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[21] Appl. No. **7,291**
[22] Filed **Jan. 9, 1970**
[45] Patented **Nov. 16, 1971**
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Original application May 18, 1966, Ser.
No. 550,890, now Patent No. 3,519,562,
dated Oct., 1970. Divided and this
application Jan. 9, 1970, Ser. No. 007,291

[54] **TEXTILE LUBRICANT USEFUL IN AMINOPLAST**
MODIFICATION PROCESSES
4 Claims, No Drawings

[52] U.S. Cl. **8/115.6,**
8/115.5, 8/116.2, 38/144, 8/116.3, 2/243

[51] Int. Cl. **D06m 13/34**

[50] Field of Search **8/115.6,**
116.2, 116.3

[56] **References Cited**
UNITED STATES PATENTS
3,519,562 10/1970 Lanner 8/115.6 X
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ABSTRACT: Lubricant compositions comprising certain oxidized Fischer-Tropsch waxes, and selected quaternary ammonium compounds alone or in combination with ethoxylated alcohols or phenols, are used as lubricants in the application of durable-press treating agents to cotton or cotton-synthetic fabrics.

TEXTILE LUBRICANT USEFUL IN AMINOPLAST MODIFICATION PROCESSES

This application is a division of U.S. Application Ser. No. 550,890, filed May 18, 1966, now U.S. Pat. No. 3,519,562. The latter was a continuation in part of U.S. Application Ser. No. 494,910 filed Oct. 11, 1965, and now abandoned, which in turn was a continuation in part of U.S. Application Ser. No. 363,288 filed Apr. 28, 1964, and now abandoned.

This invention relates to a textile lubricant composition. More particularly, it relates to a composition which is especially suitable for imparting sewing lubricity to cellulose. The composition is particularly applicable for use with cotton.

In the conventional manufacture of cotton textiles, raw cotton is subjected to the opening, blending and picking operations in the course of which by strictly mechanical action of the cotton from the bale is loosened, freed from grosser impurities and formed into a continuous web for delivery to the card. In the following carding operation the cotton fibers are oriented parallel to each other and formed into a thin sliver or untwisted strand of fibers. The card sliver is formed into warp and filling yarn in the spinning process and the yarns are woven into fabric. The fabric is then ready for finishing which consists of a variety of processes designed to clean and impart special characteristics to the fabric. Among the finishing processes are desizing, scouring, bleaching, mercerizing and the application of physical and chemical modifiers such as sewing lubricants, softeners, firming agents, scrooping agents, water repellents and wash-wear resins. Following the application of the chemical finishes further modifications may be obtained by additional mechanical operations, such as, calendaring to produce smoothness or luster, napping to raise the surface of the fabric and Sanforizing to produce dimensional stability.

The application of a sewing lubricant facilitates the cutting of the fabric, helps the fabric lie flat and fold more easily in the sewing operation, and most important provides surface lubricity between the sewing needle and the fabric and thereby decreases the number of yarns cut during high-speed commercial sewing operations and provides a stronger and better appearing finished fabric. The lubricating agent is usually applied to the fabric in the form of a water suspension and an anionic, nonionic or cationic emulsifying agent is commonly added to aid in the formation of the lubricating agent water suspension. A cationic lubricant composition can be applied to fabric by exhaustion, a method of application depending upon cationic attraction, which is most efficient for batch runs. On the other hand, cationic lubricant compositions would normally be expected to give rise to compatibility problems if applied together with anionic textile chemicals, such as a wash-wear resin cross-linking catalysts including, for example, magnesium chloride and zinc nitrate. Nonionic emulsified polyethylene is most commonly applied as a sewing lubricant and provides lubricity and some softness. Heretofore, there has been no textile lubricant composition which imparts both outstanding sewing lubricity and softness to fabric, particularly at low fabric moisture content, and which may be applied to fabric by cationic attraction and together with anionic textile chemicals.

It is an object of this invention to provide a textile lubricant composition which imparts outstanding sewing lubricity and softness to fabric and at the same time is versatile in its method of application to fabric.

The textile lubricant composition of the present invention also advantageously increases fabric tear strength, tensile strength and abrasion resistance and provides sewing lubricity at fabric moisture content as low as 0.6 percent.

Other objects and advantages of the present invention will be apparent from the description of the invention which follows.

It has been found that a textile lubricant composition can be made using certain oxidized Fischer-Tropsch waxes, certain cationic quaternary ammonium emulsifiers and water. Certain nonionic emulsifiers are optional ingredients. Parts and percentages herein are by weight.

The oxidized Fischer-Tropsch waxes within the scope of the present invention are synthesized from carbon monoxide and hydrogen under pressure and in the presence of a cobalt, thorium or ruthenium catalyst and are oxidized catalytically with air in conventional fashion. They have a chain length ranging from about 40 to about 55 carbon atoms, a melting point ranging from about 200° F. to about 220° F., an acid value ranging from about 10 to about 35 and a penetration ranging from about 1 to about 6. The oxidized Fischer-Tropsch waxes are fully characterized to those familiar with the art by chain length range, melting point range, acid value range and penetration range. Melting point determination follows the general procedure of A.S.T.M. D-127—Melting Point of Petrolatum and Microcrystalline Waxes. The acid value is the number of milligrams of potassium hydroxide neutralized by the free acids present in 1 gram of wax. The determination is done by titrating the sample in hot toluol and using phenolphthalein as an indicator. The method for needle penetration determination follows the general procedure of A.S.T.M. D-1321 for Needle Penetration of Petroleum Waxes. A penetrometer conforming to the specifications outlined in A.S.T.M. D-217 is used with a total load of 100 grams for the needle and all attachments.

Preferred oxidized Fischer-Tropsch waxes within the scope of this invention are those having chain lengths ranging from 45 to 50 carbon atoms, melting points ranging from about 205° F. to about 210° F., acid values ranging from 28 to about 34 and penetrations ranging from about 4 to about 6.

