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COMPOSITION AND PROCESS FOR TREATING FIBROUS MATERIALS

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The invention relates, in general, to the processing of textile materials, and in particular, to the treatment of textile materials during their various steps of fabrication and to correlated improvements in compositions employed in such treatments.

In the manufacture of textile yarns, threads, fabrics and the like from animal, vegetable or synthetic fibers, many mechanical and chemical steps are involved. In order to prepare the textile materials for such steps and in the process of carrying out such steps, these materials are treated with various compositions which invariably contain, among other components, sulfonated fatty substances, such as fatty acids, fatty oils, fats, waxes and the like.

The function of these sulfonated products is to provide emulsifying, dispersing, wetting, penetrating and like properties to the various compositions or baths normally employed in the fabrication or processing of textile materials. In the fabrication of natural silk, for example, the raw silk fibers normally contain, or are coated with, a natural resinous or gummy mass (sericin) which must be softened or plasticized in order to handle the same. Such softening is usually carried out by soaking the natural silk fibers in an oil soaking bath which in many instances contains a sulfonated fatty material. This soaking step also serves to coat the fibers with a lubricant which is necessary during the weaving, knitting or other fabricating steps. After the weaving, knitting or other step, the fabricated product is subjected to a boil-off in a bath containing a soap and a penetrating agent such as a sulfonated fatty material. Further, in the bleaching, dyeing, finishing and like steps, sulfonated fatty materials are employed as penetrating, wetting, dispersing and like agents.

In the processing and fabrication of artificial silks, such as rayon, acetate yarns, etc., lubricants are employed which are preferably emulsifiable so that they may be removed after weaving or knitting by scouring. Sulfonated fatty materials find use as emulsifying agents in such lubricants which may be applied as coning oils, emulsified oil baths, sprays or the like. Cotton and woolen and other animal and vegetable fibers are also processed in a somewhat similar manner. The bleaching, dyeing and finishing operations usually call for the use of sulfonated products as aforementioned.

While the sulfonated fatty materials heretofore produced have found rather extensive use in the processing and fabricating of textile materials,

many difficult problems with commensurate disadvantages have resulted therefrom. Sulfonation products of fatty animal and vegetable materials, such as, fatty acids, fatty oils, fats, waxes and their derivatives, as now produced commercially, contain large quantities of moisture and inorganic salts, which it has been found are the prime causes of the aforementioned problems and resulting disadvantages. The moisture or water present in normal sulfonated products result, in part, from the reaction effected during the sulfonation step and, in part, from that introduced during the washing, panning, neutralizing and the other conventional steps employed in the preparation of these products. The inorganic salts are introduced during the same steps and especially during the neutralizing step.

It has been found that the relatively high water and inorganic salt content of sulfonated products of the aforementioned type is undesirable and detrimental for various reasons. The high water content of these products greatly reduces their range of application and adds to the difficulty of their blending with non-sulfonated oils and/or other substances with which they are incorporated for textile treatment processes.

The inorganic salt content of the sulfonated products greatly reduces the stability of emulsions produced by use of such products, thereby deleteriously affecting the textile materials being treated. By catalytic action the salts also cause the oils to hydrolyze and to oxidize more rapidly and thereby considerably reduce their utility as oils used in the textile industry must show a minimum tendency to oxidize. It has also been found that the inorganic salts crystallize out on the textile fibers, thereby imparting a harsh feel and other undesirable properties to the material treated.

The presence of inorganic salts also deleteriously affects the processes of treating textile materials as well as the treated materials. For example, in the application of a lubricant containing the conventional sulfonated material, such as coning oil, wherein the oil is applied to the yarns by means of a wick, the salts crystallize out in and on the wick, thereby clogging the same and preventing the normal supply of oil to the yarn being treated. Various other difficulties have been encountered in the processing and fabrication of textiles which have limited the use of the otherwise desirable sulfonated fatty materials.

The general object of the invention is to obviate the foregoing and other disadvantages.

A specific object of the invention is to provide improved textile treating compositions containing sulfonated animal or vegetable fatty materials.

Another specific object of the invention is to provide improved processes for treating textile materials to impart enhanced lubricating, softening, dyeing, finishing, etc., characteristics thereto.

