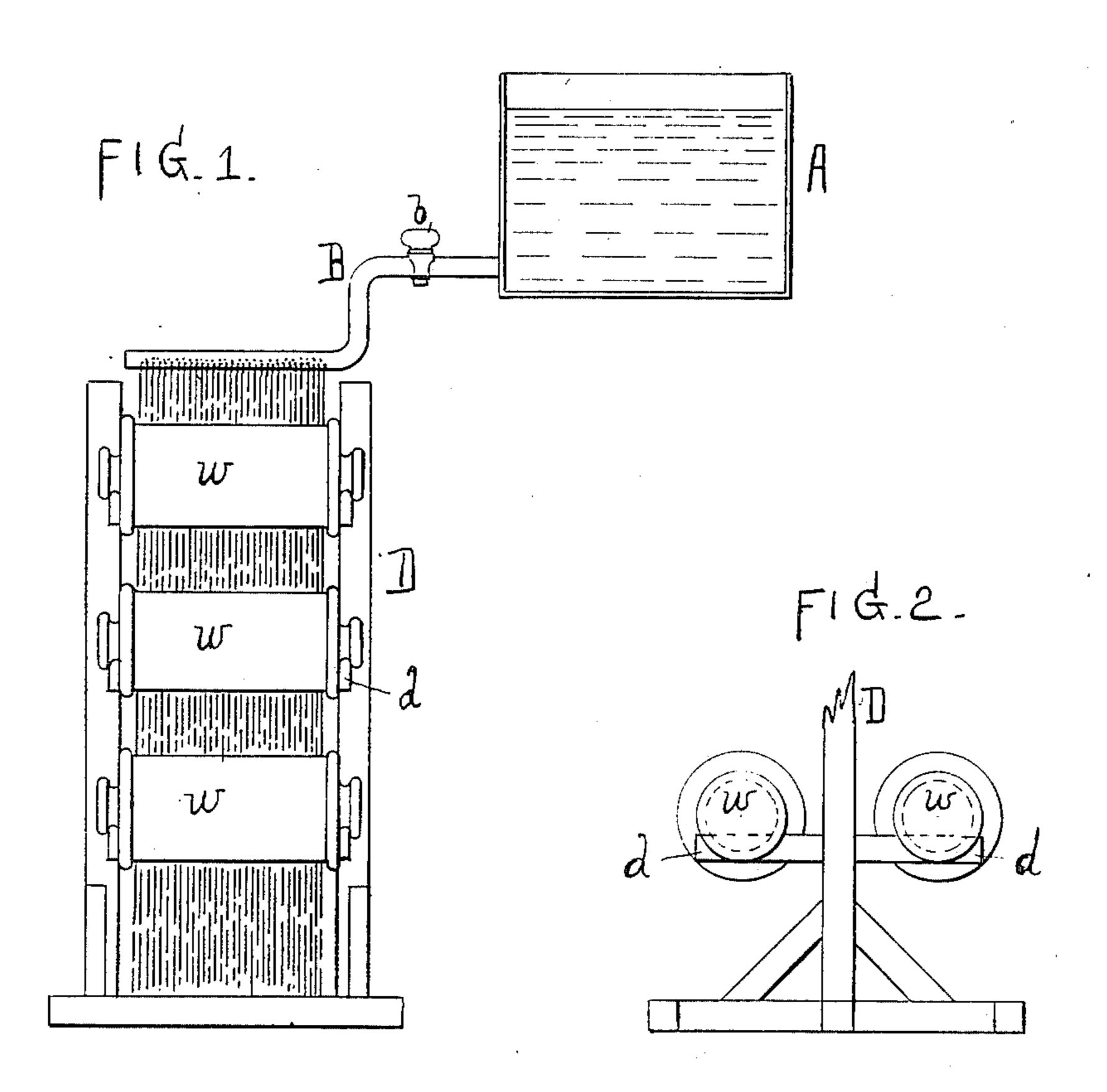
Patented Nov. 6, 1900.

