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PROCESS OF CASTING FILM EMPLOYING 2-(2-AMINOETHYLAMINE) ETHANOL AS A RELEASE AGENT

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This invention relates to release agents to be used in conjunction with the casting of film which as a final product is self-supporting, and which film is useful as a coating medium on a solid surface. More particularly the invention relates to release agents for use in the casting of a film such as is used in the electron tube industry for coating cathode base materials. The character of film is disclosed in the patent to Kerstetter and Wennin, No. 2,986,671 granted May 30, 1961, to which reference may be made for more particulars as to the film. In general, the film, as used for coating a cathode sleeve or a cathode filament, is comprised of one or more of the carbonates of barium, strontium and calcium in a suitable organic binder such as ethylcellulose or nitrocellulose, preferably admixed with a plasticizer. It is also contemplated utilizing this invention in conjunction with cast films made from nylon, methyl methacrylate and polyvinyl alcohol binders and for films containing such solids as iron oxide; electroluminescent and picture tube phosphors; graphite; and magnesium oxide. In casting the film, as on the casting wheel disclosed in application 783,787, now Patent No. 2,974,364, of the same inventors, the mixture is held in suspension or solution in suitable volatile liquids or in water, and after such casting, it is necessary to strip the casting surface or wheel of the partially set film. Since the film, in accordance with the process disclosed herein and in application 783,787, now Patent No. 2,974,364, when removed from the casting surface is still very sleazy, it is apt to rupture unless an appropriate release agent is applied to the casting surface.

Early research into conventional release agents such as the heteropolysaccharides—gum tragacanth, guar, agar, karaya—and other starches and sugars which have the desirable characteristics of being poor adhesives either in aqueous solution or when plasticized, showed that these materials were unsatisfactory because of the formation of a non-uniform film, which in turn affected the uniformity of thickness of the cast film, or because of solubility in the solvents of the casting suspension, if a water type suspension be used. Also such conventional release agents showed their general undesirabiltiy in a film which must subsequently be processed, purified or activated on a vacuum exhaust system. In general liquids are employed as release agents in preference to solids.

For somewhat similar reasons, the usual fatty acids or fatty acid derivatives cannot be used satisfactorily. This is particularly true when carbon residues cannot be tolerated as in the case of electron emissive coatings. Likewise since the halides of the alkaline earth metals are more difficult to decompose thermally than the carbonates, the release agents must be free of the halides and for somewhat similar reason, must be free of sulphur and phosphorus as well as alkali metals. In general the solvents, or solvent combinations, necessary to give optimum film strength, desired porosity, or practical drying or evaporation rates are also solvents for the fatty acid derived compounds, and they are therefore removed from the casting surface and absorbed into the film. This both destroys their usefulness as release agents and incorporates a detrimental and unwanted material into the film.

It is an object of this invention to provide a release agent which shall avoid the use of the above agents and still have the following desirable properties: a low vapor pressure under normal atmospheric condition whereby the agent would be slow in evaporating while it is on the casting surface; a moderately high heat of vaporization; high viscosity; capability of forming a smooth continuous layer on the casting surface; insolubility in the casting suspension or have sufficient viscosity to dissolve or diffuse only very slowly into the suspension; and one which should be non toxic in either the liquid or vapor state.

The effectiveness of the release agent for the type of film set forth above and its selection depends on the nature of the casting surface. The surface of course should be very smooth. It may have a polished chrome finish, or be of stainless steel, aluminum, nickel, brass, copper, silver, mylar (a Du Pont polyester), Teflon (a Du Pont tetrafluorethylene), cellulose acetate, polyvinyl alcohol, polyethylene, crystal plate glass, Pyrex, glazed paper, Bakelite, porcelain enamel, or Micarta. All of these have been tried and found satisfactory, with the polished chrome surface best.

The amount of release agent required to give the desired thickness of film on the casting surface, between 0.0001" and 0.001", depends on the ratio of binder in the suspension to the surface area of the carbonates or other inorganic filler in the suspension. In general less release agent is required for larger binder-pigment ratios although the amount of such agent to secure the desired thickness on the casting surface also depends upon the porosity of the film and the degree and rate of film penetration by the release agent. It has been found that the releasee agent should have under normal pressure and temperature conditions (760 mm. and 20° C.), a vapor pressure of about 0.01 mm., a boiling point of about 244° C., a viscosity of from 100 to 500 centipoises and preferably about 120. In general the release agent should be an amine derivative containing hydroxyl groups or other oxygen bonded side chains. The preferred release agent is 2-(2-aminoethylamino) ethanol

$$(NH_2.CH_2.CH_2.NHCH_2CH_2OH)$$

in a concentration from 5 to 20% in ethanol, as will be set forth in greater detail hereinafter.