A further object of the invention is to provide an improved textile lubricating and softening composition.

An additional object of the invention is to provide an improved coning oil.

Another object of the invention is to provide an improved silk soaking composition.

Another object of the invention is to provide an improved composition for finishing textile materials.

A further object of the invention is to provide an improved process for lubricating textile materials that may be easily scoured.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

It has now been discovered that the foregoing and other objects may be realized by the use of substantially anhydrous and inorganic salt-free sulfonated products in the processing and fabrication of textile fibers, yarns, etc., into woven, knitted, etc., products. According to the invention the process comprises treating textile materials with compositions comprising substantially anhydrous and salt-free sulfonated fatty materials. These compositions may, according to the invention, include various other ingredients, such as animal, vegetable or mineral oils, fats, waxes, fatty alcohols, etc., as well as other known materials used in such compositions.

The invention accordingly comprises the several steps and relation of one or more of such steps with respect to each of the others, and a composition of matter possessing the characteristics, properties and relation of components, all of which will be exemplified in the following detailed disclosure, and the scope of the invention will be indicated in the claims.

The substantially anhydrous and salt-free sulfonated products referred to and employed in the present invention may be produced according to any suitable process which will result in a sulfonated product that is substantially free from water and inorganic salts. These products are, however, preferably prepared according to the processes disclosed in any of the following co-pending patent applications, Serial No. 77,761, filed May 4, 1936; Serial No. 160,270, filed August 21, 1937; and Serial No. 197,858, filed March 24, 1938. The process, according to the first two applications, comprises sulfonating a fatty material, such as fatty acids, fatty oils, fatty waxes and fatty derivatives in the usual manner and then taking up the crude sulfonation mass in a substantially anhydrous organic solvent for the mass and neutralizing the mass by means of a concentrated aqueous solution of an alkali or alkali dissolved in an organic solvent. The inorganic salts formed during neutralization, as well as any salt that might be present in the mass, precipitate out from the sulfonated product. The volume of the solvent used must be at least equal to the volume of the mass and preferably two to four times that quantity of solvent is used. The precipitated salts are removed from

the sulfonated mass by filtration, centrifuging, decanting, or otherwise. After the salts are removed, the sulfonated mass is subjected to distillation, preferably under vacuum, to drive off the solvent and any remaining water. The latter cited application discloses a process wherein the sulfonated material is washed, neutralized and panned in the usual manner, after which it is partially dehydrated under reduced pressure at a relatively low temperature which will not deleteriously affect the sulfonated material. After the greater part of the water has been removed the mass is taken up in solvent, whereupon the salts are precipitated out. The mass is then filtered to remove the inorganic salts and the solvent and residual water are removed by distillation. Suitable fatty materials which may be sulfonated and treated according to these processes include, inter alia, oils, fats and waxes, such as castor, corn, cottonseed, linseed, olive, palm, peanut, sunflower, rape seed, teaseed, rice bran, chaulmoogra, tomato seed, sesame, soya bean, cacao butter, Chinese tallow, cod, herring, menhaden, porpoise, seal, salmon, sardine, shark, sperm, whale, tuna, neat's foot oils; beef tallow, bone fat, horse fat, mutton tallow, wool grease, etc., as well as the corresponding acids of these products. Suitable derivatives of these or other fatty materials may also be used as alkyl, esters of the higher fatty acids—alkylolamides or aralkylolamides of fatty acids; and, in general, any fatty material or derivative thereof which is capable of sulfonation due to unsaturated linkages or hydroxyl groups.

