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VISCOSE COATING AND FILLING

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The present invention relates to filled or coated textiles, and to a process for producing the same; more particularly, it is concerned with the provision of a "permanent" filling or coating upon a relatively light weight, open construction, woven cotton fabric.

It has long been known that fibrous textile materials can be filled or coated by means of viscose solutions, and that such fillings or coating are "permanent"—that is to say, are so tenaciously retained by the textile material as largely to persist upon the latter for its natural "life". However, the use of viscose, alone or with known additions (as for example, a mineral "filler") in the manner heretofore followed, has not always afforded the most desirable results. For instance, when used in thin solutions, usually of relatively low cellulose content (e. g., a viscose content equivalent to about 4 to 3% cellulose content) the viscose dressing has unduly penetrated the yarn of the fabric, producing a thin filling and an undesirable stiffness of the product. Such pronounced penetration of the yarn by the viscose tends to leave any mineral filler of the dressing composition on the outside of the fabric and insufficiently bonded thereto after the fixation of the viscose by the small amount of regenerated cellulose which has not penetrated into the yarn. Heavier (i. e., thicker) solutions of relatively high cellulose content—while less penetrative of the yarn and, therefore, productive of a more nearly complete filling of the fabric—yield a heavy "boardy" non-extensible finish which generally is subject to cracking in an unsightly manner when stretched or flexed. Furthermore prior fillings or coating on textiles have not always possessed the desired resistance to washing or laundering.

An object of the present invention is to provide a process for producing, from a relatively light weight, open construction fabric, a filled fabric which (in comparison with the original material) is heavy yet soft and pliable, has the appearance of being more closely woven, and has a pleasant "mellow" feel—all of which qualities persist through repeated launderings. A second object is to provide a pleasingly soft coated fabric having a permanent high luster, the dressing of which may be more or less transparent, insoluble in water and in the aqueous media used in laundering, and extensively and freely pliable without detrimental cracking. A third object is to provide upon an extremely light weight and open fabric, such as tobacco cloth, a highly

transparent, pliable and flexible coating which possesses a high permanency toward water.

It has now been found that if substances suitable in nature and in amount, such as certain water soluble organic or inorganic materials—among which, glucose, sodium chloride and ammonium acetate may be mentioned—are added to a filling or coating composition consisting essentially of an aqueous viscose solution in amounts necessary to produce a suitable partial coagulation of the viscose and the viscose mixture in this transitory, induced critical state is applied and fixed on a textile material in a known manner, a filled or coated textile is produced which possesses very superior qualities. In the case where the object is primarily that of filling a low grade textile to enhance its fullness, weight and appearance, these superior qualities include: (a) a pleasant, soft, flexible feel or handle; (b) an appearance in nature entirely similar to that of a high grade but unfilled textile; (c) a good extensibility, as shown by freedom from cracks when stretched; (d) a high degree of permanency toward repeated laundry washings. In the case where luster or brilliancy is a primary or added consideration, it is readily possible to produce coated or filled textiles which possess in addition to the above mentioned qualities a high brilliancy and luster of good permanence. In the case where it is desired to produce a transparent coating such as upon a very open or low grade textile or textile fabric, the qualities conferred by the process include: (a) a highly uniform transparent brilliant coating; (b) a high pliability and suppleness; (c) an excellent permanence toward water.

Partial coagulation of the viscose filling or coating mixture by heat or aging at room temperature or above or below, is difficult to control, is irreversible, and filled or coated textiles produced therefrom lack certain merits, conferred by the presence of a coagulant. Thus the presence in the dressing of the coagulating agent used to produce the aforesaid incipient coagulation favorably modifies the action of the fixing bath (e. g., solution of acid, acid salt, or the like), giving in part to the product produced the desirable properties previously mentioned. Also, during an acid fixation of the viscose, sulphur-containing compounds are much more easily removed, thereby greatly facilitating any subsequent bleaching process. The amount of reagent required to bring about a certain degree of coagulation of the viscose will depend upon the chemical and physical nature of the reagent and also

upon the quality, temperature and age of the viscose. While the action of the added coagulating or precipitating agent is mainly physical in nature, it may be accompanied by chemical effects such, for instance, as the displacement of ammonia from added ammonium salts by interaction with caustic soda present in the viscose solution. The fact that the partial coagulation induced by added reagents is often reversible between limits, is of no small practical advantage in manufacture.

