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- (54) Wool scouring.
- Wool containing wool grease is scoured by a method including a hot suint bowl treatment, which method comprises the steps of (a) treating the wool with an aqueous medium containing a building agent capable of reducing the level of free calcium and/or magnesium therein and having a temperature of at least 45°C and thereafter (b) treating the wool with an aqueous medium having a pH of at least 8, containing a detergent and having a temperature of at least 45°C.

This invention relates to the scouring of raw wool, ie wool which contains large quantities of wool grease, which wool is usually subjected to the scouring process before a carding operation.

Raw wool contaminants consist mainly of grease, suint and particulate soil. Suint is mostly a mixture of inorganic salts from evaporation of sheep sweat. The grease component is a very complex mixture containing fats, fatty acids and triglycerides. The particulate soil is, for example, dust, dirt and skin debris. The contaminants tend to divide themselves into "easy-to-remove" and "hard-to-remove" contaminants; see B.O. Bateup, Proceedings of the CSIRO Division of Textile Industry, Symposium on Wool Scouring and and Worsted Carding: New Approaches (1986), page 8.

Typically, the aim of wool scouring is to reduce the "extractables" (contaminants that can be extracted into dichloromethane, DCM) to a level between 0.3-0.5% and at the same time provide a wool having good whiteness, ie a low degree of yellowness.

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Very low levels of extractables are not required as a small amount of residual wool grease helps in the subsequent processing of the wool through the mill; very clean wool is brittle and will tend to break if processed at high speeds, thus reducing total output rate.

Raw wool is generally scoured by washing it in a hot solution of detergent to remove the contaminants followed by rinsing in slightly cooler water. The clean wool is then dried by passing it through a hot air dryer before being passed onto the next processing stage.

This scouring process is preferably carried out continuously by passing the wool through a series of "bowls". Preferably, wool passes from bowl to bowl in one direction, and liquor is pumped in the opposite direction. The raw wool enters the first bowl, and becomes cleaner as it moves towards the last. Clean water enters the last bowl and becomes dirtier as it moves towards the first. The quantities and nature of the chemicals added to the various bowls have been the subject of much work over the last few years. Raw wool has a high level of contaminants (up to 30% by weight) and this raises significant effluent disposable problems. Much of the recent work, especially in Australia, has concentrated on producing more easily treated effluent rather than on improving the efficiency of the scouring process itself. A review of this work has been published by J.R. Christoe and B.O. Bateup, Wool Science Review (1987), 64, 25.

A conventional scouring system consists of two or three scouring bowls, followed by two or three rinse bowls, depending upon the numbers of bowls, typically 46, in the range. The rate of water addition depends on the volume and type of wool being processed.

Introduction of clean water dilutes the active material in the bowls, so addition of fresh detergent and any other chemicals present (see below) are made discontinuously by hand (for example, every half hour), or continuously by means of an automatic dosing pump. The amount of detergent added to the various scouring bowls varies from high levels in bowl 1 to very low levels in bowl 3. This is because, as the wool becomes cleaner, less detergent is required to solubilise the contaminants. Wool grease melts at around 40-45°C, so in the conventional scouring system, a typical temperature for bowl 1 is in the region 65-70°C, so that the hot first bowl softens the grease and allows penetration of the grease droplets by the detergent. The temperature can then be gradually decreased on progression from bowl 3 (about 60°C) dropping to about 55°C for bowls 4 and 5.

In the simplest system, the only added chemical is the detergent. However, several workers have proposed the addition of so-called "builders" to the above system. Builders are compounds, usually inorganic salts, added to increase the effectiveness of the detergent (see the article by Bateup, supra). Thus, scour liquor often contains free calcium and/or magnesium originally present in any of the water used for scouring, the suint salts and possibly lime deposited on the wool itself. In particular, the wool may be so called "slipe" wool, or may be a blend containing such wool, this being wool removed from the hide of a dead sheep using lime. Often, a wool scourer will not know whether a particular batch to be scoured contains such slipe wool. The builders assist in removal of such unwanted calcium and/or magnesium from the scour liquor, and from the surface of the wool. Sodium sulphate is often used as a builder, but its value remains questionable where, as is usual, the detergents used are exclusively non-ionic.

