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STABLE WATERPROOFING COMPOSITION

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My invention relates to compositions for delustering, waterproofing and finishing textile fibers and fabrics, especially silk and rayon. This application is a continuation-in-part of my application, Serial No. 98,621, filed August 29, 1936, for Textile treating, method and composition.

A purpose of the invention is to produce a stable concentrated emulsion containing an inorganic inert delustrant and a waterproofing agent for textiles, and to dilute the concentrated emulsion as desired to produce a one-bath delustering and water repelling treatment.

A further purpose is to produce a concentrated stable dispersion of an inorganic inert delustrant and a wax, such as paraffin, in the presence of aluminum sulphate or an equivalent, and to dilute the concentrated emulsion to treat textile fibers and fabrics, especially silk or rayon in a single bath.

A further purpose is to subject textile fibers and fabrics, particularly silk or rayon, and more particularly stockings after washing, to a single treatment which will impart superior dull luster, water repellant properties and finish.

A further purpose is to stabilize a concentrated dispersion of an inorganic inert delustrant and/or a wax in the presence of aluminum sulphate or an equivalent by a starch of substantially uniform viscosity over the climatic temperature range encountered.

A further purpose is to avoid any reaction between aluminum sulphate and a stabilizing agent and to render the dispersion stable over a wide temperature range by employing a starch of substantially uniform viscosity over the climatic temperature range to stabilize the dispersion of a delustering, waterproofing and finishing composition.

A further purpose is to treat textile fibers and fabrics with a dispersion including a wax, an inorganic inert delustrant, aluminum sulphate or the equivalent, soap or the equivalent, gelatine or the equivalent, and starch, thereby delustering, waterproofing and finishing the fiber or fabric.

Further purposes appear in the specification and in the claims.

The invention relates both to the methods employed and to the compositions produced.

For the sake of simplicity, the invention will be explained as it is applied to fabrics such as silk or rayon, particularly ladies' stockings.

In the prior practice, where a delustrant and a waterproofing agent were applied to the textile fibers or fabrics in one bath, the bath was made up by separate additions of the delustrant and

the waterproofing agent to the diluted bath. Usually the fiber or fabric was treated with the delustrant bath before the waterproofing agent was added, or vice versa. This prior practice has been unnecessarily time consuming, particularly where a relatively small amount of fiber or fabric was undergoing treatment.

As a result of investigation, it has been found possible, in accordance with the present invention, to make up a thoroughly stable concentrated emulsion which includes both the delustrant and the waterproofing agent, and to dilute this concentrated emulsion at one operation to form a single bath which applies both the delustrant and the waterproofing agent to the fiber or fabric. The dilution is usually made by adding water until the dilute bath is 50 to 400 times the volume of the concentrated emulsion. In any case the dilute bath will have more than ten times the volume of the concentrated emulsion.

A particular application of the present invention is to the delustering, waterproofing and finishing of ladies' silk or rayon stockings which have lost, by repeated washing, the delustering, waterproofing and finish applied by the manufacturer. It is not possible for the housewife to treat the stockings by the methods used at the time of manufacture, because of the complexity, the need of equipment and the special conditions required. But it is possible for the housewife by the present invention to restore the deluster, the waterproofing and the finish in a single bath without complex equipment. The concentrated emulsion of the invention will keep indefinitely in any climate, and satisfactorily treat silk, rayon, wool, cotton, linen, etc., fibers and fabrics, either at the time of manufacture or to restore the properties lost by wear and washing.

Similarly, the invention may be used by the textile manufacturer.

The treating composition is applied to the textile material in the form of a dispersion in water. As sold, the dispersion will be quite concentrated, but for use it may be diluted to say fifty to four hundred volumes or more by adding water. By a concentrated dispersion I mean one which is diluted to at least ten times (ordinarily at least fifty times) the volume of the concentrated dispersion before use. The dispersion contains among its disperse phases an inorganic inert delustrant, and a wax which assists in waterproofing and finishing. Aluminum sulphate or the equivalent is also present to cooperate with other ingredients and assist in waterproofing and finishing.

