ABSTRACT OF THE DISCLOSURE

A process for treating fibers before carding and combing to form yarn to provide increased strength and elongation of the fibers and to reduce the buildup of static electricity during handling is provided which comprises applying to the fibers a reaction product which is obtained from a compound having an alkyl group containing from 2 to 22 carbon atoms at least one nitrogen atom and at least one reactive hydrogen atom, and from two alkylene oxides, one being ethylene oxide containing two carbon atoms, and the other having at least three carbon atoms in the molecule.

This invention relates to methods of treating textile fibers prior to forming them into yarn and particularly to a process of treating textile fibers prior to carding and combing.

In the processing of woolen and worsted systems, it is usual to treat the textile fibers with various auxiliaries which tend to facilitate carding, combing and subsequent spinning. These textile aids are usually dispersed, emulsified or dissolved in water. Some of these textile aids, e.g., oleic acid or mineral oil, are, under normal conditions, practically insoluble in water and have been used as emulsified systems. In more recent years, textile aids, which are soluble or dispersible in water without the addition of dispersing agents, have been used. One such system is a water solution of alkylene oxide addition products of carboxylic acids.

There are different opinions about the working mechanism of these textile aids. A current opinion regarding this mechanism is that these aids should act as fiber lubricants. However, in the case of wool fibers, it is probable that the fiber/fiber friction is comparatively low, and thus it should not be necessary to add lubricating agents for carding, combing and subsequent spinning of wool fibers. In such cases it is obvious that some other function is served. On the other hand, for those fibers, such as terylene and acrylic fibers, in which the fiber/fiber friction is comparatively high, the addition of a lubricating agent would seem necessary or at least desirable for carding, combing and subsequent spinning.

It is probable that one of the most important functions of the textile aids would be to provide some desired adhesion between fibers. An additional function, particularly in the case of synthetic fibers, would be to prevent or counteract the build-up of static electricity. Further, in cases where these textile aids are not removed from the fabric, as in the manufacture of certain furniture cloths and carpets, there should be no product breakdown or deterioration resulting, for example, in unpleasant odors. It is also desirable if the auxiliary improves the hand of the fabric.

These textile aids may be liquid or solid at room temperature. It is usually preferred to apply liquid aids, often denoted as spinning oils, but it is also possible to utilize solids or fats, called spinning fats. For the matter of simplicity, these agents used for carding, combing and subsequent spinning will be called spinning oils whether they are liquid or solid.

It has been discovered that essential advantages can be achieved in carding, combing and subsequent spinning, if these processes are carried out in presence of a spinning oil which is a reaction product obtained from a compound having at least one nitrogen atom and at least one reactive hydrogen atom, and from two alkylene oxides, one of which contains two carbon atoms, ethylene oxide, and the other at least three carbon atoms in the molecule.

One can obtain one or more of the following advantages by use of these new spinning oils:

1. Increased adhesion between the fibers;
2. More uniform yarn;
3. Increased strength of the yarn;
4. Increased elongation of the yarn;
5. Improved yield;
6. Increased spinning speeds without increased number of breakages;
7. Increased smoothness of the yarn;
8. Improved yarn properties at weaving;
9. Dyeing of yarns without previous scouring;
10. Good dye fastness without dullness;
11. Improved lustre of the fabric;
12. Improved hand of the fabric;
13. Reduction of static electricity;
14. Elimination of objectionable odor in those cases where the spinning oil is not removed from a fabric;
15. The interval between cleaning of card clothing can be extended.

The favorable properties in use of these new spinning oils can be ascribed, I believe, to the simultaneous presence of at least one nitrogen atom, ethylene oxide units, and alkylene oxide units having at least three carbon atoms in the molecule. Yarns manufactured in accordance with the new process can be used for clothes, carpets and the like.

These favorable properties in use of these new spinning oils can be explained in a number of ways. One possible and seemingly plausible explanation follows. Of course, the favorable properties of these new oils are not intended to be restricted by the validity of the explanation which is simply a possible theory for their effectiveness.