In the preparation of a viscose filling or coating mixture according to the present invention, a coagulating agent is added to the viscose mixture until a degree of coagulation and thickening is achieved, such that the desired distribution of deposited regenerated cellulose between the exteriors and interiors of the yarns and fibers of the textile will occur when the filling or coating composition is applied and fixed in a known manner. What distribution or positioning of the coating or filling is desired, will depend in a measure upon the textile and its proposed uses. Where the permanent filling of a low grade fabric is the primary object, partial coagulation is brought to such a point that the viscose filling mixture penetrates into the yarns and fibers of the fabric only enough to firmly secure the filling to them and not enough to render the fabric unpleasantly harsh and stiff, while on the surfaces of the yarns there remains ample substance to firmly bond talc or particles of a like filler to the fabric. By working in this manner with an incipiently coagulated viscose, a filling composition of 3 to 4% cellulose content may be used to produce a product which is completely filled, yet has a pleasant handle—not too firm—and is extensible and flexible without cracking or splitting of the filling, and these properties remain substantially unchanged through repeated laundry washings. These properties are enhanced if, prior to fixing, the filled or coated fabric is heated and/or dried, whereby coagulation is slowly completed and a regeneration of cellulose begins due to the accelerated action of the added coagulating agent at the higher temperature and as its concentration is increased during drying. The use of a non-coagulated viscose of the equivalent thickness and viscosity would require a cellulose content in the neighborhood of 7-9% and would lead to the production of a heavy, stiff, non-extensible filling, subject to unsightly cracks. Without incipient coagulation, the use of a viscose solution of 3-4% cellulose content would yield, due to excessive penetration, a stiff, harsh product with any filler insecurely bonded to it and, therefore, possessing poor washing qualities. The devices of the prior art have proved inadequate to remedy the foregoing defects to a substantial degree. The incorporation of such foreign materials as soaps, oils, fats, waxes, and softeners have proved generally inadequate. There may be used an insoluble metallic soap such as that produced according to the process of our co-pending application Serial No. 283,734 in conjunction with the incipiently coagulated viscose filling or coating mixture described in the present invention.

When the production of silk-like or lustrous effects is an added or primary object the present invention is particularly applicable. The ready control of the positioning and distribution of the regenerated cellulose coating or filling with respect to the supporting textile which the process affords, makes possible the production of highly

lustrous, thin, flexible, extensible, coatings whose properties persist through numerous washings. Luster or silk effects are usually produced in the absence of a filler such as talc.

Where the production of a transparent coating or filling on a very low count open fabric is desired, the present invention is also of especial application. By the use of partially coagulated viscose a thickness and viscosity can be imparted to the coating mixture, even though it be of only 3-4% cellulose content, such that large interstices between yarns are readily bridged when the coating mixture is spread and fixed on the cloth in a known manner. There results a fabric covered with a thin, uniform, flexible, highly transparent coating which possesses good resistance toward water. And since all the openings or interstices between the yarns have been closed, the fabric is rendered substantially impervious and substantially air-tight. By water-proofing this material in a known manner the fabric is readily rendered impervious so that aqueous solutions as well as vapors and solids cannot pass through it.

Among operable coagulating or precipitating agents are: glucose, monohydric alcohols such as ethyl alcohol and methyl alcohol, polyhydric alcohols such as glycerine and glycol, sugars, weak acids typified by acetic acid and water-soluble salts, including chlorides, nitrates, acetates, thiocyanates and sulfates of the alkali and alkaline earth metals. It is preferred to use, however, for many purposes sodium chloride or ammonium acetate as coagulating agent because of their relative cheapness and efficiency. The viscose dressing mixture is suitably partially coagulated by the addition of one or more of such coagulating or precipitating agents or the like and when in this critical state, applied in a known manner to the fabric after which the viscose readily may be completely coagulated by heat, steam, or treatment with appropriate acid or acid salt solutions or the like.