Another builder which is often used is sodium carbonate, often in the form of soda ash. This does effectively remove calcium. However, its calcium binding capacity and its rate of binding calcium is limited. Sodium silicate acts as a much better builder, but is highly alkaline and may cause wool damage if present at too high a concentration. Similarly, sodium hydroxide may be used as a builder, but with the same disadvantage; see B.O. Bateup, Textile Research Journal (January 1985), 55, 50. The best calcium binders are sodium tripolyphosphate (STP) and zeolites, but these are expensive in comparison with the builders mentioned above.

In addition to the detergents and builders, it is also possible to include, for example, metal ion sequestrants (which can also be called "builders"), for example citrates, ethylene diamine tetraacetic acid (EDTA) and nitrilotriacetic acid (NTA) in the final rinse bowl to increase the whiteness of the scoured wool; see B.O. Bateup, Text. Res. J. (1985), 55, 50.

A conventional wool scouring system may utilise five bowls in the order: detergent, detergent, mild deter-

gent (or rinse), rinse, rinse. However, there are several modifications to this system, many of which are described in the abovementioned review by Christoe and Bateup, Wool Science Review (1987) <u>64</u>, 25. One such modification is known as multi-stage scouring in which, for a five bowl system, the order of the bowls is: detergent, rinse, detergent, rinse, rinse. The role of the first bowl is to remove the particulate salts and the more easily removed grease and suint salts, while the second bowl, also at 65°C, continues to soften the wool grease before the wool is reintroduced to a detergent solution to effect removal of this grease. It is generally accepted that two rinse bowls are required at the end of the cycle, so the system is in practice applicable only to five or six bowl ranges.

Another modification is the use of a so-called "suint bowl". In one system, this consists merely of cold, or slightly warm water at a temperature well below the melting point of the wool grease; see for example, US-A-2552944. Here, the cold water which contains no additives, merely removes suint, which itself has, in its own right, a detergent effect and allows this to contribute to removal of wool grease in the subsequent detergent stages. In order to improve the efficiency of dirt removal (by improving dirt settling efficiency), it is known to include in the cold suint bowl a builder, for example soda ash or sodium metasilicate, or a low concentration of a non-ionic, anionic or cold water laundry detergent; see C.A. Anderson et al, Proc. 7th Int. Wool Text Conf., Tokyo (1985) 5, 255, and B.O. Bateup and J.J. Warner, Text. Res. J. (1986), 56, 489.

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In another modification, a hot suint bowl is used, which contains no chemicals. The bowl order is thus: suint bowl, detergent, detergent, rinse, rinse. Here the hot suint bowl is used to remove particulate soils and soluble suint salts. It also allows the wool grease to be softened by the hot water before coming into contact with the detergent in the main scouring bowl. As with the abovementioned multistage scouring, the requirement for two rinse bowls usually means that this system requires five or six bowls in total.

Some scourers, especially those with six bowl ranges, have used a combination of the above to give a bowl configuration of (cold) suint bowl, detergent, rinse, detergent, rinse, rinse. It is considered that the late introduction of detergent into the system allows for greater softening of the wool grease, especially wool grease containing coloured impurities and so gives the scoured product a much better (ie less yellow) colour.

Each of US-A-2655428 and US-A-2629723 describes a three-stage system in which the suint alone is effectively employed as scouring agent, and a neutral, polar, oxygen containing organic compound and preferably a neutral electrolyte is added to enhance the effectiveness of the suint in emulsifying the wool grease. Thus, for example, wool is agitated in an aqueous solution containing 2% normal butanol and sodium chloride while maintaining the temperature at 60°C. The wool is then squeezed free of the solution and rewashed in a fresh batch of the same solution at the same temperature. The wool is then again squeezed free of the solution, washed with water and dried. However, the scouring efficiency of the suint above has not proved sufficient for the process to achieve at least widespread commercial acceptance.

We have developed a scouring process which, as compared with conventional wool scouring processes, is capable of providing an improved whiteness while maintaining acceptably low values of residual wool grease.

Surprisingly, we have found that this improvement can be achieved by including, prior to a detergent scouring step, a hot de-suinting step in which the de-suinting liquor contains a builder.