One can start from the assumption that the polyglycol chains, which are formed by the ethylene oxide units and the alkylene oxide units having at least three carbon atoms in the molecule, are mainly responsible for the desired textile technological properties in facilitating carding, combing and subsequent spinning of the fibers. Of these properties, good adhesion between the fibers is of prime importance. The role of the nitrogen atoms appears to be to make it possible for the spinning oil film to adhere more firmly to the fibers and thus the favorable textile technological properties of the film can be asserted to a much higher degree. Some of the above mentioned advantages, such as increased smoothness of the yarn and a better hand of the fabric, can possibly also be ascribed to an interaction of the nitrogen atoms with the other constituents of the molecule.

In the following, as well as previously, a reactive hydrogen atom means a hydrogen atom which reacts with an alkylene oxide under ring opening.

A compound having at least one nitrogen atom and at least one reactive hydrogen atom can react with alkylene oxides in different ways. (a) The compound in question can first react with an alkylene oxide having at least three carbon atoms in the molecule. Subsequently, ethylene oxide can be added to the obtained reaction product. (b) Another possibility consists of first reacting a compound having at least one nitrogen atom and at least one reactive hydrogen atom with ethylene oxide, and thereafter adding an alkylene oxide, containing at least three carbon...
3,723,173 atoms in the molecule, to the reaction product. Further possibilities are that a compound having at least one nitrogen atom and at least one reactive hydrogen atom reacts (c) before or (d) after addition of ethylene oxide with a mixture of alkylene oxides containing at least 40% b.w. alkylene oxides having at least three carbon atoms in the molecule, and at most 60% b.w. ethylene oxide. Of course, other modes of procedure can also be considered.

The textile technological properties, changes in the chemical composition of the resulting molecules, consistency and the like in the new spinning oils can be effected by the different modes of procedure in their manufacture.

The reaction of a compound with ethylene oxide and alkylene oxides, containing at least three carbon atoms in the molecule, can be carried out in accordance with known methods in presence of alkaline catalysts at elevated temperature and pressure. The final product so obtained contains a mixture of homologues containing a different number of ethylene oxide units, and alkylene oxide units having at least three carbon atoms in the molecule.

As examples of compounds having at least one nitrogen atom and at least one reactive hydrogen atom may be mentioned: Mono- and dialkyldiamine having from 2 to 22 carbon atoms in the alkyl group, cyclohexylamine, monoethanolamine, diethanolamine, mono propyleneamine, dipropyleneamine, hexamethylene amine, acetamide, benzeneammonium; ethylenediamine, N-alkyl ethylenediamine having from 2 to 22 carbon atoms in the alkyl group, piperazine, imidazolines, propylenediamine, N-alkyl propylenediamine having from 2 to 22 carbon atoms in the alkyl group, 1,3-diaminopropane, hexamethylene diamine, phenylenediamine, diethylenetriamine, triethylenetetramine, tetraethylenepentamine, dipropylenetriamine.

Examples of alkylene oxides having at least three carbon atoms in the molecule which may be used are: propylene oxide, butylene oxide, amylene oxide.

To obtain the required effect, it is desirable that the number of nitrogen atoms, ethylene oxide units, and alkylene oxide units, having at least three carbon atoms in the molecule, are in a certain proportion to one another. Thus, in particular cases, one nitrogen atom, especially if it is tertiary, may be sufficient in the molecule. However, for a general applicability, it is often of advantage if two tertiary nitrogen atoms are in the molecule. Such compounds can be obtained from e.g. polyalkylene polyamines having the general formula:

\[ \text{H}_2\text{N}-\text{C}_n\text{H}_{2n+1}-(\text{NH}-\text{C}_m\text{H}_{2m+1})_r-\text{NH}_2 \]

wherein \( n \) is an integer from 2 to 6, and \( r \) is zero or an integer from 1 to 3. If compounds of this type react with ethylene oxide, and an alkylene oxide containing at least three carbon atoms in the molecule, the reaction can be carried out according to one of the modes of procedure (a) to (d).

