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PRESERVATIVE

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6 Claims. (Cl. 167—31)

This invention relates to regenerated cellulose. More particularly the invention relates to regenerated cellulose pellicles and in particular to regenerated cellulose pellicles in the form of caps and bands. The invention will be described in its application to caps and bands but this description is not limitative of the invention.

Caps and bands are made from a viscose solution by, for example, coating blanks of the desired shape with the viscose solution, or by extrusion of the viscose solution through suitable orifices, and by regenerating, purifying, and otherwise treating the pellicles thus formed. Wet or "gel" regenerated cellulose shrinks when it is dried. This property has been taken advantage of in the caps and bands industry, wherein gel or wet regenerated cellulose caps and bands are applied, for instance, over the cork and neck of a bottle. As the caps dry they shrink and grip the neck of the bottle, forming an attractive, tightly-stretched seal.

The prior art has attempted to use these caps and bands by drying the cap at the factory before shipping to the customer. This method is unsatisfactory in that the user is put to expense and trouble to swell and soften the cap, and in that regenerated cellulose, once dried, is inferior in this use to undried regenerated cellulose and cannot be restored to its original condition.

Another method which has been tried consists in packing the caps in an aqueous solution of formaldehyde. Here the advantage of an undried cap is obtained, but the method has the disadvantages that it involves trouble and expense in washing out the formaldehyde, that the caps must be glycerinated or otherwise softened before use, and that the work people who are in contact with the formaldehyde solution are often affected with severe dermatitis.

The tendency of industry has been, consequently, to ship the caps to the consumer submerged in water or impregnated with an excess of the softener. These methods have the advantage that the cap is better than a cap which has been dried, but have the disadvantages that the purchaser must adjust the softener content to obtain that percentage of softener whose presence produces the cap best suited to the user's purpose. Where considerable moisture is present, furthermore, the cellulose hydrate is subject to attack by a fungus.

An object of this invention is to provide a method of preparing caps or bands of regenerated cellulose which will provide for the preservation and correct conditioning of such caps or bands. Another object is to provide simultaneous preservation and conditioning. Another object is to provide a method of packing which will permit the consumer of the caps and bands to use them directly without any intermediate washing or impregnation and to store them indefinitely without deterioration. Another object is a medium for preserving or shipping regenerated cellulose which is inexpensive, efficient, and non-toxic. Other objects of the invention will be in part apparent, and in part set forth elsewhere herein.

The objects of the invention are attained, generally speaking, by immersing the wet or "gel" regenerated cellulose pellicles, whether in cap or other form, in an aqueous solution containing a preserving agent and a softening agent.

In carrying out the invention regenerated cellulose caps or other pellicles are formed after any known method but are not dried. These pellicles, containing approximately 80% by weight of water, are packed in waterproof containers. The spaces between the caps thus packed are then filled by adding enough preservative solution to submerge all the caps. The preserving solution contains water, a softener for cellulose hydrate, and a non-toxic preserving agent. The preserving agent is present in a quantity which will suffice to prevent the growth of mold and to preserve the regenerated cellulose from deterioration in other ways. A softening agent is present in such quantity that, after it has diffused throughout the wet caps or pellicles, it will reach equilibrium at a concentration equal to the optimum for correct functioning of the pellicle.

As a softening agent I have employed any of the known softeners for regenerated cellulose. These include, among others, glycol, diethylene glycol, trimethylene glycol, erythritol, pentaerythritol, diglycerine, and glycerine. In general, glycerine is to be preferred because of its abundance, cheapness, efficiency and harmlessness.

As preservatives I may use chlorinated cresol, which is preferred: alkaline earth salts of fluorides or silicofluorides, such as sodium silicofluoride; hexyl resorcinol, either as such or in the form of its sodium salts; and salicyl anilide. Acetic acid has some preservative qualities but is inferior in many ways to the substances named above. Of the chlorinated cresols, all of which are useful, I prefer a mono-chlor cresol, which can be obtained by direct chlorination, having physical characteristics somewhat as follows: melting point around 60° C., chlorine content around 24–25%, and which need not be a pure product.

