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**Aqueous structured liquid detergent composition comprising aminocarboxylate sequestrant**

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<p>(21) International Application Number: PCT/EP97/04925 (22) International Filing Date: 4 September 1997 (04.09.97) (30) Priority Data: 08/722,546 27 September 1996 (27.09.96) US (71) Applicant (for all designated States except AU BB CA GB GH IE KE LK LS MN MW NZ SD SG SZ TT UG): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). (71) Applicant (for AU BB CA GB GH IE KE LK LS MN MW NZ SD SG SZ TT UG only): UNILEVER PLC (GB/GB); Unilever House, Blackfriars, London EC4P 4BQ (GB).</p>	<p>(72) Inventors: WU, Shang-Ren; Unilever Research Shanghai, Institute of Organic Chemistry, 354 Fenglin Road, Shanghai (CN). BROUWN, Lili, Paucia; Unilever Research Vlaardingen Laboratory, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL). VAN DE PAS, Johannes, Cornelis; Unilever Research Vlaardingen Laboratory, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL). VERHAGEN, Jaap; Unilever Research Vlaardingen Laboratory, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL). WISSENBURG, Petronela, C.; Unilever Research Vlaardingen Laboratory, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL). (74) Agent: KAN, Jacob, H.; Unilever N.V., Patent Division, P.O. Box 137, NL-3130 AC Vlaardingen (NL). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). <b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	
<p>(54) Title: AQUEOUS STRUCTURED LIQUID DETERGENT COMPOSITION COMPRISING AMINOCARBOXYLATE SEQUEST-RANT</p>		
<p>(57) Abstract The present invention is directed to aqueous structured liquid detergent compositions comprising surfactant material, water and electrolyte, wherein the electrolyte comprises aminocarboxylate sequestrant and builder material. The invention further relates to a process of preparing liquids.</p>		

AQUEOUS STRUCTURED LIQUID DETERGENT COMPOSITION COMPRISING  
AMINOCARBOXYLATE SEQUESTRANT

5 Technical Field

The present invention relates to aqueous liquids, in particular structured liquids and processes of preparing aqueous liquids.

10 Background & Prior Art

Liquid detergent compositions are well-known in the art and offer several advantages over solid compositions. For example, liquid compositions are easier to measure, to dispense and to dissolve into a laundering liquor and  
15 liquid compositions give more confidence to the consumer of being safer and less harsh to the washed or laundered textile and environment than solid compositions. This may be the reason that heavy and light duty laundry liquid detergent products are gaining in popularity ever since  
20 their introduction on the market at the expense of powdered detergent products, in particular for coloured fabrics.

We have however found that liquid detergent compositions may suffer from extensive physical instability, chemical  
25 instability and/or high viscosity problems as compared to solid detergent compositions and these problems are even more imminent for concentrated aqueous liquids. This is probably due to the high number of possible interactions of ingredients in liquid detergent compositions. Moreover,  
30 these interactions will affect not only the stability and viscosity, but also the wash performance.

EP 137474 and EP 137475 disclose aqueous prespotting compositions comprising surfactant and chelating agent. The compositions are said to have good water-borne and oil-borne stain removal characteristics.

5

EP 267,653 discloses detergent compositions comprising surfactant, builder and biodegradable EDDS which is said to possess excellent stain removal properties.

10 WO 92/06164 discloses the use of polyhydroxy fatty acid amide surfactants in detergent compositions and refers to the use of EDDS as chelating agent.

WO 94/03553 discloses detergent compositions comprising  
15 bleach and EDDS, as a biodegradable bleach stabiliser.

WO 94/03554 discloses detergent compositions comprising EDDS, percarbonate and layered silicate. The compositions are said to have benefits on bleachable and enzymatic stain  
20 removal even when reduced amounts in total of builder, bleach and chelant are used.

WO 94/03572 discloses detergent compositions comprising EDDS and crystalline layered silicate to reduce the  
25 deposition of salt encrustations and multi-cycle build up of such encrustations. As indicated in the abstract and disclosed in the example, the document relates to granular compositions. The compositions comprise perborate and layered silicate material.