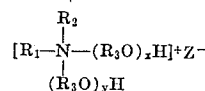
Other oxidized Fischer-Tropsch waxes within the scope of the present invention are listed in the following Table:

TABLE I

	Melting point, ° F.	Acid value	Penetration	Chain length
Wax A.....	212-215	10-12	1-2, 5	45-50
Wax B.....	211-215	14-16	1-3	45-50
Wax C.....	209-213	18-22	2-4	45-50
Wax D.....	207-212	24-26	3-5	45-50

The oxidized Fischer-Tropsch waxes with melting points below about 200° F.-205° F., acid values above about 35 and penetrations above about 6 impart poor lubricity to textiles. Oxidized Fischer-Tropsch waxes with melting points above about 220° F., acid values below about 10 and penetrations below about 1 are suspended with difficulty in the aqueous application medium.

The cationic emulsifier within the scope of the present invention which provides stability against separation of components for at least 1 week, complements the previously described Fischer-Tropsch waxes in imparting sewing lubricity and softness and also provides the previously described benefits of the cationic emulsified system, is a quaternary ammonium compound having the formula:



wherein R₁ is an alkyl group containing 14 to 20 carbon atoms; R₂ is an alkyl group containing 1 to 3 carbon atoms, a phenyl, a naphthyl or a C₁-C₃ alkyl-substituted phenyl group; R₃ is an alkylene group containing 2 to 4 carbon atoms; Z represents an anion; and x plus y ranges from 1 to 6, x ranges from 1 to 6 and y ranges from 0 to 5. R₃ can be different in the same compound. Z is usually an anion from a strong inorganic acid. Preferably x plus y has a value of 2 to 4, x a value of 1 to 4 and y a value of 0 to 3. x, y and x plus y are average values by the nature of the reaction forming the emulsifiers; therefore, x, y and x plus y can have fractional values.

Preferred quaternary ammonium compounds within the scope of the present invention include (a) a compound formed by reacting methyl chloride with ethoxylated stearyl

amine and having the above-described structural formula wherein R_1 is a stearyl group, R_2 is a methyl group, R_3 is an ethylene group, Z is a chloride ion and x and y are each 1; and (b) a compound formed from "Armeen TD," made by Armour Industrial Chemical Company, by ethoxylating "Armeen TD" with 3 ethylene oxide groups and quaternizing with benzyl chloride and having the above-described structural formula wherein R_1 is derived from hydrogenated tallow (65 percent stearyl, 33 percent cetyl and 2 percent myristyl), R_2 is a methyl phenyl group, R_3 is an ethylene group, Z is a chloride ion, x plus y equals 3 and x equals about 1.5.

Other quaternary ammonium compounds within the scope of the present invention are shown in the following table wherein R_1 , R_2 , R_3 , Z and x plus y are applied in the above-described structural formula:

TABLE II

R_1	R_2	R_3	Z	x plus y
Myristyl.....	Methyl.....	Ethylene.....	CH_3SO_4^-	2
Do.....	Propyl.....	do.....	Cl^-	4
Cetyl.....	Methyl.....	do.....	Cl^-	2
Do.....	Ethyl.....	Butylene.....	SO_3^-	4
Do.....	Propyl.....	Propylene.....	Br^-	5
Stearyl.....	Naphthyl.....	Ethylene.....	I^-	3
Do.....	Phenyl.....	Propylene.....	SO_4^-	1
Eicosyl.....	Ethylphenyl.....	Butylene.....	NO_3^-	1
Do.....	Propyl.....	Ethylene.....	Cl^-	6
Do.....	Phenyl.....	do.....	Cl^-	2
Hydrogenated tallow ¹	Methyl.....	Propylene.....	SO_4^-	2
Do ¹	Ethyl.....	do.....	SO_4^-	2

¹ Hydrogenated tallow consists of 65% stearyl, 33% cetyl and 2% myristyl.

In the above table the value of x can be 1 or range up to x plus y minus 1.

A nonionic emulsifier containing at least 8 moles of ethylene oxide is optionally and preferably included in the lubricant composition to aid in imparting stability against separation of components and to improve compatibility with ionic finishing chemicals. The nonionic emulsifiers that can be used within the scope of the present invention include n -alkyl (C_{10} - C_{14}) ethoxylated alcohols, ethoxylated rosin alcohols and C_8 - C_{10} alkyl phenol ethylene oxide ethers. Preferred nonionics are n -alkyl (C_{10} - C_{14}) ethoxylated alcohols containing about 20 to about 50 moles of ethylene oxide. Optimal nonionics include coconut alcohol ethoxylated with 45 moles of ethylene oxide and "Hercules Ad 400," a polyethylene glycol ether of "Abitol" containing 40 moles of ethylene oxide. "Abitol," made by the Hercules Powder Company, is a mixture of tetra-, di-, and dehydroabietyl alcohols made from rosin and has a specific gravity of 1.01 at 25/25° C. and a viscosity of 30 poises at 200° C. Other useable nonionics can contain up to about 50 moles of ethylene oxide and include hydrogenated tallow ethoxylated with 40 moles of ethylene oxide, stearyl alcohol ethoxylated with 30 moles of ethylene oxide, nonyl phenol condensed with 9.5 moles of ethylene oxide and isooctylphenylpolyethoxy ethanol containing 10 moles of ethylene oxide.

The nonionic emulsifier content ranges from about 0 to 7 parts for every 3 parts of cationic emulsifier and the ratio of oxidized Fischer-Tropsch wax to total emulsifier ranges from about 10:1 to about 3:7. A ratio of slightly more than 0 part, and preferably at least 0.25 parts nonionic to 3 parts cationic provides compatibility with most finishing chemicals. If the ratio of nonionic emulsifier to cationic emulsifier exceeds about 7:3 there is a loss of emulsifiability, lubricity, softness and the previously described versatility of application. If the ratio of wax to total emulsifier exceeds about 10:1 there is a loss in the suspension's stability against separation of components; if the ratio of wax to total emulsifier is less than about 3:7 there is a loss in the ability to impart lubricity. A preferred lubricant composition within the scope of the present invention has the following proportions of ingredients: 1.6 parts

nonionic emulsifier to 3 parts cationic emulsifier; 8 parts wax to 1 part total emulsifier.

