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SIZING AND FINISHING COMPOSITION

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This invention relates to sizing and finishing compositions, and has particular reference to compositions suitable for treating textile materials, such as natural or synthetic yarns, filaments, fibers, and fabrics of animal or vegetable origin. The invention also relates to a method of producing the compositions.

This application discloses subject matter in common with my application Serial No. 312,229, filed January 3, 1940, which application is a continuation-in-part of application Serial No. 277,889, filed June 7, 1939.

Sizes and finishes are widely used in the textile industry for such purposes as imparting desired body to fibers and to loosely woven cloths, to strengthen and to lubricate them, particularly in the case of warp and weft for weaving operations. Fibers and fabrics properly sized and finished have a more desirable feel, are more resistant to wear and dirt and may be handled with greater facility in many operations.

Many fibers and fabrics, particularly delicate fibers, are advantageously treated with sizing and finishing compositions sufficiently soluble in water to permit subsequent removal thereof by immersing the material in mild aqueous baths. Again, other textile materials exhibit superior qualities when treated with compositions which are not intended to be removed or are insoluble in water, particularly when the material will be subjected to excessive moisture or humid atmospheres. In the case of a large class of organic fibers it is also desirable to apply the composition in an unheated condition. In still other instances, after a definite sizing or finishing characteristic has been imparted to a fiber or fabric, it is desirable to alter this characteristic to meet the requirements of different uses to which the material may be put from that originally contemplated.

It is an object of the invention to provide sizing and finishing compositions that fulfill the above and other requirements, and that have superior and desirable properties in sizing yarns and finishing fabrics.

It is also an object of this invention to provide compositions easily applied to textile material in a cold state, thereby allowing treatment of materials whose fibers are injuriously affected by subjection to high temperatures.

Another object of the invention is to provide compositions particularly adaptable for the treatment of organic derivatives of cellulose, such as viscose rayon fibers and cellulose esters, to render the fibers more amenable to textile operations by increasing their strength, flexibility and suitability for weaving.

A further object of the invention is to provide sizing and finishing compositions which will remain a water-soluble form if desired, so that the composition can be readily washed out of the material in cold aqueous baths.

It is a further object of this invention to provide sizing and finishing compositions having water solubility characteristics which may be modified, if desired, after their application to fibers or fabrics to provide a water-insoluble composition.

In accordance with an embodiment of the present invention, it has been discovered that an unusually effective sizing and finishing composition for treating yarns and fabrics is comprised of urea; an aqueous dispersion of an animal or vegetable protein, preferably a water-insoluble protein dispersible in the dispersing agent mentioned hereinafter, for example, proteins such as casein from milk, soya bean protein, and castor bean protein; and an alkaline dispersing agent selected from the group consisting of ammonia, and derivatives thereof having a pH of 8 or above in a dilute solution, for example, about 1%. Such ammonia derivatives includes amines and quaternary bases, such as the alkylolamines, for example, mono-, di-, and triethanolamine, substituted alkylolamines, for example, diethylethanolamine and phenylethanolamines, aliphatic amines, such as ethylene diamine, diethylene triamine, quaternary bases obtained by substituting one or more of the hydrogens of ammonium hydroxide, for example, tetramethylammonium hydroxide. An organic solvent or thinning agent, having a high boiling point may be added to the sizing and finishing composition to impart a smooth liquid consistency thereto.

A composition of this type may be applied to warp or fabric in a cold condition, thus eliminating the necessity of heating the composition or maintaining it at a critical temperature during application. The composition is a relatively clear solution or dispersion and is free from slimy adhesive qualities often found in compositions containing casein or protein; it dries readily to a smooth and resistant surface, and enables its use with a large class of artificial organic fibers which react unfavorably with sizing and finishing compositions applicable only in a heated condition.

