EUROPEAN PATENT APPLICATION

Method of conditioning fabrics.

A method of conditioning fabrics in running water includes adding to the water a fabric conditioning article having a dimension greater than 1 cm, the article comprising a conditioning agent such as a fabric softener or an anti-static agent together with physical or chemical means for restraining the release of the conditioning agent. The article may be in the form of a sachet or block coated with an ethoxylated alcohol for restraining release. An electrolyte may be present to improve dispersibility of the conditioning agent and an antifoam material may also be present. The method enables fabrics to be conditioned in a running rinse while minimising loss of the conditioner.
METHOD OF CONDITIONING FABRICS

This invention relates to a method of conditioning fabrics, in particular to a method of conditioning fabrics in a washing machine following the washing step of a fabric laundering process.

The washing of fabrics with conventional detergent compositions can lead to harshening of the fabrics and this is traditionally overcome by the treatment of the fabrics with a fabric softening agent. Since many fabric softening agents are incompatible with detergent compositions, this treatment is usually carried out in a post-washing step, in which the fabric softening agent is added to the rinse water used for rinsing the fabrics. Other fabric conditioning agents are often included, such as anti-static agents and perfumes.

When a washing machine of the type which is common in Europe is used, i.e., a machine in which the fabrics are rinsed in a tub which is filled with rinse water, agitated to achieve efficient rinsing and then drained, it is a simple matter to add the fabric conditioning agent to the rinse water in the tub, either by hand or by way of an automatic dispensing device. However, some washing machines are designed in such a manner that the fabrics are rinsed in running water. The use of conventional fabric conditioning compositions added at the start of the rinse in such machines may not be satisfactory and the addition of a conventional fabric conditioning composition towards the end of the rinse is inconvenient. Thus, for example, when the conditioning composition is in the form of a liquid or a powder, a large proportion of the composition can be lost from the machine with the running rinse water without having the chance to come into contact with the fabrics and condition them, and some desorption of conditioning agent from the fabric can occur as the rinse continues.

We have now discovered a novel method by which fabrics can be conditioned in such machines, while minimising the loss of the composition from the machine. The method relies on a specific physical form of the conditioning product which avoids the need to add the product near the end of the running rinse.

Thus, according to the invention, there is provided a method of conditioning fabrics which includes the steps of

(i) placing fabrics in a rinsing tub; and
(ii) continuously running rinse water into and out of the tub to contact the fabrics in the presence of a fabric conditioning article, having at least one dimension greater than 1.0cm and comprising a conditioning agent and means for restraining the release of the conditioning agent in water.

This method enables the conditioning composition to be retained in the tub even when the rinse water is running continuously and to effectively condition the fabrics during the rinsing process, thereby minimising the loss of the conditioning agent from the tub.

The fabrics will normally have been previously washed in the same tub of the machine, or they may be placed in the tub specifically for this purpose. When previously washed, they will have at least some of the wash liquor containing a detergent active still adsorbed on or otherwise associated with them, the running water then serving to flush this wash liquor out of the fabrics. The rinse water running into the tub will be substantially free of any detergent actives. Any fabric types may be used, but fabrics comprising natural fibres such as cotton are particularly susceptible to treatment in this way when the conditioning agent is a fabric softening agent, while fabrics comprising synthetic fibres such as nylon are susceptible to treatment in this way when the conditioning agent is an anti-static agent.

The running water flow rate is determined by the design of the machine, but we have found that this method is successful when the flow rate is equivalent to about 5 to about 50 tub-full changes of water per hour, which, when the tub capacity is about 35 litres, is equivalent to from 3.5 to 30 litres per minute. Any water temperature may be used. Cold water having a temperature of 0° to 30°C has been found to be suitable. The rinsing time used will be determined in part by the design of the machine, i.e. its rinsing efficiency, and the degree of rinsing which is desirable, but we have found success with rinsing times of up to 20 minutes, such as from 5 to 15 minutes.

