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COMPOSITION AND PROCESS FOR SOAKING
SILK AND TEXTILE MATERIALSDale S. Chamberlin, Bethlehem, Pa., assignor to
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corporation of Rhode IslandNo Drawing. Application July 23, 1935,
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The present invention relates to a process and material for treating yarns and it particularly relates to a process and product for soaking natural silk yarns.

Although the present invention will be particularly described in connection with its application to natural silk in gum yarns preparatory to textile processing of such yarns, either for throwing, weaving or knitting operations, it will be understood that the process and the product to be described herein is also applicable with advantageous results to other types of yarn, such as rayon yarns.

Natural silk as it comes upon the market from Japan or other sources of supply usually consist of a plurality of strands of pure silk fiber, termed fibroin, which strands are more or less encased with a gummy material, termed sericin.

This sericin must eventually be removed since it gives the yarn and fabric woven therefrom a rather harsh feel and a somewhat dull appearance, but it has been found most advantageous in the weaving and knitting processes to retain such gum or sericin upon the fibers for its body-giving properties and to render the silk more readily handled in textile processing machinery.

However, this gum often tends to become tough and brittle, when processed in textile machinery tends to give rise to static electric charges. For these reasons, it has been found desirable to soak it to make it more pliable and suitable for textile processes and also to reduce the static electric charge generated when it is used.

In soaking silk it has been customary to immerse the silk for several hours or overnight in a bath containing an emulsifying or emulsifiable oil, a sodium salt of a fatty acid or soap being used to emulsify the otherwise immiscible oil or the oil itself being so sulphonated and combined with other sodium compounds as to be emulsifiable in itself.

However, it was found particularly difficult to assure economic utilization of the oil in these baths and to assure effective absorption thereof by the natural soaked in gum, since mere dipping of the skeins or immersion thereof was usually not satisfactory to assure a satisfactory absorption and in such case only such oil would be carried away by the silk fibers as would be contained in the liquid which would mechanically adhere thereto, most of which would afterwards be lost if the silk were subjected to a centrifugal extraction or whizzing operation.

To assure an effective absorption of the oil attempts have been made to coagulate the emul-

sion, as for example with a coagulating agent such as alum, and also attempts have been made to salt out the emulsion by adding a soluble alkali metal salt, such as a sodium salt, with resultant precipitation of the sodium salts of the sulphonated oils upon the fibers.

However, in case of either coagulation or salting out, as above described, the emulsion would be broken down so quickly as to give insufficient time for absorption of the oil by the natural silk in gum fibers and with the result that the oil would more or less form a smear or heavy greasy deposit upon the surface of the skein, which not only gives an undesirable unctuous hand to the yarn, but in addition permits of removal of substantial quantities of the oil when the skeins were subsequently subjected to vacuum extraction or hydro-extraction.

On the other hand, if too little of the coagulating or salting out agent were added, an unsatisfactory absorption would be obtained and there was no method in which the absorption could be controlled by either salting out or coagulating process so that it would take place over a satisfactory length of time to enable most satisfactory penetration.

Among the objects of the present invention are to provide an improved soaking process which will permit the natural silk in gum to be placed in a bath for any desired length of time, and preferably from eight hours to overnight with assurance, that a most effective and complete absorption of the oil from the soaking bath would be attained without forming a smear or unctuous deposit on the surface of the yarn, and with further assurance that the absorption would be so complete that mere centrifugal extraction would not suffice to remove any substantial amounts of oil from yarns so prepared.

Another object is to provide a natural treating bath for natural silk in gum and similar yarns which may be inexpensively made up and which may be most effectively utilized to assure most satisfactory absorption of the lubricating oil upon the silk yarns, and most effective use of the oil employed.

Other objects will be in part obvious or will appear during the course of the following specification.

In accomplishing these objects, it has been found most satisfactory to accomplish a gradual discharge of a negatively charged disperse phase in the bath by a material which will over a period of time take up the negative charge main-

taining the dispersion, or which will gradually reduce the pH or alkalinity.

Although the addition of many chemicals may be employed to achieve this end, it has been found most satisfactory to utilize materials which will gradually release or make available an acid, or which will gradually take up the alkaline concentration in the bath upon standing until substantially all of the disperse phase has been discharged and has been caused to deposit upon the silk skeins placed in the bath.

Among the most satisfactory compounds for this purpose are salts of weak base and strong acids, such as ammonium sulphate, ammonium chloride, and ammonium silico-fluoride.

In preparing the bath it has been found generally most satisfactory to utilize a sulphonated oil such as sulphonated olive oil, and to neutralize this oil until its sulphonic or carboxylic acid groups and its sulphated groups have been in large part or wholly converted into sodium salts with sodium carbonate.