Thus, the composition of the present invention consists essentially of a homogeneous water suspension of a specific oxidized Fischer-Tropsch wax emulsified by a specific cationic emulsifier and optionally by a specific nonionic emulsifier, all of which have been previously described.

When the lubricant composition is prepared, the wax can be suspended in at least about 55 percent water by weight of the total composition. If less than about 55 percent water is used the composition will not be readily pourable and mixing will be difficult. After being mixed, a composition containing at least about 55 percent water will be stable against separation of components for at least about a week. If separation occurs, homogeneity can be reobtained with only a moderate amount of mixing. The composition preferred for sale is one which contains a high percentage of solids so that it can be shipped inexpensively and at the same time, one that is liquid or pourable so that it is easily handled and readily diluted with water.

A lubricant composition within the scope of the present invention preferred for sale has the following formula (the preferred ingredients have hereinbefore been set forth): oxidized Fischer-Tropsch wax 34.8 percent, nonionic emulsifier 1.5 percent, cationic emulsifier 2.85 percent and the balance water.

A lubricant composition within the scope of the present invention can be prepared in an exemplary manner described as follows; it is the method used in the examples. The wax is melted and heated to about 220° F. The emulsifiers are added to the melt and the mixture heated to a temperature ranging from about 250° F. to about 290° F., e.g., 270° F., for addition to 190° F. to 195° F. water. The cationic emulsifier can be conveniently added to the melt in the form of an isopropanol solution containing 25 percent isopropanol. (The isopropanol evaporates when the emulsifier is added to the wax melt.) Since the nonionic emulsifier does not dissolve in the melt, the melt must be stirred continuously during addition to the water with mixing to ensure uniform distribution of nonionic. To obviate this uniformity problem the nonionic can optionally be dissolved in water to which the melt is added. The melt must be added quickly to the water because the cationic emulsifier is not indefinitely heat stable; preferably within a period of 3 to 10 minutes either by pouring it directly into the water at the vortex caused by rapid stirring or by adding it beneath the water surface by means of suitable piping, pumping and mixing devices. However, adding the melt at too fast a rate can cause surface foaming because of localized overheating or because the melt has been added at a rate faster than mixing will blend it in and will result in objectionably large suspension particle size. Particle size is preferably less than about 5 microns. When the melt has been suspended, the resulting suspension is cooled at a rapid rate with uniform mixing; a temperature drop of greater than 1° F. per minute is preferred to prevent objectionably large particle size, such as 5 to 30 microns. This cooling rate must be very rapid to produce ordinarily a product having a viscosity ranging from about 200 to 500 c.p.s. at room temperature and which is therefore readily pourable from its container by consumers and/or readily further diluted with, and dispersed in, water. For example, the suspension can be uniformly cooled from a temperature on the order of about 190° F. or 195° F. to the packing temperature of 70° F. to 100° F. in a time period ranging from about 5 seconds to about 100 seconds and preferably in a time period on the order of about 15 to 30 seconds to produce ordinarily the above-described benefits of rapid cooling. This rapid cooling can be achieved, for example, by passing the 190°-195° F. suspension through a heat exchanger or in a jacketed cooling tank or by the addition of cold water, for example at 40° F., or ice or other cooling agent. A closed system is desirable for the suspending and cooling steps in order to prevent water evaporation which may result in the formation of a scum which will not readily resuspend. The cooled suspension is filtered and packed into drums when it reaches 70° F. to 100° F.

The contents of the drum are ordinarily further diluted with cold water, such as at a temperature of 60° F., until the solids content ranges from about 0.1 percent to about 1 percent for application to the fabric. Concentrations in this range provide a solids content applied to the fabric in the range of about 0.1 percent to about 1.0 percent by weight of the fabric. The present lubricant composition can be applied to fabric by direct pickup by passing fabric through a pad, an apparatus for applying finishing chemicals to fabric in which the fabric is passed through the finishing chemicals and between two or more rolls one of which is usually rubber, or similar finishing equipment containing the present lubricant composition alone or together with other finishing chemicals. For instance, the lubricant composition of the present invention can be applied by direct pickup together with a wash-wear resin finish formula by passing the scoured, bleached, possibly mercerized and/or dyed fabric through a pad or similar finishing equipment containing both the wash-wear finishing chemicals and the present lubricant composition; the previously described amounts of nonionic must be present for compatibility purposes when wash-wear chemicals are coapplied to the fabric. Or the present lubricant composition can be applied by direct pickup in a pad as a top dressing; the application of lubricant composition to the fabric takes place after the fabric has been resin treated. Or the present lubricant composition can be applied by exhaustion; in the exhaustion method the fabric is placed in intimate contact with a lubricant composition in acidified cationic form for 0.5 minutes to 15 minutes in a batch operation, and the wax is attracted to the fabric by ionic attraction. For application by exhaustion the concentrated lubricant composition of the present invention, preferably containing at least about 60 percent water, is diluted with 10 to 40 times the fabric's weight of water at room temperature and the dilute composition is then made slightly acidic, such as pH from 6.5 to 3.5, with a weak organic acid, such as, acetic acid. The concentration of concentrated lubricant composition of the present invention in the finish bath can range from about 0.5 percent to about 4 percent by weight of the fabric.

Fabric sewability generally improves, that is, fewer yarns are cut during sewing, with increased percent applied solids, wax and emulsifiers, deposit of which in turn depends on increased concentration of solids in the applied suspension.

The following examples are illustrative of this invention and are not to be construed in any way as limiting the scope of the present invention.

