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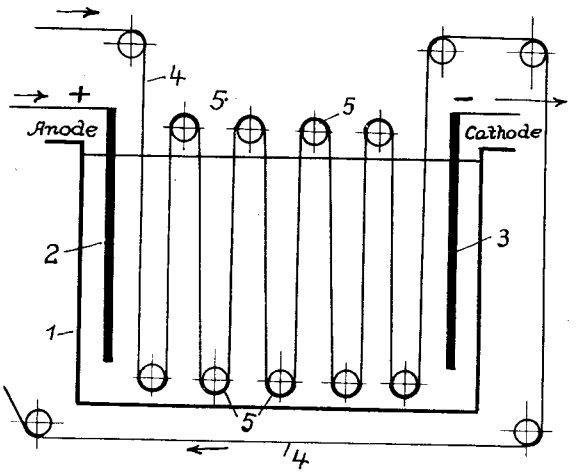



Fig. 1

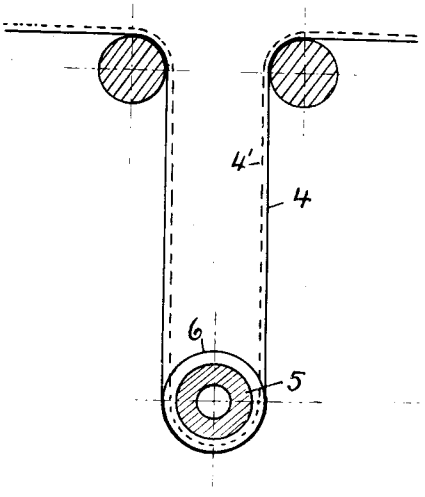



Fig. 2

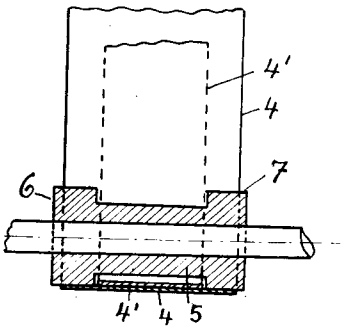



Fig. 3.

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# UNITED STATES PATENT OFFICE.

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## PROCESS FOR THE PRODUCTION OF FILMLIKE BANDS FROM CELLULOSE SOLUTIONS AND SIMILAR INITIAL MATERIALS.

Application filed January 15, 1926, Serial No. 81,557, and in Germany October 15, 1921.

This invention relates to the production of film-like bands from cellulosic or similar viscose substances, the cellulosic material being applied to a conveying band, which band with the cellulosic material thereon is passed through a precipitating bath, in which the material on the band is subjected to the precipitating action of an electric current, the electric current acting progressively on the material applied to the band during the travel of the band through the bath.

According to this invention the action of an electric current is employed for the production of insoluble film-like ribbons from cellulosic solutions, viscose or gelatine and the like, in a new and advantageous manner, being applied so that electrolysis and precipitation may take place in the alkaline, then in the neutral and finally in the acid portions of a bath situated between the electrodes, the film material as a cellulosic solution being first coagulated in a weakly alkaline portion of a salt solution; it then reaches the neutral portion of the bath and is thereafter submitted to the action of the acid portion of the bath. The alkaline, neutral and acid portions of the precipitating bath act upon the film on the carrier in progressive order and under the influence of the electric current, the precipitating bath serves as an electrolyte, exhibiting an alkaline reaction at the cathode or negative pole and an acid reaction at the anode or positive pole while a continuous graduation from the alkaline to the acid zone exists during the passage of the current between the two electrodes, so that the central portion of the bath becomes neutral. There is no need to fear any mixing of the precipitating bath if a suitable, not too rapid, speed of passage of the film is maintained.

Inasmuch as according to the invention, the film upon the conveying band is passed at the requisite speed, i. e., sufficiently slowly through the region of action of the electric current a very slow and exceedingly uniform coagulation and a particularly favourable hardening of the film takes place. A further advantage is that the alkaline and the acid treatment of the film are not effected in the usual manner in two separate baths but in a single precipitating bath. A precipitation on both sides is also attained because the bath liquor penetrates the pores of the conveying band and also because the conveying band is already, on application of the cellulose or viscose solution, moistened with the thin layer of precipitating agent, the action of which is increased and activated by the electric current. By virtue of the employment of a conveying band, tearing, rolling up of the film and shrinkages are avoided which disadvantages can easily occur during precipitation on both sides with the aid of small closely adjacent rollers for alternately guiding the film.

The present process can be applied to all cellulosic solutions which contain inorganic electrolytes such as sodium hydrates, sulphuric acid, zinc chloride, calcium rhodanite, cuprammonia and the like. A fabric impregnated with celluloid, "bakelite" or similar substance stable in the conditions of comparatively low alkalinity and acidity of the bath is preferably employed as conveying band. The conveying band and the film are preferably guided between the electrodes transversely to the direction of the current in several windings. Since highly viscous solutions such as viscose and the like are employed in the production of film-like bands, there is no fear of the solution running off the conveying band.