Thus, the present invention provides a method of scouring raw wool comprising the steps of (a) treating the wool with an aqueous medium containing a building agent capable of reducing the level of free calcium and/or magnesium therein and having a temperature of at least 45°C and thereafter (b) treating the wool with an aqueous medium having a pH of at least 8, containing a detergent and having a temperature of at least 45°C.

Preferably, the temperature of the aqueous medium in step (a), namely the hot de-suinting liquor, lies between 45-75°C, more preferably 60-70°C.

The pH of the hot de-suinting liquor is preferably at least 8, more preferably from 8-10.5, especially 9-10. Builders which may be used in a method embodying the invention may be inorganic or organic builders.

Examples of inorganic detergency builders include water-soluble alkali metal carbonates, bicarbonates, silicates and crystalline and amorphous aluminosilicates. Specific examples include sodium carbonate (with or without calcite seeds), potassium carbonates, sodium and potassium bicarbonates and silicates.

Examples of phosphorus-containing inorganic detergency builders in particular include water-soluble salts, especially alkali metal pyrophosphates, orthophosphates and polyphosphates. Specific examples of inorganic phosphate builders include sodium and potassium tripolyphosphates, phosphates and hexametaphosphates.

Examples of organic detergency builders include the alkali metal, ammonium and substituted ammonium polyacetates, carboxylates, polyacetal carboxylates and polyhydroxysulphonates. Specific examples include sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilotriacetic acid, oxydissuccinic acid, melitic acid, benezene polycarboxylic acids and citric acid.

Typical examples of the above are ethylene diamine tetraacetic acid (EDTA), nitrilotriacetic acid (NTA), citrates, zeolites, sodium tripolyphosphate, sodium carbonate and sodium silicate, though for those builders

providing an acid pH, a compound capable of providing a pH of at least 8 is preferably added.

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However, a preferred builder, in addition to its role in removing free calcium and/or magnesium from the aqueous medium, is preferably also capable of controlling the pH of the de-suinting liquor to provide a pH of at least 8.

More preferably the builder comprises at least one inorganic ionic compound, especially a mixture of inorganic ionic compounds, more especially a mixture of an alkali metal, for example sodium, carbonate and an alkali metal, for example sodium, silicate, preferably in a proportional amount such as to provide a sodium silicate:sodium carbonate weight ratio of from 20:80 to 80:20, more preferably 30:70 to 50:50, especially about 40:60.

Preferably, the builder concentration in step (a) is at least 0.2g/l and more preferably lies within the range from 0.5-10g/l, especially 1-5 g/l, more especially about 2 g/l.

A preferred liquor to fibre ratio is from 60:1 to 90:1, especially 75:1 to 85:1, more especially about 80:1.

In a preferred method, the preliminary de-suinting step (a) is followed by a detergent scouring step (b) in which the detergent composition contains the same builder as that employed in the de-suinting step, though preferably at a lower concentration, more preferably 0.1-1 g/l, especially 0.3-0.7 g/l, more especially 0.5 g/l.

Because of the oily mixture of wool grease, its removal is best effected by the use of a non-ionic, rather than an anionic, surfactant.

Examples of suitable nonionic surfactants that can be included in the detergent active system include:

- 1. The polyethylene oxide condensates of alkyl phenol, eg. the condensation products of alkyl phenols having an alkyl group containing from 6 to 12 carbon atoms in either a straight chain or branched chain configuration, with ethylene oxide, the said ethylene oxide being present in amounts equal to 3 to 30, preferably 5 to 14 moles of ethylene oxide per mole of alkyl phenol. The alkyl substituent in such compounds may be derived, for example, from polymerised propylene, di-isobutylene, octene and nonene. Other examples include dodecylphenol condensed with 9 moles of ethylene oxide per mole of phenol; dinonylphenol condensed with 11 moles of ethylene oxide per mole of phenol; and nonylphenol and diisooctylphenol condensed with 13 moles of ethylene oxide.
- 2. The condensation product of primary or secondary aliphatic alcohols having 8 to 24 carbon atoms, in either straight chain or branched chain configuration, with from 2 to about 40 moles, preferably 2 to about 9 moles of ethylene oxide per mole of alcohol.
- Preferably, the aliphatic alcohol comprises between 9 and 18 carbon atoms and is ethoxylated with between 2 and 9, desirably between 3 and 8, moles of ethylene oxide per mole of aliphatic alcohol. The preferred surfactants are prepared from primary alcohols which are either linear (such as those derived from natural fats) or prepared by the Ziegler process from ethylene, eg. such as the Lutensols, Dobanols and Neodols which have about 25% 2-methyl branching (Lutensol being a Trade Name of BASF, Dobanol and Neodol being Trade Names of Shell), or Synperonics, which are understood to have about 50% 2-methyl branching (Synperonic is a Trade Name of ICI) or the primary alcohols having more than 50% branched chain structure sold under the Trade Name Lial by Liquichimica. Specific examples of nonionic surfactants falling within the scope of the invention include Dobanol 45-4, Dobanol 45-7, Dobanol 45-9, Dobanol 91-2.5, Dobanol 91-3, Dobanol 91-4, Dobanol 91-6, Dobanol 91-8, Dobanol 23-6.5, Synperonic 6, Synperonic A7, Synperonic 14, the condensation products of coconut alcohol with an average of between 5 and 12 moles of ethylene oxide per mole of alcohol, the coconut alkyl portion having from 10 to 14 carbon atoms, and the condensation products of tallow alcohol with an average of between 7 and 12 moles of ethylene oxide per mole of alcohol, the tallow portion comprising essentially between 16 and 22 carbon atoms. Secondary linear alkyl ethoxylates are also suitable in methods embodying the present invention, especially those ethoxylates of the Tergitol series having from about 9 to 15 carbon atoms in the alkyl group and up to about 11, especially from about 3 to 9, ethoxy residues per molecule.
- 3. The compound formed by condensing ethylene oxide with a hydrophobic base formed by the condensation of propylene oxide with propylene glycol. The molecular weight of the hydrophobic portion generally falls in the range of about 1500 to 1800. Such synthetic nonionic detergents are available on the market under Trade Name of "Pluronic", supplied by Wyandotte Chemicals Corporation.

The detergent active system may comprise a mixture of anionic and nonionic detergents, in which case the preferred nonionic material or mixtures thereof will have an HLB (hydrophilic-lipophilic balance) of not more than 10.5, preferably in the range of from 6 to 10, most preferably in the range of 8 to 9.5. As explained, the detergent active system can contain one or a mixture of more than one nonionic detergent-active material. The mixture can contain one or more nonionic materials having an HLB of more than 10.5, providing the average HLB of the mixture of nonionic materials is not more than 10.5. The HLB scale is a known measure of hydrophilic-lipophilic balance in any compound. It is fully defined in the literature, for example in "Nonionic Surfactants", Volume 1, edited by M J Schick. A method of determining the HLB of a mixture of nonionic materials is

also defined in this reference.

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Preferred nonionic materials are the alkoxylate adducts of fatty compounds selected from fatty alcohols, fatty acids, fatty esters, fatty amides and fatty amines. The fatty compound contains at least 10 carbon atoms and the nonionic material contains an average of less than 8 alkylene oxide groups per molecule.

Alkylene oxide adducts of fatty alcohols useful in methods embodying the present invention preferably have the general formula:

$$R^{10}$$
-O- $(C_nH_{2n}O)_yH$

wherein R^{10} is an alkyl or alkenyl group having at least 10 carbon atoms, most preferably from 10 to 22 carbon atoms, y is preferably not more than 10, such as from 0.5 to about 3.5, and n is 2 or 3. Examples of such materials include Synperonic A3(ex ICI), which is a C_{13} - C_{15} alcohol with about three ethylene oxide groups per molecule, and Empilan KB3 (ex Marchon) which is lauric alcohol 3EO.

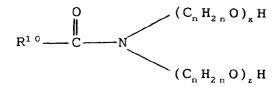
Alkylene oxide adducts of fatty acids useful in methods embodying the present invention preferably have the general formula:

$$R^{10}$$
-C-O $(C_n H_{2n} O)_v H$,

wherein R¹⁰, n and y are as given above. Suitable examples include ESONAL 0334 (ex Diamond Shamrock), which is a tallow fatty acid with about 2.4 ethylene oxide groups per molecule.