EXAMPLE I

Sixty gms. of oxidized Fischer-Tropsch wax having a chain length ranging from 45 to 50 carbon atoms, a melting point range of 205° F. to 209° F., an acid value of 32 to 34 and a penetration ranging from 4 to 6 (Shanco W-1123 manufactured by Shanco Plastics and Chemicals, Inc.), 2.0 gms. of nonionic emulsifier consisting of rosin fatty alcohols reacted with about 40 moles of ethylene oxide (Hercules AD 400 which was previously described in detail) and 18 gms. of "Ethoquad 18/12," a commercial cationic emulsifier, were made into a lubricant composition (the wax and emulsifiers comprising 20 percent of the lubricant composition and water comprising 80 percent of the lubricant composition) by the previously described procedure. The commercial cationic emulsifier "Ethoquad 18/12," is a solution consisting essentially of 25 percent isopropanol and 75 percent quaternary ammonium compound. The quaternary ammonium compound was previously described and has the hereinbefore described structural formula wherein R₁ is a stearyl group, R₂

is a methyl group, R₃ is an ethylene group, Z is a chloride ion, and x and y are each 1. The lubricant composition in the form of a homogeneous suspension contained about 0.45 parts nonionic to 3 parts cationic and about 3.9 parts wax to 1 part total emulsifier.

A finish bath was prepared as follows: three 100 ml. portions were made up; one by diluting 30 gms. of 50 percent cyclic ethylene urea (50 percent water), a wash-wear resin-water solution, with water; one by dissolving 4.5 gms. MgCl₂·6 H₂O, a wash-wear resin cross-linking catalyst, in water; and one by diluting 16 gms. of the above-described lubricant composition, containing 80 percent water, with water. The dissolved magnesium chloride catalyst was added to the cyclic ethylene urea with stirring at room temperature; then the diluted lubricant composition was added giving a total volume of 300 ml. The mix containing about 1 percent lubricant solids was stirred for about 5 minutes and was then ready for application to fabric.

Unfinished, bleached cotton sheeting, 80 by 80 and containing about 0.1 percent ethyl ether extractables, was cut into eight lengths of about 10 inches by about 80 inches, each length weighing about 56 gms., and the lengths passed through a pad having two rubber squeeze rolls adjusted for a finish pickup of about 70 percent by weight of the fabric, 39 gms., and containing the above-described finish bath. The fabric was passed through the pad bath and between the squeeze rolls two times with the ends reversed each time and at a rate of 9 yards per minute. The finished fabric was air dried at room temperature and cured in a circulating air oven for one minute at 340° F.

Other similar cotton sheeting was finished as above in 300 ml. baths containing (1) water only; (2) water, resin and catalyst only; and (3) water, resin and catalyst and 13 gms. of commercial nonionic polyethylene wax composition, containing 30 percent solids and having a wax-to-emulsifier ratio of 3:1, as a lubricant. The emulsifier was made up of nonyl phenol condensed with 9.5 moles of ethylene oxide and 20 percent potassium hydroxide (80 percent water) solution and contained 10 parts of nonyl phenol for each 3 parts of potassium hydroxide solution. The polyethylene wax had a melting point range of 213° to 221° F., an acid value range of 14 to 17, and a penetration range of 3 to 6.

Softness of the above-finished fabrics was graded by three experts using an absolute scale ranging from 1 to 10, 1 being very harsh and 10 being very soft. The grades between 1 and 10 are degrees of softness between the above-described extremes; the higher the grade, the softer the fabric. A softness grade of 2 is assigned to a cotton control padded through water with a roll pressure allowing 70 percent pickup by weight of the fabric. A grade of 8 represents outstanding softness.

Sewability was tested by determining the number of filling yarns cut per 12-inch seam in the sewing operation. A Singer Sewing Machine No. 253-211 with a No. 16 Needle and cotton thread (50/3 top, 60/3 bottom) was used to sew the fabric folded to four thicknesses with 12 stitches per inch at a machine speed of 5200 r.p.m. The yarns cut in the third thickness from the top during the above sewing operation were counted and the results reported in yarns cut per 12-inch seam.

Tear strength of filling yarns was tested according to Federal Specification CCC-T-191b, Method 5132 (May 15, 1951). Results are in terms of number of grams required to tear the sheeting tested.

The following table shows the results obtained upon evaluation of the fabrics for softness, fabric sewability and tear strength.

TABLE III

	Unfinished fabric (water only)	Resin treated (only)	Resin and nonionic emulsified polyethylene in bath	Resin and present lubricant in bath
Percent lubricant solids on fabric.....	0.0	0.0	0.9	0.75
Softness.....	2	1	4	5
Sewability ¹	360	440	100	25
Tear strength, gms.....	345	205	320	315

¹ Filling yarns cut per 12-inch seam.

As can be seen from the above table the lubricant composition of the present invention imparted outstanding lubricity; in fact, one-fourth as many yarns were cut in fabric treated by the composition of the present invention as compared to nonionic emulsified polyethylene-treated similar fabric. Anionic and cationic emulsified polyethylenes provide sewability results similar to or worse than nonionic emulsified polyethylene. The composition within the scope of the present invention also advantageously increased softness and tear strength of resin-treated fabric. Furthermore, the composition within the scope of the present invention was compatible with the magnesium chloride cross-linking catalyst.

EXAMPLE II

Six-foot lengths of cotton sheeting were resin treated by padding through a 300 ml. finish bath containing 30 gms. of 50 percent cyclic ethylene urea (50 percent water), 4.5 gms. $MgCl_2 \cdot 6H_2O$ and water. Padding was done at room temperature with 70 percent pickup by weight of the finish bath. The resin-treated lengths of cotton sheeting were air dried and cured for 1 minute at 340° F.

