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MANUFACTURE OF VISCOSE FILMS, ETC.

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The object of this invention is to provide a process of manufacturing, in a continuous manner, cellulose strips, films, and the like derived from aqueous solutions of sodium cellulose xanthate generally termed "viscose".

The expression "in a continuous manner", is intended to indicate that the operation is conducted in such a way that the liquid viscose, running uninterruptedly into a coagulating bath, is transformed without any interruption into a finished product: strips, films, etc., consisting of regenerated cellulose obtained from sodium cellulose xanthate.

In certain former processes of making film, the xanthate solution was subjected to the coagulating action of a bath composed of an aqueous solution of ammonium sulphate or of other salts endowed with coagulating properties. The film so obtained was then treated by means of various successive baths for the purpose, first, of purifying it by means of an aqueous solution of sodium chloride, then of causing the xanthate to be transformed into cellulose under the action of a mineral acid, and lastly of washing it first with cold and next with hot water.

The present invention consists of a new process for manufacturing in a continuous manner threads, filaments, strips, films, tubes, etc., starting from viscose. Its essential difference from the one hitherto used—mention of which is to be found above—lies in this; that after the sodium cellulose xanthate has been coagulated in the first bath, its transformation into cellulose by means of a mineral acid follows immediately in the next bath, and the regenerated cellulose is then subjected to the action of certain baths, as will be explained later, for the purpose of purifying, dyeing, glycerinizing, etc. The application is a division of my prior application, No. 646,478, filed June 19, 1923, which has been issued as Patent 1,548,864 dated Aug. 11, 1925.

In the following description, there will be discussed, for convenience, the application of the present process to the manufacture of films, which films are obtained by extruding viscose in the form of a sheet from a slotted hopper and allowing it to pass into the first coagulating bath; if it is desired to obtain threads, films, strips, tubes, etc., it would be necessary to use instead of the slotted hopper, a suitable device containing apertures

through which the viscose is extruded, which apertures are adapted to make the kind of product desired.

In manufacturing film, the viscose or solution of sodium cellulose xanthate, as it issues from the slotted extruding hopper, passes into a suitable coagulating bath. The film so formed then travels, in a continuous manner, through a series of vats wherein it is supported by suitable guide rollers, the successive vats containing baths adapted for subsequent operations; but there is no occasion to emphasize the mechanical features of the apparatus.

On issuing from the first or coagulating bath, the film passes through a converting bath which causes the cellulose xanthate to be converted to cellulose; next the film is subjected either to cold or to hot water washing and then to the action of a desulphuring bath; thereafter, the film is subjected to a second washing in either hot or cold water, at any rate hot at first, and finally is subjected to the action of a dyeing bath. After this last operation, the film is successively washed in running water, squeezed, put through a glycerine bath and ultimately dried either immediately or later.

Reference will again be made to each one of the various aforementioned operations, specifying the operating method and the characteristics for and of every one of them.

Coagulation.—Coagulation of the sodium cellulose xanthate is effected, as in former processes, by means of a bath containing, for instance, an aqueous solution of sulphate of ammonia; I can also utilize aqueous solutions containing either a mixture of sulphate of ammonia and an acid, or a mixture of sulphate of ammonia and a base, or bisulphate of soda or sulphate of soda with an acid added thereto, or magnesium sulphate or zinc sulphate; furthermore I may utilize any two normal sulphates in combination.

When I use an acid coagulating bath or a bath containing bisulphates, I find that in this coagulating bath the xanthate immediately becomes partly transformed into cellulose.

The composition (proportion of constituents) of the coagulating bath actually used is determined by a number of factors, viz: the temperature of this bath, the degree to which the viscose has matured or ripened, the composition of the viscose, the thickness

of the film desired and the duration of contact of the viscose with the coagulating solution—which is determined by length of travel in the bath and machine speed.

5 There are, for instance, certain working conditions which hold good for all the following explanations and to which the example given will refer.

I will assume that the working speed is 25 meters per minute, and that the film to be obtained is to weigh from 25 to 30 grams per square meter.

Under these conditions, and utilizing in the first bath as a coagulating solution a mixture of sodium sulphate and sulphuric acid, the best results are obtained with the following proportions, viz: 10 grams of sodium sulphate and 10 grams of sulphuric acid per 100 cubic centimeters, the film remaining in the coagulating bath for about 35 seconds.

Transforming bath.—This bath, which is designed to effect the transformation of the xanthate into cellulose and which is utilized immediately after coagulation and previous to all other operations, consists of an aqueous solution of a mineral acid such as, for instance, hydrochloric acid, sulphuric acid etc.

Under the aforesaid conditions (speed 25 meters per minute; film weight 25 to 30 grams per square meter) good results are obtained with a bath containing about 4½ grams of sulphuric acid in 100 cubic centimeters of water, with a contact-duration of about 35 seconds.

Washing.—On issuing from the transforming bath, the film is subjected to a cold or hot water washing to remove acid and salts. The film after acid treatment is opaque and white. If the washing is thorough, the film becomes somewhat transparent, but it still contains a certain amount of sulphur which makes it somewhat opaque. This sulphur may be removed, however, by means of the desulphuring bath.

