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(54) **HIGH SPEED TEXTILE FIBRE PREPARATIONS**

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(76) Inventors: **Marta Domingo**, Barcelona (ES);
Maria Dolors Lopez Gomez,
Barcelona (ES); **Joaquin Bigorra**
Llosas, Sabadell (ES); **Rafael Pi**
Subirana, Granollers (ES)

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Correspondence Address:

COGNIS CORPORATION
2500 RENAISSANCE BLVD., SUITE 200
GULPH MILLS, PA 19406

(57) **ABSTRACT**

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A composition containing a saponin present in an extract of an *Argania spinosa* plant.

HIGH SPEED TEXTILE FIBRE PREPARATIONS

FIELD OF THE INVENTION

[0001] This invention relates generally to textiles and more particularly to new high-speed spin finishes and to the use of certain mixtures for the production of such preparations.

PRIOR ART

[0002] Both in the production and in the processing of synthetic fibers, full control of the forces acting on the fibers is extremely important. Basically, the influencing factors may be divided into two groups, namely: acceleration forces which are transmitted, for example, from rotating rollers to the fibers and friction forces which are generated by rubbing of the fibers on hard surfaces. In order to counteract friction, i.e. yarn breakage, one of the established remedies is to treat the yarn with a so-called spin finish which, in simple terms, is an aqueous preparation which on the one hand has a lubricating effect and on the other hand reduces static charging both between the fibers themselves and between the yarn and the roller.

[0003] Although many spin finishes are already known from the prior art and although numerous articles and books have been published on the subject of avoiding friction in yarn manufacture, there are various problems which have not yet been satisfactorily solved. One disadvantage of known spin finishes, for example, is that, although they develop an adequate lubricating effect in the medium speed range, they are unsuitable for modern high-speed processes in the 100,000 to 110,000 r.p.m. range. Another particular disadvantage is that conventional finishes can only be used for processing undyed fibers because they tend to dissolve dyes under the usual texturing conditions, so that the dyes "bleed" from the fibers. This also increases the deposits on the rollers.

[0004] Accordingly, the problem addressed by the present invention was to provide new spin finishes which would enable even dyed fibers or yarns to be processed and which, at the same time, would be suitable for high-speed processes.

DESCRIPTION OF THE INVENTION

[0005] The present invention relates to high-speed spin finishes containing

[0006] (a) esterquats,

[0007] (b) fatty acid amidoamines and

[0008] (c) ethoxylated nonionic surfactants.

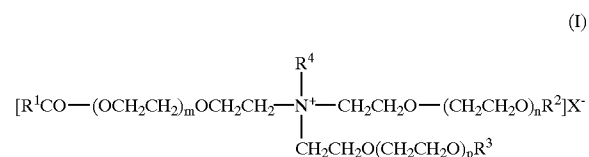
[0009] It has surprisingly been found that not only do the preparations according to the invention significantly reduce static charging, have an excellent lubricating effect and, hence, are even suitable for the high-speed processing of yarns without yarn breakages or roller deposits occurring, the unwanted "bleeding" of dyed synthetic fibers or yarns, particularly those based on polyacrylate or polyester fibers, is also reliably prevented.

[0010] Esterquats

[0011] "Esterquats" are generally understood to be quaternized fatty acid triethanolamine ester salts. They are

known substances which can be obtained by the relevant methods of preparative organic chemistry. Reference is made in this connection to International patent application WO 91/01295 (Henkel), according to which triethanolamine is partly esterified with fatty acids in the presence of hypophosphorous acid, air is passed through the reaction mixture and the whole is then quaternized with dimethyl sulfate or ethylene oxide. In addition, German patent DE 4308794 C1 (Henkel) describes a process for the production of solid esterquats in which the quaternization of triethanolamine esters is carried out in the presence of suitable dispersants, preferably fatty alcohols. Overviews on this theme have been published by R. Puchta et al. in *Tens. Surf. Det.*, 30, 186 (1993), by M. Brock in *Tens. Surf. Det.*, 30, 394 (1993), by R. Lagerman et al. in *J. Am. Oil Chem. Soc.*, 71, 97 (1994) and by I. Shapiro in *Cosm. Toil.* 109, 77 (1994).