It is to be understood that the present process is also applicable to other sulphonated or sulphated oils, such as menhaden oil, palm oil, mustard seed oil, pea seed oil, coconut oil, cottonseed oil, rape seed oil, castor oil, neat's-foot oil, peanut oil, lard oil, tallow oil, elaine oil, corn oil, sperm oil, and/or fish oil or mixtures thereof, and that other alkaline metal compounds may be utilized for neutralizing the sulphonated compound, as for example potassium carbonate and so forth.

As a general rule, the discharging salt and the sulphonated oil material are added together to the soaking bath in such proportions and concentrations that the discharge will be substantially completed after a predetermined number of hours, say from eight to fifteen hours, and so that the oil will be largely if not completely removed from the bath and caused to be absorbed by the yarns.

Preferably the temperatures during this treatment in the soaking bath with the natural silk yarns is maintained between 80 to 100° F., although the temperatures for other yarns such as rayon may be maintained between 125 to 140° F. for every 100 parts by weight of natural silk in gum, 500 parts by weight of water are utilized in the soaking bath, to which bath is added between 10 to 20 parts by weight of the sulphonated oil, between 1½ to 3½ parts by weight of potassium carbonate and between 1 to 2 parts by weight of discharging salt or discharging salt mixture.

To give several illustrative examples without restricting the invention thereto, for 100 pounds of natural silk in gum to be utilized in making knitting yarns for stockings a bath is made up of 60 gallons or 500 pounds of water, in which is included 16 pounds of a sulphonated olive oil, 2½ pounds of potassium carbonate and 1½ pounds of the discharging salt mixture of the present application.

For treating yarns to be utilized in making crepe silks, on the other hand, 100 pounds of natural silk in the forms of skeins are immersed in 60 gallons or 500 pounds of water, including 12 pounds of sulphonated neat's-foot oil, 2½ pounds of sodium carbonate and 1½ pounds of the salt mixture of the present application.

Sufficient sodium or potassium carbonate or other alkaline reagents should be added to neutralize the free fatty acids or other acid components and it is most desirable that at the end

of the treatment in the soaking bath, which may be from 10 hours to overnight, the bath does not change to acid condition. The treatment should end with the bath having a pH of about 7, or above.

Generally in one hour's treatment according to the present invention, it has been found that at least 25% of the oily material has been completely absorbed by the silk in gum, while with 8 hours and the above proportions, the absorption is substantially completed.

During this treatment with natural silk the temperature should be maintained between 70 to 100° F., although slightly higher temperatures may be utilized in the soaking of rayon.

The preferred ammonium salt mixtures, which may be utilized according to the above examples, are those composed of ammonium sulphate and ammonium silicofluoride.

For example, in the case of natural silk for hosiery, where the hygroscopic effect is important, it may be desirable to utilize a salt mixture containing 30% of ammonium sulphate and 70% of ammonium silicofluoride.

On the other hand, in the case of broad silk yarns, where the throwster is not particularly interested in hygroscopicity the salt mixtures may be conveniently made up of 75% of ammonium sulphate and 25% of ammonium silicofluoride.

When the salt and the bath has been properly proportioned it has been found that during the treatment, the pH will gradually drop off from 9 to 10 until it reaches about or is slightly above 7, when the reaction is substantially complete and all of the oil has been absorbed.

When the proportions have been as above stated, the silk which has been treated will have absorbed substantially all of the oil from the bath and it will be satisfactorily lubricated for textile processes, will not tend to become harsh or tough upon standing, but will be readily flowable and will not tend to generate any static charge.

In addition, the silk will be most satisfactory from a handling viewpoint of the silk throwster.

Although ammonium silicofluoride appears to affect most favorably the hygroscopic properties of the silk, nevertheless other salts or discharging compounds may be employed such as compounds of triethanolamine, diaminopropanol, methyl, ethyl and amyl-amines, together with various acids such as acetic acid, lactic acid, phosphoric acid and citric acid. Ammonium salts of these acids may also be employed.

In the baths as above stated it is also possible that glycerine, glycols or other polyhydroxy materials be employed to assist the hygroscopic action of the silicofluoride or in lieu of the hygroscopic action in the silicofluoride.

In making a discharge bath agent it is generally desirable that the bath initially have a pH of 9.8 to 10.

Although the exact mechanism of the above mixture in discharging the bath has not been definitely ascertained, it is possible that positively charged ions or anions, such as sodium and potassium ions, which maintain a negatively charged disperse phase in the emulsion are gradually combined with or taken up by the cations or negatively charged ions of the ammonium salts, with the formation of only slightly ionized ammonium hydroxide or ammonia. This ammonia comes off so gradually and slowly during

the discharge as to be substantially unobservable.