The substantially anhydrous and inorganic salt-free sulfonated products derived from the foregoing processes exhibit marked and distinct improvements over the prior known sulfonated products, especially with respect to textile treatments. Ordinary sulfonated products available on the market contain from about 10% to 30% or more water and varying quantities of inorganic salts ranging from about one percent and up. In the processing of textiles, various blended oils or compositions possessing properties peculiar to the specific treatment under consideration are employed. In most cases the composition comprises a so-called soluble or dispersible oil which readily dissolves or disperses in water. The use of raw or untreated animal, vegetable and/or mineral oils or suitable derivatives are desirable for imparting various characteristics to textile materials. Due to operating conditions and for other reasons, these oils cannot be applied directly to the textiles but must be blended with water-soluble or water-dispersible products which will impart these same characteristics to the oils. Heretofore, use was made of the ordinary sulfonated fatty oils and the like (which normally contain about 10% to 30% moisture and 1% or more inorganic salts) to render the oils dispersible in water. However, in producing a clear or homogenous mixture of the raw and sulfonated oils, soaps and the like, it was necessary to make use of various agents to effect the proper blending of the oil and dispersing agent. The general procedure employed in producing a so-called "soluble" mineral oil, for example, was to admix 60 to 70 parts of mineral oil with 20 to 25 parts of an ordinary sulfonated oil of the aforementioned type, after which various stabilizing and blending agents, such as alkalies, fatty acids, organic solvents, etc., were added to bring about a suitable product. It has now been found that an improved, readily-dispersible, blended oil

may be produced by merely admixing a suitable quantity of a substantially anhydrous and salt-free sulfonated oil with a raw oil. In some cases it is desirable to add a very small quantity of moisture (.25%-2.0%) to ensure a clear, homogeneous product, but the use of the ordinary blending agents is entirely dispensed with.

In carrying out the process of the invention, textile materials are treated with a substantially anhydrous salt-free sulfonated fatty material with which there may or may not be blended a vegetable, animal or mineral oil or wax and/or such other suitable substances, such as fatty alcohols, synthetic esters of fatty acids and other water-immiscible fatty derivatives. In the fabrication of natural silk, for example, the silk fibers may, according to the invention, be soaked in an aqueous bath having dispersed therein a composition comprising a substantially anhydrous salt-free sulfonated fatty material blended with a raw vegetable oil. During such soaking the sericin on the fiber, as well as the fiber itself, will absorb the composition, whereby the fiber is rendered pliable so that it may be spun or thrown and/or woven or knitted into the desired textile material. The soaking oil will also provide the necessary lubrication during the fabrication operation. After weaving or knitting, the silk product is scoured to remove the sericin and the lubricant. In some cases the blended oil composition may be applied to the yarn directly as by spraying or otherwise coating the fiber, thus eliminating the aqueous bath.

Woolen, cotton, rayon and other textile fibers or materials may also be treated with the so-called soluble type oil comprising, according to the invention, a mineral or other suitable oil with which there is blended a substantially anhydrous and salt-free sulfonated fatty material. Such a composition will impart softness to the fiber and provide the necessary lubrication for weaving, knitting, spinning or other fabricating operations. The oil will be removed from the fiber by scouring after it has served its purpose, and in many instances the oil is self-scouring in view of the high efficiency of emulsifying agents employed herein.

The soluble oils may be applied to the fibers in any suitable manner. In the treatment of artificial yarns or yarns produced from artificial silk fibers, for example, the oil may be applied as a coning oil by means of wicks. The oil may also be dispersed in a water bath and the yarns immersed therein or passed therethrough or the composition may be applied by an applicator roll or by means of spraying, etc.

The anhydrous and salt-free sulfonated fatty materials may be included in the scouring bath to assist in removing the oil, wax or other substance from the fibers being scoured. Moreover, these sulfonated products may also be incorporated in bleaching and dyeing baths to enhance the penetrating and wetting qualities of such baths.

When the yarns or fabrics have been bleached, dyed and otherwise completed, a finishing oil comprising an oil and/or a wax or other fatty substance and a substantially anhydrous and salt-free sulfonated fatty material is applied thereto. It has been found that the use of these sulfonated products in finishes produces a fabric having excellent softness, draping qualities, hand, and the outstanding feature is that these products are salt-free and are produced in a media that is anhydrous, the quantity of free

fatty acid resulting from their production being very low. As for the oil or wax employed in the finishing oil, it may comprise mineral oil, vegetable oils, raw fatty alcohols, synthetic fatty acid esters, etc. or any suitable mixture of these or other products. Thus, on oxidation tests which have been conducted, it has been found that fabrics treated in the aforementioned manner exhibit considerably less oxidation on storage than the fabrics treated according to prior practices.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following examples which are given merely to illustrate further the invention and are not to be construed in a limiting sense:

Example I

In the process of softening and lubricating artificial silk yarns (fibers derived from cellulose acetate, viscose, casein, cuprammonium solutions of cellulose, etc.), a coning oil of the following composition may be used:

	Parts by weight
White mineral oil (50 sec.)	70
Raw olive oil	15
Substantially anhydrous and salt-free sulfonated olive oil	15
Such a composition is admirably suited for application to the yarn by means of wicks. In view of the salt-free nature of the oil, the wicks may be used for long periods of time without clogging, due to the crystallizing out of inorganic salts.	
The composition may alternatively be applied to the yarns by means of a spray, applicator roll or the like composition may be dispersed in a water bath and applied as such.	

Example II

In carrying out the process described under Example I, the following composition may be employed:

	Per cent
White mineral oil	68.5
Raw sperm oil	11.0
Sulfonated teaseed oil (anhydrous and salt-free)	18.5
Moisture	2.0
The above components may be blended together in any order and usually a clear, soluble (self-emulsifiable) oil results. In some instances it may be necessary to slightly vary the above constituents to render the oil clear and emulsifiable in tepid water.	

Example III

The following composition may be employed in softening and lubricating rayon, silk, cotton and wool yarns:

	Per cent
White mineral oil	80
Sulfonated sperm oil (anhydrous and salt-free)	20
This soluble oil is readily dispersed in luke warm water.	

Example IV

The following composition may be employed in the soaking of natural silk or in the finishing of rayon, silk, cotton, woolen or other fibers or fabrics:

	Per cent
Sulfonated olive oil (anhydrous and salt-free)	50
Raw olive oil	50

This product is readily dispersed in water and is preferably applied as an aqueous dispersion. When used for soaking silk, a small quantity of the alkaline agents usually employed may be added.

Example V

In the process of soaking natural silk, the following soluble oil may be employed:

	Per cent
Sulfonated olive oil (anhydrous and salt-free)	50
White mineral oil	50

This composition may also be used in the finishing of textile materials.

Example VI

The following composition may be used in the soaking of natural silk or in the finishing of woolen, cotton, silk or rayon fabrics or yarns:

	Per cent
Sulfonated olive oil (anhydrous and salt-free)	80
Raw sperm fatty alcohols	20

In preparing a silk soaking bath, any one of the foregoing compositions, adapted for silk soaking, may be used in the following proportion:

Silk soaking oil	lbs. 16-18
Potassium carbonate	lbs. 1½
Sodium bicarbonate	lbs. 3
Water	gals. 60

This bath is suitable for a still tub, circulating tub or machine soaking and is capable of soaking about 100 pounds of raw silk.

Example VII

The following composition has shown merit in the finishing of textiles:

	Per cent
Sulfonated tallow (anhydrous and salt-free)	65
Paraffin wax	35

A fabric treated with this composition displays an improved softness and drape.

Example VIII

Another successful finishing composition has the following formula:

	Per cent
Sulfonated tallow (anhydrous and salt-free)	70
Raw coconut oil	10
Raw tallow	15
Paraffin oil	5

Fabrics finished with this composition exhibit a pleasing hand and excellent draping qualities.

Example IX

Another suitable finishing oil may have the following general formula comprising:

	Per cent
Sulfonated sperm oil (anhydrous and salt-free)	20
Paraffin oil	80

This composition likewise imparts desirable characteristics to the finished fabric. This composition, as well as the foregoing finishing compositions, may be added to the final rinse water in laundries to replenish, in the washed fabrics, the finishing removed therefrom during the washing operation.

While reference has been made as to the treatment of textile materials, it is within the purview

of the invention to also include the treatment of fibrous materials in general, such as leather, paper and the like. The textile materials which may be treated, according to the invention, may be in the form of loose fibers, yarns, staple-fiber, threads, felts, knitted fabrics, woven fabrics, etc., and which may be composed of any one or combination of the following: cotton, wool, ramie, hemp, flax, cellulose hydrate, cellulose esters, cellulose ethers, cellulose oxy or hydroxy ethers, casein, glass, synthetic resins, or other suitable materials whether of animal, vegetable, mineral or synthetic origin. The substantially anhydrous and salt-free sulfonated materials referred to contain only a trace of inorganic salts (up to .12%) and may contain up to 3 or 4% moisture, but a salt and moisture content below these values is preferred. The expression "sulfonated" is used herein to connote the neutralized sulfonation product, i. e. the salt of the sulfuric acid ester of the particular fatty material treated.