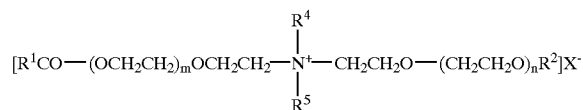
[0012] The quaternized fatty acid triethanolamine ester salts correspond to formula (I):



[0013] in which R¹CO is an acyl group containing 6 to 22 carbon atoms, R² and R³ independently of one another represent hydrogen or have the same meaning as R¹CO, R⁴ is an alkyl group containing 1 to 4 carbon atoms or a (CH₂CH₂)_qH group, m, n and p together stand for 0 or numbers of 1 to 12, q is a number of 1 to 12 and X is halide, alkyl sulfate or alkyl phosphate. Typical examples of esterquats which may be used in accordance with the present invention are products based on caproic acid, caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, isostearic acid, stearic acid, oleic acid, elaidic acid, arachic acid, behenic acid and erucic acid and the technical mixtures thereof obtained, for example, in the pressure hydrolysis of natural fats and oils. Fatty acids containing 16 to 22 carbon atoms, more particularly stearic acid, behenic acid and partly hydrogenated C_{16/18} tallow fatty acid, are preferably used. To produce the quaternized esters, the fatty acids and the triethanolamine may be used in a molar ratio of 1.1:1 to 3:1. With the performance properties of the esterquats in mind, a ratio of 1.2:1 to 2.2:1 and preferably 1.5:1 to 1.9:1 has proved to be particularly advantageous. The preferred esterquats are technical mixtures of mono-, di- and triesters with an average degree of esterification of 1.5 to 1.9 and are derived from technical C_{16/18} tallow or palm oil fatty acid (iodine value 0 to 40). In performance terms, quaternized fatty acid triethanolamine ester salts corresponding to formula (I), in which R¹CO is an acyl group containing 16 to 18 carbon atoms, R² has the same meaning as R¹CO, R³ is hydrogen, R⁴ is a methyl group, m, n and p stand for 0 and X stands for methyl sulfate, have proved to be particularly advantageous. Corresponding products are commercially

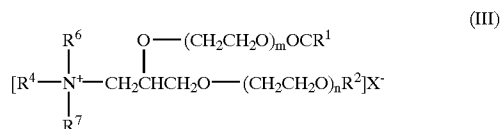
available under the name of Dehyquat® AU (Cognis Deutschland GmbH).

[0014] Besides the quaternized fatty acid triethanolamine ester salts, other suitable esterquats are quaternized ester salts of fatty acids with diethanol-alkylamines corresponding to formula (II):



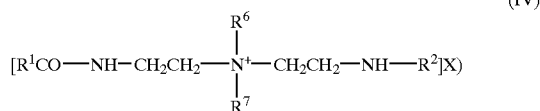
[0015] in which R¹CO is an acyl group containing 6 to 22 carbon atoms, R² is hydrogen or has the same meaning as R¹CO, R⁴ and R⁵ independently of one another are alkyl groups containing 1 to 4 carbon atoms, m and n together stand for 0 or numbers of 1 to 12 and X stands for halide, alkyl sulfate or alkyl phosphate.

[0016] Finally, another group of suitable esterquats are the quaternized ester salts of fatty acids with 1,2-dihydroxypropyl dialkylamines corresponding to formula (III):



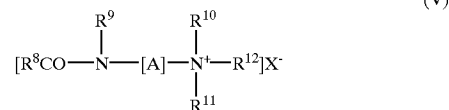
[0017] in which R¹CO is an acyl group containing 6 to 22 carbon atoms, R² is hydrogen or has the same meaning as R¹CO, R⁴, R⁶ and R⁷ independently of one another are alkyl groups containing 1 to 4 carbon atoms, m and n together stand for 0 or numbers of 1 to 12 and X stands for halide, alkyl sulfate or alkyl phosphate.

[0018] In addition, other suitable esterquats are substances in which the ester bond is replaced by an amide bond and which—preferably based on diethylenetriamine—correspond to formula (IV):



[0019] in which R¹CO is an acyl group containing 6 to 22 carbon atoms, R² is hydrogen or has the same meaning as R¹CO, R⁶ and R⁷ independently of one another are alkyl groups containing 1 to 4 carbon atoms and X is halide, alkyl sulfate or alkyl phosphate. Amide esterquats such as these are commercially obtainable, for example, under the name of Incroquat® (Croda).