A modification of the present invention, and one that is usually preferred, comprises the addition of a hardening agent, such as an aldehyde, to

the composition above described. If desired, the hardening agent may be mixed in with the other ingredients in the aqueous dispersion or it may be applied after application of the composition to the material to be treated. In most instances it is preferable to include the hardening agent as an ingredient of the composition. If the composition containing the hardening agent as an ingredient thereof is applied, or if the composition without the hardening agent is applied and the hardening agent applied to the sized or finished material in a separate operation, and the material is then dried at a moderate temperature, it remains substantially soluble in water. However, if the fabric thus treated is subjected to temperatures of about 250° F. to 280° F., or above, during drying or later, the constituents of the composition react chemically to render the same insoluble in water.

If it is desired to avoid the use of high temperatures, the composition comprising a hardening agent, which has been applied to the textile material, may be insolubilized by subjecting the treated material to an acid bath treatment at ordinary temperatures which will insolubilize the composition. The bath may be prepared by forming a solution, preferably dilute, of any commercially available mineral or organic acid or acid reacting salt. After immersion in the bath the fabric is preferably washed and soaped to remove excess acid.

Referring to an illustrative example of the sizing or finishing composition of the invention not containing the hardening agent, it is comprised primarily of protein from a vegetable source such as soya bean, or an animal source, such as milk, and an alkylolamine, such as triethanolamine, and urea, which are in thoroughly mixed aqueous solution. An organic wetting or thinning agent may be included.

The following example of a sizing and finishing composition in accordance with the invention is given merely as illustrative. The parts are by weight.

	Parts
Casein	15 to 30
Triethanolamine	10 to 20
Urea	20 to 35
Water	30 to 55
Octyl alcohol	.2 to .4

As a more specific illustration, a composition may contain 92.5 parts of casein, 55 part of triethanolamine, 115 parts of urea, 136.5 parts of water and one part of octyl alcohol. The parts of water may vary from 125 to 150.

Vegetable or animal proteins of the type described of reasonably low ash content, or mixtures of the two, have been found equally adaptable for use as the protein constituent of the sizing composition, but I will refer to casein herein as illustrative.

Of the dispersing agents mentioned, triethanolamine is described as illustrative as it has proven particularly suitable for use in the sizing composition because of its action with the casein in softening and dispersing the same, and its commercial availability. Monoethanolamine is suitable, and if used, a lesser amount is required due to its greater alkalinity. Similarly, when other dispersing agents having a high alkalinity are used, such as some of the ammonium bases, a lesser amount is required to disperse the protein.

The use of a sufficient quantity of the dispersing agent to solubilize all of the casein is important. The casein, which is acidic, is believed to react with the basic dispersing agent to form a caseinate, which is a salt that is soluble in any concentration of water. Accordingly, the composition may be diluted readily to the extent required for its use as a sizing or finishing composition. The amount of dilution, in general, will vary from 4 ounces to 3 pounds of the composition per gallon of water, depending upon the type of product desired.

It is well known that casein is dispersed by urea, but, probably because of the weak basic characteristics of the urea (its aqueous solutions are substantially neutral), the reaction product of casein and urea is not soluble in dilute solutions. Heretofore, it has been usual to form glues and adhesives comprising casein and urea, or casein, urea and a small amount of an alkylolamine, but in such compositions there is not sufficient alkylolamine present to solubilize all of the casein. As a consequence, part of the casein is dispersed by the urea, and compositions of this type may not be diluted to the extent required in a sizing or finishing composition without causing undesirable precipitation.

Although the casein is capable of reacting with both the dispersing agent and the urea in the composition, the reaction is preferential in nature and tends to take place between the casein and the dispersing agent, probably because of the greater alkaline characetristics of this latter material. Accordingly, because sufficient dispersing agent is present in the composition to react with all of the casein, the reaction between these two materials will be substantially complete and substantially all of the urea present will remain in an uncombined state. It will be apparent that in order to insure the presence of sufficient dispersing agent to react with all of the casein, it is possible to add the dispersing agent in excess, but such an excess serves no useful purpose in the composition and therefore is avoided for economical reasons. In general, in order to fully neutralize the casein when triethanolamine is used as the dispersing agent, for example, it is necessary to use from about 60% to 65% of triethanolamine based on the weight of the dry casein. It will be apparent that this amount may vary, depending upon the acidity of the casein. As a specific example, a composition prepared in accordance with the invention may comprise casein and triethanolamine in the ratio of 15.6 grams of casein and 9.5 grams of triethanolamine. The amount of urea added is not regarded as critical.

