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TEXTILE TREATING METHOD

Joseph Edward Smith, Wilmington, Del., assignor
to E. I. du Pont de Nemours & Company, Wil-
mington, Del., a corporation of Delaware

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This invention relates to the application of positively charged colloidal dispersions to textile fibers and more particularly to the application of positively charged colloidal dispersions to wool, silk, and nylon.

This invention has as an object the application of positively charged colloidal dispersions to textile fibers. A further object is the uniform application of positively charged colloidal dispersions to wool, silk and nylon in the presence of cationic surface active agents or glue. Other objects will appear hereinafter.

These objects are accomplished by the following invention. A positively charged colloidal dispersion having a high affinity for the textile fabric to be treated is dispersed in water. A relatively small proportion of a cationic surface agent or glue is added to the bath. The textile fabric is entered in the bath and agitated under selected conditions of pH and temperature to obtain a uniform deposition of the dispersion on the goods. The addition of the cationic surface active agent or glue to the bath retards markedly the rate of exhaustion of the colloidal dispersion onto the goods.

The preparation of positively charged resin dispersions which exhaust from aqueous baths onto textile fibers including animal fibers, nylon, and cellulosic fibers has been described in my copending applications, Serial Nos. 356,083, 351,084, 351,086, and 351,087, filed on this same day. The preparation of positively charged colloidal dispersions containing paraffin waxes and petroleum hydrocarbons has been described in U. S. Patent 2,047,217. The colloidal dispersions prepared in accordance with these applications and patent must have a high affinity for the textile fabrics to be treated if economical use of the dispersed material is to be obtained. Dispersions with a high capacity for exhausting onto the textile material frequently are absorbed so rapidly as to be deposited unevenly on the surface of the fibers unless steps are taken to regulate the rate of exhaustion. The rate of exhaustion may be regulated in part by control of the temperature and pH of the bath. However, it has been found more desirable and effective to use, in addition, cationic surface active agents as additions to the bath to regulate the rate of exhaustion. The addition of glue to the bath instead of a cationic surface active agent is also effective but is not as desirable as the addition of a cationic surface active agent since it is harder to obtain economical exhaustion of the bath in the presence of the glue.

It is usually the case in treating large batches of textiles that different portions of the goods are alternately in contact with the bath. If exhaustion of the bath is to produce a uniform deposition on the textiles the rate of exhaustion must be relatively slow so that all of the goods are treated in essentially the same concentrations of the dispersion during processing. It also frequently happens that the goods are fairly tightly packed and as a result considerable time is required to produce penetration of the dispersed phase through the mass. It obviously is necessary that the rate of exhaustion be slow enough so that the larger part of the deposition of the dispersed phase does not occur at the surfaces of the textiles first reached. It is of particular value to control the rate of exhaustion of a dispersion during the treatment of hosiery, since it is well known that hosiery becomes badly tangled in the dyeing and finishing operations.

It is essential that a retarding agent, to have its best effect, shall be highly effective in decreasing the rate of exhaustion during the initial stages of exhaustion but shall eventually permit adsorption of the greater part of the dispersion to obtain economical use. The cationic surface active agents described in this invention are ideal for use since at room temperature they exert a strong retarding action yet permit almost complete exhaustion of the dispersion from the bath in 30 minutes. The effectiveness of the retarding agent is decreased with increase in temperature; it is possible completely to exhaust the dispersion from a bath onto animal fibers at temperatures above 100° F. The cationic retarder may be selected from quaternary ammonium compounds, long chain alkyl pyridinium compounds, long chain alkyl betaines, and the condensation product of diethanol amine and stearic acid condensed with dimethyl sulfate. The quantity of cationic agent used may vary from 0.02% to 0.5% and preferably from 0.02% to 0.2% on the weight of the goods. The addition of from 1% to 5% animal glue on the weight of the goods, to the bath, also acts to retard the exhaustion of positively charged dispersions. The use of glue is accompanied by the disadvantage that it is more difficult to obtain complete exhaustion of the bath.

In carrying out the process, the positively charged colloidal dispersion or mixture of dispersions is diluted with water to obtain the desired volume and the cationic surface agent or glue is added in solution to the bath. In applying the dispersion to animal fibers, the pH in the bath

is preferably in the range 4.0 to 6.5 although it is satisfactory to carry out the process under any conditions of pH where the dispersions are stable and have an affinity for the fibers. The goods are entered in the bath at room temperature and agitated until well impregnated; after partial exhaustion of the dispersion from the bath the temperature may be raised if desired to obtain complete exhaustion of the bath.