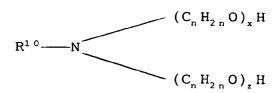
Alkylene oxide adducts of fatty esters useful in methods embodying the present invention include adducts of mono-, di- or triesters of polyhydric alcohols containing 1 to 4 carbon atoms; such as coconut or tallow oil (triglyceride) 3EO (ex Stearine Dubois).

Alyklene oxide adducts of fatty amides useful in methods embodying the present invention preferably have the general formula:



wherein R^{10} is an alkyl or alkenyl group having at least 10 carbon atoms, most preferably from 10 to 22 carbon atoms, n is 2 or 3 and x and z in total are not more than 4.0, preferably from about 0.5 to about 3.5, while one of x and z can be zero. Examples of such materials include tallow monoethanolamide and diethanolamide, and the corresponding coconut and soya compounds.

Alkylene oxide adducts of fatty amines useful in methods embodying the present invention preferably have the general formula:



wherein R¹⁰ and n are as given above, and x and z in total are preferably not more than 4.0, most preferably from about 0.5 to about 3.5. Examples of such materials include Ethomeen T12 (tallow amine 2EO, available from AKZO), Optameet PC5 (coconut alkyl amine 5EO) and Crodamet 1.02 (oleylamine 2EO, available from Croda Chemical).

Cationic detergent-active materials suitable for use herein include quaternary ammonium surfactants and surfactants of a semi-polar nature, for example amine oxides.

Amounts of amphoteric or zwitterionic detergent compounds can also be used in a method embodying the invention, but this is not normally desired owing to their relatively high cost. If any amphoteric or zwitterionic detergent compounds are used, it is generally in small quantities relative to the much more commonly used anionic and/or nonionic detergent-active compounds.

The detergent is preferably present in a concentration of 0.5 to 10 g/l, especially 1-5 g/l, more especially about 2 g/l.

A preferred method is carried out continuously, more preferably with the wool and water travelling in countercurrent flow.

An especially preferred scouring system includes an additional subsequent detergent treatment (c), using

an aqueous medium at a temperature of at least 45°C, though using a lower concentration of detergent, preferably 0.1-1, more preferably 0.3-0.7, especially about 0.5 g/l. Steps (a) to (c) may be carried out successively, though preferably excess liquor in the wool is removed between the successive stages.

Typically, a method embodying the invention comprises the successive steps of

- (a) treating the wool with an aqueous medium containing a building agent capable of reducing the level of free calcium and/or magnesium in the aqueous medium, having a pH of at least 8 and having a temperature of at least 60°C,
- (b) treating the wool from step (a) with an aqueous medium having a pH of at least 8 and a temperature of at least 60°C and containing a detergent,
- (c) treating the wool from step (b) with an aqueous medium having a temperature of at least 55°C and containing a detergent,
- (d) treating the wool from step (c) with an aqueous medium having a temperature of at least 50°C to provide a first rinse and
- (e) treating the wool from step (d) with an aqueous medium having a temperature of at least 50°C to provide a second rinse.

A preferred continuous system for carrying out a method embodying the invention will now be described with reference to the accompanying drawing, which is a schematic representation of a continuous countercurrent flow system.

Thus, the system preferably contains four to six, especially five bowls, indicated as 1-5 in the drawing. Arrows A and B represent entry of raw wool into and exit of wool out of the system and arrow E represents the direction of travel of the wool. Arrow C represents the water inlet and arrow D the water outlet, arrow F representing the direction of travel of the water. After all of the bowls have been filled with water and the appropriate detergents and chemicals added, the raw wool is introduced into bowl 1 and moved along the bowl by a series of rakes suspended above the bowl. At the end of the bowl, the wool passes up a ramp onto a pair of squeeze rollers which removes most of the liquor entrained between the wool fibers and so prevents conveyance of contaminants removed in bowl 1 into bowl 2. At the same time as the wool moves down the line of bowls, clean water is introduced into bowl 5 and a countercurrent flow system is used to pump this water back down the line of the bowls.

The de-suinting operation takes place in bowl 1, preferably at a temperature from 60-70°C, more especially 65°C, and a builder concentration of about 2 g/l. Typically the builder is a mixture of sodium silicate/sodium carbonate in a ratio of 40/60. Such a builder is commercially available from Crosfield Textile Chemicals, Warrington, England, as Croscour P100.