[0020] Finally, other suitable esterquats are compounds based on ethoxylated castor oil or hydrogenation products thereof which correspond to formula (V):



[0021] in which R⁸CO is a saturated and/or unsaturated ethoxylated hydroxyacyl group containing 16 to 22 and preferably 18 carbon atoms and 1 to 50 oxyethylene units, A is a linear or branched alkylene group containing 1 to 6 carbon atoms, R⁹, R¹⁰ and R¹¹ independently of one another represent hydrogen or a C₁₋₄ alkyl group, R¹² is a C₁₋₄ alkyl group or a benzyl group and X is halogen, alkyl sulfate or alkyl phosphate.

[0022] So far as the choice of the preferred fatty acids and the optimal degree of esterification are concerned, the examples mentioned for (I) also apply to the esterquats corresponding to formulae (II) to (V).

[0023] The esterquats corresponding to formula (I) to (V) may be obtained both from fatty acids and from the corresponding triglycerides. One such process, which is intended to be representative of the relevant prior art, is proposed in European patent EP 0750606 B1 (Cognis). The condensation of the alkanolamines with the fatty acids may also be carried out in the presence of defined quantities of dicarboxylic acids, for example oxalic acid, malonic acid, succinic acid, maleic acid, fumaric acid, glutaric acid, adipic acid, sorbic acid, pimelic acid, azelaic acid, sebacic acid and/or dodecanedioic acid. In this way, the esterquats are obtained with a partly oligomeric structure which can have an advantageous effect on the clear solubility of the products, particularly where adipic acid is used. Corresponding products are commercially available under the name of Dehyquat® D 6003 (Cognis Deutschland GmbH) and are described, for example, in European patent EP 0770594 B1 (Cognis). The esterquats are normally marketed in the form of 50 to 90% by weight alcoholic solutions which may readily be diluted with water as required.

[0024] Fatty Acid Amidoamines

[0025] The fatty acid amidoamines suitable as component (B) are known pseudocationic compounds which represent condensation products of C₁₂₋₂₂ fatty acids with ethylenediamine, diethylenetriamine, triethylene-tetramine, propylenediamine, dipropylene triamine and/or tripropylene tetramine. Typical examples are the reaction products of the di- and oligoamines mentioned with lauric acid, isotridecanoic acid, myristic acid, palmitic acid, palmitoleic acid, stearic acid, isostearic acid, oleic acid, elaidic acid, petroselic acid, linoleic acid, linolenic acid, elaeostearic acid, arachic acid, gadoleic acid, behenic acid and erucic acid and technical mixtures thereof. From the performance perspective, it is particularly preferred to use the condensation product of behenic acid with diethylenetriamine (DETA).

[0026] Ethoxylated Nonionic Surfactants

[0027] Suitable ethoxylated nonionic surfactants which form component (c) are products of the addition of on

average 20 to 150, preferably 30 to 100 and more particularly 40 to 75 mol ethylene oxide onto primary C₈₋₂₂ alcohols or C₈₋₂₂ triglycerides. The ethoxylates may be prepared in known manner, i.e. by base-catalyzed addition of ethylene oxide onto the hydroxyl group of the alcohols or by hydrotalcite-catalyzed insertion of ethylene oxide into the carbonyl ester group, and may have both a conventional broad homolog distribution and a narrow homolog distribution.

[0028] Typical examples are the adducts of on average 20 to 150, preferably 30 to 100 and more particularly 40 to 75 mol ethylene oxide with fatty alcohols or oxoalcohols containing 8 to 22 and preferably 12 to 18 carbon atoms, such as for example caproic alcohol, caprylic alcohol, 2-ethylhexyl alcohol, capric alcohol, lauryl alcohol, isotridecyl alcohol, myristyl alcohol, cetyl alcohol, palmitoleyl alcohol, stearyl alcohol, isostearyl alcohol, oleyl alcohol, elaidyl alcohol, petroselinyl alcohol, linolyl alcohol, linolenyl alcohol, elaeostearyl alcohol, arachyl alcohol, gadoleyl alcohol, behenyl alcohol, erucyl alcohol and brassidyl alcohol and technical mixtures thereof. Products of the addition of on average 40 to 75 mol ethylene oxide onto stearyl alcohol, tallow fatty alcohol and behenyl alcohol are particularly preferred.

[0029] Other typical examples are the adducts of on average 20 to 150, preferably 30 to 100 and more particularly 40 to 75 mol ethylene oxide with synthetic or natural triglycerides derived from C₈₋₂₂ and more particularly C₁₂₋₁₈ fatty acids. Typical examples of suitable vegetable oils and animal fats are palm oil, palm kernel oil, coconut oil, olive oil and bovine tallow. However, it is particularly preferred to use adducts of on average 20 to 300, preferably 50 to 250 and more particularly 100 to 200 mol with castor oil and hydrogenated castor oil; here, the addition largely takes place onto the secondary hydroxyl group in the fatty chain.

