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PROVISIONAL SPECIFICATION.

Improvements in or relating to the Manufacture of Sheets or Films
of Composition Containing Cellulose Esters or Ethers.

We, SPICERS LIMITED, a British company, of 19, New Bridge Street, London, E.C. 4, and HENRY JAMES HANDS, a British subject, of 100, Duke's Avenue, Chiswick, London, W. 4, do hereby declare the nature of this invention to be as follows:—

This invention relates to the manufacture of sheets or films of compositions containing esters or ethers of cellulose, such as cellulose acetate, by spreading a solution of such composition in a volatile solvent on a surface, drying off the solvent and removing the sheet or film from the surface.

In such manufacture, it is found that the sheet or film after leaving the surface on which it is formed carries an electric charge which is liable to cause sparking and consequent ignition of the vapour of the solvent or other combustible matter in the vicinity.

It is one of the principal objects of the invention to provide methods of and means for removing this electric charge.

According to one feature of the invention, the product, after it leaves the aforesaid surface, is passed through an aqueous bath (preferably an electrolyte) capable of wetting the product, which bath is electrically connected to earth.

Preferably, the bath is one which slightly alters the chemical composition of the surface layers of the product, for example, it reduces an extremely thin layer thereof to cellulose, thereby more effectually removing the electric charge therefrom.

Such a bath may consist of an aqueous solution of a caustic alkali, or of an acid or an acid salt. Preferably, the product is afterwards immersed in a neutralising bath and is then washed with water and dried by means of hot air.

A further object of the invention is to provide methods of and means for removing the last traces of solvent from the product; this residual solvent being apt to cause cockling or distortion of the product and to impart odour thereto.

According to this further feature of the invention, the product, after it leaves the surface on which it is formed, is passed

through an aqueous oxidising bath, for example, a bath containing hydrogen peroxide.

The treatments for conducting away the electric charge and for removing residual solvent may be effected, if desired, in a single bath having the necessary characteristics.

The invention is particularly advantageous when applied to the treatment of very thin films.

The invention may be carried into effect in the following manner, assuming that the product is manufactured by applying continuously a solution of its ingredients to an endless travelling band from which the product, substantially dry, is continuously removed. The product when stripped from the endless band is passed around rollers in a downwardly-directed loop through a bath consisting of a solution of caustic soda in water which is electrically connected to earth. The strength of this solution may vary within wide limits, depending partly upon the time of immersion. For example, the strength may be $\frac{1}{2}$ —1 per cent. by weight for a time of immersion of about 10 seconds. The caustic soda may, if desired, be dissolved in an oxidising bath consisting of an aqueous solution of about $\frac{1}{2}$ per cent. by weight of sodium peroxide and $\frac{1}{4}$ to $\frac{1}{2}$ per cent. by weight of sulphuric acid. In this case the bath is alkaline but contains hydrogen peroxide which it has been found assists in removing the last traces of solvent from the product. Alternatively, the oxidising bath may be separate from the alkaline bath.

After leaving the alkaline bath the product may be passed in a similar manner through an acid bath (for example, a bath of very dilute sulphuric acid, one part of acid to four hundred parts of water) for the purpose of neutralising the alkali adhering to the product. It is then washed by being passed through a bath of water, or between jets of water directed upon both sides thereof, or both, and dried by means of currents of hot air playing upon both sides.

During the treatment, the product is maintained in an extended or stretched

condition by means of suitable guide-rollers.

The solution of caustic soda has the property of wetting the surface of the product. It also reacts chemically with it, reducing a very thin surface layer thereof to cellulose. It is believed that this chemical action assists materially in the removal of the electric charge and that it renders the product less liable to acquire and hold a charge after treatment as it is liable to do owing (it is thought) to molecular changes undergone in the process of "ageing". It is advantageous also to add a softening agent such as methylated spirit to the alkaline bath, for the purpose of assisting the aforesaid chemical action. The amount of spirit may be about one-tenth of the total volume of water.

