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LOOPY, GAS JET BULKED YARN AND METHOD
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This invention relates to a method of obtaining an improved bulked yarn.

It is often necessary to apply a lubricating and conditioning composition to filaments, yarns, threads and the like in order to make them more amenable to the various physical operations to which they are subjected e.g. spinning, winding, weaving, knitting etc. The presence of the composition is intended among other things to reduce friction between the yarn and the various pieces of apparatus with which it comes in contact, e.g. guides, rollers, and the like to prevent tearing of the filaments and to minimize excessive attraction or repulsion of the filaments caused by electrostatic charges.

Various yarn lubricating compositions have been developed for use on conventional yarns spun from staple fiber. While these compositions are more or less satisfactory with these staple yarns, they often leave something to be desired when applied to bulked, textured, or voluminous yarns made, for example, from continuous multifilament yarns. The bulked yarns may be formed by subjecting a continuous multifilament yarn or bundle to a turbulent stream of a compressible fluid such as air. As disclosed in application S. No. 356,349, filed May 23, 1953, now U.S. Patent 2,942,402, which corresponds to British Patent 790,911, and in Patent No. 2,783,609 of Breen, the bulking of yarn in this manner is caused by the loops, whorls and convolutions formed by the interlocking of filaments in the surface and in the interior of the finished yarn. The quality of bulked yarn of this type depends to some extent on the number, uniformity and permanence of the filament loops which in turn depend on the static filament to filament friction properties, i.e. the ability of separate filaments to lock to each other after the loops are formed. The permanence of the loops is particularly important in view of the tendency of the loops to pull out when the yarn is subjected to the normal stresses of yarn and fabric-treating operations e.g. coning, weaving, knitting etc.

Other factors to be considered in the production of a loopy type of bulked yarn are the efficiency of the operation as indicated by the number of doffs produced yielding 100% high quality bobbins, the degree of adjustment of the jets required during the operations, uniformity of bulking from jet to jet, the cover and hand of the fabric produced from the bulked yarn and the amount of unaccounted for variations in the denier of the yarn produced.

It has been found that the foregoing factors are affected by the nature of the lubricating compositions applied to the yarn before the bulking operation is carried out. In particular, the nature of the yarn lubricant may have an appreciable effect on the loss of bulkiness caused by the stresses to which the yarn may be subjected after bulking.

It is an object of this invention to provide an improved bulked, voluminous, or textured yarn. It is a further object of this invention to provide a bulked yarn which contains greater and more uniform loopiness and which loses a minimum of its loopiness when subjected to the stresses associated with normal yarn-handling operations. It is a still further object of this invention to provide a more efficient process for the production of continuous multifilament bulked yarn which may be woven

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or knitted into fabrics having improved hand and cover. Further objects will become apparent from the following description of the invention.

In accordance with one aspect of this invention, a continuous filament yarn or bundle which is intended to be bulked by causing loops, whorls or convolutions to be formed among the filaments, is treated with a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, an alkanolamine, a sulfated fatty oil, and a sulfated long chain aliphatic carboxylic acid. It has been found that when a composition of this type is present as lubricant in a yarn which is bulked by contacting it with a jet stream of a compressible fluid, e.g. air, the bulked yarn possesses greater and more uniform loopiness which has a greater tendency to remain after the yarn has been subjected to ordinary stress than if other, conventional yarn lubricants are used. Moreover, the use of the lubricating composition of this invention results in greater efficiency of operation as indicated by the doffs produced having 100% high quality bobbins, reduced need for jet adjustment, less variations of product when different jets are used, improved fabric cover and hand, and fewer unaccounted for variations in yarn denier.

The mineral oil in the lubricating composition of this invention is a refined paraffinic petroleum product and is present in the composition in the range of 40 to 85% by weight. It suitably has a viscosity at 100° F. in the range of about 50 to 100 Saybolt Universal seconds.

The long chain aliphatic carboxylic acid is preferably a liquid at room temperature and may suitably contain 8 to 18 carbon atoms. The acid may be monoethylenically unsaturated such as oleic, caproic (9-decenoic), myristoleic (9-tetradecenoic), linderic (4-decenoic), palmitoleic (9-hexadecenoic) and petroselinic (6-octadecenoic) acids, or saturated such as caprylic (octanoic) and pelargonic (nonanoic) acids. Oleic acid is preferred because of its general availability. The long chain aliphatic carboxylic acid may be present in the composition for example in the range of 5 to 20% by weight of the composition.

