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TREATMENT OF CELLULOSE SOLUTIONS

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My invention relates to the treatment of cellulosic materials and particularly to processes for the coagulation and purification of dissolved cellulose to form a sheet of precipitated homogeneous cellulose.

An object of my invention is to provide a series of chemical treatments for the precipitation of cellulose from solution, and for the purification thereof.

Another object of my invention is to provide a process for producing a sheet of cellulosic material of superior uniformity of thickness, purity, transparency and strength.

In the prior art of the manufacture of transparent sheets of cellulosic material it has been customary to dissolve the natural cellulose fibres in a mixture of caustic and carbon disulphide solutions according to the well-known viscose process, to reprecipitate the material in a sheet form and then coagulate and purify it. Difficulty has been experienced in obtaining a sheet of uniform thickness, satisfactory transparency, satisfactory strength and adequate freedom from impurities, solvents, and re-agents.

My invention provides an improved series of treatments for the precipitation, coagulation, and purification, of cellulose from the dissolved state.

Other objects and structural details of my invention will be apparent from the following description.

In the operation of my process the cellulose fibres are dissolved in the caustic and carbon disulphide solutions in the well-known fashion. The prepared solution is then applied to a traveling belt according to the disclosure of my copending application, Serial No. 137,462, filed September 24th, 1926. As shown in that disclosure, the cellulose solution after coating upon the traveling belt is given a primary coagulating treatment on the belt, which is the first step in the process of my invention. This treatment consists of the application of a saturated solution of sodium acid sulphate containing about 5% of animal glue or similar colloidal strengthening substance. The application of this solution causes a coagulation of the cellulose upon the belt and produces a sheet having a sufficient degree of physical strength to permit it to be stripped from the belt. After removal from the coating belt the sheet is attached to a cross-bar member as described in my copending application, Serial No. 137,464, filed September 24th, 1926, and drawn directly into a vat containing a solution for producing a complete coagulation of the sheet, for the second step in my process.

The treatment of the second step consists in immersion for complete coagulation, in a solution of sodium acid sulphate and sodium lactate to which has been added a very small proportion of nitrate of iron. The sodium lactate is mentioned specifically as a constituent of the bath but it is desired that this be understood as being representative of any of the typical acids of the fatty acid series. Likewise, while iron nitrate is mentioned, other iron compounds are also usable. The sodium acid sulphate aids in the complete coagulation of the cellulosic sheet, and the coagulating effect is further aided by the sodium lactate which produces a greater strength in the finished sheet. The iron has a marked affinity for sulphur compounds, and it aids materially in expediting the removal of the sulphur compounds from the coagulum and in the production of a finished sheet of greater strength and greater transparency.

The sheet is then drawn through a second vat, the third step in the process, containing a hot solution of Glauber's-salt (or neutral sodium sulphate). This solution continues the purification of the coagulated cellulose by the removal of reaction products from the preceding step.

The sheet is then drawn into a third vat, containing a very weak solution of acetic acid, as the fourth step in the process.

From the vat containing the acetic acid solution the sheet is then drawn to another vat containing a solution of common salt (or sodium chloride) which continues the purifying operation, for the fifth step in the process.

The sheet is then desirably continued through a vat of cold water to remove fur-

ther portions of the re-agents and re-action products of the preceding steps, as the sixth step.

From the cold water, the sheet is desirably drawn through a solution of trisodium phosphate for the further removal of sulphur compounds and reaction products, as the seventh step of the process.

Following the trisodium phosphate treatment the sheet is desirably given a second treatment in a solution of common salt (or sodium chloride), for the eighth step in the process.

I have found that additional gains of strength in the finished sheet and in the transparency thereof may be obtained by passing the sheet from the common salt solution into a solution of a soluble oil, such as sulphonated oil, or I may use olive oil, or paraffin oil, or a common soap solution. This bath aids in the removal of the sulphur compounds and is desirable, but is not an essential step in the process.

From the oil or soap bath the sheet is desirably washed in hot water to remove traces of the oil, and then in cold running water, for the further removal of traces of oil, or soap, for the tenth, and eleventh steps in the process.

It is desirable that the sheet be bleached in the course of the processing and it has been found convenient to make the bleaching operation the twelfth step in the process. It is done by drawing the sheet through a vat containing an oxalic acid solution. After bleaching, the sheet is washed in hot water in another vat for the thirteenth step, and the bleaching may then be completed by a second treatment in oxalic acid solution for the fourteenth step. The second bleaching is followed by another wash in a vat of cold water for the fifteenth step, and a treatment in a vat of glycerine solution for the sixteenth step.

The sheet may then be dusted with French chalk and rubbed off as a final operation, the seventeenth step, after which the wet sheet may be reeled and sent to the drier, or may be led to the drier without reeling.

By this process of my invention I am enabled to coagulate a solution of cellulose into a sheet form, and by further treatment to produce therefrom a strengthened, pure, and transparent sheet of homogeneous cellulosic material.

While I have described but a single embodiment of my invention it is capable of numerous modifications therefrom without departure of the spirit thereof, and it is desired therefore that only such limitations shall be imposed thereon as are required by the prior art or indicated by the appended claims.