Although it is preferred to employ a caustic alkali, an acid bath may alternatively be used. This may consist of a weak solution of an acid salt, for example, sodium-hydrogen sulphate.

It is within the scope of the invention

to employ a wetting and conducting bath which exerts no chemical action on the product, although in general a longer time of immersion is necessary with such a bath. Solutions of sodium chloride or ammonium sulphate or sodium sulphate may, for example, be used.

The invention may advantageously be carried out in conjunction with that described in the Specification of our copending Application, No. 32,582 of 1926. In this case, the product is first subjected to the action of moist air or steam and is then passed through the aqueous bath or baths described herein.

When the surface layers of the sheet or film are reduced to cellulose, as hereinbefore described, it is found that gelatine coatings adhere more readily thereto than to the surface of the same material which has not been thus treated.

Dated this 30th day of August, 1927.

BOULT, WADE & TENNANT,
111 & 112, Hatton Garden, London,
E.C. 1,

Chartered Patent Agents.

COMPLETE SPECIFICATION.

Improvements in or relating to the Manufacture of Sheets or Films of Composition Containing Cellulose Esters or Ethers.

We, SPICERS LIMITED, a British company, of 19, New Bridge Street, London, E.C. 4, and HENRY JAMES HANDS, a British subject, of 100, Duke's Avenue, Chiswick, London, W. 4, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the manufacture of sheets or films of compositions containing esters or ethers of cellulose, such as cellulose acetate, by spreading a solution of such composition in a volatile solvent on a surface, drying off the solvent and removing the sheet or film from the surface.

In such manufacture, it is found that the sheet or film after leaving the surface on which it is formed carries an electric charge which is liable to cause sparking and consequent ignition of the vapour of the solvent or other combustible matter in the vicinity.

It is an object of the invention to provide methods of and means for removing this electric charge.

According to the invention, the product, after it leaves the aforesaid surface, is passed through an electrically conducting aqueous bath capable of wetting the pro-

duct, which bath is electrically connected to earth.

Preferably, the bath is one which slightly alters the chemical composition of the surface layers of the product, for example, it reduces an extremely thin layer thereof to cellulose, thereby more effectually removing the electric charge therefrom.

Such a bath may consist of an aqueous solution of a caustic alkali, or of an acid or an acid salt. Preferably, the product is afterwards immersed in a neutralising bath and is then washed with water and dried by means of hot air.

Further according to the invention the sheet or film may, after treatment in the electrically conducting aqueous bath, be passed through an aqueous oxidising bath, for example a bath containing hydrogen peroxide.

This further treatment has the effect of removing the last traces of solvent from the product; this residual solvent being apt to cause cockling or distortion of the product and to impart odour thereto.

The treatments for conducting away the electric charge and for removing residual solvent may be effected, if desired, in a single bath having the necessary characteristics.

The invention is particularly advantageous when applied to the treatment of very thin films.

5 The accompanying drawing illustrates diagrammatically apparatus with which the invention may be carried into effect in the case of film manufacture by applying continuously a solution of the ingredients thereof to an endless traveling band from which the film, substantially dry, is continuously removed.

10 The film 10 when stripped from the endless band (not shown in the drawing) is passed around rollers 12 in a downwardly-directed loop through a tank 14 containing a solution of caustic soda in water which is electrically connected to earth as indicated at 16. The strength of this solution may vary within wide limits, depending partly upon the time of immersion. For example, the strength may be $\frac{1}{2}$ to 1 per cent. by weight for a time of immersion of about 10 seconds. The caustic soda may, if desired, be dissolved in an oxidising bath consisting of an aqueous solution about $\frac{1}{2}$ per cent. by weight of sodium peroxide and $\frac{1}{4}$ to $\frac{1}{2}$ per cent. by weight of sulphuric acid. In this case the bath is alkaline but contains hydrogen peroxide which it has been found assists in removing the last traces of solvent from the film. Alternatively, the film may be treated in an oxidising bath separately and after treatment in the alkaline bath.

