Jan. 28, 1930. E. CZAPEK ET AL 1,745,247

MANUFACTURING FOILS, FILMS, RIBBONS, AND THE LIKE FROM VISCOSE AND SIMILAR CELLULOSE SOLUTIONS

Filed Sept. 1, 1927

Fig. 1.

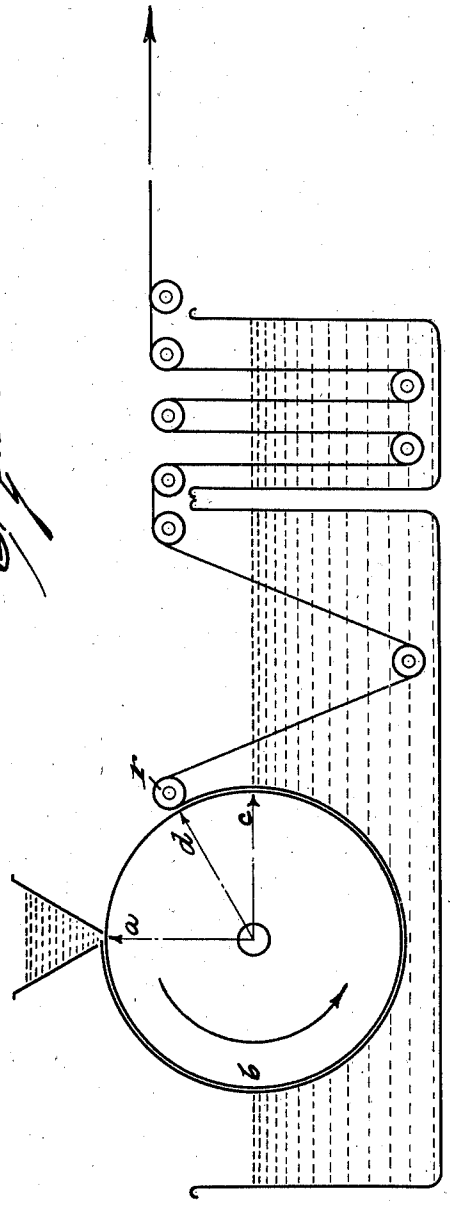
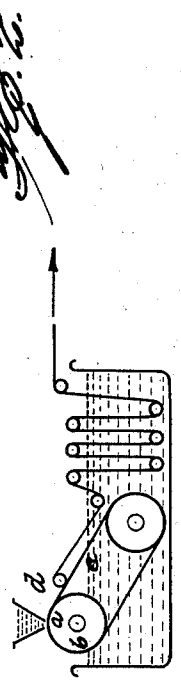


Fig. 2.



Inventors

Emil Czapek et al

Richard Weingand.

By Pennie Edmonds

Attorneys

Patented Jan. 28, 1930 1,745,247

UNITED STATES PATENT OFFICE

EMIL CZAPEK AND RICHARD WEINGAND, OF BOMLITZ, NEAR WALSRODE, GERMANY

MANUFACTURING FOILS, FILMS, RIBBONS, AND THE LIKE FROM VISCOSE AND SIMILAR CELLULOSE SOLUTIONS

Application filed September 1, 1927, Serial No. 217,012, and in Germany September 13, 1926.

This invention relates to a process for the production of foils, films, bands, and the like, from viscose and similar cellulose solutions.

At the present time two fundamentally different processes are employed for the production of cellulose foils from cellulose solutions.

One of these processes is disclosed in the specifications of German Patents No. 237,152, No. 249,256 and No. 257,227, and consists in extruding the cellulose solutions through a wide funnel-like nozzle into the precipitating liquid. The cellulose solution which congeals in the precipitating liquids is at first still very sensitive to mechanical influences, for which reason it must be passed through the subsequent hardening baths with the aid of a large number of closely adjacent rollers. In this process the mounting of the numerous rollers which has partly to be effected within acid liquids, offers exceedingly grave difficulties. Moreover, as the shrinking of the cellulose does not proceed uniformly, no uniformly smooth and transparent film is produced, but, on the contrary, irregular formations occur in the foil, which, held to the light, exhibits, in addition, irregular streaks, so that the quality of the product is both mechanically and optically depreciated.

The second type of process differs from the former in so far as a pouring base is employed. In contradistinction to the previously described process a brilliant very homogeneous film is obtained. Such processes employing pouring bases have however the disadvantage that it is extremely difficult to carry them into practical effect. Incompletely precipitated remnants of the material to be precipitated too easily remain adhering to the pouring base. Attempts have therefore been made to provide the pouring base with various kinds of cleaning devices, such as for example with scrapers and brushes. A further process aims at preventing the adherence of impurities by rubbing fat into the pouring base prior to the application of the cellulose solution. These methods of cleaning render the carrying out of the process very difficult and are never completely successful since even very small remnants of for example half precipitated material or locally diffused portions of the precipitating bath are sufficient to disturb the entire operation.