[0030] Spin Finishes

[0031] Preparations containing

[0032] (a) 40 to 65, preferably 45 to 55% by weight esterquats,

[0033] (b) 10 to 20, preferably 12 to 18% by weight fatty acid amidoamines and

[0034] (c) 15 to 50, preferably 30 to 40% by weight ethoxylated nonionic surfactants,

[0035] with the proviso that the quantities shown add up to 100% by weight, optionally together with other auxiliaries and additives,

[0036] have proved to be successful for the production of high-speed spin finishes distinguished by particularly small deposits on the rollers. Suitable auxiliaries are, for example, antistatic agents, yarn compacting agents, pH adjusters, bactericides and/or corrosion inhibitors. In general, the total quantity of additives, based on the finishes, is up to 15% by weight and preferably from 1 to 10% by weight.

[0037] According to the invention, the finishes are preferably used in the form of aqueous emulsions containing 1 to 30% by weight and preferably 15 to 25% by weight of the finish. These emulsions are applied to the fibers or yarns in the usual way, i.e. for example by lick rolls or dosing pumps and applicator pins. The emulsions may also be applied by passing the fibers or yarns through immersion baths. The finishes may be used at various stages of yarn production or yarn finishing, i.e. both immediately after extrusion, during texturing or during winding. In this connection, the term "spin finish" may be regarded as synonymous with "texturing aid" or "winding oil".

[0038] Commercial Applications

[0039] Finally, the present invention relates to the use of mixtures containing

[0040] (a) esterquats,

[0041] (b) fatty acid amidoamines and

[0042] (c) ethoxylated nonionic surfactants

[0043] for the production of high-speed spin finishes.

EXAMPLES

[0044] Red-dyed polyacrylic fibers were passed at 68° C. through immersion baths containing 1% by weight aqueous solutions of various spin finishes. The fibers were then subjected for 6 h to high-speed texturing at a roller speed of 100,000 r.p.m. Yarn breakage, roller deposits and fiber bleeding were then evaluated (Yarn breakage: +=less than 1, o=1 to 5, -=more than 5. Roller deposits: +=no deposit, o=slight deposit, -=large deposit. Bleeding: +=no bleeding, o=slight bleeding, -=serious bleeding). The results are set out in Table 1. Examples 1 and 2 correspond to the invention, Examples C1 and C2 are intended for comparison.

TABLE 1

Composition and performance of the spin finishes				
	1	2	C1	C2
<u>Composition/performance</u>				
Cetyl phosphate potassium salt	—	—	80	—
Esterquat I ¹⁾	—	—	—	80
Esterquat II ²⁾	50	70	—	—
Behenic acid/diethylenetriamine condensate	15	10	20	—
Castor oil + 200 EO	35	20	—	—
<u>Performance test results</u>				
Yarn breakage	+	+	o	o
Deposit formation	+	o	o	—
Bleeding	+	+	—	—

¹⁾Dehyquart ® AU 57; basis C_{12/18} palm oil fatty acid;

²⁾Dehyquart ® AU 18; basis stearic acid

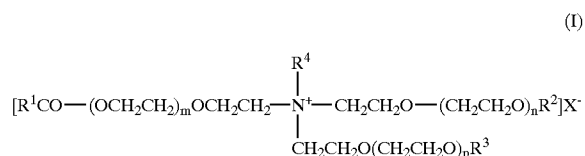
1. Spin finishes containing

(a) esterquats,

(b) fatty acid amidoamines and

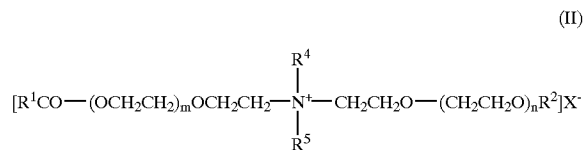
(c) ethoxylated nonionic surfactants.