Soap is very desirably included in the composition, since it is itself an emulsifying agent, and it will combine with the aluminum salt or the equivalent, producing an aluminum or equivalent salt of the fatty acid which also functions as an emulsifying and waterproofing agent and imparts a finish to the fabric.

In order to prevent coagulation or flocculation of the colloid on long standing, to maintain the dispersion stable at the high dilutions employed during use and to prevent precipitation with marked change in temperature, it is important to add a stabilizing agent in addition to the emulsifying agent which assists in forming the dispersion and has the property of keeping the wax and the delustrant in stable dispersion. The presence of the aluminum sulphate or the equivalent makes it much more difficult to obtain a satisfactory stabilizer. Gums of various sorts such as gum tragacanth, which might otherwise be used, react with the aluminum sulphate or the equivalent and lose their efficiency as stabilizers. Emulsifying agents such as glue and gelatine liquefy and lose body at elevated temperatures, and therefore they will not withstand the wide variations in temperature offered by climatic conditions in various parts of the inhabited world.

After extensive experimentation, the present inventor has discovered that certain starches are not affected by aluminum sulphate or the equivalent, and also have substantially uniform viscosity over the temperature ranges encountered in various climates. As the starches include also many substances which flocculate in the presence of aluminum sulphate or the equivalent and which possess markedly varying viscosity over the climatic temperature ranges, it is intended to include only those starches of substantially uniform viscosity over the climatic temperature ranges.

The present inventor has discovered, furthermore, that starches of substantially uniform viscosity over the climatic temperature range have desirable finishing properties, imparting body and sealing to textile fibers and fabrics.

The most satisfactory starch known to applicant for the present purpose is tapioca, since it does not flocculate in the presence of aluminum sulphate or the equivalent and does not substantially change its viscosity over the climatic temperature ranges. It also possesses finishing properties.

The delustrant use should be inorganic and inert to the other ingredients. Organic delustrants (such as zinc or aluminum stearate, oleate or palmitate) can only be dispersed in very small concentrations to produce stable suspensions. As the wax (such as paraffin) tends to increase the luster, the small quantities of organic delustrants which could be placed in stable dispersion are ineffective to overcome the lustering influence of the wax and impart substantial delustering properties to the composition.

Certain inorganic delustrants such as zinc oxide, bismuth oxide, barium sulphide or barium carbonate are not inert, but react with some other ingredient (for example tapioca) and form an insoluble substance which spots the hosiery.

While the invention may be embodied in a wide variety of different compositions, one example of the ingredients and one example of the manner of compounding and the desirable

proportions will be given, recognizing that other compositions and other methods of compounding may be devised by those skilled in the art.

As a delustrant any suitable finely divided insoluble inorganic metallic salt, inert to the other ingredients, may be employed. It is preferred to use titanium oxide (dioxide), barium sulphate or a mixture of the two such as Titanox B. Less satisfactory but commercial results may be obtained with barium oxide, lead oxide, zirconium oxide or lead sulphate.

The delustrant is ground to a suitably fine state of subdivision before incorporating in the treating composition of the invention, and then is dispersed in water in suitable manner, using an emulsifier (machine) for example.

The quantity of delustrant must be more than sufficient to overcome any lustering effect of the wax, where wax is present.

A dispersion of wax is also used in the composition. The wax is preferably paraffin, although any suitable wax such as chlorinated naphthalene, petroleum jelly or petrolatum, Japan wax, montan wax, candelilla wax, ozokerite or carnauba wax might be used. The wax is dispersed in a suitable manner, as for example by melting it, incorporating a protective colloid, and passing the water mixture containing the molten wax through an emulsifier.