A first detergent scouring operation is carried out in bowl 2, preferably at a temperature of from 60-70°C, more especially 65°C, using a non-ionic detergent, typically at a concentration of 2 g/l and the same builder as that used in bowl 1, but at a lower concentration of about 0.5 g/l.

A subsequent detergent treatment is carried out in bowl 3, which contains the same detergent as bowl 2, but at a lower concentration of about 0.5 g/l and no builder. The scouring temperature is preferably 55-65, more preferably 60°C.

In each of bowls 4 and 5, rinsing operations are carried out, using clean water at a temperature preferably from 40-60°C, more preferably about 55°C.

Usually, the throughput of wool will be around 1,000 kilo/hour, with a residence time in each bowl of about 2 mins.

Example

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The following experiment was carried out in order to illustrate the advantages of a method embodying the invention.

A 300g batch of English 56's Arran wool was hand blended to ensure an even distribution of soiling. This 300g was divided into 6×50 g lots which were then further divided into 5×10 g samples. To simulate an industrial scouring range 5×1 litre beakers were used. Each beaker contained 800 ml of water giving a liquor to fibre ratio of 80:1. The temperature of the water in each beaker was as shown in Table 1 below.

Additions of builders and detergents were made to various beakers at outlined in Table 1.

Run Nos. 1-4 and 6 represent comparative methods, while Run No. 5 represents a method embodying the invention.

In this experiment, the samples of wool were treated consecutively as separate 10g samples with a residence time of 2 minutes in each beaker/bowl. The wool was passed through a laboratory mangle between each beaker to simulate the squeeze rollers used on industrial scouring ranges.

The first 30g of each 50g sample was carded on a Shirley Miniature laboratory cotton card to produce an

evenly distributed array of aligned fibres from which reflectance measurements could be made to assess the colour of wool. A Zeiss Elrepho Spectrometer was used for this operating at 460 nm and using a Zeiss supplied white disc as a reflectance standard. Data are presented as R460 values, ie reflectance at 460 nm on a scale of 1 to 100, where 100 represents a perfect "white". Measurements were made at ten different places on the wool sample and results averaged.

The remaining 20g of each 50g sample was treated in a similar manner to provide a measure of the change in the observed data as soil and other contaminants accumulated in the beakers. Approximately 5g of this wool was accurately weighed, extracted with dichloromethane for 2 hours and reweighed to assess the residual grease level on the wool.

The results of the reflectance and residual grease level tests are shown in Table 2 below.

a) Colour (whiteness) of the Wool

The poorest result (Run 1) is found when using detergent only in Bowls 1 and 2. A small improvement can be obtained by using a hot suint bowl (Run 3). If a mixed builder system is included with the detergents in Bowls 1 and 2 then, as Run 2 shows, some improvement in colour is obtained.

Run 4 shows that the use of a suint bowl in combination with detergent and builder in Bowls 2 and 3 produces a further improvement in colour (whiteness). However, the best colour is found for Run 5 where the major portion of the builder is included in the first suint bowl. This improved whiteness is especially apparent for the results from the last 20g of wool from the 50g sample. At this stage the scouring solutions in the beakers have started to "mature" and scouring efficiency is becoming optimal. Thus, it is recognised in the industry that after cleaning and refilling the bowls, scouring efficiency is somewhat reduced for about the first 30 minutes until the bowls begin to "mature". We believe that such maturation may be due to improved wetting resulting from the formation of soaps of wool grease.

Run 6 shows the results for a prior art method of B.O. Bateup and J.J. Warner, Text. Res. J. (1986), <u>56</u>, 489, where a cold suint bowl is used with sodium carbonate as the builder. As discussed above, this method is usually used to facilitate effluent disposal and not to optimise the quality of the scour. The reflectance values show it to be inferior to the built suint bowl used in the method embodying the invention

b) Residual Wool Grease

All six runs gave acceptably low values of residual wool grease. It is common practice to try and optimise the wool scour process to give residual values in the range 0.35-0.45% by weight. Lower residual levels than this can give problems in subsequent processing.

