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FILM

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This invention relates to the stripping of dry cast polyvinyl alcohol substitution derivative films, especially continuous webs, from the casting surface. More particularly it appertains to the incorporation of materials, for convenience called "stripping agents," in polyvinyl acetal dopes for facilitating separation of the cast film from the casting wheel.

Polyvinyl acetals which have been applied in solution to the surface of such materials as wood, metals, ceramics, plastics and the like, and the solvent evaporated, possess considerable adhesion therefor. If applied for protective purposes this adhesion, between the polyvinyl acetal and the base object upon which it has been coated, would be a desirable property. Such an inherent adhesion is very undesirable when the base is employed only as a temporary support during the solvent removal as in the casting of self-supporting film such as sheet wrapping and similar material.

In general dry casting procedures involve the laying of a film of dope (solution of the cellulose derivative) on to a casting wheel mounted for rotation and arranged for the removal of the solvent from the dope during its rotation. The means for the solvent removal may include heating the surface of the wheel or surfaces adjacent thereto, together with devices such as fans for carrying the solvent or solvent-laden atmosphere away from the film. Ordinarily the speed of the wheel and the degree of heating are adjusted so that the desired amount of solvent is removed in somewhat less than one revolution of the casting wheel. Nickel, chromium, stainless steel and other metals and alloys are generally used for surfaces of the casting wheels.

In attempting to carry out a continuous casting operation of this type with polyvinyl acetal dopes, it was found that the adhesion between the partially dried polyvinyl acetal film and the casting surface was a frequent source of trouble. It caused the film to break or tear at the place where the film was being stripped from the wheel. This action was especially troublesome at the edges of the film. Even when the film did not tear or break, distortion and deformation were quite common due to the high tension required for pulling the film away from the casting surface.

A group of materials has now been discovered which greatly decreases the adhesion between such partially dried film and the metal casting surface. With the preferred members of the group the adhesion is reduced to such an extent that only a very slight tension is required to strip the film from the casting surface.

This invention had for an object the manufacture of transparent, strong and flexible polyvinyl acetal sheets or films which were undeformed and/or undistorted and/or unmarred, and the production of compositions of matter suitable for the casting thereof. A further object was to dry cast benzene methanol (60:40) and methanol soluble polyvinyl acetals into thin, strong, smooth, flexible sheets capable of being easily stripped from the casting wheel while retaining 5% to 15% of the solvent of the casting dope. Further objects were to devise a means of decreasing the adhesion between a dry cast, organic solvent soluble, polyvinyl alcohol substitution derivative and the casting surface, to facilitate the removal of partially dried, dry cast, polyvinyl alcohol substitution derivatives from casting wheels and belts, and to find materials which could be added to polyvinyl alcohol dry casting solutions to make removal of the film easy when only a small proportion of the solvent remains. A general advance in the art, and other objects which will appear hereinafter, are also contemplated.

It has now been discovered that monobasic esters and their inorganic salts, may be incorporated in casting dopes with the result that the film formed therefrom can be easily and satisfactorily removed from the casting surface.

From the following description and specific examples, in which are disclosed certain embodiments of the invention as well as details of what is believed to be the best mode for carrying out the invention, it will be apparent how the foregoing objects and related ends are accomplished. The parts are given by weight throughout the application, unless otherwise specified.

Example I

A film was prepared by evaporating the solvent from a composition consisting of 17% polyvinyl formal (hydroxyl 2%, formal 90%, acetyl 8%), 3% of castor oil and 80% solvent (benzene-methanol-3:2), in which had been incorporated 3% dipalmityl phosphate (on the basis of the polyvinyl formal plus the castor oil). The resulting extremely durable film was stripped from the metallic casting surface while it still contained 15% of the solvent. The stripping operation was easily carried out. Similar results were obtained with monolauryl phthalate, monomyristyl phthalate, and dilauryl phosphate.

Example II

To a solution containing 20% polyvinyl acetal (prepared from polyvinyl alcohol and butyraldehyde) dissolved in methanol, was added 3% dilauryl phosphate (on the basis of the polyvinyl acetal). Film cast from this solution onto a nickel surface and dried possessed only a slight adhesion to the nickel surface, when compared to film cast from a similar polyvinyl acetal solution to which no dilauryl phosphate had been added.