The resin-treated lengths of cotton sheeting were then padded through a 300 ml. finish bath containing 16 gms. of the 80 percent water-containing Fischer-Tropsch lubricant composition of example I. The finish bath contained about 1 percent lubricant solids. Other lengths of cotton sheeting, resin-treated as above, were padded through a 300 ml. finish bath containing 13 gms. of the nonionic emulsified, 30 percent solids-containing, polyethylene wax composition of example I. Padding was done at room temperature with 70 percent pickup by weight of the fabric. The fabric was padded through the pad bath and between the squeeze rolls two times with ends reversed each time and at a rate of 9 yds. per minutes and conditioned at 65 percent relative humidity and 70° F. The following table compares, in terms of softness, sewability and tear strength, water-treated sheeting, resin-treated sheeting top-dressed with nonionic emulsified polyethylene and resin-treated sheeting top-dressed with a lubricant composition of the present invention according to the tests described in example I.

TABLE IV

	Unfinished fabric (water treated)	Resin treated (only)	Resin finished and then top dressed with nonionic emulsified polyethylene in bath	Resin finished and then top dressed with present lubricant in bath
Percent Lubricant solids on fabric.....	0	0	0.9	0.7
Softness rating.....	2	1	2	6
Sewability ¹	360	440	155	60
Tear strength, gms.....	345	205	300	340

¹ Filling yarns cut per 12-inch seam.

As can be seen from the above table the lubricant composition of the present invention imparted outstanding softness, lubricity and tear strength when applied as a top dressing. Less than half as many yarns were cut in fabric treated with the composition of the present invention as in fabric treated with

nonionic emulsified polyethylene. Anionic and cationic emulsified polyethylene provide sewability results similar to nonionic emulsified polyethylene. Similar results are obtainable if the nonionic emulsifier is omitted from the Fischer-Tropsch wax-containing composition.

EXAMPLE III

One thousand grams of lubricant composition were prepared according to the previously described method from 255 gms. of Shanco W-1123, 36 gms. Ethoquad 18/12 and 18 gms., Hercules AD 400; these materials are described in example I. The composition contains about 2.6 parts nonionic to 3 parts cationic emulsifier and about 5.7 parts wax in homogeneous suspension to 1 part total emulsifier. The water percentage was 70 percent.

The above lubricant composition was applied by exhaustion to two 10- by 18-inch strips of 3.8 oz./sq. yd. treated cotton broadcloth. Each strip weighed 14.5 gms. and was treated in a 435 ml. bath which was 30 times the fabric's weight. The bath contained 4 percent of the above-70 percent water-containing lubricant composition, based on the weight of the fabric. The strips were treated at immersion times of one-half minute and 5 minutes, respectively. The strips, wet in soft water, were placed in the dilute, lubricant suspension baths acidified to pH of 4 with acetic acid; the baths were constantly stirred during the application period. The lubricated strips were air dried, heated for 15 minutes in a circulating air oven at 260° F. and conditioned at 65 percent relative humidity and 70° F. overnight. Two other similar strips of the same cotton broadcloth were treated in the same manner as above except that a 70 percent water-containing commercial nonionic-emulsified polyethylene lubricant composition as in Example I was substituted for the 70 percent water-containing lubricant composition of the present invention.

The following table compares in terms of sewability resin-treated cotton broadcloth exhausted as above with the above-

described lubricant composition, i.e. the present lubricant, and resin-treated broadcloth exhausted as above with the above-described commercial nonionic emulsified polyethylene wax and a control which was treated similarly in a lubricant-free bath.

TABLE V

Lubricant Composition	Time in Bath (mins.)	Fabric Sewability*
None	5	380
Nonionic polyethylene (30% solids)	½	260
Present Lubricant (30% solids)	½	210
Nonionic polyethylene (30% solids)	5	170
Present Lubricant (30% solids)	5	60

*Filling yarns cut per 12-inch seam according to the test described in example I.

As can be seen from the above table the lubricant composition of the present invention imparted outstanding lubricity when applied by exhaustion, especially with immersion times of 5 minutes; in fact, only about one-third as many yarns were cut in fabric exhausted with a composition of the present invention as compared to fabric similarly exhausted with nonionic emulsified polyethylene.

EXAMPLE IV

A lubricant composition containing 25.5 parts Shanco W-1123 (described in example I), 4.05 parts cationic emulsifier, 0.45 parts Hercules AD 400 (described in Example I) and 70 parts water was made by the procedure described previously, forming a homogeneous suspension. The cationic emulsifier was previously described and was formed by ethoxylating 1 mole of Armeen TD with 3 moles of ethylene oxide and quaternizing with benzyl chloride. The cationic emulsifier has the hereinbefore-described structural formula wherein R_1 is derived from hydrogenated tallow (65 percent stearyl, 33 percent cetyl and 2 percent myristyl), R_2 is a methyl phenyl group, R_3 is an ethylene group, Z is a chloride ion, x plus y equals 3 and x equals about 1.5. The composition contained about 0.33 parts nonionic to 3 parts cationic and about 5.7 parts wax to 1 part total emulsifier. Another similar lubricant composition was prepared by substituting emulsifiable polyethylene with a melting point range of 213° to 221° F., an acid value range of 14 to 17 and a penetration range of 3 to 6 for the Shanco wax W-1123 and Ethoquad 18/12 (described in example I) for the ethoxylated quaternized Armeen TD in the above composition. The above compositions were applied to cotton broadcloth in a pad together with a wash-wear resin as in example I at about a 0.6 percent lubricant solids, wax or polyethylene and emulsifiers, concentration.

The following table compares, in terms of sewability and tear strength, a resin-treated fabric control, the above resin-treated, cationic-nonionic emulsified polyethylene-finished fabric and the above resin-treated, cationic-nonionic emulsified Fischer-Tropsch wax-finished fabric according to the tests described in Example I.

TABLE VI

	Resin treated (only)	Resin and emulsified polyethylene treated	Resin and present lubricant treated
Percent lubricant solids on fabric.....	0	0.6	0.5
Sewability ¹	290	140	60
Tear strength, gms....	370	530	610

¹ Filling yarns cut per 12-inch seam.

