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PROCESS OF REMOVING SOLVENTS FROM PLASTIC COLLOIDS

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This invention relates to a process of removing solvents from semi-solid or plastic colloids and in its specific embodiments relates particularly to the removal of solvents from cellulose ester plastic compositions, such as cellulose nitrate plastics.

The process set forth herein is an improvement on the process described and claimed in my copending application Serial No. 433,191, filed March 4, 1930.

In my copending application above referred to, I describe a process of removing solvents from plastic colloids containing the same by immersing the colloid in a bath of a nonsolvent of the colloid having an osmotic pressure at least as great as the osmotic pressure within the colloid, and gradually reducing the osmotic pressure of the bath below that within the colloid. The process described is very suitably adapted for removing solvents from plastic colloids such as vulcanized fiber and the like but is particularly adapted for removing solvents from cellulose nitrate plastic compositions, such as viscoloid, fiberloid, and the like. In the preferred practice of such process, freshly formed sheets of cellulose esters are immersed in a bath consisting of a 2% to 40 per cent. solution of alcohol in water and gradually replacing the alcohol present with water over a relatively short period, say five hours. This treatment results in so-called "ageing" of cellulose ester plastics and was devised for overcoming the disadvantages of the former practice of air drying over long periods of ageing.

Several modifications of the preferred practice of the process were described in said copending application. For example, it is disclosed that various treating baths other than an aqueous alcohol solution could be employed to secure an osmotic pressure at least as great as or greater than the osmotic pressure within the colloid to be treated. For example, it was stated that a concentrated aqueous solution of common salt or sugar might be employed. However, the preference was indicated for a solution of the desired osmotic pressure of the solvent or one of the ingredients of the solvent present in the plastic colloid, an aqueous solution of alcohol being especially adapted for use where cellulose ester plastics or like nitro-cotton plastic colloid was being treated. Moreover, it was disclosed in said copending application that the plastic colloid might be extruded directly into a treating bath of the character described. Furthermore various methods for effecting the gradual reduction of osmotic pressure of the bath were set forth.

The above described process has proved to be of great practical value in commercial use, especially in connection with the manufacture of cellulose ester sheets for use in the preparation of "shatter-proof glass". However, I have discovered that the above described process may be

improved whereby sheets containing less stresses and strains may be prepared.

In the practice of my present process, I first treat the plastic colloids substantially in the manner disclosed in my copending application Serial No. 433,191, and as described above, after which the treated products are placed in a drier and are subjected, over a period of several days, to gradually increasing temperatures up to approximately 150° to 160° F., depending upon the nature of the product being treated. When this improved process is employed it is not necessary to use in the liquid treating bath as large a percentage of alcohol or the like as in the process described in my copending application referred to where the sheets were not heated in a drier after the ageing in the liquid bath. However, the present process may be practiced, in the initial part thereof, in substantially the same manner as described in my copending application, if desired.

In the preferred practice of the present process, a freshly formed cellulose ester sheet, or other plastic colloid, is treated in an aqueous bath containing approximately from 2 to 25 per cent., and preferably 10 per cent. alcohol, for several days. If the alcohol content of the bath is increased, the time of treatment in the bath may be correspondingly decreased. Following this initial treatment, the alcohol present is replaced with water and the partially treated plastic colloid is then soaked in running cold water for from several hours to several days. Thereafter, the sheets are suspended in a drier and the temperature is raised to about 100° F. and gradually increased until a temperature of from about 150° to 160° F. is reached. The time of treatment in the drier varies with the gauge of the stock under treatment.

The following treatment suitable for removing solvent from a 215 gauge cellulose ester sheet may be considered as illustrative of the invention:

- 3 days soaking in 10 per cent. alcohol, 90 per cent. water bath;
- 2 days soaking in running cold water bath;
- 1 day in dryer----- temperature 100° F.
- 1 day in dryer----- temperature 115° F.
- 1 day in dryer----- temperature 130° F.
- 1 day in dryer----- temperature 150° F.
- 2 days in dryer----- temperature 160° F.

While I have described in detail the preferred practice of my process, it is to be understood that the details of procedure may be variously modified without departing from the spirit of the invention or the scope of the subjoined claims.

I claim:

1. The process of removing solvent from a cellulose ester plastic composition containing the same and also containing a plasticizing agent, 60

without substantial removal of said plasticizing agent, which comprises immersing such plastic composition in a bath of a nonsolvent of the plastic composition having an osmotic pressure at least as great as the osmotic pressure within the plastic composition, the amount of nonsolvent in said bath being insufficient substantially to remove the plasticizing agent from said plastic composition, reducing the osmotic pressure of the bath below that within the plastic composition, introducing the thus treated product into a drier, and gradually raising the temperature in said drier to approximately from 150° to 160° F. over a period of several days.