Example III

A dope consisting of:

	Parts
Polyvinyl butyral	6.3
Polyvinyl isobutyral	6.8
Polyvinyl formal	3.8
Castor oil (crystal grade)	3.1
Methanol	70.0
Ethyl formate	10.0

in which had been incorporated 3.2% of zinc lauryl phthalate (based on the total film-forming and plasticizing ingredient material), was cast continuously on the polished surface of a rotating nickel cylinder ten feet in diameter. The volatile materials were evaporated, and the resulting film stripped from the surface of the casting wheel. The film, which had a thickness of 0.0013 inch, was very clear, could be satisfactorily sewed, had excellent tear resistance, and was easily removed from the surface of the casting wheel. Similar results were obtained with the sodium, potassium and calcium salts of monolauryl phthalate and monomyristyl phthalate. Zinc myristyl phthalate was also very satisfactory.

Example IV

A film was prepared by evaporating the solvent from a composition consisting of 17% polyvinyl formal (hydroxyl 2%, formal 90%, acetyl 8%), 3% castor oil, 64% methyl acetate, 2% water and 14% methanol, to which had been added zinc oleyl phthalate. The film, which stripped easily from the polished metal casting surface, was found to be extremely durable and not to tear readily under a wide variety of temperatures.

Example V

A dope consisting of:

	Per cent
Polyvinyl butyral*	10
Polyvinyl formal (Example I type)	5
Castor oil	3
Methanol	33
Benzene	49

*(That is, hydroxyl corresponding to 19% polyvinyl alcohol, butyral corresponding to 80% polyvinyl butyral and acetyl corresponding to 1% polyvinyl acetate.)

in which had been incorporated 2% (based on the polyvinyl acetal) lithium dipalmityl phosphate, was cast continuously on the polished surface of a rotating nickel cylinder six feet in diameter. The volatile materials were evaporated, and the resulting film stripped from the surface of the casting wheel. The film, which had a thickness of .00088 inch, was very clear, could be satisfactorily sewed, and had excellent tear resistance. It was undeformed, unmarred, and undistorted. It was stripped from the casting wheel very easily. Similar results were obtained with the sodium, potassium, calcium, zinc, and like salts of dilauryl phosphate.

The dope or polyvinyl acetal solution containing the strip agents of the invention may be handled and cast in the same manner as the dry casting dopes previously known to the art.

The most marked decrease in adhesion between the film and the casting wheel surface has been noted when the casting surface was nickel.

In the preferred mode of operation the film is stripped from the casting wheel while it still contains 5% to 15% of the solvent.

This invention is not limited to the polyvinyl acetals of the specific examples, although it peculiarly appertains thereto. In general it is, with considerable efficacy, applicable to all kinds of polyvinyl acetals which are soluble in organic liquids (solvents). The preparation and properties of polyvinyl acetals are disclosed in U. S. A. Patents Nos. 2,035,939 and 2,036,092 (Morrison).

Although many plasticizers for polyvinyl acetals have been suggested heretofore, castor oil is preferred for thin films.

For convenience of consideration, the materials of this invention which decrease the adhesion between a dry cast polyvinyl acetal film and the surface upon which the film is cast and dried (or partially dried), may be divided into sub-generic groups as follows: mono basic esters and inorganic salts of mono basic esters.

The mono basic esters comprehended by the present invention are those obtained from alcohols containing at least seven carbon atoms. Preferably the alcohol contains 12 to 20 carbon atoms or more. Especially suitable for the purposes of the present invention are the phthalic acid and phosphoric acid esters. The results obtained with mono-lauryl phthalate, monomyristyl phthalate, dilauryl phosphate and dipalmityl phosphate are outstanding.

The salts of mono basic esters comprehended by the present invention are the sodium, potassium, calcium, zinc and like metal salts of the mono basic esters mentioned in the preceding paragraph.

In the preferred embodiments of the invention the stripping agent is present in an amount within the range of 0.5% to 5% by weight on the basis of the polyvinyl acetal. Higher concentrations are not excluded, and may be used in order to secure specific results.

The strip agents may be used singly or in any combination, and it is to be understood that the invention is not limited to the specific materials listed above or in the specific examples.

The stripping agents of this invention may be incorporated in the casting dope in any desired manner. If preferred they may be predissolved and the solvent used for dissolving the polyvinyl acetal. Ordinarily they are incorporated into the solvent simultaneously with the polyvinyl acetal. In some instances it has been found convenient to add the stripping agent to the solution of the polyvinyl acetal.