The urea present in the composition acts as a plasticizer for the caseinate when the composition is used in this form. When a hardening aldehyde is added, the urea reacts chemically with the aldehyde to impart additional and desirable characteristics to the composition, as will be described more fully hereinafter.

One of the higher boiling point alcohols, such as butyl or octyl alcohol, an alkyl ether of a mono- or di-alkylene glycol, an ester or a ketone is preferably added to the composition to insure a smooth liquid consistency. Other ingredients, such as sulfonated wetting agents, emulsifying agents and the like, may be added to the composition, the desirability of these added constituents being determined primarily according to the purpose for which the composition is to be used.

In preparing the sizing and finishing composition of the invention, the protein and ethanolamine are added to the water and mixed. The

urea is then added and the substances thoroughly mixed by a suitable stirring device. The composition thus prepared is soluble in any amount of water, which may be added in amounts determined by the viscosity of the composition which is desired. The composition prepared in accordance with the example given is particularly adaptable for sizing delicate fibers and fabrics, being applicable thereto in a cold state, and easily washed from the woven fabric with wash solution of cold water.

In another preferred embodiment of the invention, in which a hardening agent is employed, a lower molecular weight aldehyde or compound or polymer yielding the aldehyde in the composition is added to the above described composition. Polymers, analogs or other compounds of formaldehyde and of other aldehydes may be used, such as trioxymethylene, paraformaldehyde, hexamethylenetetramine, acetaldehyde, paraldehyde, and crotonaldehyde. Formaldehyde has been found to be particularly suitable as a hardening agent; it is readily available, and will be referred to hereinafter as illustrative. When formaldehyde is used it is gradually added to the composition in the proportion of 100 parts by weight of the above described composition to 20 to 55 parts by weight of a 40% solution of formaldehyde. When other aldehydes or compounds or polymers thereof are used, the proportions may be varied depending on the combining weights.

As the formaldehyde is added to the composition, the protein is at first precipitated therefrom, but upon continued addition of formaldehyde the precipitate redissolves. The addition of the formaldehyde causes an exothermic chemical reaction between the constituents in the aqueous solution. This is indicated by the development of heat and the freedom of the solution from the characteristic odor of free formaldehyde. The formaldehyde is thoroughly stirred in with the other constituents of the composition and the solution set aside until the chemical reaction of the formaldehyde on the constituents of the solution has gone to completion.

It is believed that the formaldehyde combines chemically with the caseinate in the composition to coagulate or polymerize the same and that the reaction is analogous to the reaction of any aldehyde with a caseinate.

The above reaction being preferential in nature, the formaldehyde added to the composition tends to react with the caseinate present until all such caseinate is combined with formaldehyde. Any formaldehyde added in excess of that required to combine with the caseinate may then react with the urea present in the composition to form a urea formaldehyde initial condensation product. In accordance with the invention, it is preferred to add sufficient formaldehyde to the composition to react with all of the caseinate and to add an additional or excess amount to react with the urea. In the example given above of a mixture comprising 15.6 grams of casein and 9.5 grams of triethanolamine, it is necessary to add 15 cc. of formaldehyde to insure that sufficient formaldehyde is present to combine with all of the ethanolamine caseinate. When more than 15 cc. of formaldehyde is added, the excess combines with the urea as set forth above.

The composition formed by the combination of formaldehyde with an alkylolamine caseinate is soluble in dilute solutions in the presence of urea or the urea formaldehyde condensation product. Accordingly, the composition comprising casein and alkylolamine, urea and formaldehyde, formed in accordance with the invention, may be diluted to the extent required for sizing and finishing operations without any appreciable precipitation from the solution.

If the composition is to be compounded prior to use, it is preferable to add enough formaldehyde to react will all of the caseinate and to add the remainder of the formaldehyde, that which is to react with the urea, prior to the application of the composition to the textile.