If polyalkylene polyamines react with two alkylene oxides of the above mentioned type according to mode of procedure (a), compounds having the general formula are obtained:

\[ \text{H}(\text{CH}_2\text{O})_n(\text{CH}_3\text{OH})_m(\text{CH}_2\text{O})_k(\text{CH}_3\text{OH})_l \]

wherein \( n \) is an integer from 2 to 6, \( r \) is zero or an integer from 1 to 3, \( m \) is an integer from 3 to 5, \( a \) is an integer which is in average from 2 to 120, and \( b \) is an integer which is in average from 1 to 40.

Starting from ethylenediamine and in case that \( m=5 \), compounds having the following general formula are obtained:

\[ \text{H}(\text{CH}_2\text{O})_n(\text{CH}_3\text{OH})_m(\text{CH}_2\text{O})_k(\text{CH}_3\text{OH})_l \]

wherein \( a \) is an integer which is in average from 2 to 120, and \( b \) is an integer which is in average from 1 to 40.

The integrals \( a \) and \( b \) in the above formulas denote average numbers and are only an indication regarding the total content of ethylene oxide units, and alkylene oxide units, having at least three carbon atoms in the molecule, in a given product but not about their distribution in the homologues present in this product.

In the following, some points of view are given regarding the properties of these new spinning oils which are desirable.

It is usual for the spinning oil to have a liquid consistency at the temperatures at which it is applied. Products, which are liquid above 8° C., are obtained if in the last above mentioned general formula \( a \) is an integer from 5 to 15, and \( b \) an integer from 2 to 13.

Another property which must be taken into consideration is the solubility of the spinning oil in water. Before application to the fiber systems, the spinning oil is, in general, dissolved in water. Consequently, the number of hydrophilic ethylene oxide units in the molecule has to be large enough to bring about solubility in water. At a certain concentration, the solution of the spinning oil in water becomes cloudy upon warming to a certain temperature. This so called cloud point is a good measure of the hydrophobic-hydrophilic balance in the molecule. It is of advantage in this application, if the cloud point in a 10% solution of the spinning oil is between 40° and 80° C.

An additional very important property is, as already pointed out, the adhesive force exerted by the spinning oil or its solution in water on the textile fibers. This adhesive force can be measured by various physical methods and it is possible to obtain some preliminary information regarding the magnitude of these forces. However, in view of the many kinds of textile materials and various other factors which may be of importance in this connection, it is recommended that these measurements be made and evaluated in conjunction with actual trial runs.

In some cases which are met in actual runs of carding, combing and subsequent spinning of wool fibers, synthetic fibers and their blends, it may occur that a product having at least one nitrogen atom, ethylene oxide units and alkylene oxide units, containing at least three carbon atoms in the molecule, does not exhibit all the required properties which in particular cases may be desirable for its use as spinning oil. In such cases, the product can be combined with one or more other substances without loss of effectiveness. Since the products, containing at least one nitrogen, ethylene oxide units, and alkylene oxide units, in their solutions in water are used for oiling of fibers before carding, combing and subsequent spinning, are mainly of nonionic character, said products can be combined with other nonionics, as well as with anionic and cationic substances. For example, if the product containing at least one nitrogen atom, and alkylene oxide units as described above does not counteract the build-up of static electricity satisfactorily, the addition of an antistatic agent may be necessary. Examples of such addition compounds could be: sulfated derivatives of fatty alcohols or ethoxylated fatty alcohols having from 8 to 20, preferably from 12 to 18 carbon atoms in the fatty chain, sulfated derivatives of vegetable oils, e.g. castor oil, and sulfated ethoxylated alkylphenols having from 6 to 12 carbon atoms in the alkyl group. Examples of other types of addition compounds with antistatic properties may be mentioned phosphoric acid esters of fatty alcohols.
containing from 8 to 20, preferably from 12 to 18 carbon atoms, phosphoric acid esters of ethylene oxide adducts of these fatty alcohols, and phosphated ethoxylated alkyl-phenols having from 6 to 12 carbon atoms in the alkyl group, such as ethoxylated nonynonylphenol.