Many of the advantages of the present invention will be apparent from the preceding portion of the specification. Specific mention may be made of the fact that by this invention it is possible to easily, speedily and satisfactorily remove the partially dried, dry cast polyvinyl acetal film from a metal or alloy casting wheel at elevated temperatures, and that all danger of tearing or deforming the dry cast film at the stripping point (or line) due to the pull required to separate the film from the casting surface has been removed.

The advantage of incorporating in a casting

solution a material which decreases the adhesion between the polyvinyl acetal film cast therefrom and the casting surface without undesirably affecting the transparency and surface characteristics of the film, will be obvious to those skilled in the art.

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 this invention is not limited to the specific embodiments thereof except as defined in the appended claims.

I claim:

1. A plasticized, organic solvent soluble polyvinyl acetal film, containing 0.5% to 5% of di-lauryl phosphate.

2. A film of polyvinyl acetal containing 0.5% to 5% of diester of phosphoric acid, the esterifying radicals being aliphatic and containing at least 7 carbon atoms.

3. The product of claim 2 when the alcohol radical contains 12 to 20 carbon atoms.

4. An organic solvent, dry casting solution of polyvinyl acetal containing di-ester of phosphoric acid, the esterifying radicals being aliphatic and containing more than 7 carbon atoms, as strip agent.

5. The product of claim 4 when the strip agent contains 12 to 20 carbon atoms.

6. A plasticized, organic solvent soluble polyvinyl butyral film, containing 5 to 15% of solvent therefor and 0.5% to 5% of di-lauryl phosphate.

7. A composition of matter comprising organic solvent soluble polyvinyl acetal, a solvent therefor and 0.5% to 5% of di-ester of phosphoric acid, the esterifying radicals being aliphatic and containing at least 7 carbon atoms.

8. A plasticized, organic solvent soluble polyvinyl butyral film, containing 0.5% to 5% of di-lauryl phosphate.

9. A plasticized, organic solvent soluble polyvinyl butyral film, containing 0.5% to 5% of di-ester of phosphoric acid, the esterifying radicals being aliphatic and containing at least 7 carbon atoms.

10. A plasticized, organic solvent soluble polyvinyl butyral film, containing 0.5% to 5% of di-palmityl phosphate.

11. A plasticized, organic solvent soluble polyvinyl acetal film, containing 0.5% to 5% of di-ester of phosphoric acid, the esterifying radicals being aliphatic and containing more than 7 carbon atoms.

12. An organic solvent dry casting solution of polyvinyl acetal containing di-lauryl phosphate as strip agent.

13. An organic solvent dry cast polyvinyl butyral film containing 5% to 15% of a solvent therefor, and 0.5% to 5% of diester of phosphoric acid, the esterifying radicals being aliphatic and containing more than 7 carbon atoms, as strip agent.

14. An organic solvent dry casting solution of polyvinyl butyral containing di-ester of phosphoric acid, each of the esterifying radicals being aliphatic and containing 12 to 20 carbon atoms as strip agent.

15. A plasticized organic solvent soluble film of polyvinyl butyral containing 0.5% to 5% of di-ester of phosphoric acid, each of the esterifying radicals being aliphatic and containing 12 to 20 carbon atoms.

16. An organic solvent dry casting solution of polyvinyl butyral containing di-lauryl phosphate as strip agent.

17. A film of polyvinyl butyral containing 0.5% to 5% of di-ester of phosphoric acid, the esterifying radicals being aliphatic and containing at least 7 carbon atoms.

18. A plasticized organic solvent soluble film of polyvinyl acetal containing 0.5% to 5% of di-ester of phosphoric acid, each of the esterifying radicals being aliphatic and containing 12 to 20 carbon atoms.

19. A cast film of polyvinyl acetal containing 5% to 15% of solvent therefor and 0.5% to 5% of diester of phosphoric acid, the esterifying radical containing 12 to 20 carbon atoms.

20. A cast plasticized organic solvent soluble polyvinyl acetal film containing 5% to 15% of a solvent therefor and 0.5% to 5% of di-lauryl phosphate.

21. A film of polyvinyl acetal containing 5% to 15% of solvent therefor and 0.5% to 5% of di-ester of phosphoric acid, the esterifying radicals containing at least 7 carbon atoms.

22. A film of polyvinyl butyral containing 0.5% to 5% of diester of phosphoric acid, the esterifying radicals containing 12 to 20 carbon atoms.

23. An organic solvent dry casting solution of polyvinyl butyral containing diester of phosphoric acid, the esterifying radicals containing at least 7 carbon atoms.

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