30

WO 94/20599 discloses detergent compositions comprising surfactant and EDDG or HPDDS. The compositions are said to have good stain removal and cleaning performance.

5 US 3,956,198, US 5,221,496, US 5,186,856, US 4,560,492 and EP 233,010 disclose the use of aminocarboxylates for improving stain removal.

Aminocarboxylates have further been disclosed in US  
10 4,663,071 and FR 2,156,080.

We have found that a way of formulating detergent compositions which have good stain removing performance and environmental profile. The compositions have the advantage  
15 of being physically and chemically stable with low viscous and good dispersing properties.

Consequently, the present invention is directed to an aqueous structured liquid detergent composition comprising  
20 surfactant material, electrolyte and water, wherein the electrolyte comprises aminocarboxylate sequestrant and builder material.

The present invention is further directed to a process of  
25 preparing an aqueous structured liquid detergent composition comprising surfactant material, electrolyte and water, wherein the electrolyte comprises aminocarboxylate sequestrant, and wherein the process involves mixing the ingredients whilst keeping the pH above 6 and keeping the  
30 temperature below 80°C, preferably after addition of the carboxylate.

The present invention is further directed to a process for washing coloured fabric by using a liquid detergent composition comprising an aminocarboxylate sequestrant.

#### 5 Detailed Description of the Invention

In view of the consumer confidence, liquids preferably only contain safe and non-harsh ingredients. Consequently, liquids of the invention are preferably free of bleach, of layered silicate and contain up to 10%, more preferably up to 5%, most preferably up to 3%, in particular preferred substantially no solvent material.

We have found that that liquids of the present invention which meet one or more of these criteria are in particular 15 preferred for the washing of stained, coloured clothes.

#### Liquid compositions

Two general and separate classes of liquids compositions, isotropic and structured liquids, are known in the art. 20 Isotropic liquids are liquids in which all ingredients are dissolved and, contrary to structured liquids, there is no structure present in isotropic liquid.

Structured liquids are well-known in the art. They can 25 either be internally structured, whereby the structure is formed by primary ingredients, preferably by surfactant material, and/or by providing a three dimensional matrix structure using secondary additives, preferably polymers and/or silicate material. Structuring may be brought about 30 to endow properties such as consumer preferred flow properties and/or turbid appearance. Many structured liquids are also capable of suspending particulate solids,

such as particles of clay that may be used to provide a fabric-softening effect to fabrics. Examples of structured liquids without suspended solids are given in US-A-4,244,840, whilst examples where solid particles are 5 suspended are disclosed in EP-A-160 342; EP-A-38 101; EP-A-140 452 and also in the aforementioned US-A-4,244,840. We have found that structured liquids are preferred over isotropic liquids as they provide more formulation flexibility, e.g. as regards inclusion of ingredients and 10 concentration. Structured liquids may however suffer from viscosity and stability problems.

Preferably, liquids of the invention have a viscosity of from 200 to 2500mPa.s at  $21 \text{ s}^{-1}$  at 25°C, more preferably 15 the viscosity is lower than 2000, most preferably the viscosity is lower than 1200.

Preferably, liquids of the invention have a stability of at most 5mm volume phase separation after 1 month storage at 20 room temperature, more preferably at most 2mm, most preferably there is substantially no phase separation.

#### Surfactants

Liquid detergent compositions of the invention comprise 25 surfactant materials.

In the widest definition the surfactant material in general, may comprise one or more surfactants, and may be selected from anionic, cationic, nonionic, zwitterionic and 30 amphoteric species, and (provided mutually compatible) mixtures thereof.

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Suitable nonionic surfactants include for example aliphatic alcohols, acids, amides or alkyl phenols with alkyl oxides, especially ethylene oxide. Specific nonionic detergent compounds are alkyl (C<sub>6</sub>-C<sub>18</sub>) primary or secondary linear or 5 branched alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylene-di-amine.