The adhesion between textile fibers can be increased in certain cases by the addition of polyethylene glycol esters of resin acids, e.g. ethoxylated abietic acid, and polyethylene glycol esters of resin and fatty acids, e.g., tall oil. Products obtained by the addition of ethylene oxide to polypropylene glycols having an average molecular weight from 900 to 3,500 are other additives which can modify adhesion. Of the additional substances mentioned above, one or more may be incorporated in the spinning oil.

The spinning oil is usually dissolved in water before application. The concentration of the solutions varies, depending on the material which is to be treated, but is preferably between 5% and 35% by weight.

In order to examine the applicability of the reaction products as spinning oils, laboratory tests have been carried out taking into account the points mentioned previously. Moreover, fabric-scale trials have been carried out on machines normally used in textile mills. Since different textile materials were used in these plant scale trials, those products were selected, which, based on the laboratory tests, were considered to be generally applicable as spinning oils, namely reaction products from polyethylene polymer, propylene oxide and ethylene oxide. Of these products, specific products obtained from the reaction of ethylenediamine, propylene oxide and ethylene oxide (in the examples denoted as alkoxylated ethylenediamine) were examined.

At the fabric-scale trials, comparison tests were carried out with the same textile material treated in some cases with solutions of spinning oils in water according to this new application, and in others with solutions of spinning oils in water which are previously employed in the textile mills. The latter spinning oils included a conventional water-soluble nonionic compound, a conventional miner oil-based spinning oil emulsified in water system, and a water-soluble anionic (alkyl ether sulphate) product.

The results are given in the Examples 1 to 9. The concentration of spinning oil is calculated on the weight of the material.

**EXAMPLE 1**

A lot of dyed 100% glossy rayon consisting of a blend of 60% (denier) and 40% 12 den rayon was oiled with 2.7% of an oil blend containing 1.7% of the water-soluble nonionic compound, and 1% of the conventional mineral oil-based spinning oil. Another lot of the same rayon was oiled with 2% of alkoxylated ethylenediamine according to this invention. With the latter spinning oil, a yarn was obtained in which the strength was about 15% and the elongation about 11% higher than that yarn which had been treated with the conventional oil-based spinning oil. No difficulties were encountered with regards to static electricity.

**EXAMPLE 2**

A lot of dyed 100% acrylic fibers was oiled with 1.1% of conventional oil blend containing 0.6% water-soluble nonionic spinning oil, and 0.5% alkyl ether sulphate as antistatic agent. Another lot of dyed 100% acrylic fibers was oiled with an oil blend containing 1% alkoxylated ethylenediamine and 0.3% alkyl ether sulphate according to this invention. Strength and elongation of the yarns were similar in both cases. No difficulties were encountered with regards to static electricity.

**EXAMPLE 3**

A lot of dyed 100% rayon containing 60% material of 7 den and 40% material of 12 den was oiled with 1.8% of a conventional oil blend containing 1.3% water-soluble nonionic spinning oil, and 0.5% alkyl ether sulphate. Another lot of the same material was oiled with 1.5% alkoxylated ethylenediamine according to this invention. The strength of the yarn increased by 19% in the latter case treated according to this invention. The elongation was similar in both cases.

**EXAMPLE 4**

A lot of dyed 100% wool was oiled with a conventional oil blend containing 2% of water-soluble nonionic, and 1% of mineral oil-based spinning oil. Another lot of the same material was oiled with 2% of alkoxylated ethylenediamine according to this invention. In the latter case, using this invention the strength of the yarn increased by about 8%, the elongation by about 10% and, in addition, the yarn had a much better hand.

**EXAMPLE 5**

A lot of undyed 100% rayon of 4 den was oiled with a conventional oil blend containing 1.5% of water-soluble nonionic spinning oil, and 0.5% of alkyl ether sulphate. Another lot of the same material was oiled with a 1% of alkoxylated ethylenediamine according to this invention. In the latter case, the strength of the yarn increased by about 19% and the elongation by about 14%.

**EXAMPLE 6**

A lot of undyed 100% wool of the same sort as in Example 4 was oiled with a conventional oil blend containing 2% of water-soluble nonionic, and 1% of mineral oil-based spinning oil. Another lot of the same material was oiled with 1.8% of alkoxylated ethylenediamine according to this invention. In spite of the lower oil content in the latter case (1.8% against 3%), its elongation increased by about 8% and the strength of the yarn was practically unchanged.