Various modifications of known filling and coating technique may be used in conjunction with the present invention. Limits to the concentration and composition of viscose plus coagulant filling or coating mixtures are not set and may be varied between fairly wide limits and yet produce desirable results. The degree to which the viscosity is increased by the coagulating agents may be varied to best suit the fabric to be filled. Unbleached, non-absorbent fabrics need not have the filling coagulated to such an extent as when in the bleached or absorbent state. We have observed that in many cases the coagulation treatment should increase the time it takes a ball to fall a given distance, twenty to thirty times. However, in many instances the filling mixture must be so much thickened that even a large (1" diameter) heavy ball will not fall through it. In no case, however, is coagulation carried so far that a glassy, non-sticky filling or coating composition is placed upon the goods. In the following examples are given a limited number of representative applications of the invention.

Example 1

The viscose used in this example (also those following) is made as follows: 25 lbs. bisulphite wood pulp is impregnated for 3 hours with caustic soda (18.5% NaOH) at 21° C.—pressed to 77 lbs. and disintegrated for 2 hours at about 20° C. After about 14 hours the soda cellulose is sul-

phurized with 12 lbs. carbon bisulphide for 5 hours at 22° C. Water and caustic are added until a solution containing 10% cellulose and 4.6% NaOH (on the pulp) is obtained. For this example, the viscose is aged for 70 hours at about 12° C. (This aging is not necessary but may be convenient.) 25 lbs. of the above 10% viscose is mixed with 25 lbs. talc wet out with 35 lbs. water and 90 lbs. sodium chloride solution (containing 14 lbs. NaCl) added slowly, making a 4% cellulose solution. The mixing is continued at a suitable temperature until the mass thickens. The viscosity is taken with a 1" solder ball weighing 64.2 grams. The time required to fall 6" is about 60 seconds. To fall the same distance in a 4% viscose solution containing an equal amount of talc, but no NaCl, it takes 2 seconds. The fabric (68 x 72 4.75)—print cloth—having been boiled, bleached and dried, is filled on a backfilling machine, dried, fixed with a 5% H₂SO₄ solution at about 60° C., for a few minutes, then washed, dried and finished.

Example 2

Into 25 pounds of 10% viscose solution there is added and thoroughly mixed 5 lbs. of talc which has been wetted out with 5 lbs. of water. To this mixture there is slowly added, with agitation, a solution of 1.5 lbs. of calcium thiocyanate (B. P. 135° C.) 1 lb. of acetic acid 28%, and 25 lbs. of water. The resulting thick mass containing about 4% of cellulose is applied to the fabric (bleached or unbleached) by means of a backfilling machine, dried, fixed in a 5% sulfuric acid bath, washed, bleached and finished.

Example 3

To 25 lbs. of 10% viscose and 5 lbs. of talc $\frac{3}{4}$ lb. of calcium chloride dissolved in 30 lbs. of water is slowly added and the resulting thickened mixture is applied as indicated in Example 2.

Example 4

25 lbs. of 10% viscose is mixed with 40 lbs. of water containing 1.5 lbs. of ammonium thiocyanate. After a short time (say, 1 hour) the mixture begins to thicken. The viscosity is taken with a $\frac{3}{8}$ " steel ball weighing 3.13 grams, falling 11 $\frac{1}{3}$ ". The time required is 387 seconds. In a 4% viscose solution containing no ammonium chloride, this ball fell in 17 seconds. The mixture is then spread upon cloth, dried, fixed with sulfuric acid, washed, and run through a solution of hot sodium sulphide (to desulfurize it). On drying under tension there results a transparent film which may be kept flexible by an application of some hygroscopic agent such as glycerine.

Example 5

25 lbs. of 10% viscose is mixed with 40 lbs. of water containing 2 lbs. of ammonium acetate, and the resulting mixture is treated and used in the manner set out in Example 7.

In connection with the foregoing examples, it is to be noted that it may be found desirable for certain purposes, to further modify the fillings or coatings by incorporation therewith of oils, fats, waxes, soaps, sulfonated oils or fats, glycerine, softeners, pigments, dyestuffs, metals, minerals, wood pulp and other substances whose use is known to the art.