The alkanolamine in the composition is preferably a lower alkanolamine e.g. in which the alkylol groups contain 2 to 3 carbon atoms. Some suitable alkanolamines are 2-amino-2-methyl-1-propanol, diethanolamine, triethanolamine, diethyl ethanolamine, dimethyl isopropanolamine and dimethyl ethanolamine. The alkanolamine may be present in an amount of 1 to 10% by weight of the composition.

The sulfated fatty oil and sulfated long chain aliphatic carboxylic acid in the composition may suitably be sulfated peanut oil and sulfated oleic acid. Other sulfated fatty oils which may be used are sulfated soybean oil and sulfated corn oil and other sulfated acids which may be used are sulfated caproic, sulfated myristoleic, sulfated linderic, sulfated palmitoleic and sulfated petroselinic acids. The sulfated fatty oils may be present in an amount of 1 to 10% and the sulfated acid to 1 to 10% by weight of the composition. The sulfated fatty oil and sulfated acid may be in the form of their alkali metal salts e.g. sodium salts.

The composition may also contain a polyhydric alcohol which is preferably saturated and contains, for example, 5 to 8 carbon atoms. Some polyhydric alcohols which may be used are octylene glycol, 2-ethyl-1,3-hexanediol, hexylene glycol, and pentanediol. The polyhydric alcohol may be replaced in whole or in part by a long chain monohydric alcohol which may suitably contain 11 to 18 carbon atoms. Some monohydric alcohols which may be used are oleyl alcohol, 2,6,8-trimethylnonanol-4, 2-butyl octanol, 5-ethylnonanol-2, 7-ethyl-2-methylundec-

anol-4 and 3,9-diethyltridecanol-6. The polyhydric and/ or monohydric alcohol may suitably be present in a total amount of 5 to 20% by weight of the composition.

An alkyl phenol such as ditertiary amyl phenol may also be present. Other alkyl phenols which may be used are ditertiary-butyl phenol, ditertiary butyl paracresol (butylated hydroxytoluene), and butylated hydroxy anisole. The alkyl phenol may be present in amount of 0.01 to 5% by weight of the composition.

To obtain the sulfated components of the composition a mixture of unsulfated oil and acid, diluted if necessary with mineral oil, may be treated with concentrated or fuming sulfuric acid while preventing the temperature from reaching too high a level. The mixture may then be neutralized with an alkali such as caustic, dehydrated under vacuum, and separated from the salt formed during the neutralization. The dehydrated, desalted composition may then be mixed with desired quantities of the other components to obtain the finished lubricant. If desired, the sulfated mixture may be mixed with a further quantity of mineral oil and a small amount of a long chain monohydric alcohol prior to separation of the salt. If an alkyl phenol is used, it may be added to the sulfated mixture after neutralization but before dehydration.

Since the long chain acid and the alkanolamine form an organic soap when the final composition is prepared, a preformed soap made from these materials may be added in lieu of all or part of the separate components.

In carrying out the process of this invention the lubricating composition described above is preferably applied to a continuous multifilament yarn or untwisted bundle before bulking. The lubricated bundle is then bulked by contacting it with a turbulent stream of a compressible fluid e.g. air from a jet in a manner described for example in application S. No. 356,349, filed May 23, 1953, or in Patent No. 2,783,609 of Breen. The lubricating composition may also be applied after the yarn is bulked to improve the ability of the bulked yarn to retain its loopiness under stress.

The jetted fluid which travels lengthwise in the direction of the movement of the filament causes transverse loops, whorls etc. to form along the length of the filament bundle. The filaments are then further treated e.g. by twisting to form a bulked yarn. As disclosed in application S. No. 356,349 the jet outlet is preferably offset from the path of the jet inlet through which the filament bundle enters.

The process of the invention is applicable to continuous filament yarn made from a wide variety of materials, for example organic derivatives of cellulose e.g. cellulose esters such as cellulose acetate, cellulose propionate, cellulose butyrate, cellulose acetate-propionate, and cellulose acetate butyrate, cellulose ethers such as ethyl cellulose and benzyl cellulose, polyesters such as polyethylene terephthalate, polyamides such as polyhexamethylene adipamide and regenerated cellulose. The process is particularly advantageous in the production of bulked yarn of cellulose acetate. The supply yarns or filament bundles treated may suitably have a denier of 200 to 4800 with a denier per filament of 1.9 to 20. It may be substantially untwisted prior to being contacted with the jet stream of fluid, or may contain up to 20 turns per inch and possibly greater. During or after the bulking operation the yarns may suitably be further twisted in an amount up to 25 additional turns per inch. The amount of lubricating composition in the yarn may suitably be in the range of 0.25 to 10.0 percent by weight of the yarn or filament bundle.