It is of course understood that if the full effect of the retarding agent is to be obtained the goods and the finishing agent should not both be entered into the bath prior to the addition of the cationic surface active agent.

The following examples are illustrative of the invention but are not intended as limiting it in any way.

EXAMPLE 1

Composition A

	Per cent
Refined paraffin wax	16.7
Acetate of deacetylated chitin	1.3
Basic aluminum acetate	3.0
Acetic acid	0.5
Technical dodecyl diethyl cyclohexylamine sulfate	0.09
Water	78.41

Composition B

	Per cent
Methyl methacrylate	13.9
Partially saponified polyvinyl acetate, Saponification No. 110	1.39
The acetate of deacetylated chitin	0.695
Acetic acid	0.208
Technical dodecyl diethyl cyclohexylamine sulfate	0.014
Water	83.793

0.125 gram of Composition A and 0.125 gram of Composition B were dispersed in water at 85° F. Glue dissolved in water was added to the bath. A number of baths containing from 0.025 to 0.25 gram of glue were prepared and the volume adjusted to 200 cc. The pH in the bath was in each case approximately 4.3. The baths were contained in pint Mason jars. Five gram samples of pure silk piece goods were added to each bath and the baths were agitated in a Launder-Ometer at 85° F. The following table contains a summary of the amounts of the dispersed phase adsorbed by the silk at intervals during a thirty-minute application.

Effect of glue on adsorption—Amount of glue on weight of goods

Time in minutes	0.5%	1%	2.5%	5%	0%	0%	0%
	Percent of dispersed phase exhausted from bath						
5	60	50	30	30	70	65	70
10	70	60	40	40	70	75	90
15	80	70	50	50	85	85	95
20	90	70	50	50	90	90	90
30	95	80	60	50	95	95	95

The glue exerts a retarding action on the adsorption of the dispersed phase by the silk.

EXAMPLE 2

0.125 gram of Composition A and 0.125 gram of Composition B (see Example 1) were dispersed in water at 85° F. in each of seven baths. Varying amounts of stearyl trimethyl ammonium bromide dissolved in water were added to the baths.

The bath volumes were made up to 200 cc. and the experiment continued as in Example 1.

Effect of stearyl trimethyl ammonium bromide on adsorption—Amount of stearyl trimethyl ammonium bromide on weight of goods

Time in minutes	0.02%	0.05%	0.1%	0.2%	0%	0%	0%
	Percent of exhaustion of dispersed phase from bath						
5	50	50	20	15	70	65	70
10	75	60	40	30	70	75	90
15	80	70	60	50	85	85	95
20	90	85	70	55	90	90	95
30	95	95	75	70	95	95	95

The stearyl trimethyl ammonium bromide exerts a marked retarding action on the adsorption of the dispersed phase in the range covered.

EXAMPLE 3

The reaction product of alpha brom stearic acid and trimethylamine (see Example 1 of U. S. Patent 2,129,264) was used in the same manner as stearyl trimethyl ammonium bromide in Example 2. The results are summarized below.

Effect of reaction product of U. S. Patent 2,129,264 on adsorption—Amount of reaction product on weight of goods

Time in minutes	0.125%	0.25%	0%
	Percent of dispersed phase exhausted from bath		
5	40	35	60
10	60	50	75
15	70	65	90
20	90	75	90

The reaction product of alpha brom stearic acid and trimethylamine exerts a marked retarding action on the adsorption of the dispersed phase by the silk.

EXAMPLE 4

A condensation product of diethanol amine and stearic acid reacted with dimethyl sulfate (see U. S. Patent 2,096,749) was used in place of the stearyl trimethyl ammonium bromide used in Example 2.

Effect of condensation product of U. S. Patent 2,096,749 on adsorption—Weight of condensation product on weight of silk

Time in minutes	0.07%	0.15%	0.3%	0%
	Percent of dispersed phase exhausted from bath			
5	50	45	35	60
10	60	65	50	75
15	70	75	70	90
20	80	80	65	90

The condensation product exerts a marked retarding action on the adsorption of the dispersed phase by the silk.

EXAMPLE 5

0.25 gram of Composition A and 0.25 gram of Composition B (see Example 1) were dispersed in water in each of several baths at 85° F. Varying amounts of the condensation product used in Example 4 were added to the baths. The bath volumes were adjusted to 200 cc. and each had a pH of approximately 5.5. The baths were con-

tained in pint Mason jars and a ten-gram piece of pure silk was placed in each jar. The baths were agitated in a Launder-Ometer 15 minutes at 85° F. The temperature was raised to 105° F. and the agitation continued for an additional 15 minutes.