Aluminum sulphate imparts considerable acidity, and it is usually desirable to reduce the acidity somewhat by an alkali. Instead of aluminum sulphate, another soluble salt of a polyvalent (having a valence of two, three or more) metal, possessing the property of imparting water-repellency and finish to the textile, may be used. Of course, if the solution of the salt is highly colored or stains the fiber or fabric, it would be objectionable. Among the equivalents which might be used, instead of aluminum sulphate, alum, aluminum acetate, aluminum formate, basic aluminum formate, zinc sulphate, zinc acetate, zinc formate, etc., may be suggested.

A very useful ingredient of the treating composition is soap or the equivalent. Among the soaps which may be used, sodium or potassium stearate, oleate or palmitate is particularly recommended. As an equivalent of soap, a sulfonated oil, a fatty alcohol, a fatty alcohol sulphate, etc., may be employed.

As a protective colloid it is preferred to use gelatine, although glue may be employed instead of gelatine. A preservative is desirably employed along with the gelatine or glue; for example, formaldehyde, benzoic acid or sodium tribenzoate. One of these preservatives is suitably used with the starch, also. The point at which the preservative is added is not critical.

According to the preferred method of compounding and in the preferred proportions, to make about 100 pounds of a mixture used as an intermediate in making the composition, the following ingredients are employed:

Liquid A		Pounds	
Soap	-----	1.0	
Water	-----	20.0	70
Gelatine	-----	5.5	
Water	-----	23.0	
Paraffin	-----	20.0	
Total	-----	69.5	69.5 75

Liquid B

Aluminum sulphate	8.0
Water	20.0
Soda ash	0.5
Water	2.0
Formaldehyde	0.2
Total	30.7

100.2

In Liquid A, the soap solution (1 pound anhydrous soap in 20 pounds of water) is made up separately from the gelatine solution (5.5 pounds gelatine in 23 pounds of water) and the soap and gelatine solutions are mixed preferably at about 160° to 170° F. (61° to 76.5° C.). Melted paraffin (20 pounds) is incorporated and the mixture is passed through an emulsifier until it is thoroughly emulsified.

Liquid B is made up separately by dissolving the aluminum sulphate (8 pounds) in water (20 pounds), separately dissolving the soda ash (0.5 pound) in water (2 pounds) and mixing the soda ash solution and the aluminum sulphate solution. The formaldehyde (0.2 pound) is then added to Liquid B, Liquids A and B are mixed and the mixture is run through an emulsifier.

The emulsified mixture of Liquids A and B is used in making up about 100 pounds of the finished composition:

Emulsified mixture of Liquids A and B	30.0
Tapioca	7.0
Water	50.0
Titanium oxide (dioxide)	5.0
Water	5.0
Benzic acid or formaldehyde	2.0
Total	99.0

The tapioca (7 pounds) is dissolved in hot water (50 pounds), the titanium oxide (5 pounds) is separately made into a paste with water (5 pounds) and the emulsified mixture of Liquids A and B, the starch solution, the titanium oxide paste and the benzoic acid or formaldehyde are mixed together while hot and run through an emulsifier until thoroughly dispersed.

In the finished composition as sold (concentrated emulsion) and before dilution for use, it has been found that the quantity of paraffin or the like should be from about 2 to 10%, preferably about 6%, that of inorganic inert delustrant should be from about 1 to 8%, preferably about 5%, that of aluminum sulphate or the equivalent should be from about 0.5% to 5%, preferably about 2.5%, that of stabilizer should be from about 3 to 10%, preferably about 7%, that of soap or the equivalent should be from about 0.1 to 0.5%, preferably about 0.3% and that of a protective colloid (gelatine) should be from about 0.05 to 0.5%, preferably about 0.2%.

It will of course be understood that the quantities mentioned above may be varied, and, as noted in the claims and elsewhere in the specification, some of the ingredients may be omitted. Other ingredients which do not harmfully affect the composition may be added. The above description of the actual composition as preferably made according to the invention is intended as a specific example merely, subject to variation by those skilled in the art.

