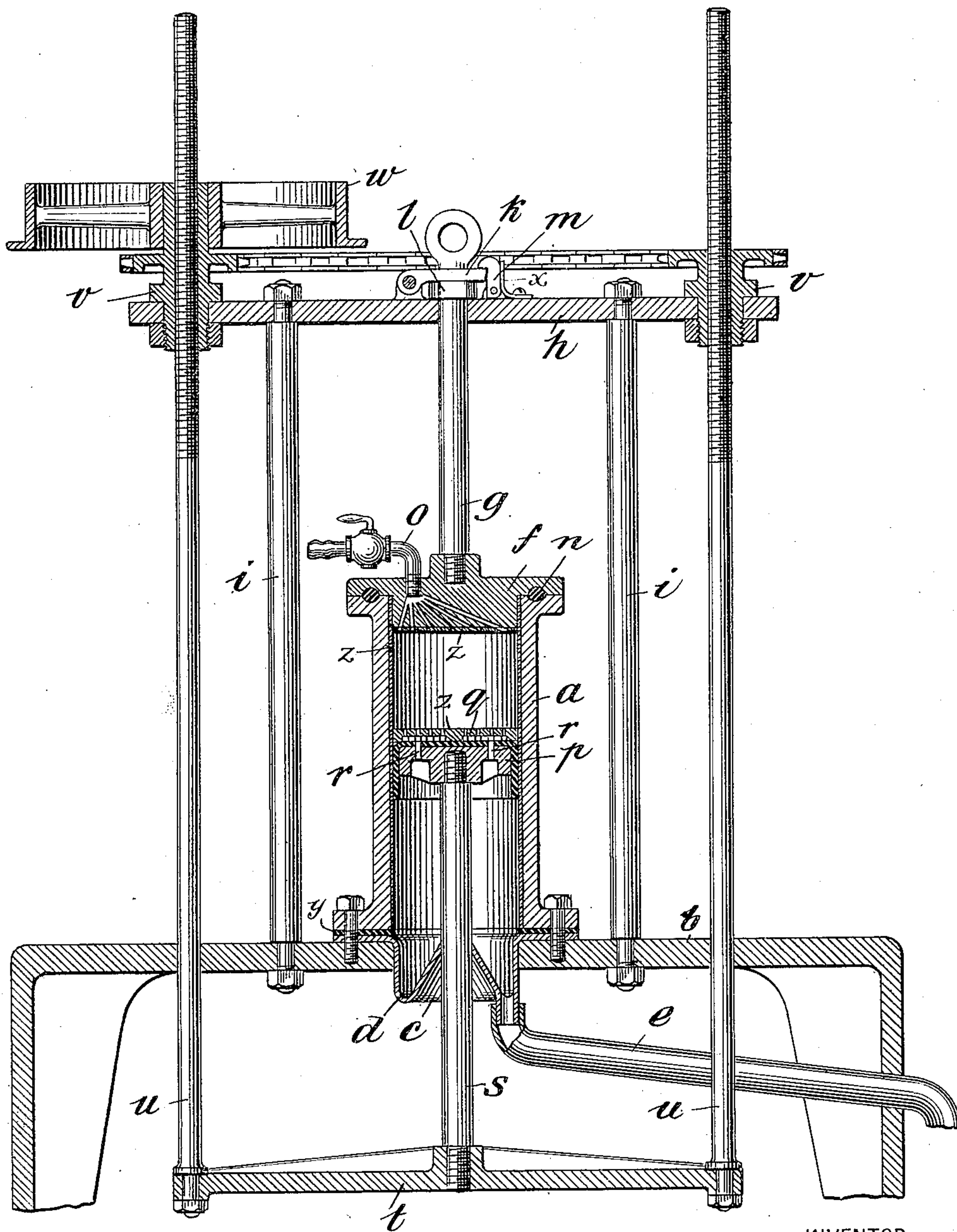
No. 648,147.

Patented Apr. 24, 1900.

F. I. DU PONT.
PROCESS OF MAKING GUNCOTTON.

(Application filed Apr. 15, 1899.)

(No Model.)



WITNESSES:

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FRANCIS I. DU PONT, OF WILMINGTON, DELAWARE.