A lubricating composition under this invention was prepared as follows:

A mixture of 65 gallons of paraffinic mineral oil having a viscosity at 100° F. of 50 Saybolt Universal seconds, 22.5 gallons of refined peanut oil and 47.7 gallons of oleic acid was cooled to 5 to 8° C., 108 pounds of fuming sulfuric acid containing 20% by weight of SO₃ were added

over a 30 minute period, holding the temperature at a maximum of 20° C., after which the mixture was stirred for an additional 45 minutes. An aqueous solution of 50% by weight of sodium hydroxide in an amount of 150 pounds was added and after the neutralization was complete, 10.9 gallons of ditertiary amyl phenyl were added. The mixture was then stirred for an additional 15 minutes and subsequently dehydrated under vacuum. To the dehydrated mixture were added 289 gallons of mineral oil and 58 pounds of oleyl alcohol. The resulting composition was kept in a settling tank for 3 to 4 days after which the supernatant liquid was decanted and filtered to remove the last traces of sodium sulfate.

The above composition was composed essentially of 80% by weight mineral oil, 5% by weight of the sodium salt of sulfated peanut oil, 12% by weight of the sodium salt of sulfated oleic acid and 3% by weight of ditertiary amyl phenol. It was mixed in an amount of 8.33 parts by weight with 66.67 parts by weight of white mineral oil having a viscosity at 100° F. of 50 Saybolt Universal seconds, 12.92 parts by weight of oleic acid, 8.33 parts by weight of 2-ethylhexanediol-1,3. The resulting finished composition is designated as Lubricant A.

The performance of Lubricant A in connection with bulked yarn was compared with three standard mineral oil-based textile lubricating compositions conventionally used on cellulose acetate and designated as Lubricants B, C, and D.

Each lubricant was tested in an identical manner by applying it to a cellulose acetate yarn of about 200 denier, 105 filaments and having a twist of 0.15 turn per inch. The yarn was further twisted to 5.5 turns per inch prior to bulking after which it was bulked in apparatus such as that described in application S. No. 356,349 using a yarn inlet orifice size of 0.029 inch and a yarn outlet orifice size of 0.052 inch, the center of the outlet being offset by 0.010 inch of the center of the tube through which the yarn passed. Air was supplied at 26 p.s.i.g. and the yarn entered the jet at a rate of 66 yards per minute. The yarn was bulked while twisting to a total of 7.5 turns per inch.

In testing the results of the bulking of yarns containing different lubricants it may be assumed that variations in denier of the supply or parent yarn, would affect the denier of the bulked yarn. To account for these variations, the bulked yarn was characterized in terms of "Bulkability" defined in terms of the supply yarn as follows:

Percent Bulkability=(Bulked yarn denier before draw-down minus Supply yarn denier)×100 divided by Supply yarn denier

The yarns containing the different lubricants were bulked using three different rates of overfeed, defined as the percent by which the velocity of the yarn entering the jet is greater than the leaving the jet. The denier of the bulked yarn was measured without subjecting the yarn to any appreciable degree of tension. Results are shown in Table I. Each of the denier and bulkability figures represents an average of ten samples.

Table I

Lubricant	Amount of Lubricant on Yarn, Percent w.	Supply Yarn Denier	Percent Bulkability at Different Rates of Overfeed		
			23.0%	25.0%	31.0%
A-----	1.06	198.7	19.5	26.9	27.1
B-----	1.81	194.6	18.2	19.8	22.4
C-----	2.99	196.9	16.6	17.7	20.9
D-----	2.30	194.2	16.5	18.1	21.4

It can be seen from the results in Table I that the use of Lubricant A resulted in bulked yarn of superior bulkability at all rates of overfeed, despite the fact that a

smaller amount of Lubricant A was used than of the other lubricants.