We have found that the wash performance of liquids is in 10 particular improved when nonionics are included in compositions of the invention. Preferably, the level of nonionic surfactant materials is from 1 to 40% by weight of the composition, more preferred from 2 to 25%.

15 Compositions of the present invention may contain synthetic anionic surfactant ingredients. Suitable anionic surfactants are usually water-soluble alkali metal salts of organic sulphates and sulphonates having alkyl radicals containing from about 8 to about 22 carbon atoms. Examples 20 of suitable synthetic anionic surfactant compounds are sodium and potassium C<sub>8</sub>-C<sub>18</sub> alkyl sulphates, sodium and potassium alkyl (C<sub>9</sub>-C<sub>20</sub>) benzene sulphonates and ether variants thereof. Generally, the level of the above mentioned non-soap anionic surfactant materials is from 1 25 to 40 % by weight of the composition, more preferred from 2 to 25%.

It is also possible, and sometimes preferred, to include an alkali metal soap of a C<sub>12</sub>-C<sub>18</sub> carboxylic acid. Preferably, 30 the level of soap is from 1 to 35% by weight of the composition, more preferred from 5 to 25%.

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Preferably, surfactant material is present at a level of at least 1% by weight of the composition, more preferred at least 5% by weight, most preferred at least 10% by weight of the composition; and preferably at a level of at most 70% by weight, more preferably at most 40%, most preferably at most 35% by weight.

Preferably, compositions according to the invention comprise anionic and nonionic surfactants, more preferably each at a level of higher than 5% by weight of the composition.

#### Electrolyte Material

Compositions according to the invention comprise electrolyte material. It is noted that for the purpose of the invention, the term electrolytes includes sequestrants and builder material.

Preferably, the total level of electrolyte is from 1 to 60% by weight of the composition, more preferably from 5 to 45% by weight, most preferably from 10 to 30% by weight.

#### Aminocarboxylate Sequestrants

Compositions of the invention comprise an aminocarboxylate sequestrant.

Most preferably, the aminocarboxylate sequestrant is selected from 1N carboxylates, 2N carboxylates, 3N carboxylates and mixtures thereof.

30

Preferably, aminocarboxylates of the invention are selected from the amino(poly)carboxylates.

Preferably, the sequestrant is selected from aminocarboxylate comprises an  $-N(MOOCCH_2)_2$  group and/or an  $-N-CH_2(CH_2COOM)(COOM)$  group, wherein M is hydrogen, metal (e.g. alkali metal), ammonium or a mixture thereof, wherein the sequestrant is optionally substituted with one or more  $-SO_3Na$  groups.

in the liquid detergent

Preferably, the sequestrant of the invention is selected from salts of glutamic acid N,N-diacetic acid, beta-alanine diacetic acid, ethylenediamine triacetic acid, methyl glycine diacetic acid, diethylenetriamine penta-acetic acid, ethylenediamine-N,N-disuccinic acid, ethylenediamine di(sulfosuccinate) and mixtures thereof.

15

Within this group, the preferred sequestrant is selected from salts of glutamic acid N,N-diacetic acid, beta-alanine diacetic acid, methyl glycine diacetic acid, ethylenediamine-N,N-disuccinic acid and mixtures thereof.

20

Within this group, beta-alanine diacetic acid, methyl glycine diacetic acid, ethylenediamine-N,N-disuccinic acid and mixtures thereof are preferred.

25 Within this group, methyl glycine diacetic acid, ethylenediamine-N,N-disuccinic acid and mixtures thereof are preferred. S,S ethylenediamine-N,N-disuccinic acid is in particular preferred.

in the liquid detergent

30 Preferably, the level of the sequestrant of the invention is from 0.1 to 5% by weight of the composition, more



preferably from 0.5 to 4% by weight, most preferably from 1 to 3% by weight of the composition.

#### Builder

5 In any event, compositions according to the present invention include detergency builder material, some or all of which may be electrolyte. In this context it should be noted that some surfactant materials such as for example soaps, also have builder properties.