**EXAMPLE 7**

A lot of dyed 100% wool was oiled with an oil blend containing 2% of water-soluble nonionic, and 1% of mineral oil-based spinning oil. Another lot of the same material was oiled in accordance with this invention with 2% of alkoxylated ethylenediamine. The strength of the yarn oiled with the alkoxylated ethylenediamine increased by about 11% and its elongation by about 8%.

**EXAMPLE 8**

A lot of dyed material containing 40% wool and 60% rayon was oiled with 2.7% of an oil blend containing 1.7% of the water-soluble nonionic, and 1% of the mineral oil-based spinning oil. Another lot of the same dyed material was oiled with 1.7% of the alkoxylated ethylenediamine according to this invention. Spinning proceeded satisfactorily and the yarn, in the latter case, exhibited the same strength as in the former. Moreover, the yarn in the latter case exhibited good fastness of the dye without dullness.

**EXAMPLE 9**

A lot of dyed material containing 30% wool, 40% reconstituted wool, and 30% rayon was oiled with 2% of the water-soluble nonionic, and 1% of the mineral oil-based spinning oil. Another lot of the same material was oiled by the process of this invention with 1.75% of the alkoxylated ethylenediamine. Strength and elongation of the yarns were the same in both cases in spite of the lower oil content in the latter case (1.75% against 3%). It should be pointed out particularly that the presence of reconstituted wool with its comparatively high residual fat content, which may amount to 5-7%, often gives rise to difficulties in carding and spinning operations.

The foregoing examples show the marked improvement which can be achieved by the practice of this invention in all types of textile fibers. The process of this invention will, in general, increase both the strength and elongation of the fiber as well as eliminate the problems of static electricity with a considerable improvement in handling textile fibers.

While I have illustrated and described certain preferred practices and embodiments it will be understood that this
invention may be otherwise embodied within the scope of this disclosure.

I claim:

1. A process for treating textile fibers before carding, combing and spinning to form yarn to provide increased strength and elongation of the fibers and to reduce the build-up of static electricity during handling which comprises applying to the fibers a reaction product which is obtained by reacting an organic compound having an alkyl group containing from 2 to 22 carbon atoms, at least one group which contains a nitrogen atom and at least one reactive hydrogen atom, with two alkylene oxides, one being having at least two carbon atoms, and the other having at least three carbon atoms in the molecule.

2. A process in accordance with claim 1 in which the treatment is carried out by applying to the fibers an ethylene oxide adduct of a product obtained by treating an organic compound containing at least one nitrogen atom and at least one reactive hydrogen atom with an alkylene oxide containing at least three carbon atoms in the molecule.

3. A process in accordance with claim 1 in which the treatment is carried out by applying to the fibers a reaction product obtained by addition of ethylene oxide to an organic compound having at least one nitrogen atom and at least one reactive hydrogen atom and thereafter treating the product with an alkylene oxide containing at least three carbon atoms in the molecule.

4. A process in accordance with claim 1 in which the treatment is carried out by applying to the fibers an ethylene oxide adduct of a product obtained by reacting an organic compound having at least one nitrogen atom and at least one reactive hydrogen atom with an alkylene oxide mixture containing at least 40% by weight alkylene oxide having at least three carbon atoms in the molecule and at most 60% by weight ethylene oxide.

5. A process in accordance with claim 1 in which the treatment is carried out by applying to the fibers a reaction product obtained by adding ethylene oxide to an organic compound having at least one nitrogen atom and at least one reactive hydrogen atom and thereafter treating the reaction product with an alkylene oxide mixture containing at least 40% by weight alkylene oxide having at least three carbon atoms in the molecule and at least 60% by weight ethylene oxide.

