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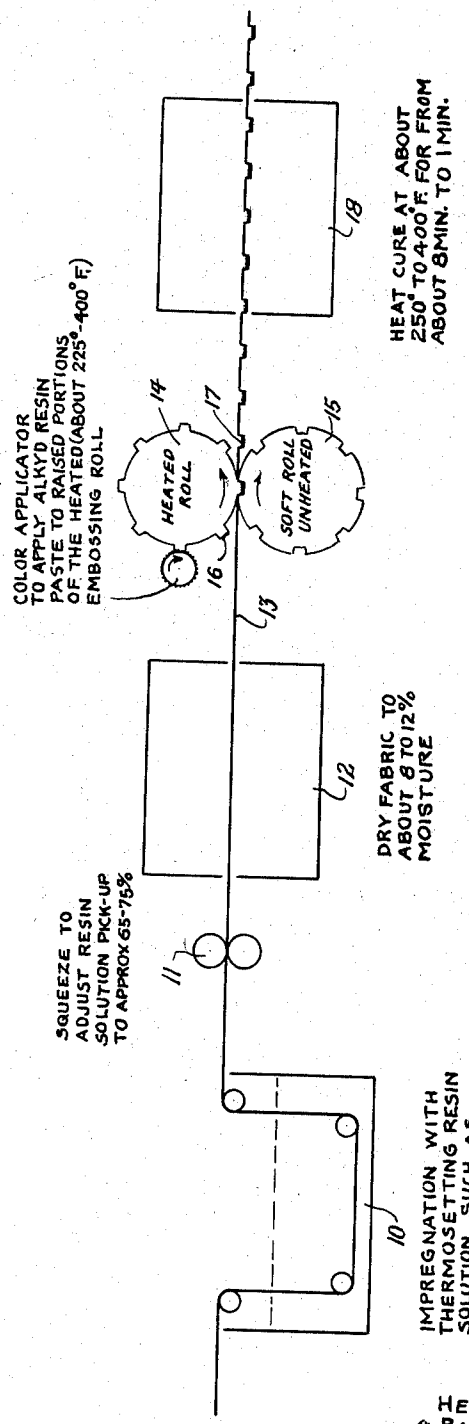

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PROCESS OF COATING FABRICS TO PRODUCE DURABLE INLAY EFFECTS AND RESULTANT ARTICLE

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13 Claims. (Cl. 117—11)

This invention relates to the production of inlay effects on embossed fabrics, and its main objective is to obtain inlay effects which are durable to repeated washings and dry cleanings.

Inlay effects are produced by applying color to the depressions in the embossed fabric on the face side thereof, as, for example, in the bottom, i. e., the shallow-most portions of the valleys. In order to obtain correct registration of the embossed pattern and the color, the process must be carried out simultaneously on the embossing machine by first applying the color to the outer raised surfaces of the engraved steel embossing roller (as by means of a printing roll) which, in turn, embosses the fabric and simultaneously deposits the color in the depressions.

Durable embossed finishes have long been known, but, insofar as we are aware, we are the first to provide inlay effects in embossed finishes which are durable to repeated washings and dry cleanings and which are also fast to crocking. Moreover, in the carrying out of the process, we are enabled to secure inlay effects which are clear and distinct. In other words, the color does not migrate, and what is called "build-up" is avoided, i. e., the paste can be run indefinitely without solidifying to a hard gummy mass on the application roller.

Generally speaking and as shown in the schematic flow sheet, Fig. 1, in carrying out the process, we first impregnate the fabric with a solution of thermosetting resin-forming materials (desirably a precondensate of resin, such as melamine formaldehyde, urea formaldehyde, and similar thermosetting resin precondensates and mixtures thereof, well known and understood in the art of producing durable embossed finishes), squeeze 11 so that the fabric retains approximately 65–75% pick-up over and above its dry weight, and thereafter we dry 12 the fabric, preferably to the point that it contains some moisture, desirably from about 8% to about 12%. Alternatively, we may dry to remove free moisture and then either "condition" by adding moisture, or allow the fabric to regain moisture before calendering by letting it come into equilibrium with the surrounding atmosphere. A suitable catalyst, of which there are many konwn, is employed, desirably by incorporation in small amounts in the impregnating solution. Ordinarily an acidic type catalyst is used.

The impregnated fabric 13 is now passed through an embossing calender consisting essentially of two rolls, one of which is usually made of metal 14 and is engraved to produce elevations and depressions, giving the desired pattern, and the other of which is the so-called "soft" roll 15 which, after running in, conforms to the pattern of the engraved roll. The metal roll or bowl is heated, usually to a temperature of from about 225° F. to about 350° F. The fabric is run between the "nip" of these two rolls and the embossed pattern is thereby imparted thereto.