10

Examples of phosphorous containing inorganic detergency builders include the water-soluble salts, especially alkali metapyrophosphates, orthophosphates, polyphosphates and phosphonates. Examples of non-phosphorus-containing  
15 inorganic detergency builders, when present, include water-soluble alkali metal carbonates, bicarbonates, silicates and crystalline and amorphous aluminosilicates. Examples of organic detergency builders, when present, include the alkaline metal, ammonium and substituted  
20 ammonium polyacetates, carboxylates, polycarboxylates, polyacetyl carboxylates and polyhydroxysulphonates.

Preferably, builder material is selected from sodium citrate, sodium tripolyphosphate, zeolite and mixtures  
25 thereof.

Preferably, the builder is included in level to obtain a  $\text{Ca}^{2+}$  concentration in the wash liquor is below  $10^{-3}$  mol/l, more preferably below  $10^{-4}$  mol/l, most preferably below  $10^{-5}$   
30 mol/l, e.g. from 0 to  $10^{-6}$  mol/l. We have found that this favourably influences the objective of the present invention.

Preferably, the level of non-soap builder material is higher than 5%, more preferably higher than 7%, most preferably higher than 8% by weight of the composition and 5 preferably lower than 40%, more preferred lower than 38% by weight of the composition.

Surprisingly, we have found that in particular the combination of a sequestrant according to the invention and 10 the builder material according to the invention at the levels indicated, leads to stable and high performing liquids.

#### Salting-out electrolyte

15 Compositions according to the invention preferably contain a salting-out electrolyte that is able to bring about internal structuring of the liquid, preferably in the form of lamellar droplets of the surfactant material.

Salting-out electrolyte has the meaning ascribed to in 20 specification EP 79,646, i.e. salting-out electrolytes have a lyotropic number of less than 9.5, preferably less than 9.0. Examples are sulphate, citrate, NTA and carbonate.

We have found that the aminocarboxylate of the compositions 25 of the invention is preferably combined with another salting-out resistant electrolyte in order to provide stable, low viscous structured liquids.

Preferably, the compositions contain from 1% to 60%, 30 especially from 10 to 45% of salting-out electrolyte.

Optionally, some salting-in electrolyte (as defined EP 79,646) may also be included.

#### 5 Water

It is preferred to prepare highly concentrated liquids, although this is in particular difficult in view of the physical properties of the liquid. Liquid compositions of the invention therefor preferably comprise water at a level  
10 of from 30 to 60%, more preferably from 32 to 50%, most preferably from 35 to 45% by weight of the composition.

Preferably, liquids of the invention have a pH of from 7 to 10, more preferably of from 7.5 to 9.

15

#### Optional ingredients

Apart from the ingredients already mentioned, a number of optional ingredients may also be present, for example lather boosters, lather depressants, inorganic salts such  
20 as sodium sulphate, and, usually present in very minor amounts, fluorescent agents, perfumes, enzymes such as proteases, amylases and lipases (including Lipolase (Trade Mark) ex Novo), enzyme stabilisers, anti-redeposition agents, germicides and colorants.

25

#### Processing

Generally, liquid detergent compositions of the invention may be prepared by any method described in the art. An example is to disperse of electrolyte ingredient together  
30 with the minor ingredients except for the temperature and pH sensitive ingredients, such as enzymes, perfumes, etc. - if any- in water of elevated temperature, followed by the

addition of the builder material ~~if any~~, the surfactant material (possibly as a premix) under stirring and thereafter cooling the mixture and adding any pH and temperature sensitive minor ingredients.

5

However, as indicated above, we have found a particularly preferred process of preparing liquids of the invention.

Consequently, the present invention is directed to a  
10 process of preparing a liquid detergent composition comprising surfactant material, electrolyte and water, wherein the composition has a structure and the electrolyte comprise aminocarboxylate sequestrant and mixtures thereof, by mixing the ingredients and keeping the pH above 6 and  
15 the temperature below 80°C.