The fabric conditioning article may take many forms, provided that it has at least one dimension greater than 1cm. If the article is smaller than this, we have found that it can be lost from the machine with the running water. The article may consist of or alternatively contain the conditioning composition. Clearly, when the article consists of the composition it is not possible for the composition to be in the form of a liquid or fine powder since it would not have the required dimension of greater than 1cm. However, it is possible for the composition to be in the form of a block or tablet having the required dimension, such as a disk or sphere shaped block having a diameter of from 2 to 10 cm.

When the article contains the composition then it is in the form of a container having at least one dimension greater than 1cm. The container is constructed in such a manner as to retain the conditioning composition, which in this case may be in any suitable physical form, including liquids and powders. A suitable such container is a sachet formed of water permeable material, such as a square sachet having a side length of at least 3 cm.

The term "fabric conditioning agent" is used here in the broadest sense to encompass any material conferring a benefit, for example softness, reduced static charge, perfume, drape, crease resistance, ease of ironing or improved bleaching, but fabric softening or antistatic agents are preferred. The invention is particularly beneficial when the conditioning agent is a cationic material.

Suitable cationic softening agents include water-insoluble cationic fabric softeners.
The water-insoluble cationic fabric softener can be any fabric-substantive cationic compound which has a solubility in water at pH 2.5 and 20° C of less than 10 g/l. Highly preferred materials are quaternary ammonium salts having two C_{12}-C_{24} alkyl of alkenyl chains, optionally substituted or interrupted by functional groups such as -OH, -O-, -CONH, -COO-, etc.

Well known species of substantially water-insoluble quaternary ammonium compounds have the formula

$$[R_1 R_3 X][R_2 R_4]$$

wherein $R_1$ and $R_2$ represent hydrocarbyl groups from about 12 to about 24 carbon atoms; $R_3$ and $R_4$ represent hydrocarbyl groups containing from 1 to about 4 carbon atoms; and X is an anion, preferably selected from halide, methyl sulfate and ethyl sulfate radicals. Representative examples of these quaternary softeners include ditallow dimethyl ammonium chloride; ditallow dimethyl ammonium methyl sulfate; dihexadecyl dimethyl ammonium chloride; di (hydrogenated tallow alkyl) dimethyl ammonium chloride; dioctadecyl dimethyl ammonium chloride; didocosyl dimethyl ammonium chloride; di (hydrogenated tallow) dimethyl ammonium methosulfate; dihexadecyl diethyl ammonium chloride; di (coconut alkyl) dimethyl ammonium chloride. Ditallow dimethyl ammonium chloride, di (hydrogenated tallow alkyl) dimethyl ammonium chloride, di (coconut alkyl) dimethyl ammonium chloride and di (coconut alkyl) dimethyl ammonium methosulfate are preferred.

Another class of preferred water-insoluble cationic materials are the alkylimidazolinium salts believed to have the formula:

$$[R_6 R_7 N][C_2H_4 R_8 R_9 R_{10}]$$

wherein $R_6$ is an alkyl or hydroxyalkyl group containing from 1 to 4, preferably 1 or 2 carbon atoms, $R_7$ is an alkyl or alkenyl group containing from 8 to 25 carbon atoms, and $R_8$ is hydrogen or an alkyl containing from 1 to 4 carbon atoms and $A^-$ is an anion, preferably a halide, methosulfate or ethosulfate. Preferred imidazolinium salts include 1-methyl-1-(tallowylamido)-2-tallowyl-4,5-dihydro imidazolinium methosulfate and 1-methyl-1-(palmitoylamido)ethyl-2-octadecyl-4,5-dihydro-imidazolinium chloride. Other useful imidazolinium materials are 2-heptadecyl-1-methyl-1-(2-stearylamido)ethyl-imidazolinium chloride and 2-lauryl-1-hydroxyethyl-1-oleyl-imidazolinium chloride. Also suitable herein are the imidazolinium fabric softening components of US Patent No 4 127 489, incorporated by reference.