The disadvantage of the adherence is to be solved. According to German specification No. 421,383 it is proposed to overcome the aforesaid disadvantage by uniformly sprinkling the pouring base with precipitant prior to the application of the cellulose solution so that on applying the cellulose solution, a precipitation immediately takes place from below which is intended to avoid adherence to the pouring base. In practice however it has been found that it is not so easy, particularly at a high speed of operation, to render the sprinkling of the pouring base with precipitant so small or so uniform, that the process always proceeds uniformly and normally. In particular, care must be taken to avoid the sprinkling producing a precipitation beneath the pouring funnel to such an extent that the nozzle of the funnel becomes choked.

Numerous lengthy and costly experiments have now led to the hereinafter described simple process which also employs a pouring base but does not exhibit any of the aforesaid disadvantages so that it constitutes an extremely simple process which can be easily carried into practical effect.

In describing the invention in detail, reference will be had to the accompanying drawing forming part of this application, in which—

Figure 1 illustrates a diagrammatic view showing how the web of material is formed and removed from the drum; and

Figure 2 illustrates a similar view showing the results when a belt is employed as a substitute for the drum.

The process employs the customary spreader device consisting of a rotating drum which dips into a coagulating bath. Near the highest point of the drum there is a slot-shaped spreader. The viscose passes through the spreader device *a* to the cylindrical surface of a large drum, where, by means of the spreader device, it is laid out in a thin flat layer. The drum revolves in the direction of the arrow, by reason of which the viscose layer is carried forward from *a* to *b*, during

which time its outer portion is exposed to the air. But at *b* it enters a precipitating bath, through which it passes as far as *c*. The coagulation occurs between points *b* and *c*, by the action of the precipitating bath, with the result that the viscose is hardened into a film or foil. At point *c*, the hardened film, still supported by the pouring base, leaves the precipitating bath and at *d* it is stripped from the supporting drum surface by means of the roller *r* and carried forward through the ensuing baths, as indicated in the drawing, for further treatment. From *d* to *a*, the drum surface remains empty and at *a* it receives a fresh covering of viscose; which, being constantly repeated, maintains the continuity of the process uninterrupted.

In place of the drum above described, there may be used as a pouring base an endless belt stretched over two smaller drums and functioning in exactly the same manner as described.

The outstanding difficulty in every process like the present one, in which a pouring base is employed, is to secure the removal of the foil or film complete and uninjured from the supporting base, so that the latter is not soiled by any adhering, still liquid, viscose or newly formed film. The pouring base, moreover, must not be given too heavy a sprinkling or an uneven sprinkling of the coagulating bath, because any one of these factors may be the cause of trouble at the spreader slot.

Other processes have attempted to rid the supporting base of adhering film by means of scrapers and to clean it thoroughly thereafter with brushes. The present application, however, solves the question in quite another way, a simple and effective one.

By careful study and observation of the process of coagulation it was found that in the course of coagulation a moment of time occurred during which the film can be readily and neatly removed from the pouring base. The situation in question may be regarded as arising in something like the following manner: the pouring base is first covered with the syrup-like viscose, a very sticky substance that glues itself to the base; then the bath solution penetrates and is diffused in the viscose; the salts in the bath solution cause the viscose to curdle or coagulate; simultaneously there occurs an adjustment of the uneven osmotic pressures in the viscose solution and in the salt solution, of the precipitating bath, respectively; finally the alkali viscose solution is neutralized by the acid in the bath solution to form H_2O. These reactions proceed simultaneously and are reciprocally affected by each other. They bring it about that the viscose solution coagulates just a moment before that in which the reciprocating solution becomes so completely diffused as to spread out to the supporting base; the congealing viscose upon the supporting base hardens into a thin skin, which, to start with, is completely dry and contains no trace whatever of any excess of the precipitating solution. Since, in addition, the skin, in forming, shrivels together, it has a tendency all its own to draw away from the supporting base.

A single moment after the juncture just described, the precipitating solution will have diffused itself through the thin layer of skin and will have spread upon the supporting base in an uneven and uncontrollable manner. But if the moment of coagulation above described is turned to account, the film can be continuously removed from the supporting drum and the drum can be kept dry all the while without any special care.

The crucial moment in question is, of course, an extremely brief one, yet practical tests have demonstrated that it is altogether feasible to grasp that moment in practical operation. If we assume the working speed of the machine to be about 25 meters per minute, the surface of the supporting drum and the film covering it will move at a rate of about 42 cm. per second. The space of one-fifth of a second will therefore be measured upon the supporting surface by a traversed distance of 8.5 cm. Therefore if the moment for a favorable removal of the film lasted for only one-fifth of a second, such removal can be effected within a span of 8.5 cm.; so that the process may be regarded as being projected upon the supporting base in much the same manner as a slow-motion picture upon the screen; and that momentary, crucial instant of time can positively be established, controlled and turned to account in practical operation.