I claim as my invention:

1. The step in a process of the treatment of cellulosic materials which comprises interaction between a solution of cellulose and another solution containing both a sodium acid salt and glue.

2. The step in a process of the treatment of cellulosic materials which comprises interaction between a solution of cellulose and another solution containing both sodium acid sulphate and glue.

3. The step in a process of the treatment of cellulosic materials which consists in the complete coagulation of a solution of cellulose by a solution containing both a mineral acid salt and a salt of a fatty acid.

4. The step in a process of the treatment of cellulosic materials which consists in the complete coagulation of a solution of cellulose by a solution containing both sodium acid sulphate and sodium lactate.

5. The step in a process of the treatment of cellulosic materials which consists in the complete coagulation of a solution of cellulose by a solution containing both sodium acid sulphate and sodium lactate.

6. The step in a process of the treatment of cellulosic materials comprising a treatment of a cellulosic material by a solution containing an acid salt, a normal salt of a fatty acid and an iron salt.

7. The step in a process of the treatment of cellulosic materials comprising a treatment of a cellulosic material by a solution containing sodium acid sulphate, sodium lactate and iron nitrate.

8. The sequence of steps in the treatment of cellulosic materials which consists of partial coagulation of a solution of cellulose by treatment with a solution containing an acid salt and a colloid, followed by complete coagulation thereof by a solution containing an acid salt and a neutral salt of a fatty acid.

9. The sequence of steps in the treatment of cellulosic materials which comprises partial coagulation of a solution of cellulose by treatment with a solution containing an acid salt and a colloid, followed by complete coagulation by a solution containing an acid salt, a neutral salt of a fatty acid and an iron salt.

10. The sequence of steps in the treatment of cellulosic materials which comprises partial coagulation of a solution of cellulose by treatment with a solution containing an acid salt and a colloid, followed by complete coagulation by a solution containing an acid salt, a neutral salt of a fatty acid, and a further treatment with a neutral salt.

11. The sequence of steps in the treatment of cellulosic materials which comprises partial coagulation of a solution of cellulose by treatment with a solution containing an acid salt and a colloid, followed by complete coagulation by a solution containing an acid salt, and a neutral salt of a fatty acid, and further treatment with a saturated solution of normal sodium sulphate.

12. The sequence of steps in the treatment of cellulosic materials which consists of interaction between a solution of cellulose and a solution containing an acid salt and a colloid, followed by complete coagulation in a solution containing an acid salt and a salt of a fatty acid, and the further step of treatment in a solution of a phosphate salt.

13. The sequence of steps in the treatment of cellulosic materials consisting of a preliminary precipitation of cellulose from solution as a film, the complete precipitation thereof, the removal of the precipitants, a treatment with a phosphate salt, and the treatment with a soluble oil.

14. The sequence of steps in the treatment of cellulosic materials consisting of a preliminary precipitation of cellulose from solution as a film, the complete precipitation thereof, the removal of the precipitants, a treatment with a phosphate salt, and the treatment with a soluble oil and a bleaching.

15. The steps in a treatment of cellulosic materials comprising precipitation, a purification by means of a phosphate, a further purification by a soluble oil treatment, and a cooperating bleaching by oxalic acid solution.

16. The steps in a treatment of cellulosic materials comprising precipitation, a purification by means of a phosphate, a further purification by a soluble oil treatment, and a cooperating bleaching by oxalic acid solution, and a treatment with glycerine solution.

17. The steps in the treatment of homogeneous cellulosic films which comprise dusting with chalk and rubbing off.

18. The steps in the treatment of homogeneous cellulosic films which comprise dusting the wet film with chalk and rubbing off.

19. The steps in the treatment of cellulosic materials which consist of solution of the raw cellulose in a mixture of carbon disulphide and caustic solution, coating upon a support, partially coagulating the coating, stripping the partially coagulated film from the coating support, completely coagulating the film, simultaneously removing sulphur compounds, removing a portion of the coagulatants, treating with weak acetic acid solution, treating with common salt solution, washing in cold water, treating with a phosphate solution, treating again with common salt solution, treating with a soluble oil, washing in warm water, washing in running cold water, bleaching by oxalic acid solution, washing in hot water, rebleaching in another oxalic acid solution, washing in cold water, treating in a glycerine bath, dusting with chalk, rubbing off excess chalk, and drying.

20. The step in a process for the treatment of cellulosic material which comprises interaction between a solution of cellulose and another solution containing both an acid salt and a glue.

21. The step in a process for the treatment of cellulosic material which comprises interaction between a solution of cellulose and another solution containing both an acid salt and an animal glue.

22. The step in a process for the treatment of cellulosic materials which consists in the treatment thereof with a neutral solution of a soluble oil.

23. The steps in the treatment of cellulosic material which consists of a preliminary coagulation, a final coagulation, a preliminary purification, and a second purification step consisting of the application of a neutral solution of a soluble oil.

24. The process of removing sulphur compounds from precipitated cellulose which consists in the combination therewith of a neutral solution of a soluble oil, and the removal by solution in water.

In witness whereof, I hereunto subscribe my signature.

CHARLES E. HETZEL.