Desulphuring.—The desulphuring bath consists either of a hot aqueous solution of sodium sulphide, the temperature of which may be as high as 100° C., or of a solution of caustic soda or of a solution of both or of any other body or substance endowed with desulphuring properties.

The composition of this bath depends on the same factors as those already mentioned. There may be used, for instance, under the aforesaid conditions, a solution of about 0.4 gram or 0.5 gram of caustic soda for about 100 cubic centimeters of water, and the film may be allowed 15 seconds contact with the bath.

Washing.—After desulphuration, the film is subjected to a washing with water in two vats. This washing is preferably effected hot, at any rate in the first vat, and at as high temperature as possible, even up to

100° C. The desulphuring agent, which is carried over with the film, is in fact of an alkaline nature and therefore very difficult to remove with cold water. The hot washing may, if desired, be followed by a cold water washing.

Dyeing.—The film is next subjected to a dyeing treatment, which may be carried out by passing it through a vat or vats. The dyeing baths should have the same composition as those used for dyeing cotton, artificial silk or any other cellulosic fiber, in so-called continuous dyeing apparatus, the colors usually used for this purpose being the so-called "direct" colors. Dyeing may also be effected at the same time as desulphuration by adding to the desulphuring bath coloring materials such as the so-called "sulphur" colors. It is also to be clearly understood that viscose may be used which has been colored previous to coagulation either by means of sulphur colors or of any other coloring materials that will dissolve in viscose without decomposing.

Washing.—After being dyed, the film is subjected to a washing in rapidly running water. This washing may be effected hot or cold, or even in two different baths, one hot and the other cold, with about 15 seconds stay in each bath.

Glycerine bath.—After washing, the film is subjected to a squeezing action between two smooth presser rollers, and is then treated in a glycerine bath. The purpose of this treatment is to impart the required pliancy to the cellulose film.

With the assumed speed of 25 meters per minute, the film has a length of time of immersion in the glycerine bath of about eight seconds, and the concentration of the glycerine bath is determined by the use for which the film is intended. This concentration is greater when it is intended to make a more pliable film, and less when it is intended to make a less pliable film, but ordinarily the concentration is maintained so that the specific gravity of the glycerine solution is between 1.009 and 1.012.

After this treatment, the film may be squeezed and then placed in a suitable drying apparatus to dry.

It is to be understood that the process applies equally to the case where inert substances such as barium sulphate, kaolin etc., have been introduced into the viscose.

I claim as my invention:

1. A process for the continuous manufacture of cellulose films and the like, comprising the steps of coagulating an aqueous solution of sodium cellulose xanthate; passing the coagulated product through a transforming bath to convert it into cellulose; washing the transformed product and passing it through a solution containing about 0.5 gram of caustic soda per 100 cubic centi-

meters of water to desulphur it; and washing, dyeing, washing, glycerinating and drying said product.

2. A process for the continuous manufacture of cellulose films and the like, comprising the steps of coagulating an aqueous solution of sodium cellulose xanthate by introducing it into an aqueous mixture of sodium sulphate and sulphuric acid, such mixture having a content of 10 grams of sodium sulphate and 10 grams of sulphuric acid per 100 cubic centimeters of the mixture; maintaining the xanthate in motion in said mixture for about 35 seconds; transforming the coagulated product into cellulose; and desulphuring and dyeing the transformed product; and then glycerinating and drying the same.

3. A process for the continuous manufacture of cellulose films and the like, comprising the steps of coagulating an aqueous solution of sodium cellulose xanthate; passing the coagulated product through an aqueous mineral acid solution to transform it into cellulose; washing the transformed product with a solution containing about 0.5 gram of caustic soda per 100 cubic centimeters of water to desulphur the same and dyeing it; and then, glycerinating and drying the product.

4. A process for the continuous manufacture of cellulose films and the like, comprising the steps of coagulating an aqueous

solution of sodium cellulose xanthate; passing the coagulated product through an aqueous mineral acid solution to transform it into cellulose; washing the transformed product with an aqueous solution of caustic soda containing about 0.5 gram of caustic soda per 100 cubic centimeters of water to desulphur it, and maintaining said product in such solution for about 15 seconds; dyeing the product; and then glycerinating and drying said product.

5. A process for the continuous manufacture of cellulose films and the like, comprising the steps of coagulating a solution of sodium cellulose xanthate; transforming the coagulated product into cellulose; and then washing the transformed product with an aqueous solution of caustic soda containing about 0.5 gram of caustic soda per 100 cubic centimeters of water to desulphur it.

6. A process for the continuous manufacture of cellulose films and the like, comprising the steps of coagulating a solution of sodium cellulose xanthate; transforming the coagulated product into cellulose; washing the transformed product with an aqueous solution of caustic soda containing about 0.5 gram of caustic soda per 100 cubic centimeters of water to desulphur it; dyeing the product; and then glycerinating and drying said product.

In testimony whereof I affix my signature.
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