

No. 764,776.

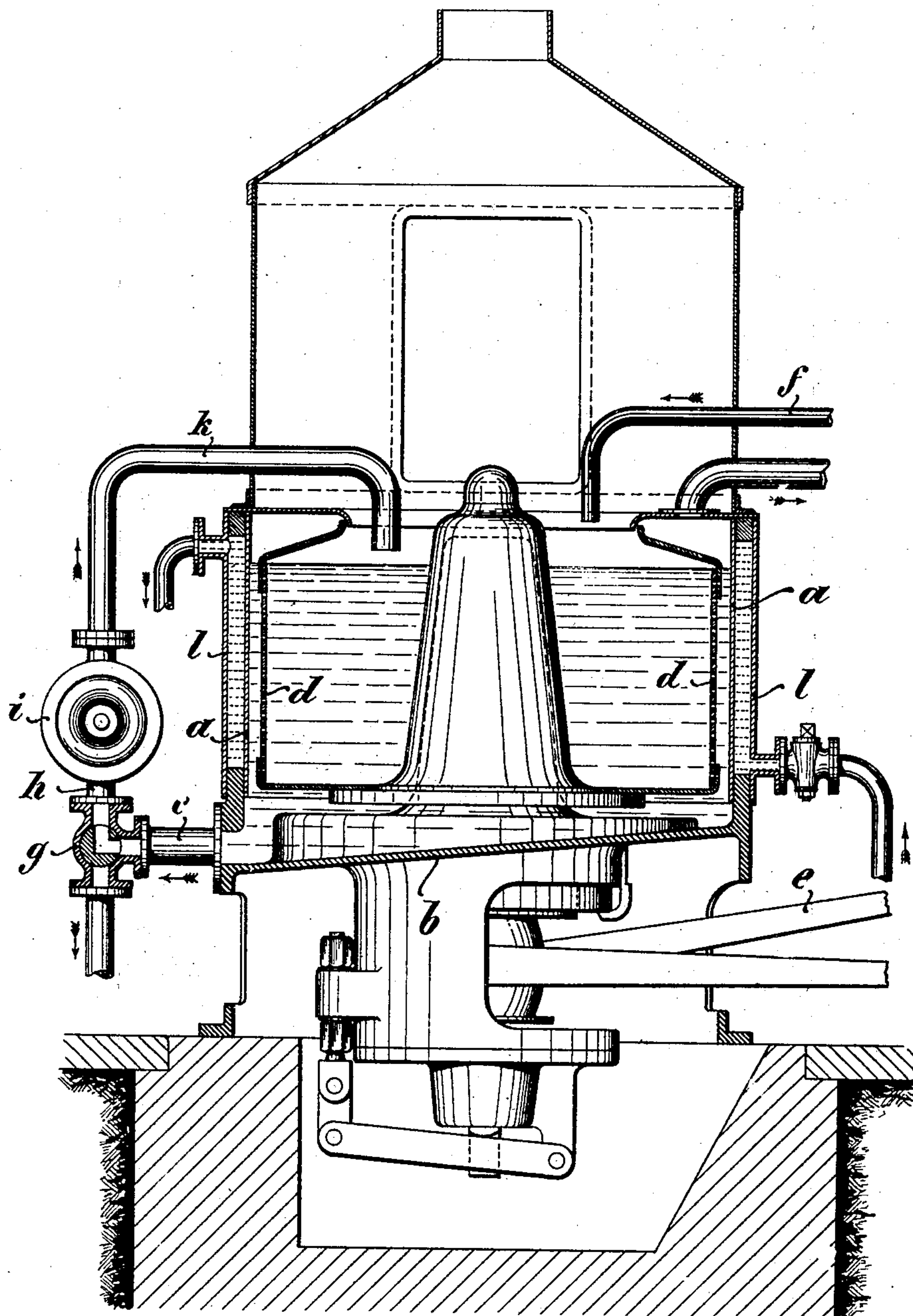
PATENTED JULY 12, 1904.

J. SELWIG.

PROCESS OF NITRATING FIBROUS MATTER.

APPLICATION FILED APR. 6, 1904.

NO MODEL.



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UNITED STATES PATENT OFFICE.

JOHANNES SELWIG, OF BRUNSWICK, GERMANY.

PROCESS OF NITRATING FIBROUS MATTER.

SPECIFICATION forming part of Letters Patent No. 764,776, dated July 12, 1904.

Application filed April 6, 1904. Serial No. 201,938. (No specimens.)

To all whom it may concern:

Be it known that I, JOHANNES SELWIG, a subject of the German Emperor, and whose post-office address is No. 24 Petritorpromenade, Brunswick, German Empire, have invented a new and useful Process of Nitrating Fibrous Matter; and I do hereby declare that the following is a full, clear, and exact description of my invention, which will enable others skilled in the art to which it appertains to make and use the same.

This invention refers to the process of treating fibrous matter—such as cotton, cellulose prepared from wood, and others—with nitric acid or mixtures of nitric and sulfuric acids in order to obtain nitrated cellulose or nitrocellulose; and its object is to so improve upon said process that self-decomposition is obviated and an evenly-nitrated product obtained.