As can be seen from the above table the present lubricant composition imparted outstanding lubricity and tear strength

when compared to a control and to a cationic-nonionic emulsified polyethylene lubricant composition.

EXAMPLE V

About 1200 lbs. of lubricant composition was prepared according to the previously described procedure from 417.5 lbs. of Shanco W-1123 (described in example I), 44.5 lbs. Ethoquad 18/12 (Ethoquad 18/12 is described in Example I), 18 lbs. of coconut alcohol ethoxylated with 45 moles ethylene oxide and about 730 lbs. water. This lubricant composition is the preferred one within the scope of the present invention and contains about 1.6 parts nonionic to 3 parts cationic and about 8 parts wax to 1 part total emulsifier. The water concentration was about 60 percent. The composition was a homogeneous suspension.

The above lubricant composition was applied in a pad to 80-by-80-inch cotton sheeting.

The following table compares as to softness and sewability unfinished fabric, fabric treated with the present lubricant and fabrics treated with the commercial nonionic emulsified polyethylene mentioned in example I according to the tests described in example I.

TABLE VII

	Unfinished control	Nonionic polyethylene treated, percent	Present lubricant composition treated, percent
Percent solids in product.....		30	30
Suspension concentration used.....		4	2
Percent solids deposited on the fabric at 70% pickup.....		0.84	0.55
Softness rating.....	2	5	80
Sewability ¹	290	9	0

¹ Filling yarns cut per 12 inch seam.

As can be seen from the above table the preferred present lubricant composition imparted outstanding lubricity and softness when compared to a control and to a nonionic emulsified polyethylene.

The following oxidized Fischer-Tropsch waxes can be substituted for the Fischer-Tropsch wax in example I with similar results: (1) an oxidized Fischer-Tropsch wax having a chain length ranging from 45 to 50 carbon atoms, a melting point ranging from 212° to 215° F., an acid value ranging from 10 to 12 and a penetration ranging from 1 to 2.5; (2) an oxidized Fischer-Tropsch wax having a chain length ranging from 45 to 50 carbon atoms, a melting point ranging from 211° to 215° F., an acid value ranging from 14 to 16 and a penetration ranging from 1 to 3; (3) an oxidized Fischer-Tropsch wax having a chain length ranging from 45 to 50 carbon atoms, a melting point ranging from 109° to 213° F., an acid value ranging from 18 to 22 and a penetration ranging from 2 to 4; and (4) an oxidized Fischer-Tropsch wax having a chain length ranging from 45 to 50 carbon atoms, a melting point ranging from 207° to 212° F., an acid value ranging from 24 to 26 and a penetration ranging from 3 to 5.

The following cationic emulsifiers can be substituted for the Ethoquad 18/12 in example I with similar results: the cationic emulsifiers having the previously described structural formula wherein (1) R_1 is a myristyl group, R_2 is a methyl group, R_3 is an ethylene group, Z is a CH_3SO_4 ion, x is 2 and y is 0; (2) R_1 is a stearyl group, R_2 is a phenyl group, R_3 is a propylene group, Z is a SO_4 ion, x is 1 and y is 0; (3) R_1 is a stearyl group, R_2 is a naphthyl group, R_3 is an ethylene group, z is an iodide ion, x is 2 and y is 1; (4) R_1 is an eicosyl group, R_2 is a propyl group, R_3 is an ethylene group, Z is a chloride ion, x is 4 and y is 2; and (5) R_1 is an eicosyl group, R_2 is an ethylphenyl group, R_3 is a butylene group, Z is an NO_3 ion and x is 1 and y is 0.

Hydrogenated tallow ethoxylated with 40 moles of ethylene oxide, stearyl alcohol ethoxylated with 30 moles of ethylene oxide, nonyl phenol condensed with 9.5 moles of ethylene oxide and isoctylphenylpolyethoxy ethanol containing 10

moles of ethylene oxide can be substituted for the Hercules AD 400 in example I with similar results.

The lubricant composition of the present invention can also be applied to fiber, yarn, thread, knit fabric and nonwoven fabric. It also has utility as a fiber lubricant for the picking, carding, spinning, plying, twisting, winding, weaving, knitting, napping, and calendering processes. It is further applicable to rayon and synthetics such as nylon, polyamide, polyester, polyethylene, polypropylene, and acrylics; and to cellulosic materials, such as paper, when surface lubricity is important as in calendering, coating and impregnating. The lubricant composition of the present invention has further utility as a wax or polish for coating exposed surfaces of paint, wood, metal.

The present composition can also be employed to impart increased tear strength, wear and abrasion resistance, and softness to durable-press finished cotton and cotton-synthetic fiber blends, especially cotton-polyester and cotton-nylon blends.

In conventional durable-press finishing, a cotton or cotton-synthetic fabric is first impregnated with a solution, applied as a finish bath, comprising durable-press reactant, acidic catalyst, wetting agent, and water. The impregnated fabric is then partially dried under time and temperature conditions which do not cause reaction between the fabric fibers and applied reactant. The dried fabric can be stored for up to 6 months before it is made into a garment by cutting and sewing. The garment, in turn, is creased and shaped by pressing. This pressing operation causes a reaction between the reactant and fibers and permanently sets the creases and shape of the garment. The pressed garment can also optionally be cured additionally after pressing, thereby causing a further reaction to take place. The garment so processed is resistant to the wrinkling ordinarily caused by wearing or laundering, and retains the shape and creases permanently set therein despite numerous launderings and without the necessity of ironing the garment after each of these numerous launderings.

The above-mentioned durable-press finish bath ordinarily comprises, by weight, from about 3 percent to about 15 percent durable-press reactant, from about 0.25 percent to about 2 percent acidic catalyst, from about 0.1 percent to about 0.4 percent wetting agent, and water. This finish bath is applied to the fabric by conventional textile treating methods, for example, by padding. The amount of durable-press reactant applied to the fabric in this manner ranges from about 3 percent to about 15 percent by weight of the fabric.