35 After leaving the tank 14, the film passes between squeegees 18 which remove the bulk of the liquid adhering thereto. It then passes around rollers 20 through a tank 22 containing an acid solution (for example very dilute sulphuric acid, one part of acid to four hundred parts of water) for the purpose of neutralizing the alkali adhering to its surface.

40 After passing between a second pair of squeegees 24 the film is passed around rollers 26 through a tank 28 containing water. On emerging from the water, the film passes between two pipes 30 supplied with water and provided with holes through which jets of water are directed upon both sides of the film. The excess of water escapes from the tank through a waste-pipe 32.

55 The film then passes between a third pair of squeegees 34 and enters a drying chamber 36 supplied with hot air through ducts 38 which enter the drying chamber from above and below. Within the chamber 36 the film passes in a zig-zag manner over and under a series of guide-rollers 40, and is finally wound on a reel 42, or cut into lengths, as desired.

65 The solution of caustic soda has the property of wetting the surface of the pro-

duct. It also reacts chemically with it, reducing a very thin surface layer thereof to cellulose. It is believed that this chemical action assists materially in the removal of the electric charge and that it renders the product less liable to acquire and hold a charge after treatment as it is liable to do owing (it is thought) to molecular changes undergone in the process of "ageing". It is advantageous also to add a softening agent such as ethyl alcohol (or methylated spirit) to the alkaline bath, for the purpose of assisting the aforesaid chemical action. The amount of spirit may be about one-tenth of the total volume of water.

80 Although it is preferred to employ a caustic alkali, an acid bath may alternatively be used. This may consist of a weak solution of an acid salt, for example, sodium-hydrogen sulphate.

85 It is within the scope of the invention to employ a wetting and conducting bath which exerts no chemical action on the product, although in general a longer time of immersion is necessary with such a bath. Solutions of sodium chloride or ammonium sulphate or sodium sulphate may, for example, be used.

90 The invention may advantageously be carried out in conjunction with that described in Specification No. 287,635. In this case, the product is first subjected to the action of moist air or steam and is then passed through the aqueous bath or baths described herein.

105 When the surface layers of the sheet or film are reduced to cellulose, as hereinbefore described, it is found that gelatine coatings adhere more readily there to than to the surface of the same material which has not been thus treated.

110 Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. The method of treating sheets or films formed by the evaporation of the solvent from solutions of compositions containing esters or ethers of cellulose, consisting in passing the sheet or film, after it leaves the surface upon which it is formed, through an electrically conducting aqueous bath capable of wetting the sheet or film, which bath is electrically connected to earth.

2. The method according to Claim 1 wherein the bath is one which slightly alters the chemical composition of the surface layers of the sheet or film, for example it reduces the ester or ether to cellulose in an extremely thin surface layer thereof.

3. The method according to Claim 2

wherein the bath is an alkaline bath, for example a one-half per cent. to one per cent., solution in water of caustic soda.

4. The method according to Claim 2, wherein the bath is an acid bath, for example a weak solution of sodium-hydrogen sulphate.

5. The method according to any one of the preceding claims, wherein the sheet or film, after treatment in the electrically conducting aqueous bath, is passed through an aqueous oxidising bath, for example a bath containing hydrogen peroxide.

6. The method according to any of the preceding Claims 1 to 4, wherein the bath

contains an oxidising substance, for example hydrogen peroxide.

7. The method according to any of the preceding claims wherein the earthed bath contains a softening agent, for example ethyl alcohol (or methylated spirit).

8. Apparatus for carrying out the method according to any of the preceding claims, substantially as described with reference to the accompanying drawing.

Dated this 29th day of May, 1928.

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2nd Edition

[This Drawing is a reproduction of the Original on a reduced scale.]