As an indication of the ability of yarns bulked with the different lubricants to retain their bulkiness after being subjected to normal processing tension, the yarns containing the different lubricants were bulked under identical conditions using 23% overfeed as shown in Table I and were drawn down in the conventional manner using processing tensions which were varied by means of different traveler sizes. The deniers after draw-down were then determined and the results expressed in terms of "non-bulkstability" defined as follows:

Percent Non-Bulkstability

$$= \left(\frac{\text{Bulked yarn denier before draw-down} - \text{Bulked yarn denier after draw-down}}{\text{Bulked yarn denier before draw-down}} \right) \times 100$$

Results are shown in Table II. Each of the non-bulkstability figures represents an average of ten samples.

Table II

Processing Tension (grams)	Percent Non-Bulkstability with Different Lubricants at Various Processing Tensions			
	Lub. A	Lub. B	Lub. C	Lub. D
39.8	3.8		6.6	5.2
32.6	3.3	6.2	5.3	5.0
24.7	3.1	6.2	5.5	5.0
21.4	2.6		5.7	4.3
17.3	2.2	5.1	4.9	4.1
10.5	2.8	4.8	3.3	3.3
10.1	2.7	3.7	3.7	3.3
8.5	1.8	3.4	3.7	2.4
6.9	1.0	2.2	3.1	1.8
5.8	.9	2.7		1.2
4.6	.9	2.0	2.3	1.5
4.0	.9	2.5	1.6	1.5

It can be seen from Table II that yarns bulked with Lubricant A of this invention had substantially lower values of percent non-bulkstability after being drawn down at all levels of processing tension than yarns bulked with the other lubricants.

Further tests were carried out on two large sample lots of the same cellulose acetate yarn tested above and bulked in the same manner using 20% overfeed. One sample contained 1% of Lubricant A before bulking and the other sample contained 2% of the same lubricant. Denier determinations of 267 samples of the bulked yarn containing 1% Lubricant A yielded an average denier of 225 with standard denier deviation of 2.1. Similar determinations of the denier of 240 samples of the yarn containing 2% of Lubricant A yielded an average denier of 224 with a standard denier deviation of 2.1. Thus yarns lubricated with Lubricant A showed excellent denier control and moreover were significantly more uniform than yarns containing other lubricants. The low and fairly constant denier deviation obtained with yarns from both lots were much superior to the deviation when the yarn was treated with other lubricants. In addition, both lots yielded an increased number of doffs containing 100% high quality bobbins. Finally, when yarns from each lot were woven, the resulting fabrics had superior cover and uniformity.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of our invention.

Having described our invention what we desire to secure by Letters Patent is:

1. A loopy, gas jet bulked continuous multifilament yarn containing a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, an alkanolamine, a sulfated fatty oil and a sulfated, long chain aliphatic carboxylic acid, said lubricating composition having been applied prior to bulking and serving to

retain the bulk of said yarn when it is subjected to tension.

2. A loopy, gas jet bulked continuous multifilament yarn of cellulose acetate containing a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, an alkanolamine, a sulfated fatty oil and a sulfated, long chain aliphatic carboxylic acid, said lubricating composition having been applied prior to bulking and serving to retain the bulk of said yarn when it is subjected to tension.

3. A loopy, gas jet bulked continuous multifilament yarn containing a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, a member of the group consisting of polyhydric alcohols, long chain monohydric alcohols and mixtures thereof, an alkanolamine, and alkylphenol, a sulfated fatty oil and a sulfated long chain aliphatic carboxylic acid, said lubricating composition having been applied prior to bulking and serving to retain the bulk of said yarn when it is subjected to tension.

4. A loopy, gas jet bulked continuous multifilament yarn of cellulose acetate having transverse loops containing a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, a member of the group consisting of polyhydric alcohols, long chain monohydric alcohols and mixtures thereof, an alkanolamine, an alkyl phenol, and a desalted and dehydrated reaction product of sulfuric acid with a mixture of a fatty oil and a long chain aliphatic carboxylic acid neutralized with an alkali, said lubricating composition having been applied prior to bulking and serving to retain the bulk of said yarn when it is subjected to tension.

5. A loopy, gas jet bulked continuous multifilament yarn of cellulose acetate having transverse loops containing a lubricating composition comprising 40 to 85 percent by weight of a mineral oil, 5 to 20 percent by weight of a long chain aliphatic carboxylic acid, 1 to 10 percent by weight of an alkanolamine, 1 to 10 percent by weight of a sulfated fatty oil and 1 to 10 percent by weight of a sulfated long chain aliphatic carboxylic acid, said lubricating composition having been applied prior to bulking and serving to retain the bulk of said yarn when it is subjected to tension.