Release agents which have been investigated and found

to be unsatisfactory are listed hereinafter with reasons for rejection:

	Toxic	Too Volatile	Tacky When Dry	Solvent For Binder	Difficult to Handle Wet Film	Plasticizer For Binder	Absorbed by Film	Poor Release	No Release*
Dimethylamino 2-propanol	X						X		X
Dimethylaminoethanol							X		X
Ethyl aminoethanol	X	X							
Aminoethanol							X		X
1 amino-2 propanol	X								X
3,3-Diaminodipropylamine			X						X
Triethyl orthoformate							X		X
2-methyl oxyethyl acetate							X		X
Dimethyl cyanamide									X
Methyl nonanoate									X
Iso-butyl formate		X							
2,2 Dimethoxypropane		X							
2 Ethoxyethanol									X
Cyclohexylamine	X						X		X
t-Butyl amine	X								X
Diethylene triamine	X						X		X
Triethylene tetramine	X						X		X
Tetraethylene pentamine	X						X		X
Butyl carbitol				X					
Butyl carbitol acetate				X					
Ethylene glycol							X		X
Triethanol amine							X		X
Water					X				
Silicone resins									X
Stearic acid									X
Oleic acid								X	
Butyl stearate						X			
Cetyl betaine $(CH_3)_3NCH_2COO$									X

*"No release" means that material adheres so firmly that it can be removed only by mechanical scraping or by re-dissolving.

For the reasons previously set forth the use of the previously listed release agents was discarded in favor of the preferred release agent.

The preferred release agent may be applied to a surface by dipping, as by rotation of a casting wheel into a trough containing prepared release agent; by brushing the agent thereon, as through the use of a rotating brush which is supplied with the agent; by a sponge kept saturated with the agent; or by textured paper saturated with the agent, this last method being preferred where a flat glass surface is the casting surface. In the case of continuous wheel casting, the preferred method is to use a rotating wheel, as disclosed in the referred to previous application of the instant inventors. Control of film thickness is maintained by dilution of agent with the proper solvent and by the speed, direction of rotation, spacing and pressure of the rotating wheel against the casting wheel and by the character of casting wheel. Of course prior to application of the release agent, the wheel is rendered dust free in any desirable fashion as by sponging, brushing or rinsing techniques.

In the case of a polished chrome surface moving at the rate of about one and one half feet per minute and a suspension containing the following proportions:

Ethylene carbonate	gm.	200
Toluene	ml.	400
Ethyl alcohol	ml.	125
Ethyl acetate	ml.	50
Butyl carbitol	ml.	25
Ethyl cellulose N-300	gm.	30
Barium nitrate	gm.	4
C-10 triple carbonates	gm.	400

with a film cast on the surface of the wheel, 0.002" thick. the release agent is in the form of a 5% concentration of 2-(2-aminoethylamino) ethanol, in ethanol having a film thickness of about 0.0002". Substantially the same concentration of release agent in ethanol has effectively released the films from the other suspensions mentioned earlier.

The presence of both the ethylene carbonate and the butyl carbitol in the suspension aids in the release of the film. The ethylene carbonate function to retard the adsorption of the release agent by the film. The butyl carbitol acts as a solvent for the binder in the film.

Having described the invention what is claimed is:

1. A process for casting films on moving surfaces comprising applying to the surface a release agent essentially in the form of 2-(2-aminoethylamino) ethanol and then casting on the so treated surface a suspension containing finely ground solids in a binder as well as a volatile organic solvent for the binder.

2. A process for casting fi'ms on a moving surface comprising applying to the surface a release agent in the form of 2-(2-aminoethylamino) ethanol in a concentration of from 5% to 20% in ethanol and then casting on the so treated surface a suspension containing one or more of the alkaline earth carbonates in a cellulosic binder.

3. A process for casting films on a moving surface comprising applying to the surface a release agent in the form of 2-(2-aminoethylamino) ethanol in a concentration of from 5% to 20% in ethanol and then casting on the so treated surface a suspension containing one or more of the alkaline earth carbonates in a cellulosic binder and a quantity of ethylene carbonate.

4. A process for casting film on a polished stainless steel surface travelling at the rate of about one and one half feet per minute comprising applying a release agent in the form of a 5% concentration of 2-(2-aminoethylamino) ethanol in ethanol to the surface to a thickness of 0.0002 inch and casting a suspension on the treated surface of triple carbonates suspended in a cellulosic binder, the carbonates being applied at such a rate on the travelling surfaces as to result in a film thickness of about 0.002 inch.

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