Simultaneously with the embossing, the color inlay is applied, that is to say, the color which has previously

been applied to the engraved roll 16 is now transferred to the depressions of the embossed fabric, on the face, as will be hereinafter more fully described.

The embossed inlaid fabric 17 is now subjected to heat 18, as, for example, by leading it through a heated oven, the temperature of which is ordinarily from about 250° F. to 400° F., with a residence time of from 8 minutes to 1 minute or less. With respect to the resin solution with which the fabric is initially impregnated, the heating serves to cure such resin, i. e., to harden, set or polymerize it to the insoluble stage, whereby the physical deformation or pattern imparted to the fabric by the embossing calender is fixed and rendered durable to repeated washing and dry cleaning. The functioning of the heat with respect to the color inlay will be later set forth.

Considering, now, the color inlay which is to be made durable to washings and dry cleanings. It is obvious that, to secure durability, the color pigment applied should not be susceptible of physical or chemical removal by washing and dry cleaning. It will also be understood that the pigment must be applied in paste form, as distinguished from a free running solution, for the reason that the color is applied only to the end portions of the elevations on the engraved bowl in a very thin layer of film and must, therefore, have a consistency or viscosity such that it will remain upon such portions without running and with the capability of transfer to the shallow portions of the valleys or depressions in the fabric. (The inlay paste is applied to the calender in advance of the nip of the calender bowls, in the direction of rotation of the engraved bowl, by means of a printing roll or rolls made of rubber or any suitable material.)

We have discovered that if a precondensate of a modified alkyd resin be incorporated in the paste, such resin will secure the desired durability and still be compatible with and/or meet the conditions of the process. In the first place, such resins act not only as a thickener for the paste and a vehicle for the pigment, but also are an effective binder to hold the pigment to the fabric. In the next place, premature or objectionable hardening or polymerization of the resin will not occur on the calender because the time element required is considerably longer than that encountered in the inlaying operation for the hardening thereof. Despite the fact that the paste is applied in a very thin film on the surfaces of the heated calender and therefore tends rapidly to approach the temperature of the calender, we have found that premature hardening or polymerization does not occur prior to or at the time of deposition of the paste onto the fabric, but that the hardening to the insoluble stage only occurs during the heat treatment step following the calendering. Such resin will go in solution or in dispersion along with the pigment, in the paste.

Of the alkyd resins, we prefer to use the soya oil modified type. The amount of alkyd resin in the paste may range from about 10% to about 95% by weight of the paste, although one may go as low as 5% content and secure a fair degree of durability.

As the vehicle for the oil modified alkyd resin in the paste, we may employ an oil, preferably a vegetable oil. Such as oil is compatible with the resin and affects the resin and will also withstand the heat of the calender. (Water as a vehicle is incompatible with such resins, and moreover would tend to undesirably affect the resin.) The film produced by alkyd resins when hardened, is harsh, tough and stiff and would therefore impart an undesirable hand or feel. We have found that the oil counteracts this and renders the film of the resin, when hardened, more soft and flexible. The amount of oil which it is necessary to add to obtain a proper hand is dependent on the amount of modifying oil used when the

resin was initially made. Moreover, the oil also acts as a softener for the fabric, again affecting hand or feel. Therefore, we prefer to use a long oil modified alkyd resin usually with the addition of a small amount of oil. In addition, the oil minimizes if not eliminates the tendency of solids to stick on the calender, which would cause "build up." It is unnecessary to frequently clean the engraved bowl.

Oxidizing or non-oxidizing oils may be used. If oxidizing, they will assist in the final hardening of the resin. Either type of oil will act as a dispersing medium for the solids. The oil may, in part, be replaced by a high boiling point, preferably a non-toxic solvent for the resin, i. e., a solvent which will withstand the temperature of the heated bowl such, for example, as Stoddard Solvent No. 4 (B. P. ca. 300–325° F.), glycerine and the like, of which there are a large number known. The addition of such a solvent is a convenient way for adjusting viscosity. Driers may also be incorporated in the paste, such, for example, as lead naphthenate.