In carrying out the said nitrating process in the usual manner it is out of reach, as is well known to those skilled in the art, to secure a uniform progress and distribution of the nitrating action throughout the entire bulk of fibers treated, so that the process results in an unevenly-nitrated product—that is to say, one of which different portions are differing from each other as regards the percentage of nitrogen combined with the cellulose therein and their behavior toward the solvents for nitrocellulose. Moreover, the fibrous matter under treatment frequently falls a prey to decomposition, followed by or resulting in spontaneous combustion. These inconveniences are due to the nitrating acid being from any cause allowed to act at some places of the bulk of fibrous matter with greater strength than at others, whereby at such places of more energetic or quicker action a correspondingly-increased generation of heat takes place, which in being propagated to the places of slower reaction, where the acid as a consequence of its slower action is more concentrated, acts to assist the latter, which now, with this assistance of heat, acts very powerfully, and thereby contributes to a very irregular generation of heat within the fibrous bulk and the strength of the acid being very irregularly altered throughout the fibrous bulk. Now I have discovered that the said irregularities and bad effects are

fully removed by causing a constant circulation of the nitrating acid through the bulk of fibrous matter to be transformed into nitrocellulose, provided the fibers be of a uniform quality. With such constant circulation of the acid the latter when at a given time samples are taken from different portions of the acid-penetrated bulk of fibers and tested will be found to have a uniform strength and temperature in such different portions, which is proof of the nitrating action going on very regularly, and this regularity of the nitrating action throughout the bulk of fibers results in a very uniformly-nitrated product. A very important merit of my improved process is the total avoidance of such conditions as result in self-composition.

In carrying my improved process into practice the well-known centrifugal hitherto used for manufacturing nitrocellulose has proved to be a very convenient instrumentality for my purpose. As known by those skilled in the art, this centrifugal is made use of as follows: The apparatus is first filled with acid, penetrating from the casing through the perforated wall of the basket into the latter, which is then supplied with the fibrous matter. As soon as the nitration of the latter is at the desired degree the acid is withdrawn from the casing, when the basket is rapidly rotated to deprive the acid-penetrated fibers from the surplus of acid adhering thereto. If employed for my purpose, the casing and basket of said centrifugal are combined with means adapted for constantly returning the acid from the casing into the basket, and the latter is given a slow speed of rotation. This slowness of rotation is desirable, for the reason that such a rapid rotation as is necessary for finally removing the acid after it has done its work or for drying and other purposes would have the effect of packing the fibers too closely against each other, thereby preventing the acid from penetrating into and passing through the bulk of fibers in a uniform and regular manner and causing it to slide up at the inner side of the fibrous bulk and to escape over the upper edge of the basket, so that the fibers are left in a state of insufficient penetration. In order to adapt the said centrifugal for my purpose, two ways

may be followed. One consists in combining with the casing and the basket an acid-pump or a compressed-air ejector for sucking the acid which has penetrated through the fibrous matter from the bottom portion of the casing and pouring it again into the basket from above near the central portion of the same, the basket being not rotated at all or only with a very slow speed. The other way consists in causing the basket to rotate slowly and simultaneously catching the acid at the outside of the basket by means of an open channel, which is arranged outside of the basket and so as to be inclined toward the wall of the casing or by means of a pipe provided with a baling-mouth, said channel or pipe being so arranged as to feed the acid back into the central portion of the basket. On the annexed sheet of drawing, which is a vertical axial section of the apparatus, is shown a centrifugal of the said type adapted for my purpose by the combination of its casing and basket with an acid-pump.

a is the casing provided with an inclined bottom *b*, having a lateral cocked acid-exit *c*, and *d* is the basket provided with perforated wall and arranged to be turned about its axis by means of a driving-belt *e*, as with other centrifugals, the interval between the top edges of the casing and basket being closed, while the top of the basket is open to provide for the opening for charging it with the fibrous matter.

f is a pipe for supplying the basket with the acid. As shown, the acid-exit *c* is connected by a three-way cock *g* with the suction-pipe *h* of an acid-pump *i* of known construction, the delivery-pipe *k* of which is bent

inward, so as to open above the top of the basket near the central portion thereof. 40

l is a jacket to be used for cooling or heating, as the case may be, by means of a circulating fluid.

It is well understood that my invention is not dependent upon the use of said centrifugal in combination with one or the other of the alleged instrumentalities for causing the nitrating acid to circulate, but may be carried out by the aid of any centrifugal apparatus so devised as to secure the reflux of the acid from the casing to the basket, and I therefore do not limit myself to the use of the apparatus shown and described. 50

What I claim as my invention is—

1. The improved process of nitrating fibrous matter, which consists in supplying to the basket of a centrifugal fibrous matter and nitrating acid, and returning the acid from the casing directly to the basket, substantially as herein described. 55 60

2. The improved process of nitrating fibrous matter which essentially consists in placing the fibrous matter in the basket of a centrifugal, pouring nitrating acid into the basket, rotating the latter with a slow speed, and constantly returning the acid from the casing to the basket, substantially as and for the purpose set forth. 65

In testimony whereof I have hereunto set my hand in presence of two subscribing witnesses. 70

JOHANNES SELWIG.

Witnesses:

CARL KOELUNE,
RICHARD WAGNER.