2. Spin finishes as claimed in claim 1, characterized in that they contain as component (a) esterquats corresponding to formula (I):



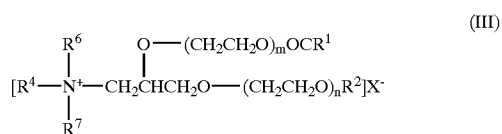
in which R¹CO is an acyl group containing 6 to 22 carbon atoms, R² and R³ independently of one another represent hydrogen or have the same meaning as R¹CO, R⁴ is an alkyl group containing 1 to 4 carbon atoms or a (CH₂CH₂O)_qh group, m, n and p together stand for 0 or numbers of 1 to 12, q is a number of 1 to 12 and x is halide, alkyl sulfate or alkyl phosphate.

3. Spin finishes as claimed in claim 1, characterized in that they contain as component (a) esterquats corresponding to formula (II):



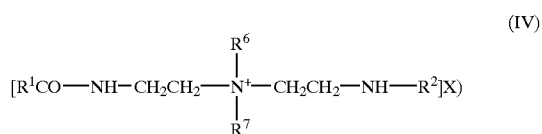
in which R¹CO is an acyl group containing 6 to 22 carbon atoms, R² is hydrogen or has the same meaning as R¹CO, R⁴ and R⁵ independently of one another are alkyl groups containing 1 to 4 carbon atoms, m and n together stand for 0 or numbers of 1 to 12 and X stands for halide, alkyl sulfate or alkyl phosphate.

4. Spin finishes as claimed in claim 1, characterized in that they contain as component (a) esterquats corresponding to formula (III):



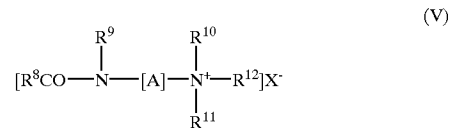
in which R¹CO is an acyl group containing 6 to 22 carbon atoms, R² is hydrogen or has the same meaning as R¹CO, R⁴, R⁶ and R⁷ independently of one another are alkyl groups containing 1 to 4 carbon atoms, m and n together stand for 0 or numbers of 1 to 12 and X stands for halide, alkyl sulfate or alkyl phosphate.

5. Spin finishes as claimed in claim 1, characterized in that they contain as component (a) esterquats corresponding to formula (IV):



in which R¹CO is an acyl group containing 6 to 22 carbon atoms, R² is hydrogen or has the same meaning as R¹CO, R⁶ and R⁷ independently of one another are alkyl groups containing 1 to 4 carbon atoms and X is halide, alkyl sulfate or alkyl phosphate.

6. Spin finishes as claimed in claim 1, characterized in that they contain as component (a) esterquats corresponding to formula (V):



in which R⁸CO is a saturated and/or unsaturated ethoxylated hydroxyacyl group containing 16 to 22 and preferably 18 carbon atoms and 1 to 50 oxyethylene units, A is a linear or branched alkylene group containing 1 to 6 carbon atoms, R⁹, R¹⁰ and R¹¹ independently of one another represent hydrogen or a C₁₋₄ alkyl group, R¹² is a C₁₋₄ alkyl group or a benzyl group and X is halogen, alkyl sulfate or alkyl phosphate.

7. Spin finishes as claimed in at least one of claims 1 to 6, characterized in that the esterquats are derived from C₁₆₋₂₂ fatty acids.

8. Spin finishes as claimed in at least one of claims 1 to 7, characterized in that they contain as component (b) condensation products of C₁₂₋₂₂ fatty acids with ethylenediamine, diethylenetriamine, triethylenetetramine, propylenediamine, dipropylenetriamine and/or tripropylene tetramine.

9. Spin finishes as claimed in at least one of claims 1 to 8, characterized in that they contain behenic acid/diethylenetriamine condensates as component (b).

10. Spin finishes as claimed in at least one of claims 1 to 9, characterized in that they contain products of the addition of on average 20 to 300 mol ethylene oxide onto primary C₈₋₂₂ alcohols or triglycerides as component (c).

11. Spin finishes as claimed in at least one of claims 1 to 10, characterized in that they contain products of the addition of on average 50 to 200 mol ethylene oxide onto castor oil as component (c).

12. Spin finishes as claimed in at least one of claims 1 to 11, characterized in that they contain

- (a) 40 to 65% by weight esterquats,
- (b) 10 to 20% by weight fatty acid amidoamines and
- (c) 15 to 50% by weight ethoxylated nonionic surfactants,

with the proviso that the quantities shown add up to 100% by weight, optionally together with other auxiliaries and additives.

13. The use of mixtures containing

- (a) esterquats,
- (b) fatty acid amidoamines and
- (c) ethoxylated nonionic surfactants

for the production of high-speed spin finishes.