Durable-press reactants for use in this finish bath include, for example, dimethylol hydroxyethyl carbamate, dimethylol ethoxyethyl carbamate, dimethylol cyclic ethylene urea, dimethylol dehydroxyethylene urea and dimethylol cyclic propylene urea.

Other useful durable-press reactants are described in U.S. Pat. No. 3,049,446, granted Aug. 14, 1962, to Herman B. Goldstein and Michael A. Silvestri. These reactants are prepared by forming an aqueous solution of 1 mole urea, 1 mole glyoxal and 2 to 2.5 moles formaldehyde, and thereafter reacting the urea, glyoxal and formaldehyde at a pH ranging from 4.0 TO 6.0 and at a temperature of 25° C. to 95° C. for a period of time ranging from 30 minutes to 72 hours. These reactants are referred to hereinafter as urea-glyoxal-formaldehyde reaction product.

Acidic catalysts for use in the durable-press finish bath include, for example, zinc nitrate, zinc chloride, and magnesium chloride.

Wetting agents for use in this durable-press finish bath include, for example, ethoxylated alkyl phenols and ethoxylated aliphatic alcohols. The ethoxylated alkyl phenols have six to 12 carbon atoms in their alkyl groups and contain from 5 to 25 moles of ethylene oxide. The ethoxylated aliphatic alcohols have eight to 18 carbon atoms in their aliphatic groups and contain from 5 to 30 moles of ethylene oxide. The alkyl and aliphatic groups can be in either straight chain or branched chain configuration.

The above-mentioned drying step is carried out by exposing the impregnated fabric to a temperature ranging from room

temperature to about 250° F. just until the residual fabric moisture content ranges from about 2 percent to about 8 percent by weight of the fabric over and above the natural moisture content of the fabric. In one conventional process, finish-bath-treated fabric is dried by exposing it to a temperature of 250° F. for 30 seconds in a circulating air oven.

In the above-mentioned pressing step, pressure is maintained for a time period ranging from about 5 seconds to about 30 seconds. The temperature maintained during pressing ranges from about 300° F. to about 340° F. Often steam is applied to the fabric at the start of pressing and a vacuum at the end. For example, a pressing procedure can consist of 5 seconds of steaming, 15 seconds of dry heat, and 5 seconds of vacuum after which the pressure is released. This step is carried out with conventional pressing apparatus, for example, hot head presses, steam presses, and hand irons.

The optional curing step is carried out by heating at a temperature ranging from about 250° F. to about 400° F., preferably 300° F. to about 340° F. for a time period ranging from about 2 minutes to about 30 minutes. This step can be carried out, for example, in a circulating air oven.

The above-described benefits of imparting increased tear strength, wear and abrasion resistance, and softness to durable-press finished fabric is achieved by applying to cotton or cotton-synthetic fabric a conventional durable-press finish bath, described above, containing additionally, the present lubricant composition in an amount sufficient to give a concentration in the finish bath of from about 0.2 percent to about 4.0 percent by weight lubricant solids (wax and emulsifiers), and carrying out the drying, pressing and curing steps conventionally. Preferably, the finish bath contains from about 1 percent to about 4 percent of the preferred lubricant composition herein which contains by weight 34.8 percent wax, 1.5 percent nonionic emulsifier, 2.8 percent cationic emulsifier, and water (the lubricant composition of example V). The lubricant composition solids content applied to the fabric ranges from about 0.25 percent to about 1.1 percent by weight of the fabric. The term synthetic is used herein to include those synthetic materials listed on lines 2 and 3 of page 25 herein.

The following example is illustrative of the application of the present lubricant composition together with durable-press reactants. The test for tear strength employed in this example is that previously described herein.

EXAMPLE VI

A finish bath is prepared as follows. One hundred grams of dimethylol cyclic ethylene urea are diluted with water to about 500 ml. at room temperature. Eighteen grams of a lubricant composition prepared as in Example V are diluted to about 200 ml. at room temperature with water containing 2.5 grams of ethoxylated nonyl phenol containing 15 moles of ethylene oxide. The diluted lubricant composition is then added to the diluted dimethylol cyclic ethylene urea with stirring. Next, 36 grams of 25 percent aqueous zinc nitrate having been diluted to 200 ml. with water at room temperature is added to the previously prepared mixture. Sufficient water is then added to make up 1 liter of finish bath.

The formed finish bath contains by weight 10 percent dimethylol cyclic ethylene urea, 0.9 percent zinc nitrate catalyst, 0.25 percent ethoxylated nonyl phenol, 0.7 percent lubricant solids, and water.

The above finish bath is applied to 100 percent mercerized cotton broadcloth, 12 feet long and 10 inches wide, in a pad at 120° F. A finish bath pickup of 55 percent is achieved. The fabric picks up 5.5 percent of its weight of durable-press reactant and 0.4 percent of its weight of lubricant composition solids.

The fabric is then allowed to dry at room temperature until it contains 12.5 percent moisture, i.e., 5 percent moisture over and above its natural moisture content. The dried fabric is cut into swatches, 11 inches by 11 inches, and each fabric swatch is then folded to produce a crease in the warp direction and

two thicknesses of fabric 11 inches by 5 5 inches. The folded swatches are then pressed at a temperature of 325° F. for 15 seconds employing two aluminum plates which impart a total pressure of about 6 pounds. The pressed swatches are cured by maintaining them at a temperature of 340° F. for 15 minutes in a circulating air oven.

Other fabric is treated as above except in one case no lubricant composition is employed in the finish bath, and in another case a lubricant composition containing the commercial nonionic emulsified polyethylene defined in Example 1 is substituted for the lubricant composition of the present invention in the finish bath.