The antistatic agents useful herein are quaternary ammonium salts of the formula $[R_9 R_{10} R_{11} R_{12} N] Y^-$ wherein at least one, but not more than two, of $R_9, R_{10}, R_{11}$ and $R_{12}$ is an organic radical containing a group selected from a C_{16}-C_{22} aliphatic radical, or an alkyl phenyl or alkyl benzyl radical having 10-16 atoms in the alkyl chain, the remaining group or groups being selected from hydrocarbyl groups containing from 1 to about 4 carbon atoms, or C_{2}-C_{4} hydroxy alkyl groups and cyclic structures in which the nitrogen atom forms part of the ring, and Y is an anion such as halide, methylsulfate, or ethylsulfate.

In the context of the above definition, the hydrophobic moiety (i.e., the C_{16}-C_{22} aliphatic, C_{10}-C_{16} alkyl phenyl or alkyl benzyl radical) in the organic radical $R_9$ may be directly attached to the quaternary nitrogen atom or may be indirectly attached thereto through an amide, ester, alkoxy, ether, or like grouping.
The fabric conditioning article also comprises means for restraining the release of the conditioning agent in water without causing the conditioning agent to gel. These may be physical or chemical means. The means for restraining the release of the conditioning agent should preferably be such as to ensure that substantially all the conditioning agent is released into the rinse liquor during the duration of the rinse and that at least 30%, most preferably 50% of the conditioning agent should be available for release during the second half of the rinse.

One suitable method of achieving sufficient restraint is to mix, coat or otherwise associate the conditioning agent with a material selected from paraffin waxes, cyclic and acyclic mono and polyhydric alcohols, substituted and unsubstituted aliphatic carboxylic acids, esters of the foregoing alcohols and acids, C1-C4 alkenyI oxide condensates of any of the foregoing materials and mixtures thereof. Tallow alcohol is particularly preferred. Other suitable materials include nonionic materials with melting temperatures similar to that of tallow alcohol ethoxylated with between 18 and 25 ethoxylate groups per molecule. Further details of suitable such materials may be found in US 3936537 (Baskerville), where they are referred to as dispersion inhibitors.

The proportions of fabric conditioning agent to release inhibitor in the composition depends upon the design of the product and the use factors such as the intended time and temperature of the rinse. The choice of release inhibitor is also dependent on the temperature of the running rinse water.

It may also be advantageous to include in the fabric conditioning composition, especially when the composition is in solid form, an electrolyte to aid the dispersion of the conditioning agent after the release inhibitor has ceased to have an effect. Preferably, this electrolyte is a water-soluble inorganic salt such as sodium chloride. The level of electrolyte in the composition may be such that the ratio of fabric conditioning agent to electrolyte is within the range of 1:0.5 to 1:10 by weight.

It may also be of advantage if the fabric conditioning composition contains an antifoam material, to suppress the foam which would otherwise occur in the rinse when the fabric conditioning agent is a surfactant. Any known antifoam material may be used, at a level sufficient for it to have the desired effect. A suitable antifoam granule may be of the type described in our European Patent Specification EP 94250-A.

The fabric conditioning article can be added to the fabrics in the tub before the rinse water flows into the tub, simultaneously with the rinse water or part-way through the rinse cycle.

**EXAMPLES 1 TO 3**

A sachet was prepared from melt blown polypropylene laminate weighing 61 grams per square metre (Kimtex SMS ex Kimberley-Clark). The sachet size was 7cm x 7cm. Inside the sachet was placed 2 grams of Arosurf TA100 which is a commercially available fabric softening agent which is approximately 100% distearyl dimethyl ammonium chloride in powder form, 4 grams of salt (sodium chloride) and 1 gram of pre-prepared antifoam granules. The sachet was then coated with 1 gram of either tallow alcohol 18 EO (Example 1) or tallow alcohol 25 EO (Example 2) as a release restraining means. For comparison purposes a sachet with no coating (Example 3) was tested.