*Effect of condensation product on adsorption—
Amount of condensation product of Patent
2,096,749 on weight of silk*

Time	Temperature Degrees Fahrenheit	0.075%	0.15%	0%
		Percent dispersed phase exhausted from bath		
5 min.....	85	50	Less than 50..	80
10 min.....	85	50	Less than 50..	85
15 min.....	85	70	Less than 50..	95
30 min.....	105	100	95.....	100

The above table illustrates that the action of a retarding agent can be minimized sufficiently to permit complete exhaustion of the dispersed phase from the bath by an increase in the temperature of the bath.

EXAMPLE 6

A batch of 42 dozen pairs of dyed silk stockings weighing 20 lbs. and contained in bags was placed in a Smith-Drum rotary-type dyeing machine containing about 60 gallons of water. A mixture of 8 oz. of Composition A and 8 oz. of Composition B (see Example 1), 0.3 oz. of the condensation product used in Example 4, and 1.6 oz. of 28% commercial acetic acid was diluted with water and added to the machine at 83° F. After agitating for about 15 minutes at 83° F. the bath was about 75% exhausted. The bath temperature was raised to 105° F. and the bath was agitated for 5 minutes to obtain complete exhaustion of the dispersed phase onto the hosiery. The pH of the bath was 6.6. The stockings were extracted, boarded and dried. The hose were well delustered and had a full, soft hand and excellent appearance. The water repellency of the stockings was excellent. The application of the finish was entirely level as judged by the appearance. The dispersed phase was entirely stable during application.

A batch of stockings treated as above without the inclusion of the condensation product used in Example 4 adsorbed all of the dispersed phase from the bath in less than 5 minutes at 83° F. The finish was distributed less evenly than from the previous treatment as could be seen by the fact that uneven delustering was obtained on a number of the stockings. The dispersed phase had a tendency to separate small wax particles during application.

Other cationic surface active agents which may be used in place of those mentioned in the preceding examples are long chain alkyl pyridinium halides, higher alkyl ammonium halides, long chain alkyl sulfonium compounds, and higher alkyl phosphonium halides. The preferred length of the long chain alkyl group is 10 to 18 carbon atoms. The retardant may be added to the application bath prior to, or simultaneously with, the dispersed phase.

The invention is useful in the treating of silk hosiery with exhausting types of dispersions.

The invention is applicable in the exhaustion of dispersions on cotton or viscose process rayon

yarn in a Franklin dyeing machine. The invention is applicable in regulating the rate of exhaustion of positively charged dispersions on wool.

The invention is applicable to the exhaustion of colloidal dispersions or emulsions on tightly packed textile fabrics or to the exhaustion of colloidal dispersions or emulsions on goods treated on a reel machine or a jig.

The present invention offers the following advantages over prior art processes:

(1) The rate of exhaustion of positively charged colloidal emulsions or dispersions onto textile fabrics or materials can be retarded to any desired extent.

(2) Better penetration and a more level application of exhausting types of colloidal emulsions and dispersions on textile fabrics and materials is obtained.

(3) The control of the rate of exhaustion of resin emulsions on silk hosiery makes possible the uniform application of resin delusterants and is a necessary requirement for obtaining commercially acceptable results.

The above description and examples are to be taken as illustrative only. Any variation or departure therefrom which conforms to the spirit of the invention is intended to be included within the scope of the claims.

I claim:

1. In a process for finishing textile materials wherein a textile finish is applied to the material as a positively charged aqueous dispersion from a dilute aqueous suspension thereof by a process of exhaustion, and wherein the finish exhausts onto the textile material at too rapid a rate to give a finish of the desired uniformity, the step which comprises retarding the rate of exhaustion of the finish from the suspension by incorporating in the dilute suspension of the dispersed finish from 0.02% to 0.5% of a cationic surface active agent, based on the weight of the textile material to be treated, the cationic surface active agent being incorporated with the positively charged aqueous dispersion subsequent to the preparation of the latter.