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5		Rowl 5 55°C	Rinse	Rinse	Rinse	Rinse	Rinse	55°C	Rinse
10		Bowl 4 55°C	Rinse	Rinse	Rinse	Rinse	Rinse	2.55	Rinse
15		Bowl 3 60°C	Rinse	Rinse	0.5 g/l Detergent	0.5 g/l Detergent 0.5 g/l Builder	0.5 g/l Datergent	U	0.5 g/l Detergent
20		RON 60	Ri	Ri	0.5 g/l	0.5 g/1 0.5 g/1	0.5 g/1	2.09	0.5 9/1
25	E 1	C 2	etergent	etergent uilder	ergent	ergent lder	ergent uilder	65°C	ergent
30	TABLE 1	Bowl 2 65°C	0.5 g/l Detergent	0.5 g/l Detergent 0.5 g/l Builder	2 g/l Detergent	2 g/l Detergent 2 g/l Duilder	2 g/l Detergent 0.5 g/l Builder	65	2 g/l Detergent
35	•	, r	rgent	cergent 11der	ions Bowl	ions Bowl	ilder ie int Bowl		bonate
40		Bowl 1 65°C	2 g/l Detergent	2 g/l Deterger 2 g/l Builder	No additic ie Suint D	No additions ie Suint Bow	2 g/l Builder ie Built Suint Bowl	20.℃	2 g/l Sodium Carbonate
45		erature	-	73	n	4	ហ	perature	9
50		Water Temperature	Run 1	Run 2	Run 3	Run 4	Run 5	Water Temperature	Run 6

In the experiments set out in Table 1, the builder employed was a mixture of sodium silicate and sodium carbonate in a 40:60 weight ratio.

The detergent employed had the following formulation:-

45.8% Synperonic NP9¹ (ex ICI)
5 14.0% Synperonic NP4² (ex ICI)

14.5% Caflon DP848³ (ex Cargo Fleet Chemicals)
 3.5% Caflon SNA⁴ (ex Cargo Fleet Chemicals)

1.2% NaOH 100°Tw (ex ICI)

10 14.0% Water

5.0% Dowanol TPM⁵ (Dow Chemicals)

Notes:

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Synperonic NP9 - nonyl phenol plus 9 moles ethylene oxide.
 Synperonic NP4 - nonyl phenol plus 4 moles ethylene oxide.

³ Caflon DP848 - 50:50 mixture of Synperonic 91/6 and Synperonic 91/8.
 Synperonic 91/6 - C₉-C₁₁ linear alcohol ethoxylate (6 moles ethylene oxide)
 Synperonic 91/8 - C₉-C₁₁ linear alcohol ethoxylate (8 moles ethylene oxide)

⁴ Caflon SNA - dodecyl benzene sulphonate as the Na salt

⁵ Dowanol TPM - tripropylene glycol mono ethyl ether

TABLE 2

R 460 VALUES

	1st 30g	Remaining 20g	Residues % by Weight
Run 1	56.23 (1.32)	56.14 (1.37)	0.42
Run 2	56.94 (1.14)	59.33 (1.86)	0.46
Run 3	56.81 (1.46)	57.57 (1.63)	0.48
Run 4	58.48 (1.63)	58.56 (1.49)	0.34
Run 5	58.56 (1.75)	59.99 (1.27)	0.43
Run 6	57.47 (1.32)	59.52 (1.42)	0.36

Figures in brackets refer to the standard deviation assesiated with the reflectance measurements.

Although not wishing to be bound by theory, it is believed that the scouring method embodying the invention exhibits the advantages illustrated above for the following reasons.

- 1. By using a hot "built" suint bowl system in accordance with the invention, most of the particulate soil and some of the greasy soils is removed in the first bowl without the addition of extra detergents. The main detergent bowl (bowl 2) therefore receives the wool in a much cleaner condition. This leads to more cost-effective use of the (comparatively expensive) detergents. In particular, as a result of extended bulk running trials, we have found that overall detergent usage is reduced and the scouring range can be operated in continuous mode for longer periods before all bowls have to be emptied for cleaning.
- 2. Because of the efficiency of sequestration of calcium and magnesium in the suint bowl by the builder, problems of calcium and magnesium soap formation are avoided. Thus, in conventional wool scouring processes, the formation of free fatty acids in the warm wool grease tends to result in the formation of a hard "skin" of calcium and magnesium soaps around the droplets of grease on the wool fibers. This skin is extremely difficult to remove and is often not properly removed at all, resulting in an undesirable grey colour being imparted to the wool.
- 3. This is especially so when using a mixture of sodium silicate/sodium carbonate, which we find is a more effective builder than sodium carbonate alone.
 - 4. The alkaline conditions created cause saponification of the fatty acids in the wool grease, so creating detergent molecules from within the wool grease, thereby yielding soaps in situ in the grease. Accordingly, the detergent action starts before additional detergent is introduced. Thus, the system begins to self-emulsify before the addition of any detergent.
 - 5. The combination of silicate and carbonate provides excellent soil suspending properties, thus giving efficient removal of particulate soils, with no tendency for redeposition.