**EXAMPLES 4 TO 7**

Blocks were manufactured by pressing fabric conditioning formulations in powder form in a 30mm dye using a hydraulic ram at a pressure level of either 1 or 1/2 tonne pressure over the surface of the block. Each block was approximately 1cm thick and had a diameter of approximately 3cm. Each formulation contained 2 grams Arosurf TA 100 and either 6 or 11 grams of salt (Examples 4 and 5 respectively) plus 1 gram pre-prepared antifoam granules. The blocks were then coated with 0.3 gram tallow alcohol 25 EO as a release restraining means. For comparison purposes Examples 4 and 5 were repeated except that the blocks were not coated (Examples 6 and 7 respectively).

The products described in Examples 1 to 7 were tested as follows. A NATIONAL NA-W 1018 twin-tub machine was used, having a capacity of 40 litres. 1.3 kg of a mixed cotton/polyester sheeting load was washed for 10 minutes at 25°C in water having a hardness of 6°FH (6 x 10^-4 molar free calcium ions per litre) using a commercially available washing powder "TOP" from the Japanese market at a dosage of 1 gram per litre in 35 litres wash liquor. After washing, the wash liquor was drained from the washing tub and the fabrics were removed therefrom and placed in the spinner and spun until no more wash liquor was being removed. The fabrics were then returned to the washing tub and rinsed for 10 minutes using a running rinse, at a temperature of 25°C, with a water input of 10 litres per minute in the presence of the fabric conditioning article to be tested. After draining the rinse water, the fabrics were removed, spun dry and then line dried at ambient temperature. The fabrics were then assessed for softness by hand and graded on a softness scale in which the value of '8' represents the harshness of desized cotton terry towelling and the value of '5' represents the softness obtained when desized terry towelling cotton is rinsed at a liquor to cloth ratio of 25:1 for 5 minutes in a tergotometer laboratory scale apparatus with an aqueous product consisting of 5% Arquad 2C (a commercially available form of dicoco dimethyl ammonium chloride) dosed at a level of 40 grams per litre, the fabrics being thereafter tumble dried.

The results obtained were as follows, lower ranking numbers representing better softness.
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<thead>
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<th>Example No.</th>
<th>Softness ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
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<td>3</td>
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<tr>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>7</td>
<td>6.5</td>
</tr>
<tr>
<td>No product</td>
<td>8.5</td>
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</table>

Arosurf/salt powder* 7

These results demonstrate the benefit of the invention not only over the use of no product at all but also over the use of a fabric conditioning product which did not include the release restraining means which is essential to the present invention. In particular the results demonstrate the benefit of coating the fabric conditioning agent with a release restraining means. Similar results are obtained if Arosurf TA 100 is replaced by

a) Varisoft 818
(ex Shearex) - distearyl dimethyl ammonium chloride modified with a small amount of dispersion aid;

b) Adogen 442-100P
(ex Rewo) - a powdered cationic based on dimethyl dihardened tallow ammonium chloride

c) Querton 442-SD
(ex Kenobel) - a powdered cationic based on dimethyl dihardened tallow ammonium chloride.

EXAMPLES 8-11

Sachets were prepared from Net X560 (ex Smith & Nephew). The sachet size was 6cm x 6cm. The sachets, after being filled with 5 grams of a non-aqueous fabric conditioning formulation, containing 52% Arquad 2T (a commercially available form of ditallow dimethyl ammonium chloride supplied as a 75% active paste) and 48% isopropyl myristate, were heat sealed. The sachet was coated with a polyvinyl acetate emulsion, as a release restraining means, to a level of 120 gram per square meter. (Example 8)

A sheet was prepared from PBS 6/85 (ex Cambrelle), a non-woven substrate. The sheet was impregnated with the above-mentioned non-aqueous formulation. (Example 9)

Both the sachet and sheet products were tested by the following method.