PROCESS OF MAKING GUNCOTTON.

SPECIFICATION forming part of Letters Patent No. 648,147, dated April 24, 1900.

Application filed April 15, 1899. Serial No. 713,135. (No specimens.)

To all whom it may concern:

Be it known that I, FRANCIS I. DU PONT, a citizen of the United States, residing at Wilmington, in the county of New Castle and State of Delaware, have invented certain new and useful Improvements in Processes of and Apparatus for Making Guncotton; and I 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.

My invention relates to improvements in processes of making guncotton or nitrocellulose, and relates more particularly to improvements in the methods employed for removing the acid from guncotton; and my invention consists in removing the acid from guncotton after nitration and in neutralizing the guncotton or rendering it slightly alkaline by a process of percolation.

The objects of my invention are to avoid loss of acid and of guncotton during the washing of the latter to remove the acid, to produce a better quality of guncotton, to avoid decomposition of the guncotton, and to keep it moist throughout the treatment, thus avoiding danger of spontaneous ignition and to neutralize it more thoroughly than has been practicable heretofore. These objects are attained in the process herein described and in the press illustrated in the drawing which accompanies and forms a part of this application, which drawing shows a central vertical section of the press.

The Reissued Patent No. 11,651 to Francis G. du Pont, dated February 15, 1898, describes a method of replacing water in guncotton by another fluid, and particularly of replacing water in wet guncotton by alcohol, which process is particularly intended for use in the manufacture of smokeless powder. In said process the wet guncotton is placed within a suitable press, mechanical pressure is applied thereto to reduce the guncotton to a condition of uniform porosity, and the fluid which is to replace the water in the guncotton is admitted to the compression-cylinder at one end thereof and is drawn through said cylinder and therefore through the guncotton therein by suction, replacing the water in the guncotton as it does so. The patent states and experience in carrying out the process shows

that alcohol when so employed does not mix with the water in the guncotton to an appreciable extent, but that a sharp line of demarcation exists between the water and the alcohol while the process is being carried out, so that the water is completely replaced by the alcohol without wasteful dilution of the latter. I have discovered that this process of replacing one liquid in the guncotton by another by percolation may be applied advantageously during the manufacture of the guncotton to the removal therefrom of the acid by which the nitration is effected and to the thorough and complete neutralization of the guncotton. Heretofore it has been considered by those skilled in the manufacture of guncotton that addition of water to guncotton while saturated with acid from the nitrating-bath must be carefully avoided, because of the heating produced by the mixing of water with the acid, which tends to cause decomposition of the guncotton, and for this reason it has been customary to wash guncotton for the removal of the acid by plunging it below the surface of water employed in such quantity that heating is prevented; but this causes great loss of the acid and also some loss of guncotton, both of which losses are prevented by the process herein described.

Referring now to the drawing, *a* is a cylinder provided with a lining *z* of lead or an alloy of lead and antimony or porcelain or other suitable material for resisting the action of the acid. The cylinder may be secured to a suitable table *b*. Its lower head or end piece *c* is provided with a gutter *d* for collecting the fluid which flows from the guncotton while the process is being carried on, and to this gutter is connected a drain-pipe *e*, which is preferably a flexible lead pipe. Suitable packing material *y*, as asbestos, is placed between the cylinder *a* and the lower cylinder-head *c*. The upper head *f* of the cylinder is removable, having attached to it a rod *g*, passing through an opening in the plate *h*, connected to the table *b* by suitable columns *i*. A suitable locking device consisting of a lever *k*, adapted to rest upon a shoulder *l* on the rod *g*, and a latch *m*, provided with a spring *x* and adapted to hold the lever *k* in place, is employed to hold the cover *f* down. A packing-ring *n*, of rubber or other suitable mate-

rial, prevents leakage between the head *f* and cylinder *a*. The head *f* should also be provided with a lining *z* of acid-proof material similar to that with which the cylinder *a* is lined. It is provided with a series of passages radiating from the end of a pipe *o* and terminating in the face of the cylinder-head.

p is the piston of the press. Like the head *f*, it is provided with a facing *z* of acid-resisting material, its face being perforated to connect a cavity *q* in the piston with the interior of the cylinder. Draining-openings *r* in the piston permit fluid collecting in this cavity *q* to flow into the lower portion of the cylinder, and so into the gutter *d* and drain-pipe *e*.

s is the piston-rod. It passes through an opening in the lower cylinder-head *c* above the level of the gutter *d* and is connected by a yoke *t* to two screw-rods *u*, having nuts *v* revolvably mounted in the plate *h*, but held against longitudinal movement. One nut is provided with a driving-wheel *w*, adapted to receive a driving-belt, and the other nut is driven from that to which the wheel *w* is connected by sprocket-gearing.