The product made in the above manner is a

heavy paste, and may be kept indefinitely in a vessel such as a collapsible tube or jar, notwithstanding the normal climatic temperature changes of the Temperate Zone or the Tropics. Freezing will not harm the composition.

The starch of substantially constant viscosity over the climatic temperature changes stabilizes the dispersion at all temperatures which are encountered and maintains its high viscosity, as well as imparting a finish to the fiber or fabric and acting as a binder for the delustrant. The aluminum salt reacts with the gelatine to give a tough, elastic compound which is soluble but imparts waterproofing properties, and reacts with the soap to give aluminum stearate or other aluminum-fatty acid compound, which is an emulsifying, waterproofing and finishing agent. The paraffin assists in waterproofing and has finishing properties, sealing the fiber or fabric and lubricating it. The gelatine, besides reacting with aluminum to produce a waterproofing compound, also serves as a protective colloid. All of the ingredients serve to seal the individual strands, filaments or fibers.

In compounding the above composition, the formaldehyde may be added with the benzoic acid instead of at the time that Liquids A and B are mixed, if so desired, although if the mixture of Liquids A and B is not to be changed immediately into the finished composition, the presence of formaldehyde in the mixture of Liquids A and B prevents decomposition. Either the formaldehyde or benzoic acid may be omitted if desired.

After the concentrated dispersion is made up as stated, it may be kept indefinitely in a container. Before use the concentrated dispersion will be diluted with water to at least ten volumes, and preferably to fifty or four hundred volumes or more. Where the composition is used to deluster, waterproof and finish stockings, a small quantity, perhaps 3 or 5 cubic centimeters or less of the concentrated emulsion, diluted with water to fifty or four hundred volumes, will satisfactorily treat a pair of stockings, which are simply dipped several times or shaken in the dilute dispersion after they have been washed and rinsed and before they are hung up or boarded to dry.

Some advantages from the present invention may be obtained even though the wax be omitted or even though the delustrant be omitted, but the satisfaction obtained by the user is greatly increased where both the wax and the delustrant are present, along with the aluminum sulphate or equivalent, the starch stabilizer, and desirably the soap or equivalent and the protective colloid.

In view of my invention and disclosure variations and modifications to meet individual whim or particular need will doubtless become evident to others skilled in the art, to obtain all or part of the benefits of my invention without copying the methods and compositions described, and I, therefore, claim all such in so far as they fall within the reasonable spirit and scope of my invention.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent is:

1. A stable concentrated dispersion in water, which on dilution with water serves as a textile-treating composition operating in a single bath, which comprises a wax, an inorganic delustrant inert to the other ingredients, in quantity more

than sufficient to overcome any lustering effect of the wax, and tapioca.

2. A stable concentrated dispersion in water, which on dilution with water serves as a textile-treating composition operating in a single bath, which comprises from 2% to 10% of a wax, from 1% to 8% of an inorganic delustrant inert to the other ingredients, and from 3% to 10% of tapioca.

3. A textile waterproofing composition comprising a stable concentrated dispersion of a wax in water, aluminum sulphate and tapioca.

4. A textile-treating composition containing a disperse phase, a soluble salt of a polyvalent metal possessing the property of imparting water-repellency and finish to the textile, and tapioca.

5. A composition for delustering silk or rayon operating in a single bath and comprising a stable concentrated dispersion in water of from 1% to 8% of an inorganic delustrant inert to the other ingredients, from 0.5% to 5% of a soluble salt of a polyvalent metal possessing the property of imparting water-repellency and finish to the textile, and from 3% to 10% of tapioca.

6. A composition for delustering silk or rayon operating in a single bath and comprising a stable concentrated dispersion in water of from 1% to 8% of an inorganic delustrant inert to the other ingredients, from 0.5% to 5% of aluminum sulphate and from 3% to 10% of tapioca.

