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ANTISTICKING CELLULOSE PELLICLES

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9 Claims. (Cl. 117—144)

1

This invention relates to improvements in smooth, non-fibrous, non-porous sheets and films. More particularly, the invention relates to the production of cellulosic pellicles which have been treated with an anti-sticking agent. The invention will be described in terms of regenerated cellulose film, but it should be understood that this is intended merely to be illustrative and not limitative.

In the manufacture of regenerated cellulose film, such as cellophane, prepared from an aqueous alkaline solution of sodium cellulose xanthate, it is necessary to maintain a certain predetermined moisture content in the film in order to produce a tough plastic film. Regenerated cellulose film will absorb moisture from the surrounding atmosphere and thereby render itself plastic when the relative humidity is sufficiently high. It is generally the practice, in order to render the regenerated cellulose film plastic at all relative humidities, to incorporate therein a hygroscopic plasticizing compound such as glycerol, glycol, or sorbitol, and the like. However, at high relative humidities the amount of moisture absorbed by the film is more than necessary to attain the desired plasticity and superimposed sheets have a tendency to stick together.

In the past the difficulty of sheets of regenerated cellulose sticking together has been overcome by coating the same with an anti-sticking lacquer. However, this necessitates an extra step in production and consequently increased cost. To overcome this the film has been impregnated, while in the wet gel state with an anti-sticking or "slip" agent such as stearamide, which is usually applied in conjunction with the plasticizer, just prior to drying.

"Slip" as used herein designates the amount of friction, or lack of it, between adjacent surfaces of superimposed regenerated cellulose films or sheets and is designated quantitatively by the coefficient of friction of two surfaces of film when subjected to a load of approximately one pound per square inch and acted on by a force at right angles to the applied load.

Stearamide is disadvantageous due to its high melting point and poor solubility. It is difficult to prepare a water emulsion or dispersion of the same. A high temperature is necessary and the

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emulsifiers, which must be employed, are affected by the hardness formers found in normal water thus leading, after a certain period of time, to an undesirably streaked film.

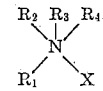
Accordingly, it is an object of the present invention to produce clear, smooth, non-fibrous, non-porous sheets and films having anti-sticking properties.

Another object of the invention is to produce softened regenerated cellulose sheets and films suffused with an anti-sticking agent and resistant to sticking together.

Other advantages and objects of this invention will be apparent from the description hereinafter.

Regenerated cellulose film or sheet is cast from viscose, an alkaline solution of sodium cellulose xanthate prepared in conventional manner, desulfurized, bleached, plasticized and dried. This is accomplished by passing the film through successive baths, by means of rollers, containing the necessary materials and in continuous operation. The film as it leaves the last or plasticizing bath before drying is wiped free of excess liquid by means of squeeze rolls, wiper blades, or the like.

The objects of the present invention are accomplished by incorporating in the plasticizer bath an anti-sticking agent of the class of quaternary ammonium compounds having the general formula—



in which:

R_1 denotes an alkyl group of at least 12 carbon atoms,

R_2 and R_3 denote alkyl groups of 1 to 20 carbon atoms,

R_4 denotes an alkyl group of 1 to 3 carbon atoms, and

X denotes an organic or inorganic anion such as Cl^-, HSO_4^-, $CH_3SO_4^-$, etc.

These compounds are prepared by the exhaustive methylation of primary, secondary, or tertiary fatty amines, such as amines from hydrogenated tallow, octadecyl amine, heptadecyl amine, nonadecyl amine, methyl octadecyl amine, dimethyl octadecyl amine, etc., with an

excess of methyl chloride or dimethyl sulfate, and the like, in a closed container at temperatures of 30 to 130° C. The primary, secondary, and tertiary fatty amines are easily prepared from the coresponding acids by well-known procedures.

Among the compounds particularly suited to the practice of the present invention may be listed octadecyltrimethylammmonium chloride and dimethyldialkylammonium chloride where the alkyl group is that produced by the hydrogenation of tallow. These compounds are self dispersible in water and hence require no emulsifiers.

In a preferred embodiment of the invention, a 5–10% dispersion in water of octadecyltrimethylammonium chloride was prepared by adding the same to water at room temperature, or higher if desired to speed up the rate of dispersion, with stirring. This dispersion was then added to the plasticizer bath, which contained 5% glycerol, to give a solids content of anti-sticking agent in the bath of 0.5%, and regenerated cellulose film in the wet gel state was passed therethrough. The excess of the bath was removed by means of squeeze rolls, or wiper blades, and the film dried in the usual manner. The resulting film showed no tendency to stick when sheets of the same were superimposed. Sheets were stacked in an oven under pressure at 120° F. for 16 hours and when removed from the oven, the sheets fell apart freely showing no tendency whatever to stick together. The film was also considerably tougher than the conventionally prepared film and did not yellow on exposure to light. The same procedure was followed using a dispersion of dimethyldialkylammonium chloride with the same desirable results.