2. In a process for finishing textile materials wherein a textile finish is applied to the material as a positively charged aqueous dispersion from a dilute aqueous suspension thereof by a process of exhaustion, and wherein the finish exhausts onto the textile material at too rapid a rate to give a finish of the desired uniformity, the step which comprises retarding the rate of exhaustion of the finish from the suspension by incorporating in the dilute suspension of the dispersed finish from 0.02% to 0.2% of a cationic surface active agent, based on the weight of the textile material to be treated, the cationic surface active agent being incorporated with the positively charged aqueous dispersion subsequent to the preparation of the latter.

3. In a process for finishing textile materials wherein a textile finish is applied to the material as a positively charged aqueous dispersion from a dilute aqueous suspension thereof by a process of exhaustion and wherein the finish exhausts onto the textile material at too rapid a rate to give a finish of the desired uniformity, the step which comprises retarding the rate of exhaustion of the finish from the suspension by incorporating in the dilute suspension of the dispersed finish from 0.02% to 0.5% of a cationic surface active quaternary ammonium compound, based on the weight of the textile material to be treated.

ed, the cationic surface active quaternary ammonium compound being incorporated with the positively charged aqueous dispersion subsequent to the preparation of the latter.

4. In a process for finishing textile materials wherein a textile finish is applied to the material as a positively charged aqueous dispersion from a dilute aqueous suspension thereof by a process of exhaustion, and wherein the finish exhausts onto the textile material at too rapid a rate to give a finish of the desired uniformity, the step which comprises retarding the rate of exhaustion of the finish from the suspension by incorporating in the dilute suspension of the dispersed finish from 0.02% to 0.5% of a cationic surface active agent, based on the weight of the textile material to be treated, the cationic surface active agent being incorporated in the treating bath separately from the incorporation in such bath of the previously prepared positively charged aqueous dispersion of the finishing agent.

5. In a process for finishing textile materials wherein a textile finish is applied to the material as a positively charged aqueous dispersion from a dilute aqueous suspension thereof by a process of exhaustion and wherein the finish exhausts onto the textile material at too rapid a rate to give a finish of the desired uniformity, the step which comprises retarding the rate of exhaustion of the finish from the suspension by incorporating in the dilute suspension of the dispersed finish from 0.02% to 0.5% of a cationic surface active quaternary ammonium compound, based on the weight of the textile material to be treated, the cationic surface active agent being incorporated in the treating bath separately from the incorporation in such bath of the previously prepared positively charged aqueous dispersion of the finishing agent.

6. In a process for finishing textile materials wherein a textile finish is applied to the material as a positively charged aqueous dispersion from a dilute aqueous suspension thereof by a process of exhaustion, and wherein the finish exhausts onto the textile material at too rapid a rate to give a finish of the desired uniformity, the step which comprises retarding the rate of

exhaustion of the finish from the suspension by incorporating in the dilute suspension of the dispersed finish from 0.02% to 0.2% of a cationic surface active agent, based on the weight of the textile material to be treated, the cationic surface active agent being incorporated in the treating bath separately from the incorporation in such bath of the previously prepared positively charged aqueous dispersion of the finishing agent.

7. In a process for finishing textile materials wherein a textile finish is applied to the material as a positively charged aqueous dispersion from a dilute aqueous suspension thereof by a process of exhaustion and wherein the finish exhausts onto the textile material at too rapid a rate to give a finish of the desired uniformity, the step which comprises retarding the rate of exhaustion of the finish from the suspension by incorporating in the dilute suspension of the dispersed finish from 0.02% to 0.2% of a cationic surface active quaternary ammonium compound, based on the weight of the textile material to be treated, the cationic surface active quaternary ammonium compound being incorporated in the treating bath separately from the incorporation in such bath of the previously prepared positively charged aqueous dispersion of the finishing agent.

8. In a process for finishing textile materials wherein a textile finish is applied to the material as a positively charged aqueous dispersion from a dilute aqueous suspension thereof by a process of exhaustion, and wherein the finish exhausts onto the textile material at too rapid a rate to give a finish of the desired uniformity, the step which comprises retarding the rate of exhaustion of the finish from the suspension by incorporating in the dilute suspension of the dispersed finish from 0.02% to 0.5% of the condensation product of diethanolamine and stearic acid reacted with dimethyl sulfate, based on the weight of the textile material to be treated, the condensation product being incorporated with the positively charged aqueous dispersion subsequent to the preparation of the latter.

JOSEPH EDWARD SMITH.

CERTIFICATE OF CORRECTION.

Patent No. 2,332,817.

October 26, 1943.

JOSEPH EDWARD SMITH.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, first column, line 30, for the serial number "356,083" read --351,083--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed, this 7th day of December, A. D. 1943.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.