7. A delustrant composition for textiles operating in a single bath at dilutions of from 50 to 400 volumes and comprising an inorganic delustrant inert to other ingredients, dispersed in water, from 0.5% to 5% of a soluble salt of a polyvalent metal possessing the property of imparting water-repellency and finish to the textile, soap and tapioca.

8. A delustrant composition for textiles operating in a single bath at dilutions of from 50 to 400 volumes and comprising from 1% to 8% of an inorganic delustrant inert to the other ingredients, dispersed in water, from 0.5% to 5% of aluminum sulphate, from 0.1% to 0.5% of soap and tapioca.

9. A textile delustering, waterproofing and finishing composition operating in a single bath and comprising a stable concentrated dispersion in water of from 2% to 10% of a wax, from 1% to 8% of an inorganic delustrant inert to the other ingredients, from 0.5% to 5% of a soluble salt of a polyvalent metal possessing the property of imparting water-repellency and finish to the textile, from 0.1% to 5% of soap and from 3% to 10% of tapioca.

10. A textile delustering, waterproofing and finishing composition operating in a single bath at dilutions of from 50 to 400 volumes and comprising a stable concentrated dispersion in water of from 2% to 10% of a wax, from 1% to 8% of

an inorganic delustrant inert to the other ingredients, from 0.5% to 5% of aluminum sulphate, from 0.1% to 5% of soap and from 3% to 10% of tapioca.

11. A textile delustering, waterproofing and finishing composition operating in a single bath at dilutions of from 50 to 400 volumes and comprising a dispersion in water of from 2% to 10% of a wax, from 1% to 8% of an inorganic delustrant inert to the other ingredients, from 0.5% to 5% of a soluble salt of a polyvalent metal possessing the property of imparting water-repellency and finish to the textile, from 0.05% to 0.5% of gelatine and from 3% to 10% of tapioca.

12. A textile delustering, waterproofing and finishing composition operating in a single bath at dilutions of from 50 to 400 volumes and comprising a dispersion in water of from 2% to 10% of a wax, from 1% to 8% of an inorganic delustrant inert to the other ingredients, from 0.5% to 5% of aluminum sulphate, from 0.05% to 0.5% of gelatine and from 3% to 10% of tapioca.

13. A delustering, waterproofing and finishing composition for silk and rayon operating in a single bath at dilutions of from 50 to 400 volumes and comprising a dispersion in water of from 2% to 10% of a wax, from 1% to 8% of an inorganic delustrant inert to the other ingredients, from 0.5% to 5% of a soluble salt of a polyvalent metal possessing the property of imparting water-repellency and finish to the textile, from 3% to 10% of tapioca, from 0.1% to 5% of soap and from 0.05% to 0.5% of gelatine.

14. A delustering, waterproofing and finishing composition for silk and rayon operating in a single bath at dilutions of from 50 to 400 volumes and comprising a dispersion in water of from 2% to 10% of a wax, from 1% to 8% of an inorganic delustrant inert to the other ingredients, from 0.5% to 5% of aluminum sulphate, from 3% to 10% of tapioca, from 0.1% to 5% of soap and from 0.05% to 0.5% of gelatine.

15. A textile delustering, waterproofing and finishing composition comprising a stable concentrated dispersion in water of a wax, an inorganic delustrant inert to the other ingredients, in quantity more than sufficient to overcome any lustering effect of the wax, a soluble salt of a polyvalent metal possessing the property of imparting water-repellency and finish to the textile, soap and tapioca.

16. A textile delustering, waterproofing and finishing composition comprising a stable concentrated dispersion in water of a wax, an inorganic delustrant inert to the other ingredients, in quantity more than sufficient to overcome any lustering effect of the wax, a soluble salt of a polyvalent metal possessing the property of imparting water-repellency and finish to the textile, gelatine and tapioca.

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