The amount of formaldehyde to be used depends primarily upon the type of finish desired. The larger the amount, the more permanent will be the finish, particularly in its resistance to washing. The finish obtained by the larger amounts also will have more resilience. The minimum amount of formaldehyde mentioned heretofore will be effective for obtaining a clear solution of all the ingredients and will give a finish that is acceptable for many purposes. The composition containing the smaller amount of formaldehyde is also more stable and resists deterioration, particularly in hot weather. For this reason it may be desirable to make the solution with the minimum amount if it is to stand before using and then to add an additional amount of formaldehyde before the composition is applied to the fabric or yarn.

The sizing or finishing composition may be applied to the fibers or textile fabrics in any conventional manner known to the art, such as brushes or applicator rolls. It can be applied to the material in the cold state and may be dried thereon at ordinary temperatures or by moderate heating at preferably not over a temperature of 150° F. The fiber or fabric thus treated carries the size or finish in a water-soluble condition, and the material may be washed substantially free therefrom.

When it is desired to impart to the material a permanent finish that is substantially water resistant, the composition containing the hardening agent is applied as above described. The treated material is then heated to a temperature of about 250° F. to 280° F. or above, in a suitable drying chamber or by the application of an iron or mangle. This heating step apparently induces a chemical reaction in the composition on the treated material which renders the composition fixed to the treated material in a water-insoluble condition. That a reaction takes place is indicated by the fact that formaldehyde is liberated during the heating.

A modification of the method of insolubilizing the textile material impregnated with a composition formed in accordance with my invention is to subject the impregnated material prepared as described above to the action of an acid bath. In this manner hardening of the finish on the product is accomplished at ordinary temperatures and does not require expensive heating equipment. However, the material which has been treated in an acid bath should be soaked subsequently and washed to remove excess acid and because of the difficulties involved in this step, the hardening method utilizing an acid bath is not preferred. A suitable hardening acid bath may be formed by preparing a dilute solution of a mineral or organic acid or acid reacting salt, such as hydrochloric acid, acetic acid, and the like. In general, any acid reacting medium will harden the composition prepared in accordance

with the invention and the exact nature of the bath is not regarded as critical.

Another significant modification of the process includes the treatment of the material with a composition not containing formaldehyde, after which the formaldehyde may be applied to the sized or finished material to react chemically with the composition in situ instead of being added originally to the composition. The treated material may then be subjected to the heat or acid conversion processes above described, if a water-insoluble size or finish is desired.

Thus it is possible in accordance with the invention to apply directly to the yarn or fabric a size or finish containing or not containing a hardening agent depending on the final properties desired in the sized or finished material. But if the size or finish not containing the hardening agent is used and it is later desired to alter its properties, the sized or finished material may be treated with formaldehyde to yield a sized or finished material which has different properties. If this size or finish in turn is to be insolubilized, the material can be treated as described. This flexibility is a very desirable feature since it enables a single composition to be used to obtain a variety of sized or finished effects.

The composition is essentially a sizing or finishing composition which, when used in treating yarns, is applied upon the surface of the yarns, for example, to form a smooth surface and facilitate weaving operations. When the composition is used in treating woven or knitted fabrics, it is applied on the surface of or in between the yarns or threads of which the fabric is composed, for the purpose of filling the fabric, rendering it more resistant to dirt and imparting a desirable "hand."

The composition also may be mixed and is entirely compatible with starch in any proportion. The starch may be added for the purpose of obtaining a modified finish or for achieving greater economies. The starch is preferably first gelatinized in any convenient way and then mixed with the composition. The viscosity of the gelatinized starch should be such that upon mixing with the composition the desired viscosity of the mixture will be obtained. The starch is more permanent in such a finish than is usually the case in starch-containing finishes. As an explanation of this, it is quite probable that a thin film of the composition is formed around the starch granules and assists in preventing their removal.

While the compositions of the present invention have been described with reference to specific ingredients, it is to be understood that other ingredients, which are the equivalent of those set forth, may be used in preparing the size. It is my intention to include all equivalent ingredients and steps in the process of this invention to be within the scope thereof as set forth in the appended claims.