The amount of oil is not critical in the following sense. If oil alone is used as the vehicle, it need only be in an amount sufficient to act as a vehicle for the solids in the paste, holding them in solution or dispersion, as the case may be. Any amount in excess thereof will depend upon the particular hand or feel that is desired in the ultimate finish. If a high boiling point solvent be employed along with the oil, the amount of the oil may be reduced. Similarly, the amount of the oil will depend upon such factors, for example, as whether the modified alkyd resin be of the long type, medium oil or short oil types. Little, or no, additional oil is required with the long oil type, more with the medium type, and still more with the short oil type. Again, it is possible to supplement the oil by incorporating in the paste in the customary small amounts a catalyst, such as copper naphthenate, lead oxide, cobalt oxide, magnesium oxide, zinc oxide, calcium oxide, barium carbonate, barium hydroxide, and the like, these being particularly useful in the case where the oil is of a non-oxidizing type. These catalysts aid in the reactions hardening the resin. Thus, again, the amount and type of oil will depend upon whether or not such catalysts are employed. We have found that in the preferred practice of our invention, from about 2% to 40% of additional oil by weight of the paste will be satisfactory. This upper limit may be somewhat exceeded, depending upon the particular hand desired, but any great excess would merely represent wastage ultimately removed in the washing. The resin, however, does retain oil in the fabric so that softness is not lost. Additionally, softening agents may be incorporated in the paste.

For the added oils we prefer, as stated, a vegetable oil, such, for example, as castor oil, dehydrated castor oil, linseed oil, tung oil and soya bean oil and the like. Synthetic oils, such as dioctyl phthalate, may also be employed.

The alkyd resin in the paste may be additionally modified, and this we prefer to do, by the incorporation in the paste of a compatible solvent soluble thermosetting type of resin, such, for example, as solvent soluble resins of the melamine type, of the urea type, of the epoxy types, and the like, such, for example, as methylol melamine, marketed by American Cyanamide Company, under the trade name, Aerotex 200, butylated urea-formaldehyde, marketed by Rohm & Haas, under the trade name, Uformite F–240, and melamine formaldehyde resin, marketed by Rohm & Haas, under the trade name, Uformite MM–40. These favorably affect the alkyd resins, especially in the direction of improvement in hand and feel and flexibility. It may be that they form copolymers or otherwise affect the reactions. Whatever the reason, they definitely improve the results. The heat treatment following the calendering cures these resins.

Where such resins are used conjointly with the alkyd resins, a conventional catalyst (usually of the delaying action type) is also incorporated in the paste. The amount of the additional resins will vary with the extent it is desired to affect the alkyd resins. A range of from about .5% to about 15% by weight of the inlay paste gives satisfactory results. One would assume that these additional resins would tend to prematurely polymerize on the hot engraved calender bowl, but surprisingly this does not take place, in all probability due to the dispersion of such resin throughout the paste. The amount of such additional resin employed should never be such as to bring about undesirable premature polymerization. The paste will include a high boiling solvent for such resin.

While we prefer the soya bean oil modified type of alkyd resin, other oil modified types are usable, such, for example, as castor, dehydrated castor, linseed, tung, and the like.

The pigment may be any compatible type of organic or inorganic pigment, suitable types of which are well known, such, for example, as titanium oxide, lamp black, chrome yellow, Aridyes, phthalocyanine pigments and vat pigments. Of course, no pigment should be employed which will not withstand the temperature or which is toxic, or which will interfere with the reactions.

It is obvious, of course, that the procedure is applicable to all types of fabric to which it has been customary to impart durable embossed effects through the use of resin, such, for example, as cotton, regenerated cellulose, mixtures thereof, etc.

Example 1

A yellow dyed cellulosic fabric is impregnated with a resin solution such as:

	Parts
Resloom HP–S	40
Resloom M–75G	40
Aerotex H	20
Elvanol 72–60	5
Catalyst AC	10

made to 100 gals. with water, squeezed so as to have a pick-up of about 65%, partially dried, embossed and inlaid with a paste of the following formula (engraved steel roll at 325°–400° F.):

	Parts
Beckosol 1307	80.0
Lead drier 25% Pb	2.0
Cobalt drier 6% Co	0.5
Aerotex P–200	9.0
Methylhydroxypropanolamine hydrochloride	1.0
Pigment paste	7.5
	100.0

The pigment paste above is made up with—

	Parts
Resoform Green GP	20.0
Synthenol	40.0
Beckosol P–96	40.0

After inlaying, the fabric is cured in a conventional ager (roller, loop or air lay) for about 2–3 minutes at from about 300° F. to about 320° F., and then washed and dried. (As is well understood in the textile art, these temperatures and times may be somewhat departed from, as it is known that the lower the temperature the longer the time, and vice versa.)