The following solution has been used with suc-

cess. The proportions and materials used in this example are illustrative of the invention, not limitative.

Example

	Parts
Glycerine	15.0
Chlorinated cresol	0.3
Water	84.7

When this formula is used, the final package contains about 5% of glycerine and about .1% chlorinated cresol. About 12 pounds of the preservative solution is satisfactory when used with about 30 pounds of wet pellicles, but almost any other ratio may be used so long as satisfactory immersion is obtained.

Any desired or preferred concentration of softener and preservative may be used. In general, the concentrations of softener and preservative are adjusted to approximately three times that which will result when the solution is poured over the wet caps or bands in order to allow for the water which is included in the pellicles themselves. This ratio of concentrations (1:3) is optional, being dependent to a large extent on the amount of water present in the caps and to a certain extent on the solubility of the softener and/or preservative. A concentration of softener to other ingredients of the solution should be chosen which will insure the presence in the wet or dry cap of enough softener to keep the cap soft without an excess which would unduly retard or even prevent drying.

An advantage of my invention is the preparation of regenerated cellulose pellicles in a condition which enables the consumer to use them without processing. Another advantage is that the preservatives do not alter the physical or chemical structure of the cellulose pellicles. Another advantage is that the preservatives are satisfactorily soluble in water and/or aqueous solutions containing softeners such as glycerine and the others herein named. Another advantage is that the preservatives are inactive toward the dye-stuffs which may be used to color the cellulose, and that the preservatives are themselves colorless, odorless, non-corrosive toward the container for the pellicles, non-toxic, mold-preventive, and of distinct germicidal value. Another advantage of the preservatives is that they can be used in extremely low concentrations, and leave no appreciable residue when the pellicle dries. Other advantages of the invention will be apparent to those skilled in the art. It will be noted from the above objects and description of the invention, that the broad scope of the invention includes a shipping solution for gel cellulosic pellicles containing a preservative and a softener. While, as has been stated, the preferred form of the invention embodies the use of preservatives which are nontoxic and otherwise harmless in themselves, it is obvious that the broad principles of the invention apply to the use of preservatives which are nontoxic and otherwise harmless under the conditions of use, e. g., in small concentrations, although great concentrations, for example, might be definitely harmful.

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

I claim:

1. The method of preserving gel regenerated cellulose pellicles which comprises packing the gel pellicle in an aqueous solution of chlorinated cresol and glycerine.

2. The method of preserving a pellicle of gel regenerated cellulose which comprises immersing the cap in an aqueous solution of chlorinated cresol and glycerine.

3. A gel regenerated celullose pellicle impregnate with chlorinated cresol and glycerine.

4. A gel regenerated cellulose pellicle impregnate with an aqueous composition containing chlorinated cresol and glycerine.

5. In a method of applying regenerated cellulose pellicles as seals for bottles and the like, the steps which comprise immersing gel regenerated celulose pellicles in an aqueous shipping solution containing a harmless regenerated cellulose preservative and a regenerated celulose softener, shipping containers of the pellicles and solution to a point of use, withdrawing the pellicles from the solution and directly applying them to the articles to be sealed, and drying the pellicles in situ.

6. The method described in claim 5 characterized in that the preservative is chlorinated cresol and the softener is glycerine.

HENRY H. WRIGHT.

CERTIFICATE OF CORRECTION.

Patent No. 2,013,739. September 10, 1935.

HENRY H. WRIGHT.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, second column, line 3, the sentence beginning with the words "It will be noted" should be read as the beginning of a new paragraph; line 30, claim 2, for "cap" read pellicle; line 32, claim 3, for "celullose" read cellulose; and lines 40 and 42, claim 5, for "celulose" read cellulose; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 8th day of October, A. D. 1935.

Leslie Frazer

(Seal) Acting Commissioner of Patents.