I claim:

1. A composition of matter comprising urea; an aqueous dispersion of a water-insoluble protein soluble in the hereinafter mentioned alkaline dispersing agent; and an alkaline dispersing agent selected from the group consisting of ammonia, and derivatives thereof consisting of lower aliphatic amines and quaternary ammonium bases having a pH value of not less than 8 in dilute solution, which alkaline dispersing agent is in an amount sufficient to disperse substantially all of said protein.

2. A composition of matter comprising an aqueous dispersion containing urea; a water-insoluble protein soluble in the hereinafter mentioned alkaline dispersing agent; an alkaline dispersing agent selected from the group consisting of ammonia, and derivatives thereof consisting of lower aliphatic amines and quaternary ammonium bases having a pH value of not less than 8 in dilute solution, said alkaline dispersing agent being in a quantity sufficient to disperse substantially all of said protein, and said composition being reacted with an aldehyde selected from the group consisting of formaldehyde, acetaldehyde, crotonaldehyde, and compounds and polymers yielding these aldehydes in the composition, said aldehyde being in a quantity sufficient to combine with substantially all of the reaction product of the protein and dispersing agent.

3. A process of producing a composition of matter which comprises dissolving in water a water-insoluble protein soluble in the hereinafter mentioned alkaline dispersing agent; urea; and an alkaline dispersing agent selected from the group consisting of ammonia and derivatives thereof consisting of lower aliphatic amines and quaternary ammonium bases having a pH value of not less than 8 in dilute solution, said alkaline dispersing agent being in a quantity sufficient to disperse substantially all of said protein; adding an aldehyde selected from the group consisting of formaldehyde, acetaldehyde, crotonaldehyde, and compounds and polymers yielding these aldehydes in the composition, said aldehyde being in an amount such that the reaction product of the protein and dispersing agent is precipitated and subsequently redissolved so that all of the ingredients are in solution.

4. A composition of matter comprising urea, an aqueous dispersion of a water-insoluble protein soluble in ammonia, and a sufficient quantity of ammonia to disperse substantially all of the said protein.

5. A composition of matter comprising urea, and an aqueous dispersion of casein reacted with a sufficient quantity of ammonia to disperse substantially all of the casein.

6. A composition of matter comprising urea, an aqueous dispersion of a water-insoluble protein soluble in the hereinafter mentioned lower aliphatic amine, and a lower aliphatic amine having a pH value of not less than 8 in dilute solution, which amine is in an amount sufficient to disperse substantially all said protein.

7. A composition of matter comprising urea, an aqueous dispersion of casein reacted with a sufficient quantity of a lower aliphatic amine having a pH of not less than 8 in dilute solution to disperse substantially all of the casein.

8. A composition of matter comprising urea, an aqueous dispersion of a water-insoluble protein soluble in the hereinafter mentioned quaternary ammonium base, and a quaternary ammonium base having a pH value of not less than 8 in dilute solution, which base is in an amount sufficient to disperse substantially all of said protein.

9. A composition of matter comprising urea, an aqueous dispersion of casein reacted with a sufficient quantity of a quaternary ammonium base having a pH of not less than 8 in dilute solution to disperse substantially all of the casein.

10. A composition of matter comprising an aqueous dispersion containing urea; a water-insoluble protein soluble in the hereinafter mentioned alkaline dispersing agent; an alkaline dis-

persing agent selected from the group consisting of ammonia, and derivatives thereof consisting of lower aliphatic amines and quaternary ammonium bases having a pH value of not less than 8 in dilute solution, said alkaline dispersing agent being in a quantity sufficient to disperse substantially all of said protein, and said composition being reacted with acetaldehyde in a quantity sufficient to combine with substantially all of the reaction product of the protein and dispersing agent.

11. A process of producing a composition of matter which comprises dissolving in water a water-insoluble protein soluble in the hereinafter mentioned alkaline dispersing agent; urea; and an alkaline dispersing agent selected from the group consisting of ammonia and derivatives thereof consisting of lower aliphatic amines and quaternary ammonium bases having a pH value of not less than 8 in dilute solution, said alkaline dispersing agent being in a quantity sufficient to disperse substantially all of said protein; and adding acetaldehyde in an amount such that the reaction product of the protein and the dispersing agent is precipitated and subsequently redissolved so that all of the ingredients are in solution.

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