The finished fabric possesses a novel, textured, clear and distinct multi-colored effect with a crease and soil resistant finish of pleasing hand that is durable to washing and dry cleaning and stabilized to shrinkage.

Both the embossed pattern and the inlay color are durable to repeated washings and/or dry cleanings.

In the foregoing example, Resloom HP–S is a melamine resin, 100% solids, marketed under that name by the Monsanto Chemical Company; Resloom M–75G is a melamine resin, 60% solids, marketed by the same

company under that name; Aerotex H is a cationic softener marketed under that name by American Cyanamid Company; Elvanol is polyvinyl alcohol, 100% solids, marketed under that name by Du Pont de Nemours; Beckosol 1307, 50% solids, is a trade name by which the Reichhold Chemical Company markets a modified soya short oil alkyd resin; Aerotex P–200 is a solvent soluble methylol melamine resin about 60% solids, marketed under that name by American Cyanamid Company; Synthenol is dehydrated castor oil, and Beckosol P–96, 100% solids, is a modified soya long oil alkyd resin.

All parts are by weight in this and the other examples. It will be seen that in the foregoing example there is present in the paste a mixture containing a short oil and a long oil type of alkyd resin, along with a thermosetting resin. The modyfying oil in the alkyd resin constitutes 18.35% by weight of the inlay paste; and the Synthenol constitutes 3% by weight of the paste.

A beautiful embossed fabric with the lower portions green is obtained which has a pleasing hand, appearance and is fast to numerous launderings and dry cleanings.

Example 2

A cellulosic fabric is treated as in Example 1, except that the following inlay paste formula was used:

	Parts
Beckosol 1307	15.0
Aerotex P–200	9.0
Methylhydroxypropanolamine hydrochloride	1.0
Pigment paste	7.5
Synthenol	60.0
Xylol	7.5
	100.0

The pigment paste above was made up with—

	Parts
Lamp black	20.0
Synthenol	40.0
Beckosol P–96	40.0
	100.0

The modifying oil in the alkyd resin constitutes 4.03% by weight of the inlay paste; and the Synthenol constitutes 63% by weight of the paste.

Example 3

A cellulosic fabric is treated as in Examples 1 and 2, except that the following inlay paste is used:

	Parts
Beckosol P–96	85.0
Uformite F–240 (50% solids)	5.0
Methylhydroxypropanolamine hydrochloride	1.0
Pigment paste	5.0
Xylol	4.0
	100.0

The pigment paste of Example 2 was used.

The modifying oil in the alkyd resin constitutes 56.55% by weight of the paste; and the Synthenol constitutes 2% by weight of the paste.

Example 4

A cotton fabric is handled as in Example 1, except that the following inlay paste is used:

	Parts
Beckosol 1307	92.5
Pigment paste	7.5

The same pigment paste as in Example 1 is used.

The modifying oil in the alkyd resin constitutes 39.9% by weight of the inlay paste; and the Synthenol constitutes 3% by weight of the paste.

Example 5

A cellulosic fabric is treated as in Example 1, except that the following inlay formula is used:

	Parts
Beckosol P–96	65.0
Aerotex P–200	11.5
Pigment paste	7.5
Methylhydroxypropanolamine hydrochloride	1.0
Xylol	15.0
	100.0

The pigment paste above was made up with—

	Parts
Resoform Green GP	20.0
Beckosol P–96	75.0
Xylol	5.0
	100.0

The modifying oil in the alkyd resin constitutes 45.8% by weight of the inlay paste; and the Synthenol constitutes 0% by weight of the paste.

Example 6

A cellulosic fabric is treated as in Example 1, except that the following inlay formula is used:

	Parts
Beckosol	82.5
Pigment paste	10.0
Synthenol	7.5
	100.0

The pigment paste above was made up with—

	Parts
Aridye Padding Green	20.0
Synthenol	40.0
Beckosol 31	40.0

The modifying oil in the alkyd resin constitutes 25.9% by weight of the inlay paste and the Synthenol constitutes 11.5% by weight of the paste.

Beckosol 1331, 50% solids, is a trade name by which Reichhold Chemical Company markets a medium oil soya modified pure alkyd resin.

Example 7

A cellulosic fabric is treated as in Example 1, except that the following inlay paste is used:

	Parts
Beckosol 1307	75.0
Dioctyl phthalate	10.0
Pigment paste	7.5
Aerotex P–200	6.5
Methylhydroxypropanolamine hydrochloride	1.0

The pigment paste of Example 2 is used above.

The modifying oil in the alkyd resin constitutes 16.33% by weight of the inlay paste; and the Synthenol constitutes 3% by weight of the paste. The total added oil constitutes 13% by weight of the paste.