The effect of the direct electric current can be simultaneously employed in carrying out the invention, to bring the precipitating bath to the required temperature. For this purpose the current strength and the distance between the electrodes or the concentration of the bath are so selected that the electric current exercises a heating action which maintains the bath at the desired temperature

The employment of the action of the electrical current for the production of films according to the invention moreover offers the possibility of simultaneously incorporating a dye into the film by means of the current, in order to produce permanently colloured films. For example there may be added to the alkaline part of the bath an acid dye which is soluble in this part of the precipi-

tating bath and which has a tendency to move towards the anode. In doing so, it must pass through the film and is taken up by the latter reaching the acid part of the bath together with the film and is rendered insoluble.

All acid dyes such as for instance alkali-blue, green, dianil black may be employed for the aforesaid purpose.

In order more clearly to understand the invention reference is made to the accompanying drawings, Fig. 1 of which illustrates diagrammatically one embodiment of apparatus suitable for carrying same into practical effect.

Fig. 2 is a detail longitudinal section taken longitudinally through two of the upper guide rollers and through a single lower roller, and Fig. 3 is a detail view partly in section of the lower guide roller, with film carrying belt thereon.

In said figures, 1 indicates the container containing the usual precipitation bath containing sodium sulphate. Two electrodes 2 and 3 of metal, graphite, carbon or the like are arranged within said container 1 and are connected to a source of direct current. The conveying band 4 upon a part thereof which is beyond the container will have applied to its upper surface, by any suitable means, as a pourer, a cellulosic or viscose solution, the band with the coating 4′ thereon being carried through the bath by means of rollers 5 in several loops to form a long path for the applied film. In consequence of the difference in potential the precipitation bath exhibits an alkaline reaction at the inlet end, thus consisting at this point of a solvent mixture of sodium sulphate and sodium hydroxide. The conveying band with the partly coagulated viscose thereon passes into the central part of the precipitation bath which is composed of neutral sodium sulphate solution. Up to this point the coagulated viscose mass is still soluble in water. However in the subsequent portion of the bath which consists of a solvent mixture of sodium sulphate and sulphuric acid it becomes insoluble in water. At the end of the bath the viscose is completely precipitated throughout and is insoluble in water so that it can be removed from the conveying band and passed through the subsequent cold and hot washing baths.

A speed regulator can be arranged in the drive of the conveying band so as to permit the speed of the conveying band to be decreased if desired, in order to utilize all the bath and the electric current action correctly. If it is intended simultaneously to heat the bath by means of the electric current, the voltage applied to the electrodes 2 and 3 must be correspondingly increased or the distance between the electrodes or also the concentration of the bath must be altered until the precipitating bath reaches and is maintained at the desired temperature.

In order to prevent the film pressing on the lower guide roller where it lies between the roller and conveying band, it may be conducted here in a recess in the rollers whilst the conveying band is maintained on the rollers and at a certain distance of the film by lateral flanges. This method of guiding the conveying band is illustrated in Figs. 2 and 3, where the lower guide roller 5 is provided with end flanges 6 and 7 upon which the conveying band is placed whilst the film 4′ is situated in the recess in the roller between these flanges. The film 4′ is thus free of the pressure of the conveying band 4 which is taken up by the flanges 6 and 7 of the roller 5. This expedient thus also prevents excessive stressing of the film when lying on the inside beneath the guide rollers.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. The method of making film-like bands from cellulosic material which contains inorganic electrolytes, which consists in passing a carrier having a film of cellulosic material thereon, in passing such carrier into and out of a precipitating bath, directing a current of electricity through the bath, and actuating the carrier at such a speed that the alkaline, neutral and acid zones of the bath act upon the film on the carrier progressively.

2. The process of producing film-like bands from cellulosic solutions which contain inorganic electrolytes capable of yielding acid, neutral and alkaline zones when subjected to electrolysis, which consists in passing an electric current through a precipitating bath heated thereby and guiding a conveying band carrying a film between electrodes and through the bath in such a manner that different zones of the bath will act upon the film progressively.

3. In the process of producing a band from cellulosic viscose solutions containing inorganic electrolytes, which consists in passing through a precipitating bath a carrier having thereon a film of said viscose, the carrier and film being caused to travel between electrodes, the precipitating strength of the bath increasing in the direction of the travel of the carrier.

4. Means for producing film-like bands of viscose solutions comprising a single tank for a precipitating bath, electrodes located at opposite ends of the tank which contains the precipitating bath, a travelling carrier to only one side of which a film of viscose solution is applied, guides for the travelling carrier which maintain the major portion thereof in vertical and parallel alinement

with the electrodes to provide means whereby the film will be progressively acted upon during its travel through different zones of the bath, the zones having different degrees of precipitating powers.

5. The method of making film-like bands from cellulosic solutions which contain inorganic electrolytes, which consists in passing an electric current through a precipitating bath, passing a carrier having a film of cellulosic material on one of its surfaces between electrodes in said bath, moving said carrier at such speed that the alkaline, neutral and acid portions of the bath act upon the film progressively, said film of cellulosic material also being affected by a dye incorporated with the bath.

6. A step in the process of forming film-like bands from a viscose solution which consists in subjecting a film applied to a carrier to travel into and through a precipitating bath, the carrier being guided so that the carrier and film thereon will travel substantially parallel to an anode and cathode located at opposite ends of the bath.

In testimony whereof we affix our signatures.

DR. EMIL CZAPEK.
RICHARD WEINGAND.