In combination with or in lieu of the above mixtures, the sulphonated oil itself may be finished or neutralized with ammonia to form an emulsifiable oil for addition to the bath. The disperse phase so formed may be gradually discharged by the addition of an alkali, such as a sodium or potassium carbonate.

In lieu of silicofluorides other double salts of halogens and non-metals may be employed in suitable environments and alkali metal double fluorides including other non-metallic element in the anion.

It is to be understood that many changes in the proportions, ingredients and treating conditions may be made without departing from the scope of the invention and it is intended to include all such alternative modifications and variations as come within the scope of the appended claims.

What is claimed is:

1. A process of soaking which comprises immersing a textile material in a bath containing a soaking oil as a negatively charged disperse phase and a discharging agent adapted to gradually discharge the bath, said discharging agent consisting of an ammonium salt.

2. A process of soaking in negatively charged oil emulsions which comprises gradually discharging emulsions by a discharging agent while a textile material is immersed therein, said discharging agent consisting of an ammonium salt.

3. The process of treating with oil which comprises immersing a textile material in a bath containing oil as a negatively charged disperse phase and then adding a discharging agent thereto which will gradually discharge the material to neutralize said negative charge, said discharging agent consisting of an ammonium salt.

4. A process of soaking which comprises immersing a textile material in a bath containing a dispersed, negatively charged sulphonated oil and an ammonium salt.

5. A process of soaking which comprises immersing a textile material in a bath containing dispersed, negatively charged sulphonated olive oil and an ammonium salt.

6. A process of discharging a negatively charged oil aqueous emulsion in the presence of a textile material to cause impregnation of the material and oil which comprises adding to said bath a discharging agent consisting of a salt composed of a base and an acid capable of generating a base which will be discharged from the emulsion and an acid, said acid neutralizing a negative charge on the emulsion.

7. The process of discharging a negatively charged aqueous oil emulsion in the presence of textile material which comprises adding an ammonium sulphate thereto.

8. The process of discharging a negatively charged aqueous oil emulsion in the presence of a textile material to cause impregnation of the material and oil which comprises adding an ammonium silicofluoride thereto.

9. The process of discharging a negatively

charged emulsion in the presence of a textile material which comprises adding an ammonium chloride thereto.

10. A discharge agent for natural silk soaking baths containing about 30% of ammonium sulphate and about 70% of silicofluoride.

11. A discharging agent for natural silk soaking baths comprising about 75% of ammonium sulphate and about 25% of ammonium silicofluoride.

12. A process of soaking silk which comprises placing about 100 pounds of natural silk in gum in the form of skeins in a soaking bath containing about 60 gallons of water, about 16 pounds of dispersed negatively charged sulphonated olive oil, about 2½ pounds of potassium carbonate, and about 1½ pounds of an ammonium salt composition.

13. A process of soaking silk which comprises immersing about 100 pounds of silk in a bath containing about 60 pounds of water, about 12 pounds of dispersed negatively charged sulphonated neat's-foot oil, about 2½ pounds of sodium carbonate and about 1½ pounds of an ammonium salt mixture.

14. A process of soaking which comprises immersing a textile material in a negatively charged oil emulsion containing ammonium salt, and maintaining a temperature of 80 to 100° C.

15. A process of soaking which comprises immersing a textile material in an aqueous emulsion of the oil containing alkali metal carbonate, dispersed negatively charged sulphonated oil and an ammonium salt, the proportions of these ingredients being such that the bath will initially have a pH of about 10 and finally a pH of about 7 at the end of the treatment.

16. A process of discharging soaking baths for silk and rayon, said soaking baths including a negatively charged dispersed oil, which comprises adding to said baths ammonium salts of strong acids.

17. A process of discharging soaking baths for silk and rayon, said soaking baths including a negatively charged dispersed oil, which comprises adding to the bath an ammonium salt selected from the group consisting of ammonium chloride, ammonium sulphate, ammonium acetate, ammonium phosphate, ammonium citrate and ammonium silicofluoride.

18. A reagent mixture for discharging soaking baths comprising a combination of ammonium sulphate and ammonium silicofluoride.

19. A reagent mixture for discharging soaking baths comprising a combination of ammonium salt of a strong acid and ammonium silicofluoride.

20. A reagent mixture for discharging soaking baths comprising a combination of ammonium salt of a strong acid and an ammonium salt of a weak acid.

21. A discharging agent for yarn soaking baths which comprises a mixture of ammonium salts containing between about 30 to 75% of ammonium sulphate and between about 70 to 25% of ammonium silicofluoride.

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