25 Claims

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- 1. A method of scouring raw wool comprising the steps of (a) treating the wool with an aqueous medium containing a building agent capable of reducing the level of free calcium and/or magnesium therein and thereafter (b) treating the wool with an aqueous medium having a pH of at least 8, containing a detergent and having a temperature of at least 45°C characterised in that step (a) is carried out at a temperature of at least 45°C.
- 2. A method according to claim 1, wherein the aqueous medium in step (a) has a pH of at least 8.
- 35 3. A method according to claim 1 or claim 2, wherein the pH of the aqueous medium in step (a) is from 8 to 10.5 inclusive.
 - **4.** A method according to claim 1 or claim 2, wherein the said building agent in step (a) is capable additionally of controlling pH so as to provide the said pH of at least 8.
 - **5.** A method according to claim 4, wherein the building agent comprises at least one inorganic ionic compound.
 - 6. A method according to claim 5, wherein the building agent is a mixture of inorganic ionic compounds.
 - 7. A method according to claim 6, wherein the said mixture comprises an alkali metal silicate and an alkali metal carbonate.
 - 8. A method according to claim 7, wherein the alkali metal is sodium.
 - **9.** A method according to claim 8, wherein the mixture comprises sodium silicate/sodium carbonate in a proportional amount of from 30/70 to 50/50.
 - **10.** A method according to claim 9, wherein the proportional amount of sodium silicate/sodium carbonate is about 40/60.
 - 11. A method according to any preceding claim, wherein the building agent present in the aqueous medium in step (a) is also present in the aqueous medium of step (b).

- **12.** A method according to any preceding claim wherein the temperature of the aqueous medium in step (a) is from 60 to 70°C inclusive.
- **13.** A method according to any preceding claim, wherein the detergent present in the aqueous medium in step (b) is a non-ionic detergent.
 - **14.** A method according to any preceding claim, which is a continuous process.

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- 15. A method according to claim 14, wherein the wool is passed in one direction so as to be immersed in (a) a first aqueous medium present in a first bowl, which first aqueous medium contains a building agent capable of reducing the level of free calcium and/or magnesium in the aqueous medium and having a temperature of at least 45°C, thereby to effect the said step (a), and thereafter is immersed in (b) a second aqueous medium present in a second bowl which second aqueous medium has a pH of least 8, contains a detergent, and has a temperature of at least 45°C thereby to effect the step (b), and water flows in the opposite direction so as to provide the aqueous medium for step (b) and thereafter for step (a).
 - **16.** A method according to any preceding claim, which includes the step (c), carried out subsequently to step (b), of treating the wool with an aqueous medium containing a detergent and being at a temperature of at least 45°C.
 - 17. A method according to claim 16, wherein steps (a), (b) and (c) are carried out successively.
 - **18.** A method according to any preceding claim, which includes at least one additional rinsing step in which the wool is treated with an aqueous medium which is at a temperature of at least 45°C.
 - 19. A method of scouring wool comprising the successive steps of (a) treating the wool with an aqueous medium containing a building agent capable of reducing the level of free calcium and/or magnesium in the aqueous medium, having a pH of at least 8 and having a tem
 - perature of at least 60°C, (b) treating the wool from step (a) with an aqueous medium having a pH of at least 8 and a temperature of at least 60°C and containing a detergent,
 - (c) treating the wool from step (b) with an aqueous medium having a temperature of at least 55°C and containing a detergent,
 - (d) treating the wool from step (c) with an aqueous medium having a temperature of at least 50°C to provide a first rinse and
 - (e) treating the wool from step (d) with an aqueous medium having a temperature of at least 50°C to provide a second rinse.

Fig.1.