A NATIONAL NA-W 1018 twin-tub machine was used, having a capacity of 40 litres. 1.3 kg of a mixed cotton/polyester sheeting load was washed for 10 minutes at 25°C in water having a hardness of 6°FH (6 x 10^-4 molar free calcium ions per litre) using a commercially available washing powder "TOP" from the Japanese market at a dosage of 1 gram per litre in 35 litres wash liquor. After washing, the wash liquor was drained from the washing tub and the fabrics were removed therefrom and placed in the spinner and spun until no more wash liquor was being removed. The fabrics were then returned to the washing tub and rinsed for 10 minutes using a running rinse with a water input of 10 litres per minute in the presence of the fabric conditioning article to be tested. The temperature of the running rinse was 10°C.

With the sachet product, the fabric conditioner formulation was released into the running rinse over a period of 3-4 minutes, after an initial 3-4 minute delay. With the sheet product the fabric conditioner formulation was released into the running rinse over a period of 3-4 minutes. With this product form there was no initial delay before the fabric conditioner was released.

In a comparative experiment (Example 10) the sheet product was added five minutes into the running rinse. In a further comparative experiment (Example 11), the non-aqueous liquid formulation was dosed at a level of 5 grams per litre at the start of the running rinse.
After the fabrics had been subjected to the 10 minute running rinse, and the rinse water drained, the fabrics were removed, spun dry and then line dried at ambient temperatures. The fabrics were then assessed for softness by the method described above. The following results were obtained.

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<td>5.5</td>
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<td>10</td>
<td>7</td>
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<td>11</td>
<td>5.5</td>
</tr>
<tr>
<td>No product</td>
<td>8.5</td>
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</table>

Claims

1. A method of conditioning fabrics which includes the steps of
   (i) placing fabrics in a rinsing tub; and
   (ii) continuously running rinse water into and out of the tub to contact the fabrics in the presence
       of a fabric conditioning article, having at least one dimension greater than 1.0cm and comprising a
       conditioning agent and means for restraining the release of the conditioning agent in water.

2. A method of conditioning fabrics according to Claim 1, wherein the means for restraining the release
   of the conditioning agent in water is such that 30% of the conditioning agent is available for release during
   the second half of the rinse.

3. A method of conditioning fabrics according to any preceding claim, wherein the restraining means is
   selected from:
      (i) paraffin waxes;
      (ii) cyclic and acyclic mono and polyhydric alcohols and esters and C1-C4 alkylene oxide
           condensates thereof;
      (iii) substituted and unsubstituted aliphatic carboxylic acids and esters and C1-C4 alkylene oxide
           condensates thereof and;
      (iv) mixtures of the foregoing materials.

4. A method of conditioning fabrics according to any preceding claim, wherein the restraining means is
   mixed with, or coats the conditioning agent in the fabric conditioning article.

5. A method of conditioning fabrics according to any preceding claim, wherein the fabric conditioning
   article is selected from
      (i) a block;
      (ii) a tablet; and
      (iii) a container containing the fabric conditioning agent.

6. A method of conditioning fabrics according to any preceding claim wherein the fabric conditioning
   article contains an electrolyte when the conditioning agent is in solid form.
# EUROPEAN SEARCH REPORT

## DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
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<tr>
<th>Category</th>
<th>Citation of document with indication, where appropriate, of relevant passages</th>
<th>Relevant to claim</th>
<th>CLASSIFICATION OF THE APPLICATION (Int. Cl.)*</th>
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The present search report has been drawn up for all claims.

**TECHNICAL FIELDS SEARCHED (Int. Cl.)***

- D 06 F
- D 06 M

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**CATEGORY OF CITED DOCUMENTS**

- **X**: particularly relevant if taken alone
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