Solutions, emulsions and dispersions of the class of compounds having the above-identified generic formula are applicable in the present invention. The percent solids of the anti-sticking agent in the plasticizer bath may be varied between 0.05 and 2%.

The compounds of the present invention are substantially non-foaming and consequently eliminate many troublesome problems often encountered with other materials, such as streaking of the film, etc. These compounds are also insensible to hard water thus avoiding the danger of the formation of deleterious salts which cause streaking of the film and contamination of the bath. The compounds are stable on storage and the pH of their aqueous dispersions can be readily varied within the range of 5.0 to 9.0 without ill effects.

It is preferred to apply the anti-sticking agent to the regenerated cellulose sheet or film while in the wet gel state, prior to any dehydration thereof, from the plasticizer or softener bath, the advantage being very little increase in cost and conventional equipment may be used with no change in the same. However, if desired, the anti-sticking agent may be applied subsequent to the addition of plasticizer to the regenerated cellulose film and prior to the drying step by means of dip rolls, sprays, or the like. When this method of application is employed, a higher solids content of anti-sticking agent in the bath must be employed due to the shorter contact time of the film with the bath. That is to say that the percent solids in the bath would more closely approximate 2% solids.

The anti-sticking agent may also be applied after the film has been completely dried but preferably before it has been dried below 69% moisture content. However, here the film must be re-wet and then dried resulting in an extra step and increased cost of production.

The present invention is further advantageous in that the application of anti-sticking agents from an aqueous solution, emulsion, or dispersion is accomplished on the film-casting machine, thus attaining a considerable saving in labor, equipment and materials over the former method of application which, as previously referred to, consisted of drying the film and coating the same with an organic solvent solution of the anti-sticking agent on a separate machine in a separate operation.

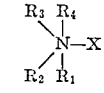
Regenerated cellulose film suffused with the anti-sticking agents of the present invention exhibit remarkably good printing properties, that is, it is more conducive to printing than ordinary regenerated cellulose film.

Although the invention has been discussed in detail in connection with the sizing of regenerated cellulose film or pellicles, it is also applicable to other smooth, non-fibrous, non-porous sheets and films which, due to their general nature, have a tendency to stick together, and especially water sensitive sheets, films, or pellicles cast from aqueous or aqueous alkaline cellulosic solutions, such as those of low-substituted cellulose ethers, esters, and ether-esters such as glycerol cellulose, methyl cellulose, ethyl cellulose, cellulose glycollic acid, cellulose phthalic acid, and the like.

It is to be understood that the description above is merely illustrative and that changes and variations may be made without departing from the spirit and scope of the invention as defined in the appended claims.

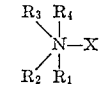
I claim:

1. As an article of manufacture a smooth, non-fibrous, non-porous, non-sticking film having all of its surfaces exposed and suffused with a compound selected from the group having the general formula



where R_1 is an alkyl group of at least 12 carbon atoms, R_2 and R_3 are alkyl groups of 1 to 20 carbon atoms, R_4 is an alkyl group of 1 to 3 carbon atoms, and X is a radical selected from the group consisting of organic and inorganic anions, said film comprising a polyhydroxy alcohol as a plasticizer therefor.

2. As an article of manufacture a non-sticking regenerated cellulose film having all of its surfaces exposed and suffused with a compound selected from the group having the general formula



where R_1 is an alkyl group of at least 12 carbon atoms, R_2 and R_3 are alkyl groups of 1 to 20 carbon atoms, R_4 is an alkyl group of 1 to 3 carbon atoms, and X is a radical selected from the group consisting of organic and inorganic anions, said film comprising a polyhydroxy alcohol as a plasticizer therefor.

3. An article of manufacture as defined in claim 2 wherein X is Cl^- and the plasticizer is glycerol.

4. An article of manufacture as defined in claim 2 wherein X is HSO_4^- and the plasticizer is glycerol.

5. An article of manufacture as defined in claim 2 wherein X is $CH_3SO_4^-$ and the plasticizer is glycerol.

6. As an article of manufacture a regenerated cellulose film containing glycerol and suffused with octadecyltrimethylammonium chloride.

7. As an article of manufacture a regenerated cellulose film containing glycerol and suffused with octadecyltrimethylammonium bisulfate.

8. As an article of manufacture a regenerated cellulose film containing glycerol and suffused with octadecyltrimethylammonium methyl sulfate.

9. As an article of manufacture a regenerated cellulose film containing glycerol and suffused with dimethyldialkylammonium chloride where the alkyl group is that produced by the hydrogenation of tallow.

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