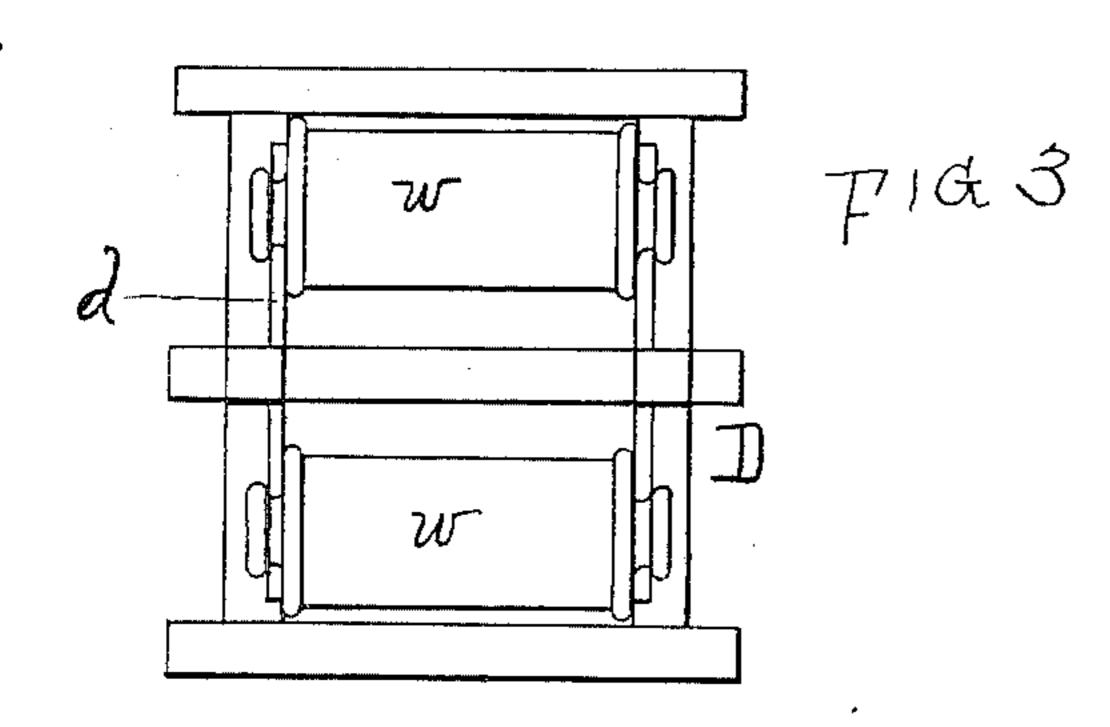
## M. FREMERY & J. URBAN.

## METHOD OF WASHING SPOOLED THREAD OR STRIPS OF CELLULOSE.

(Application filed June 20, 1899.)

(No Model.)





WITNESSES: H.W. Wright

INVENTORS:

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## United States Patent Office.

MAX FREMERY AND JOHANN URBAN, OF OBERBRUCH, GERMANY.

METHOD OF WASHING SPOOLED THREAD OR STRIPS OF CELLULOSE.

SPECIFICATION forming part of Letters Patent No. 661,214, dated November 6, 1900.

Application filed June 20, 1899. Serial No. 721,259. (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 Austro-Hungarian Emperor, both residing at Oberbruch, near Aachen, in the German Empire, have invented certain new and useful Improvements in Methods of Washing Spooled Thread or Strips of Cellulose or the Like; 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.

In the manufacture of artificial silk from solutions of cellulose it is difficult to completely free the cellulose threads when coiled or spooled on rollers from the solvent and the chemicals used for the separation of the cellulose from the solution. Even when large quantities of liquid are used and the washing is continued for several days the cellulose is not always made absolutely clean.

The object of our invention is to provide a method of thoroughly washing spooled cellulose products—such as threads, strips, and the like—which, in contradistinction to the ordinary porous cotton threads or the like, are obtained in an initial more or less gelatinous state by precipitation from solutions of cellulose in a comparatively short time and with moderate quantities of washing liquid.