The present invention, therefore, avails itself of the interval of time during which the film is in the condition above described, in order to strip the film neatly and unharmed from its supporting base and to leave the latter in so clean a state that it is prepared to receive a fresh supply of viscose from the spreader slot at once. This is effected by adjusting the speed of the pouring base and the temperature of the precipitating bath so that the brief interval of time during coagulation, to which reference has just been made, occurs while the film is passing from *c* to *a*, within which space the removal roller *r* is accordingly attached. If the conditions for coagulation are kept unchanged, the process keeps going uninterruptedly for hours, even for days and weeks, at a time, as has been demonstrated by practical experience.

Example

A solution of sodium-cellulose-xanthate containing 8% cellulose and 7% alkali and having a degree of ripening of 9, is uniformly spread upon the rotating drum above de-

scribed, which carries it forward to a coagulating bath, into which the lower half of the supporting drum dips. Let us assume the coagulating solution to be constituted by a water solution of 25% ammonium sulphate and 5% free sulphuric acid and the temperature of the bath to be maintained at 35° C. Now, if it takes, let us say, 25 seconds from the time of immersion for the cellulose solution to reach the crucial moment above described, then, by regulating the speed of rotation of the supporting drum so that a single complete revolution takes 40 to 44 seconds, the span from *b* to *c* is traversed in 20 to 22 seconds and the crucial moment will occur at a point slightly beyond *c*, at which point, therefore, the removal roller has to be placed.

The further treatment of the film so obtained from the coagulated viscose proceeds in familiar fashion, the film being carried over innumerable rollers in loops through various baths. In these baths the film is completely regenerated into cellulose, freed of all the salts, sulphur compounds and impurities it may contain, is bleached and washed, and, finally, with the addition, perhaps, of a softener, passes in the customary way over heated cylinders to be dried.

What we claim is:

1. A process for the production of foils, films, bands and the like from viscose and similar cellulose solutions by means of a pouring base dipping into a precipitating bath, which consists in removing the layer formed after coagulation, from the pouring base, at such time when the layer is already coagulated but when the coagulating bath has not yet diffused itself so far as to wet the supporting base, whereupon the foil or the like is passed unsupported through the ensuing baths for further treatment.

2. A process for the production of foils, films, bands and the like from viscose and similar cellulose solutions by means of a pouring base dipping in a precipitating bath, which consists in removing the layer formed after coagulation from the pouring base in the interval of time during which the base is completely dry in spite of the coagulation of the foil, and thereupon passing the foil or the like unsupported through the further baths, one or more or all of the factors determining the formation of foils being selected that the foil is completely coagulated at the moment of its removal from the base and that at the same time said base is dry.

3. A process for the continuous production of foils, films and the like from viscose and similar cellulose solutions by applying the cellulose solution through a suitable nozzle upon a rotating pouring base dipping into a precipitating bath, which consists in so regulating the rotation of the pouring base that the coagulation of the cellulose layer takes place upon a still perfectly dry pouring base in that section of the latter comprised within the angle of rotation between the level of the precipitating bath and the nozzle, and in removing within this space the formed cellulose layer from the pouring base.

4. A process for the production of foils, films, bands and the like from viscose and similar cellulose solutions by means of a pouring base dipping in a precipitating bath, which consists in removing the layer formed after coagulation from the pouring base in the interval of time during which the base is completely dry in spite of the coagulation of the foil, and thereupon passing the foil or the like unsupported through the further baths, fresh cellulose solution being applied to the drying base after the removal of the foil or the like without further preliminary cleaning whereby the process is rendered continuous.

5. A process for the production of foils, films and the like from viscose and similar cellulose solutions by applying the cellulose solution through a suitably shaped nozzle upon a rotating cylindrical drum partly dipping in a precipitating bath, which consists in fixing between the applying nozzle and the level of the precipitating bath the time interval during which after the coagulation of the formed cellulose layer the drum presents a completely dry part on its cylindrical surface, and in removing the formed cellulose layer during this time interval from the drum and thereupon passing it unsupported through the further baths.

6. A process for production of foils, films and the like from viscose and similar cellulose solutions by applying the cellulose solution through a suitably shaped nozzle upon a rotary cylindrical pouring drum partly dipping in a precipitating bath, which consists in choosing the factors determining the formation of the foil so that between the applying nozzle and the precipitating bath the pouring drum becomes completely dry after coagulation of the formed cellulose layer during a certain angle of rotation, during which the formed cellulose layer is removed from the pouring drum whereupon it is conducted without supporting means through the further bath.

In testimony whereof we affix our signatures.

DR. EMIL CZAPEK.
DIPL. ING. RICHARD WEINGAND.