Preferably, liquids of the invention are prepared by mixing water and electrolytes, including the sequestrant of the invention and any builder material, whereafter a premix of  
20 surfactants is added. We have found that the process of the invention ensures the best product characteristics as regards solidification and stability. The process has in particular been found to be beneficial when the sequestrant is added before the zeolite builder material, whereafter  
25 the active premix is added. This would not be expected in view of the presence of the sequestrant which would sequester metal present in the builder material.

The invention will be illustrated by way of the following  
30 non-limiting Examples.



ExamplesExamples 1-6

The tetrasodium salt of glutamic acid N,N-diacetic acid,  
5 the trisodium salt of beta-alanine diacetic acid, the  
trisodium salt of ethylenediamine triacetic acid, the  
trisodium salt of methyl glycine diacetic acid, the  
pentasodium salt of diethylenetriamine penta-acetic acid,  
the tetrasodium salt of S,S ethylenediamine-N,N-disuccinic  
10 acid, the tetrasodium salt of ethylenediamine tetra-acetic  
acid and the sodium salt of ethylenediamine  
di(sulfosuccinate) were incorporated at concentrations  
between 1 and 4% in the compositions listed in Table 1. The  
ingredients were mixed according the process of the  
15 invention, as indicated in the description.

All liquids have good physical properties and show good  
performance. The liquids with a 2/3N carboxylates were  
preferred over the 1N carboxylates as regards the  
20 performance.

Examples 7-14

The liquids of Tables 2 and 3 were prepared by mixing the  
ingredients according to the process of the invention as  
25 indicated in the description whilst keeping the pH above 6  
and the temperature below 80°C. The physical properties,  
wash performance and environmental profile of the  
compositions was determined.

Table 1

Ingredients	Ex.1	Ex.2	Ex.3	Ex.4	Ex.5	Ex.6
Na-LAS	28		7	24	17.5	
PAS 1)		10				
Nonionic 2)		18.4	3		9	18
Nonionic 3)	12					
Sodium Alkyl Ethoxy Sulphate				6		
Oleic-acid		10	1		4.5	12
NaOH and/or KOH	neutralisation up to pH 8-10					
Glycerol/borax	7.7	8.5	8.5	4.5	3.5	3.5
Mono & tri ethanolamine	4					
Sodium citrate	10	7.7		2.8	11.5	11.5
Zeolite					15	15
STP			20	22		
Deflocculating polymer 4)	1	1		1	1	1
Polymer 5)					0.5	0.5
Minors	0.8	1.1	0.8		0.9	0.9
Enzymes	1.0	0.6	0.6	0.4	0.7	0.7
PVP		0.5				
Water	up to 100					



- 1) Primary Alkyl Sulphate
- 2) Fatty alcohol ethoxylate 7EO
- 3) Fatty alcohol ethoxylate 9EO
- 4) Polymer A11 of EP 346995
- 5) Permalose, ex ICI

Table 2

Ingredients	Reference	Ex. 7	Ex. 8	Ex. 9	Ex. 10
Na-LAS	17.5	17.5	17.5	17.5	17.5
Fatty alcohol ethoxylate 7EO	9	9	9	9	9
Oleic-acid	4.5	4.5	4.5	4.5	4.5
NaOH and/or KOH	adjust pH to 8.5				
Glycerol	2.0	2.0	2.0	2.0	2.0
Borax	1.5	1.5	1.5	1.5	1.5
Sodium citrate	11.5	9.0	9.0	9	9.0
Zeolite	15	15	15	15	15
S,S EDDS 1)		2			
Trilon ES 2)			2		
Trilon GS 3)				2	

Nervanaid GBS-5 4)					2
Polymer 5)	1	1	1	1	1
Oily-Soil Release polymer	0.5	0.5	0.5	0.5	0.5
Enzyme	0.7	0.7	0.7	0.7	0.7
Minors	0.9	0.9	0.9	0.9	0.9
Water	up to 100%				
Product properties:					
Physical stability	Okay	Okay	Okay	Okay	Okay
Pourability	Okay	Okay	Okay	Border rline	Okay
Stain removal	ref	best	improved over reference		
Environment al acceptabili ty	Okay	Okay	Okay	Okay	Border line