Since certain changes in carrying out the above process and certain modifications in the composition which embody the invention may be made without departing from its scope, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all the generic and specific features of the invention herein described and all statements of the scope of the invention, which, as a matter of language might be said to fall therebetween; and that they are intended to be inclusive in scope and not exclusive, in that if desired, other materials may be added to my novel composition of matter herein claimed without departing from the spirit of the invention. Particularly it is to be understood that in said claims, ingredients or components recited in the singular are intended to include compatible mixtures of said ingredients wherever the sense permits.

Having described our invention, what we claim as new and desire to secure by Letters Patent is:

1. In the treatment of textile materials, the step which comprises applying thereto a soluble oil comprising a water-immiscible material selected from the group consisting of animal, vegetable and mineral oils, waxes and water-immiscible derivatives thereof blended with a sulfonated fatty material selected from the group consisting of animal, vegetable and marine oils, fats and their respective fatty acids and alkyl esters thereof, said sulfonated fatty material being characterized by being substantially free from moisture, inorganic salts and hydrolytic by-products.

2. In the treatment of textile materials, the step which comprises applying thereto a soluble oil comprising a water-immiscible material selected from the group consisting of animal, vegetable and mineral oils, waxes and water-immiscible derivatives thereof blended with a sulfonated fatty material selected from the group consisting of animal, vegetable and marine oils, fats and their respective fatty acids and alkyl esters thereof, said sulfonated fatty material being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

3. In the treatment of textile materials, the step which comprises applying thereto a soluble oil comprising a water-immiscible material selected from the group consisting of animal, vege-

table and mineral oils, waxes and water-immiscible derivatives thereof blended with sulfonated sperm oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

4. In the treatment of textile materials, the step which comprises applying thereto a soluble oil comprising a water-immiscible material selected from the group consisting of animal, vegetable and mineral oils, waxes and water-immiscible derivatives thereof blended with sulfonated castor oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

5. In the treatment of textile materials, the step which comprises applying thereto a soluble oil comprising a water-immiscible material selected from the group consisting of animal, vegetable and mineral oils, waxes and water-immiscible derivatives thereof blended with sulfonated teaseed oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

6. In the treatment of textile yarns, the step which comprises passing a synthetic yarn over a wick saturated with a soluble coning oil comprising mineral oil and a sulfonated fatty material selected from the group consisting of animal, vegetable and marine oils, fats and their respective fatty acids and alkyl esters thereof, said sulfonated material being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

7. A water-dispersible composition comprising a water-immiscible material selected from the group consisting of animal, vegetable and mineral oils, waxes and water-immiscible derivatives thereof blended with a sulfonated fatty material selected from the group consisting of animal, vegetable and marine oils, fats and their respective fatty acids and alkyl esters thereof, said sulfonated fatty material being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

8. A water-dispersible composition comprising a water-immiscible material selected from the group consisting of animal, vegetable and mineral oils, waxes and water-immiscible derivatives

thereof blended with a sulfonated vegetable oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

9. A water-dispersible composition comprising a water-immiscible oil and a sulfonated vegetable oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

10. A water-dispersible composition comprising a vegetable oil and a sulfonated vegetable oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

11. A water-dispersible composition comprising a mineral oil and a sulfonated vegetable oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

12. As a new composition of matter, a textile coning oil comprising a white mineral oil and a sulfonated vegetable oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

13. As a new composition of matter, a textile coning oil comprising a white mineral oil, sperm oil and a sulfonated vegetable oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

14. As a new composition of matter, a textile coning oil comprising a white mineral oil, vegetable oil and a sulfonated vegetable oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

15. A water-dispersible composition comprising a fatty alcohol and a sulfonated vegetable oil, said sulfonated oil being characterized by being substantially free from hydrolytic by-products and having a moisture content of not more than 3% and an inorganic salt content of not more than .12%.

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