6. A process in accordance with claim 1 in which the treatment is carried out by applying to the fibers an ethylene oxide adduct of a product obtained by reacting a polyelectrolypamylene with an alkylene oxide containing at least three carbon atoms in the molecule where the ethylene oxide adduct has the general formula:

7. A process in accordance with claim 1, in which the treatment is carried out by applying to the fibers an ethylene oxide adduct of a product obtained by reacting ethylenediamine with propylene oxide where the ethylene oxide adduct has the general formula:

8. A process in accordance with claim 1, in which the treatment is carried out by applying to the fibers an ethylene oxide adduct of a product obtained by reacting propylenediamine with propylene oxide.

9. A process in accordance with claim 1, in which the treatment is carried out by applying to the fibers an ethylene oxide adduct of a product obtained by reacting diethylene triamine with propylene oxide.

10. A process in accordance with claim 1, in which the treatment is carried out by applying to the fibers an ethylene oxide adduct of a product obtained by reacting tetraethylene pentamine with propylene oxide.

11. A process in accordance with claim 1, in which the treatment is carried out in presence of said reaction product of which 10 parts by weight being dissolved in 90 parts by weight water exhibit a cloud point between 40° and 80° C.

12. A process in accordance with claim 1, in which said reaction product is admixed with a sulphated derivative of an ethoxyated fatty alcohol having from 12 to 18 carbon atoms in the alkyl group.

13. A process in accordance with claim 1, in which said reaction product is admixed with a sulphotated derivative of an ethoxyated alkylphoenol having from 6 to 12 carbon atoms in the alkyl group.

14. A process in accordance with claim 1, in which said reaction product is admixed with a phosphoric acid ester of a fatty alcohol having from 8 to 20 carbon atoms in the alkyl group.

15. A process in accordance with claim 1, in which said reaction product is admixed with a phosphoric acid ester of an ethoxyated fatty alcohol having from 8 to 20 carbon atoms in the alkyl group.

16. A process in accordance with claim 1, in which said reaction product is admixed with an ethoxyated fatty alcohol having from 8 to 20 carbon atoms in the alkyl group.

17. A process in accordance with claim 1, in which said reaction product is admixed with an ethoxyated alkylphoenol having from 6 to 12 carbon atoms in the alkyl group.

18. A process in accordance with claim 1, in which said reaction product is admixed with a polypropylene glycol having an average molecular weight of about 300 to about 3,000 which ethylene oxide has been added.

19. A process in accordance with claim 1, in which said reaction product is admixed with polyethylene glycol esters of resin acids.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION
Patent No. 3,723,173 Dated March 27, 1973

Inventor(s) NIKOLAUS AUGUSTIN SCHONFELDT

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

Column 1, in the Abstract, line 15, "provide" should read -- provided -- . Column 2, line 27, "intervale" should read -- intervals -- ; line 40, after "new", insert -- spinning -- . Column 3, line 50,

\[ H_2 \cdot C_\text{H}_n \quad (NH \cdot C_\text{H}_n) \quad NH \]

should be --H_2 \cdot C_\text{H}_n \quad (NH \cdot C_\text{H}_n) \quad NH--.

Column 3, line 68, "[(C_m\text{H}_{2m}O)_b(C_\text{H}_4\text{O})_aH]_r" should be --[(C_m\text{H}_{2m}O)_b(C_\text{H}_4\text{O})_aH]_r--.

Column 4, line 7, "(C_\text{H}_6\text{O})_b(C_\text{H}_4\text{O})_aH" should be --(C_\text{H}_6\text{O})_b(C_\text{H}_4\text{O})_aH--.

Column 4, line 59, "oxide" should read -- oxide -- . Column 5, line 37, "are" should read -- were -- ; line 38, "mills" should read -- mill -- ; line 40, "miner" should read -- mineral -- . Column 6, under the heading Example 5, line 21, cancel "a". Column 7, Claim 5, line 45, "least", second occurrence, should read -- most -- .

Column 7, line 57, "[(C_m\text{H}_{2m}O)_b(C_\text{H}_4\text{O})_aH]_r" should be --[(C_m\text{H}_{2m}O)_b(C_\text{H}_4\text{O})_aH]_r--.

Signed and sealed this 20th day of November 1973.

(SEAL)
Attest:
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