The following table compares as to tear strength the fabric treated as above:

TABLE VIII

	Durable press reactant treated (only)	Durable press reactant and nonionic emulsified polyethylene in bath	Durable press reactant and lubricant in bath
Percent lubricant solids on fabric.....	0	0.5	0.4
Tear strength, gms.....	500	800	980

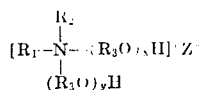
As can be seen from the above table, the present lubricant composition imparts increased tear strength to durable-press finished fabric when compared to a control and to nonionic emulsified polyethylene. The reactant-treated fabric treated with the present lubricant compositions also demonstrates increased wear and abrasion resistance and softness compared to the control and fabric treated with nonionic emulsified polyethylene.

Similar results are obtainable herein with other lubricant compositions of the present invention. In like manner, similar results are obtainable when the present lubricant composition is applied together with other durable-press reactants, catalysts and wetting agents, in that increased tear strength, wear and abrasion resistance and softness are imparted to the durable-press finished fabric. For example, dimethylol hydroxyethyl carbamate, dimethylol ethoxyethyl carbamate, dimethylol dihydroxyethylene urea, dimethylol cyclic propylene urea, and urea-glyoxalformaldehyde reaction product can be substituted for the dimethylol cyclic ethylene urea above. Zinc chloride and magnesium chloride catalysts can be substituted for the zinc nitrate catalyst employed above. Moreover, the previously described ethoxylated aliphatic alcohol wetting agents can be substituted for the ethoxylated alkyl phenol wetting agent used above.

I claim:

1. In the method of applying durable-press finish baths containing N-methylolated cellulose reactants to cellulosic fabrics, the improvement comprising including in such baths from about 10.2 to about 4 percent by weight of lubricant solids, of a lubricant composition consisting essentially of a homogeneous water suspension of an oxidized Fischer-Tropsch wax having a chain length of from about 40 to about 55 carbon atoms, a melting point ranging from about 200° F. to about 220° F., an acid value ranging from about 10 to about 35 and a penetration ranging from about 1 to about 6, emulsified by an emulsifier selected from the group consisting of:

a. a cationic quaternary ammonium compound having the formula:



wherein R_1 is an alkyl group containing 14 to 20 carbon atoms; R_2 is selected from the group consisting of alkyls containing one to three carbon atoms, phenyl, naphthyl, and C_1 - C_3 alkyl-substituted phenyls, R_3 is an alkylene group containing two to four carbon atoms, Z represents an anion, and x plus y ranges from 1 to 6 and x ranges from 1 to 6, and

b. mixtures of said ammonium compound with a nonionic emulsifier selected from the group consisting of:

- ethoxylated n-alkyl alcohols having from 10 to 14 carbon atoms,
- ethoxylated rosin alcohols, and
- ethoxylated alkyl phenols wherein the alkyl has from eight to 10 carbon atoms.

said nonionic emulsifier containing at least about 8 moles of ethylene oxide, wherein nonionic emulsifier content ranges from 0 to 7 parts for every 3 parts of cationic emulsifier, and the ratio of oxidized Fischer-Tropsch wax to total emulsifier ranges from about 10:1 to 3:7.

2. A method for imparting increased tear strength to durable-press finished fabric, said method comprising the steps of:

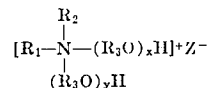
- padding said fabric through a finish bath comprising by weight from about 3 percent to about 15 percent durable-press reactant, from about 0.25 percent to about 2 percent acidic catalyst, from about 0.1 percent to about 0.4 percent wetting agent, from about 0.2 percent to about 4 percent lubricant solids supplied by a lubricant composition of claim 1, and water; said durable-press reactant being selected from the group consisting of dimethylol hydroxyethyl carbamate, dimethylol ethoxyethyl carbamate, dimethylol cyclic ethylene urea, dimethylol cyclic propylene urea, and urea-glyoxalformaldehyde reaction product, said acidic catalyst being selected from the group consisting of zinc nitrate, zinc chloride, and magnesium chloride; said wetting agent being selected from the group consisting of ethoxylated alkyl phenols having six to 12 carbon atoms in the alkyl group and containing 5 to 25 moles of ethylene oxide, and ethoxylated aliphatic alcohols having eight to 18 carbon atoms in the aliphatic group and containing 5 to 30 moles of ethylene oxide;

b. drying the padded fabric;

c. pressing the dried fabric at a temperature ranging from about 300° to about 400° F., said temperature and pressure being maintained for a period ranging from about 5 seconds to about 30 seconds to impart permanent creasing and shaping thereto.

3. The method of claim 2, wherein the pressed fabric is additionally cured by maintaining it at a temperature ranging from about 250° F. to about 400° F. for from about 2 minutes to about 30 minutes.

4. The method of claim 2 wherein the lubricant solids are supplied by a composition consisting essentially of 34.8 percent wax, 1.5 percent coconut alcohol with 45 moles of ethylene oxide, water and 2.85 percent of compound having the formula:



wherein R_1 is a stearyl group, R_2 is a methyl group, R_3 is an ethylene group, Z is a chloride ion and x and y are each 1 and (b) a compound with the above structural formula wherein R_1 is derived from hydrogenated tallow (65 percent stearyl, 33 percent cetyl and 2 percent myristyl), R_2 is a methyl phenyl group, R_3 is an ethylene group, Z is a chloride ion, x plus y equals 3 and x equals about 1.5.

* * * * *

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,620,665 Dated November 16, 1971

Inventor(s) Arthur W. Lanner

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 5, line 3, delete "01" and insert therefor --- 0.1 ---.

Column 6, line 2, after "1" and before "The", insert --- . ---.

Column 7, line 30, delete " $6bH_2O$ " and insert therefor --- $6H_2O$ ---.

Column 10, line 54, delete " 109° " and insert therefor --- 209° ---.

Column 13, line 55, delete "10.2" and insert therefor --- .2 ---.

Signed and sealed this 6th day of June 1972.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents