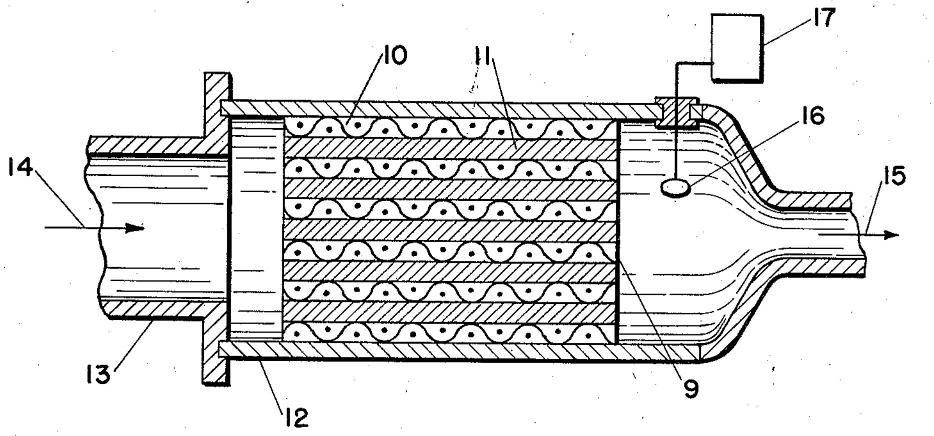
Oct. 19, 1954 J. BURNHAM 2,692,210

PROCESS OF PURIFYING AND IMPREGNATING CELLULOSIC SPACERS FOR ELECTRICAL CONDENSERS

Filed Dec. 10, 1949



JOHN BURNHAM,
INVENTOR.

BY Arthur G. Connolly
his attorney

Patented Oct. 19, 1954 **2,692,210**

UNITED STATES PATENT OFFICE

2,692,210

PROCESS OF PURIFYING AND IMPREGNATING CELLULOSIC SPACERS FOR ELECTRICAL CONDENSERS

John Burnham, Williamstown, Mass., assignor to Sprague Electric Company, North Adams, Mass., a corporation of Massachusetts

Application December 10, 1949, Serial No. 132,345

6 Claims. (Cl. 117—48)

1

My present invention relates to the purification of cellulosic spacer materials. More particularly, it concerns a method of purifying regenerated cellulose films to make them specially adapted for use in the fabrication of electrolytic capacitors.

The use of a regenerated cellulose film as a spacer material for electrolytic condensers has been repeatedly suggested in the patent literature. Unfortunately, however, successful application of the regenerated cellulose film to commercial manufacturing operations, particularly in the production of high capacity electrolytic condensers, has not been possible. This situation results from the contamination of the regenerated cellulose during its manufacture with chloride ions, sulphate ions and other ionic impurities. Such impurities in the film may lead to corrosion and failure of electrolytic capacitor structures in which the films are incorporated. In the wet state following extrusion, regenerated cellulose is extremely fragile and may be subjected only with great difficulty to thorough washing, in order to remove such ionic impurities. It has been suggested that dialysis may be employed to remove impurities of various kinds. While this process is to some extent successful, it requires extremely expensive and massive processing equipment for efficiency in operation.

It is an object of the present invention to overcome the foregoing and related disadvantages of the regenerated cellulose films. A further object is to produce improved cellulosic spacer materials for electrical devices. A still further object is to provide a simple and inexpensive process for producing regenerated cellulose film substantially free from ionic impurities. Additional objects will become apparent from the following description and claims.

These objects are attained in accordance with the present invention by employing a purification process that comprises convolutely winding a sheet of regenerated cellulose spacing material with a contacting sheet of porous spacing material into a cylindrical roll, forcing a solvent for ionic impurities through said roll in an axial direction, unwinding said roll and drying the resulting purified, regenerated cellulose film. In accordance with one of the limited embodiments of the invention there is employed a process which comprises convolutely winding a thin, regenerated cellulose film with a contacting porous paper into a cylindrical roll, forcing boiling water through said roll in an axial direction until the effluent water possesses a chloride concentration

2

below 25 parts per million by weight, forcing an electrolytic condenser electrolyte through said roll in the same axial direction, and unwinding said regenerated cellulose film from said roll. According to another limited embodiment of the invention there is employed a process which comprises convolutely winding at least two regenerated cellulose films with at least two aluminum electrodes and at least two porous paper spacers into a cylindrical roll, whereby each regenerated cellulose film is separated from the other said films by a porous paper spacer and an aluminum electrode, forcing boiling water through said roll in an axial direction until the effluent liquid has a chloride ion content substantially less than 25 parts per million and forcing an electrolytic solution in the same axial direction through said roll until said roll is saturated with said electrolyte.

According to my invention, I have devised a novel and simple process for removing chloride, sulphate and related ionic impurities from regenerated cellulose films without encountering the cost and equipment difficulties heretofore met in the purification process. According to one embodiment of the invention boiling water or some other solvent in which the ionic impurities are soluble is forced axially through a convolute roll consisting of the regenerated cellulose film and of a contacting porous spacing material, such as paper, cloth, glass, mat, etc. The axial washing process is conducted only for the length of time necessary to reduce the chloride content of the effluent solvent to the maximum desired, e. g. about 25 parts per million in the case of regenerated cellulose films for use in electrolytic condensers as spacer materials.

According to one of the limited embodiments of the invention the washing process may be followed by a treatment with a liquid which will be retained and/or absorbed in the regenerated cellulose film to act as a plasticizer and/or electrolyte. For example, a solution of ammonium borate in glycol may be passed through the washed roll of regenerated cellulose film and porous paper. Thereafter, the roll may be unwound and optionally the regenerated cellulose film may be dried prior to incorporation in capacitor assemblies.

According to one of the preferred embodiments of my invention the wash roll consists of an electrolytic capacitor assembly in which the spacing material separating the two aluminum electrodes comprises at least one layer of regenerated cellulose and at least one contacting layer of a porous paper material, such as linen paper or kraft capacitor paper. If porous aluminum electrodes, such as sprayed aluminum on gauze electrodes, are used, it is sometimes possible to eliminate the porous paper spacer. By this process it is possible to wash the cellulose spacer to a safe chloride content and subsequently to impregnate the wound capacitor with the operating electrolyte. Normally the aluminum electrodes are formed with an oxide film prior to incorporation in the wash roll.

The regenerated cellulose film obtained in accordance with my invention is useful not only as a spacing material for electrolytic capacitors, but also as a dielectric spacer for electrostatic (non-electrolytic) condensers. In such instances, the regenerated cellulose has a much higher insulation resistance and results in capacitors with lower leakage currents than corresponding capacitors made with conventional, electrical grade, regenerated cellulose films.

The regenerated cellulose employed in accordance with the invention may be made in the usual manner without special processing equipment or control. Other polar resinous films are contemplated for use in accordance with the present teachings, provided that appropriate wash solvents, wash temperatures and rates are employed.

When the basic washing process of the invention is to be practiced, it is generally desirable to wind up the contacting sheet of porous spacing material as part of the finishing operation in the manufacture of the regenerated cellulose film. The wide rolls e. g. using 56'' paper sheets may be subjected to the treating process without further handling. If a cloth or fabric spacer is used, it may, of course, be reused until failure occurs, for example, tearing, fraying, etc.

In addition to water, mixtures of water and polyhydroxy compounds, such as glycol and glycerine, may be employed as solvents. It is apparent that numerous other solvents may be employed dependent upon the ionic impurities to be removed, type of film to be processed, etc. The rate of solvent flow across the regenerated cellulose surface, the diffusion rate of ions from from the inner parts of the film, the temperature of the solvent and film are determining factors in the processing control. While I am not fully aware of the exact phenomenon involved, I believe that the primary action is a diffusion controlled process and, therefore, depends upon the diffusion rate for chloride or other ions which may be present within the regenerated cellulose film.

Reference may be made to the appended drawing which illustrates a simplified cross section of a typical washing apparatus suitable for carrying out the process of my invention. Convolutely wound roll 9 consists of a porous spacer sheet 10 and a regenerated cellulose film 11. This roll is fitted into the container 12. Cap 13 is attached to a wash water supply. The wash water 14 flows axially through the section 9 in the direction of the arrows, forming an effluent 15 which is exhausted. A conductivity cell 16 is connected to bridge 17, as a process control for the amount of ionic impurities in the effluent 15. It is generally preferable to force wash water 14 through section 9 by means of simple hydrostatic pressure. However, it is also possible to pull the wash water through the section by means of a vacuum pump.

In most instances involving large rolls of appreciable width it is desirable periodically to reverse the axial direction of the wash solution. In this way the diffusion is equalized at each end of the section.

As a specific example of the practice of my invention, 0.0005'' cellophane of 12'' width was convolutely wound with 0.003'' porous linen paper on a 1'' core to a total diameter of 5''. The core was stoppered and the wound roll placed in a column. Boiling tap water was passed through the wound roll in an axial direction at a rate of 2 liters per minute for 30 minutes. Following this, a liter of boiling distilled water was passed through the section in the same axial direction. Following the distilled water rinse, 500 cc. of a solution of 25% ethylene glycol in water was passed through the roll. The regenerated cellulose film was unwound and dried at 70° C. Prior to the washing operation the regenerated cellulose had a chloride ion concentration of 250 parts per million. After the washing process the chloride ion content was only 15 parts per million. The glycol was added as a plasticizer and electrolyte solvent for the film.

It should be noted that the final treatment with an aqueous glycol solution described in the foregoing specific example is ordinarily omitted, when electrostatic spacing material is being produced.

For some purposes the contacting sheet of porous spacing material may be composed of metallic cloth or screen. The material employed for this purpose is not critical, provided that it is porous in nature and chemically inert, i. e., does not impart ionic impurities to the regenerated cellulose film.

My new purification process requires a minimum quantity of solvent such as distilled water. It provides the user of the highly purified, regenerated cellulose film with a simple and inexpensive means for producing such film from the commercially available regenerated cellulose films containing an excessive amount of ionic impurities. If the manufacturer of the regenerated cellulose film should try to remove such ionic impurities, this would require several additional washings, a much greater amount of floor space and a great quantity of distilled water. This would greatly increase the cost of manufacture, and the relatively small demand for the highly purified, regenerated cellulose film would hardly justify the heavy investment for the additional equipment that would be required to include such purification in the regular production of the film.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope hereof, it is to be understood that the invention is not limited to the specific embodiments hereof except as defined in the appended claims.

I claim:

1. A purification process which comprises convolutely winding a sheet of regenerated cellulose having soluble impurities with a contacting sheet of porous spacing material into a cylindrical roll, and forcing a solvent for the impurities through said roll in an axial direction to cause the impurities to diffuse out of the regenerated cellulose sheet and into the solvent.

2. A purification process which comprises convolutely winding a thin, incompletely washed regenerated cellulose film with a contacting porous paper into a cylindrical roll, forcing boil-

ing water through said roll in an axial direction until the effluent water possesses a chloride concentration below 25 parts per million by weight, forcing an electrolytic condenser electrolyte through said roll in the same axial direction to saturate the resulting film with said electrolyte, and unwinding said regenerated cellulose film from said roll.

3. A purification process which comprises convolutely winding at least two incompletely washed regenerated cellulose films with at least two electrode foils and at least two porous paper spacers into a cylindrical roll having each regenerated cellulose film separated from the other of said films by a porous paper spacer and an electrode foil, forcing boiling water through through said roll in an axial direction until the effluent liquid has a chloride ion content substantially less than 25 parts per million and forcing an electrolytic capacitor electrolyte in the same axial direction through said roll until said roll is saturated with said electrolyte.

4. A process which comprises convolutely winding a sheet of regenerated cellulose having soluble impurities with a contacting sheet of porous spacing material into a cylindrical roll and forcing an aqueous aliphatic polyhydroxy solvent for the impurities through said roll in an axial direction to cause the impurities to diffuse out of the regenerated cellulose sheet and into the solvent.

5. The process of claim 4 wherein the polyhydroxy compound is glycol.

6. The process of claim 4 wherein the polyhydroxy compound is glycerine.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
643,234	Post	Feb. 13, 1900
719,973	Wotton	Feb. 3, 1903
1,315,763	Dickey	Sept. 9, 1919
1,807,375	Boyer	May 26, 1931
1,932,789	Kline	Oct. 31, 1933
1,981,352	Fruth	Nov. 20, 1934
2,095,611	McNutt	Oct. 12, 1937
2,158,981	Collins	May 16, 1939
2,199,447	Ruben	May 7, 1940
2,253,507	Clark	Aug. 26, 1941
2,290,163	Brennan	July 21, 1942
2,460,282	Gardner	Feb. 1, 1949

FOREIGN PATENTS

Number	Country	Date
670,853	France	Dec. 5, 1929