2. The process of removing solvent from a cellulose ester plastic composition containing the same and also containing a plasticizing agent, without substantial removal of said plasticizing agent, which comprises immersing such plastic composition in an aqueous solution of a nonsolvent of the plastic composition having an osmotic pressure at least as great as that within the plastic composition, the amount of nonsolvent being insufficient substantially to remove the plasticizing agent from said plastic composition, gradually decreasing the content of the nonsolvent with a miscible nonsolvent capable of reducing the osmotic pressure below that within the plastic composition, introducing the thus treated product into a drier, and gradually raising the temperature in said drier to approximately 150 to 160° F. over a period of several days.

3. The process of removing solvent from a cellulose ester plastic composition containing the same and also containing a plasticizing agent, without substantial removal of said plasticizing agent, which comprises immersing such plastic composition in a bath comprising an aqueous solution of alcohol having an osmotic pressure at least as great as that within the plastic composition, the amount of alcohol in said bath being insufficient substantially to remove the plasticizing agent from said plastic composition, gradually decreasing the alcohol content with water to reduce the osmotic pressure of the bath below that within the plastic composition, introducing the thus treated product into a drier, and gradually raising the temperature in said drier to approximately 150° to 160° F. over a period of several days.

4. The process of removing solvent from a freshly formed cellulose nitrate plastic composition containing a plasticizing agent, without substantial removal of said plasticizing agent, which comprises immersing such plastic composition in water containing alcohol, the amount of alcohol being insufficient substantially to remove the plasticizing agent from said plastic composition, decreasing the alcohol content with a nonsolvent for such plastic composition, introducing the thus treated product into a drier, and gradually raising the temperature in said drier to approximately 150° to 160° F. over a period of several days.

5. The process of removing solvent from a freshly formed cellulose ester plastic composition containing a plasticizing agent without substantial removal of said plasticizing agent, which comprises immersing such plastic composition in a bath comprising water and alcohol, the amount of alcohol in said bath being insufficient substantially to remove the plasticizing agent from said plastic composition, introducing the resulting product into a drier, and gradually

raising the temperature in said drier to approximately 150° to 160° F. over a period of several days.

6. The process of removing solvent from a freshly formed cellulose ester plastic composition containing a plasticizing agent without substantial removal of said plasticizing agent, which comprises soaking such plastic composition in a bath comprising water and alcohol, the amount of alcohol in said bath being insufficient substantially to remove the plasticizing agent from said plastic composition, soaking the thus treated product in fresh water, introducing the resulting product into a drier, and gradually raising the temperature in said drier to approximately from 150° to 160° F. over a period of several days.

7. The process of removing solvent from a freshly formed cellulose ester plastic composition containing a plasticizing agent, without substantial removal of said plasticizing agent, which comprises soaking such plastic composition for several days in a bath of alcohol and water, the amount of alcohol in said bath being insufficient substantially to remove the plasticizing agent from said plastic composition, soaking the thus treated product in running cold water, introducing the resulting product into a drier, and gradually raising the temperature in said drier to approximately 150° to 160° F. over a period of several days.

8. The process of removing solvent from a freshly formed cellulose ester plastic composition containing a plasticizing agent, without substantial removal of said plasticizing agent, which comprises soaking such plastic composition for approximately three days in an aqueous bath containing approximately 10 per cent. alcohol, soaking the thus treated product for approximately two days in running cold water, introducing the resulting product into a drier, and subjecting such product to varying temperatures increased gradually from 100° F. to approximately 160° over a period of about six days.

9. The process of removing solvent from a cellulose ester plastic composition containing the same and also containing a plasticizing agent, without substantial removal of said plasticizing agent, which comprises immersing such plastic composition in a bath of a nonsolvent of the plastic composition having an osmotic pressure at least as great as the osmotic pressure within the plastic composition, the amount of nonsolvent in said bath being insufficient substantially to remove the plasticizing agent from said plastic composition, reducing the osmotic pressure of the bath below that within the plastic composition, introducing the resulting product into a drier, and subjecting such product to varying temperatures increased gradually from approximately 100° F. to approximately 160° F. over a period of about six days.

10. The process of treating a freshly formed cellulose ester plastic composition containing a plasticizing agent, without substantial removal of said plasticizing agent, which comprises immersing such plastic composition in a bath comprising water and alcohol, the amount of alcohol in said bath being insufficient substantially to remove the plasticizing agent from said plastic composition, introducing the thus treated product into a drier, and subjecting such product to varying temperatures increased gradually to about 160° F. over a period of approximately six days.