6. A loopy, gas jet bulked continuous multifilament yarn of cellulose acetate having transverse loops containing a lubricating composition comprising 40 to 85 percent by weight of a mineral oil, 5 to 20 percent by weight of a long chain aliphatic carboxylic acid, 5 to 20 percent by weight of a member of the group consisting of polyhydric alcohols, long chain monohydric alcohols and mixtures thereof, 1 to 10 percent by weight of an alkanolamine, 0.1 to 5 percent by weight of an alkyl phenol, 1 to 10 percent by weight of a sulfated fatty oil and 1 to 10 percent by weight of a sulfated long chain aliphatic carboxylic acid, said lubricating composition having been applied prior to bulking and serving to retain the bulk of said yarn when it is subjected to tension.

7. A process for the production of loopy, gas jet bulked yarn which comprises applying to a continuous multifilament yarn a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, an alkanolamine, a sulfated fatty oil, and a sulfated long chain aliphatic carboxylic acid, and subjecting said lubricated yarn to a turbulent stream of a compressible fluid, thereby producing a yarn having loops, said loops being resistant to pulling out when subjected to tension.

8. A process for the production of loopy, gas jet bulked yarn which comprises applying to a continuous multifilament yarn a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, a member of the group consisting of polyhydric alcohols, long chain monohydric alcohols and mixtures thereof, an alkanolamine, an alkyl phenol, a sulfated fatty oil, and a sulfated long chain aliphatic carboxylic acid, and

subjecting said lubricated yarn to a turbulent stream of a compressible fluid, thereby producing a yarn having loops, said loops being resistant to pulling out when subjected to tension.

9. A process for the production of a loopy, gas jet bulked cellulose acetate yarn which comprises applying to a continuous multifilament yarn of cellulose acetate a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, a member of the group consisting of polyhydric alcohols, long chain monohydric alcohols and mixtures thereof, an alkanolamine, an alkyl-phenol, a sulfated fatty oil, and a sulfated long chain aliphatic carboxylic acid and contacting the lubricated yarn with a turbulent stream of a compressible fluid, thereby producing a yarn having loops, said loops being resistant to pulling out when subjected to tension.

10. A process for the production of a loopy, gas jet bulked cellulose acetate yarn which comprises applying to a continuous multifilament yarn of cellulose acetate a lubricating composition comprising a mineral oil, a long chain aliphatic carboxylic acid, a member of the group consisting of polyhydric alcohols, long chain monohydric alcohols and mixtures thereof, an alkanolamine, an alkyl-phenol, and a desalted and dehydrated reaction product of sulfuric acid with a mixture of a long chain aliphatic carboxylic acid and a fatty oil neutralized with an alkali, and contacting the lubricating yarn with a turbulent stream of air to form transverse loops along the length of said yarn, said loops being resistant to pulling out when subjected to tension.

11. A process for the production of a loopy, gas jet bulked cellulose acetate yarn which comprises applying to a continuous multifilament yarn of cellulose acetate a lubricating composition comprising 40 to 85 percent by weight of a mineral oil, 5 to 20 percent by weight of a long chain aliphatic carboxylic acid, 1 to 10 percent by weight of an alkanol amine, 1 to 10 percent by weight of a sulfated fatty oil and 1 to 10 percent by weight of a sulfated long chain aliphatic carboxylic acid, and contacting the lubricated yarn with a jetted turbulent

stream of air to form transverse loops along the length of said yarn, said loops being resistant to pulling out when subjected to tension.

12. A process for the production of a loopy, gas jet bulked cellulose acetate yarn which comprises applying to a continuous multifilament yarn of cellulose acetate a lubricating composition comprising 40 to 85 percent by weight of a mineral oil, 5 to 20 percent by weight of a long chain aliphatic carboxylic acid, 5 to 20 percent by weight of a member of the group consisting of polyhydric alcohols, long chain monohydric alcohols and mixtures thereof, 1 to 10 percent by weight of an alkanol amine, 0.01 to 5 percent by weight of an alkyl phenol, 1 to 10 percent by weight of a sulfated fatty oil and 1 to 10 percent by weight of a sulfated long chain aliphatic carboxylic acid, and contacting the lubricated yarn with a jetted turbulent stream of air to form transverse loops along the length of said yarn, said loops being resistant to pulling out when subjected to tension.

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