The method of carrying out the process is as follows: The upper cylinder-head *f* is removed, the piston lowered to the bottom of the cylinder, if necessary, the cylinder filled with guncotton containing the acid to be removed, the cylinder-head *f* replaced and fastened down, and pressure applied to the guncotton in the cylinder by rotating the nuts *v*, and so moving the piston upward. Sufficient pressure is applied to reduce the guncotton to a condition of uniform porosity, and for this purpose I find a pressure of from one hundred and fifty to two hundred pounds per square inch sufficient. The pipe *o* is connected to a source of supply of water under pressure, and water is admitted to the cylinder under sufficient pressure to cause it to replace the acid in the guncotton by percolation, the acid passing through the openings in the piston into the lower portion of the cylinder, falling into the gutter *d*, and then passing out through the pipe *e*.

The acid flowing from the pipe *e* may be collected in a suitable vessel. It is not diluted appreciably by the water which replaces it in the guncotton.

The termination of the process of displacement of the acid by the water is indicated by a slight rise in temperature in the pipe *e*, which may be detected by applying the hand to said pipe or by use of a thermometer. When this rise in temperature is noted, the water may be turned off, the pipe *o* connected to a source of supply of an alkaline fluid, such as is ordinarily used for neutralizing acidity in guncotton, and the end of the pipe *e* moved so as to deliver fluid flowing from it into a suitable receptacle other than the acid-receptacle. The water in the guncotton is then displaced by the alkaline fluid by percolation in a manner similar to that by which the acid was displaced by the water. The guncotton may then

be removed from the cylinder and treated as is customary or desirable.

I find that in carrying out this process no sensible heating of the guncotton within the cylinder occurs and that there is great economy, due to the saving of acid and due to the fact that none of the guncotton is lost. Treatment of the guncotton in centrifugal machines to extract the acid is avoided, a much greater quantity of acid being saved than when the centrifugal machines are employed to remove it, and at no time during the treatment is the guncotton in such condition that spontaneous ignition may occur. Moreover, a better and more stable product than that heretofore produced is obtained. I have also found that pulping of the guncotton is not required for the carrying out of this process, but that the process works well whether the guncotton be in the form of pulp or in the form of rags, the pressure sufficing to reduce the mass of guncotton in whatever form it may be to a condition of uniform porosity.

In this specification I intend to include by the term "guncotton" nitrocellulose of all degrees of nitration.

Having thus completely described my invention, what I claim, and desire to secure by Letters Patent, is—

1. The herein-described method of removing acid from guncotton, which consists in applying pressure to the guncotton and then, while such pressure is applied, replacing the acid in it with another fluid, by percolation.

2. The herein-described method of removing acid from guncotton, which consists in applying pressure to the guncotton and then, while such pressure is applied, replacing the acid in it with water, by percolation.

3. The herein-described method of removing acid from guncotton, which consists in applying pressure to the guncotton and then, while such pressure is applied, replacing the acid in it with a neutral fluid, by percolation, and then replacing the neutral fluid with an alkaline fluid, by percolation.

4. The herein-described method of removing acid from guncotton, which consists in compressing the guncotton until it has reached a condition of uniform porosity throughout, and then, while such pressure is applied, replacing the acid with another fluid by percolation.

5. The herein-described method of removing acid from guncotton, which consists in compressing the guncotton until it has reached a condition of uniform porosity throughout, and while such pressure is applied replacing the acid with water by percolation, and then replacing the water with an alkaline fluid by percolation.

In testimony whereof I hereunto affix my signature in the presence of two witnesses.

FRANCIS I. DU PONT.

Witnesses:

JOHN W. MACKLEM,
F. W. FISHER.