In the manufacture of cellulose products such as threads, &c.—obtained in an initially gelatinous condition, attempts have been 35 made to eliminate therefrom the components or parts of the dissolving agent by means of large quantities of water; but such attempts have not been successful in that such elimination was not sufficiently complete. Our 40 experiments have led us to believe that the reason for this failure was the formation of insoluble basic salts by the action of the great quantity of water upon the dissolving agents of the cellulose solvent—e. g. cupro-ammo-45 nium or chlorid of zinc—and such basic salts | cannot be eliminated by washing with water, but only with a comparatively stronger acid.

According to the present invention the initially gelatinous cellulose products above mentioned are washed out while spooled or coiled up by bringing the said products into contact only with comparatively small quan-

tities of the washing liquid or water in order not to dilute too suddenly the liquid adhering to or contained in the cellulose products 55 wound on spools or the like and in order to consequently avoid the formation of insoluble salts or combinations from the salts used for the dissolving of cellulose. We have found that sufficient washing of such products is 60 secured by using the washing-water successively in small portions.

Our present invention is practically carried out by washing the cellulose threads, strips, and the like (obtained in an initially gelati- 65 nous condition, as described) when spooled by causing the washing liquid, such as water, to flow successively over a number of spools (on which the said cellulose products are wound) arranged one over the other in which oper- 70 ation it is possible to control or regulate the supply of the washing liquid or water, so as to bring only such small quantities (and this is most important) at once into contact with the spooled cellulose products that the for- 75 mation of insoluble basic salts from the salts used for dissolving cellulose is prevented. As soon as the uppermost spools are completely washed out, they are removed and the lower spools are successively transferred from 80 below upward and nearer to the supply of washing liquid, (water.)

In carrying our invention into practice we prefer to employ the simple form of apparatus illustrated in the accompanying drawings, in 85 which—

Figure 1 is a side elevation of the apparatus with a water-reservoir indicated in section. Fig. 2 is an elevation of part of the apparatus looking from the left of Fig. 1, while 90 Fig. 3 is a plan view of the spool-stem.

A denotes a suitable reservoir or other source of supply for the washing liquid, with one or more delivery-pipes B, having cocks or valves b and perforations in those portions of the discharge-pipes which lie immediately over the superposed spools w of cellulose threads to be washed. These spools w are mounted on arms d, arranged in a suitable frame D, and they can be readily transferred from one pair of supporting-arms to another. The washing - water is allowed to flow in streams over the rollers or spools thus placed one above the other, and the flow is contin-

ued until it is ascertained by the employment of reagents that the chemicals have been washed out and completely removed from the uppermost spools of cellulose. These upper-5 most spools are then removed and the spools below are severally transferred to the next upper supporting-arms on the stand and fresh spools laid on the bottom arms and the washing continued It has been found that with 10 this mode of procedure the washing liquid completely frees the cellulose from the chemicals to be removed and this with only a moderate quantity of washing liquid. The same process and apparatus may be used for other 15 wound or spooled cellulose products—for example, strips or films.

It should be understood that the present invention has no relation to cellulose threads from cellulose having its natural organized condition—such as cotton, hemp, &c.—and consequently its natural porosity. The present invention has only relation to cellulose products obtained by dissolving natural cellulose in any suitable solvent and by then precipitating the dissolved cellulose by introducing such solution through fine apertures,

holes, or slits into a precipitating fluid, thereby obtaining initially more or less gelatinous or colloidal filaments without the original natural porosity.

We claim as our invention—

The herein-described method of freeing spooled cellulose threads, strips and the like from the chemicals used in their manufacture, the said method consisting in bringing 35 the washing liquid in small portions successively into contact with the coils of the described cellulose products arranged one over the other, so that the first coil receives the fresh small portions of the washing liquid 40 which then are brought into contact with the second and then with third coils, &c., and moving up the lower coils as the washing of the upper ones is completed.

In testimony whereof we affix our signa- 45

tures in presence of two witnesses.

M. FREMERY. JOH. URBAN.

Witnesses:

E. M. BRUNDAGE, C. E. BRUNDAGE.