Example 8

A cellulosic fabric is treated as in Example 1, except that the Synthenol of the pigment paste was replaced with soya bean oil.

Example 9

A cellulosic fabric is treated as in Example 1, except that the following inlay paste is used:

	Parts
Beckosol 1341	70.0
Soya bean oil	10.0
Pigment paste	10.0
Xylol	5.0
Synthenol	5.0
	100.0

The print paste from Example 1 is used above.

The modifying oil in the alkyd resin constitutes 13.36% by weight of the inlay paste; and the Synthenol 11.5% by weight of the paste. The total added oil constitutes 21.5% by weight of the paste.

Beckosol 1341 is a phenolated alkyd, 60% solids, linseed oil, modified 28% oil, sold by Reichhold Chemical Company.

Example 10

A cellulosic fabric is handled as in Example 1, except that the following inlay paste is used:

	Parts
Glycerol phthalate	33.0
Synthenol	57.0
Aridye printing blue FC–2G	7.5
Lead drier, 25% Pb	2.0
Cobalt drier, 6% Co	0.5
	100.0

The Synthenol constitutes 57.0% by weight of the paste. Glycerol phthalate is 100% solids.

Example 11

A cellulosic fabric is handled as in Example 1, except that Beckosol 1303 is used to replace Beckosol 1307.

Beckosol 1303 is a rosin modified alkyd 34% soya oil modified, 50% solids, sold by the Reichhold Chemical Company.

We claim:

1. Process of producing durable inlay effects in embossed fabrics which comprises applying to the raised relief-imparting surfaces of the engraved bowl of an embossing calender, which bowl is at a temperature ranging from 225° F. to 400° F., a paste which will not harden on the calender roll before being transferred to the fabric, and which has a non-aqueous vehicle, said paste having a consistency to permit retention without running on the heated embossing roll while in contact therewith, and said paste containing as essential ingredients a color pigment, an alkyd resin precondensate in an amount from 5% to 95% by weight of the paste and an oil, then simultaneously embossing the fabric and transferring the applied paste to the depressions in the fabric and heating the embossed fabric to a temperature and for a time sufficient to harden the resin.

2. The process of claim 1 in which an oil modified alkyd resin is used.

3. The process of claim 2 in which the modified alkyd resin is of the long oil type and the oil in the paste is that which is present in such alkyd resin.

4. The process of claim 1 characterized by that the total oil present constitutes from about 2% to about 60% by weight of the paste.

5. The process of claim 1 characterized by that the paste also contains solvent soluble resin of the thermosetting type.

6. The process of claim 1 characterized by that the paste also contains solvent soluble resin of the thermosetting type in an amount from about .5% to 15% by weight of the inlay paste.

7. The process of claim 1 characterized by that the oil is of the oxidizing type.

8. The process of claim 1 characterized by that the oil is of the non-oxidizing type.

9. The process of claim 1 characterized by that the oil is of the non-oxidizing type and by that the paste also embodies a catalyst which catalyzes oxidation.

10. The process of claim 1 characterized by that a high boiling solvent for alkyd resins is incorporated in the paste.

11. The process of claim 1 in which the fabric before passage through the calender is impregnated with a solution of a thermosetting resin and passed through the calender while the fabric is in a moist condition.

12. The process of producing durable inlay effects in embossed fabrics which comprises applying to the raised relief imparting surfaces of the engraved bowl of the embossing calender, which bowl is at a temperature ranging from 225° F. to 400° F., a paste, which will not harden on the calender roll before being transferred to the fabric and in which water is not used as a vehicle, said paste containing as essential ingredients a color pigment and an alkyd resin precondensate in an amount from 5% to 95% by weight of the paste, then simultaneously embosing the fabric and transferring the applied paste to the depressions in the fabric, and heating the embossed fabric to a temperature and for a time sufficient to harden the resin.

13. A durably embossed fabric having a durable inlay effect made in accordance with the proces of claim 11.

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CERTIFICATE OF CORRECTION

Patent No. 2,858,232 October 28, 1958

Henry R. Hushebeck et al.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 62, for "as" read -- an --; column 6, line 28, for "Beckosol" read -- Beckosol 1331 --; column 8, line 37, for "proces" read -- process --; line 50, for the patent number "2,657,426" read -- 2,667,426 --.

Signed and sealed this 3rd day of February 1959.

(SEAL)

Attest:

KARL H. AXLINE

Attesting Officer

ROBERT C. WATSON
Commissioner of Patents