- 1) Ethylenediamine-N,N-disuccinic acid
- 2) Methylglycine diacetic acid
- 3) Beta-alanine diacetic acid
- 5 4) Glutamic acid N,N-diacetic acid
- 5) Polymer All of EP 346995

Table 3

Ingredients	reference	Ex.11	Ex.12	Ex.13	Ex.14
Fatty alcohol ethoxylate 7EO	18	18	18	18	18
Oleic-acid	12	12	12	12	12
NaOH and/or KOH	adjust pH to 8.5				
Glycerol	2.0	2.0	2.0	2.0	2.0
Borax	1.5	1.5	1.5	1.5	1.5
Sodium citrate	11.5	11.5	11.5	11.5	11.5
Zeolite	15	15	15	15	15
S,S EDDS 1)		2			
Trilon ES 2)			2		
Trilon GS 3)				2	
Nervanaid GBS-5 4)					2
Polymer 5)	1	1	1	1	1
Oily-Soil Release polymer; Permalose	0.5	0.5	0.5	0.5	0.5
Silicon oil	0.3	0.3	0.3	0.3	0.3
Protease	0.4	0.4	0.4	0.4	0.4

Lipolase	0.2	0.2	0.2	0.2	0.2
Amylase	0.1	0.1	0.1	0.1	0.1
Fluorescent whitening agent	0.1	0.1	0.1	0.1	0.1
Perfume	0.5	0.5	0.5	0.5	0.5
Water	up to 100%				
Product properties:					
Physical stability	Okay	Okay	Okay	Okay	Okay
Pourability	Okay	Okay	Okay	Border line	Okay
Stain removal	ref	best	improved over reference		
Environmental acceptability	Okay	Okay	Okay	Okay	Border line

- 1) Ethylenediamine-N,N-disuccinic acid
- 2) Methylglycine diacetic acid
- 3) Beta-alanine diacetic acid
- 5 4) Glutamic acid N,N-diacetic acid
- 5) Polymer A11 of EP 346995

From the results it can be concluded that Trilon ES, Trilon GS and Nervanid GBS-5 give adequate product characteristics in base formulations 5 and 6. However, Trilon ES and Nervanid GBS-5 are preferred over Trilon GS

for pourability reasons. Trilon ES and GS are preferred for environmental reasons. S,S EDDS is however the most preferred sequestrant also with respect to stain removal.

Claims

1. Aqueous structured liquid detergent composition comprising surfactant material, electrolyte,

wherein the electrolyte comprises 0.1 to 5% by weight of aminocarboxylate sequestrant selected from glutamic acid N,N-diacetic acid, beta-alanine diacetic acid, methyl glycine diacetic acid, ethylenediamine-N,N-disuccinic acid and mixtures thereof; and

more than 5% by weight of builder material selected from citrate, tripolyphosphate, zeolite and mixtures thereof;

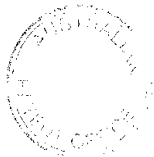
and water.

2. Composition according to claim 1, wherein the aminocarboxylate sequestrant is S,S ethylenediamine-N,N-disuccinic acid.

3. Composition according to claims 1-2, wherein the builder comprises zeolite.

4. Composition according to claims 1-3, wherein the electrolyte comprises aminocarboxylate and a salting-out resistant electrolyte.

5. Process of preparing a liquid detergent composition comprising surfactant material, electrolyte and water, wherein the composition has a structure and the electrolyte comprise aminocarboxylate sequestrant, according any of the preceding claims, by mixing the ingredients and keeping the pH above 6 and the temperature below 80EC.



AMENDED SHEET

6. Washing process for stained, coloured fabric in which a liquid detergent composition according to claims 1-4 is used.

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AMENDED SHEET