It appears that the present invention involves two factors which result directly or indirectly from the use of a viscose filling or coating mixture

incipiently coagulated or precipitated to a critical degree by the use of a coagulating or precipitating agent—suitable in nature and amount, and contained in said mixture. First: the positioning of the final deposit of regenerated cellulose with respect to the supporting textile, and any additional filler, e. g., talc, pulp, etc., is under accurate and easy control and, therefore, may be readily varied as desired, to give the most advantageous results. Second: the presence and effect of a coagulating or precipitating agent in the filling or coating composition favorably modifies the physical characteristics of the final deposit of regenerated cellulose obtained therefrom. What results are considered advantageous and what characteristics particularly to be desired, will depend in a large measure upon the product which is to be produced and such considerations have already been dealt with at length in the preceding paragraphs.

We claim:

1. In a process for the production of a permanently filled or coated textile, involving the operations of applying to the textile a dressing of a composition consisting essentially of cellulose xanthate and thereafter fixing the dressing on the textile by regenerating cellulose from the xanthate of cellulose, the improvement which consists in applying to the textile, while in a state of incipient coagulation, a dressing composition consisting essentially of a solution of cellulose xanthate containing an added coagulating agent, said solution having a cellulose content of from about 3% to about 4% and a viscosity at least equal to that of an untreated and unaged cellulose xanthate solution having a cellulose content of from 7% to 9%.

2. In a process for the production of a permanently filled or coated fabric involving the operations of applying to the fabric a coating of a composition consisting principally of viscose and thereafter fixing the coating on the fabric by regenerating cellulose from the viscose, the improvement which consists in applying to the textile, while in a state of incipient coagulation, a dressing composition consisting essentially of a viscose solution containing an added coagulating agent, said solution having a cellulose content of from about 3% to about 4% and a viscosity at least equal to that of an untreated and unaged cellulose xanthate solution having a cellulose content of from 7% to 9%.

3. Process for the production of a permanently filled or coated textile material which comprises applying to the textile material a coating of a composition consisting essentially of viscose incipiently coagulated by the presence and effect of a previously added coagulant, said viscose having a cellulose content of from about 3% to about 4% and a viscosity at least equal to that of an untreated and unaged viscose having a cellulose content of from 7% to 9%, and thereafter fixing the coating by completing the coagulation of the viscose and regenerating cellulose therefrom.

4. Process for the production of a permanently filled or coated fabric which comprises preparing a coating composition containing cellulose xanthate and of about 3% to 4% cellulose content and a coagulating agent in amount insufficient to effect precipitation of the cellulose xanthate but sufficient to materially increase its tendency to precipitate and to increase the viscosity of the xanthate to at least that of an untreated and unaged cellulose xanthate having a cellulose content of from 7% to 9%, applying a coating of the

composition to the fabric while the cellulose xanthate is in a state of incipient coagulation and fixing the coating by regenerating cellulose therefrom.

5 5. Process as defined in claim 4, characterized in that the coagulating agent is a member of the group consisting of soluble ammonium salts and soluble thiocyanates.

10 6. Process for the production of a permanently filled fabric which comprises preparing a dressing composition containing from 4% to 3% of cellulose in the form of viscose and a suitable water-soluble salt in an amount sufficient to increase the viscosity to at least that of an untreated and unaged cellulose xanthate having a cellulose content of from 7% to 9% and to materially increase the tendency of the cellulose to precipitate but insufficient to cause its complete precipitation, applying a coating of the composition to the fabric, and fixing the coating by a regeneration of cellulose therefrom.

7. As a new product, a pliable and flexible fabric of open construction the fabric having its interstices bridged and substantially filled by a water-insoluble continuous film of coating consisting essentially of regenerated cellulose adhering tenaciously to the surface of the fabric and extensible and flexible with the fabric without cracking or splitting, which product is producible by applying to a surface of the textile, while in a state of incipient coagulation, a dressing composition consisting essentially of a solution of cellulose xanthate containing an added coagulating agent, said solution having a cellulose content of from about 3% to about 4% and a viscosity at least equal to that of an untreated and unaged cellulose xanthate solution having a cellulose content of from 7% to 9%, and regenerating cellulose from the dressing composition in situ.

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