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(54) **PROCESS FOR TREATMENT OF TEXTILES.**

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Description

TECHNICAL FIELD

The invention relates to a process for treatment of textiles by use of a lipase.

BACKGROUND ART

Treatment of soiled or unsoiled textiles normally comprises washing with a detergent containing anionic and/or nonionic surfactant, followed by rinsing and drying. It belongs to the prior art that lipase can be incorporated into the detergent to improve the removal of fat soiling (EP 130,064, EP 214,761, US 4,810,414). Also, it belongs to the prior art that a non-dusting granulate containing a cellulase can be added to a main wash detergent composition in order to reduce the harshness of the laundry (US 4,435,307), that proteases are useful in detergent compositions in order to remove proteinaceous stains (US 3,723,250), and that amylases are useful in detergent compositions in order to remove starchy stains (US 3,627,688 and 4,548,727).

However, a given enzyme may be incompatible with a given detergent composition due to unsatisfactory activity and stability of the enzyme, and this imposes some limitations in the choice of enzyme and detergent formulation. It is the object of the invention to provide an alternative method of using a lipase in textile treatment, so as to obtain efficient lipolytic effects for a given lipase dosage while avoiding the above limitations.

STATEMENT OF THE INVENTION

We have found that, surprisingly, use of a lipase provides a better lipolytic effect when used in the rinse liquor than when used as a constituent of the main wash composition. The effect is particularly pronounced after repeated cycles of washing, rinsing and drying. Detergent is essentially absent during rinsing, and thus compatibility problems are avoided. The lipase may advantageously be added in the last rinse step together with a cationic surfactant.

DE 2 148 278 describes the use of a cellulase preparation containing lipase as one of the secondary activities and a cationic surfactant. However, this prior publication does only indicate the presence of lipase as a theoretical possibility and does not indicate the use of any specific lipases.

Accordingly, the invention provides a process for treatment of textiles by use of a lipase, which is used in a rinse liquor for a rinsing step after washing, characterized by the fact that the lipase is producible by means of *Humicola lanuginosa* or *Pseudomonase cepacia.*

In a preferred embodiment of the process according to the invention the lipase is used in the rinse liquor for the rinsing step after washing, together with a fabric softening agent and/or an antistatic agent. Hereby a satisfactory effect has been demonstrated.

In a preferred embodiment of the process according to the invention the fabric softening agent and/or the antistatic agent is a cationic surfactant. A satisfactory enzymatic effect is observed in this embodiment. The cationic surfactant may be a dialkyl dimethyl ammonium chloride or an alkyl imidazoline methosulfate.

In a preferred embodiment of the process according to the invention the enzyme is used in the rinse liquor for a second or later rinsing step, preferably in the last rinsing step. In this manner an even better enzymatic effect is observed.

In a preferred embodiment of the process according to the invention the concentration of lipase in the rinse liquor is in the range of 0.001 to 5, preferably 0.01 to 2 mg pure lipase protein per liter of rinse liquor. At a concentration below 0.001 mg pure lipase protein per liter of rinse liquor no significant lipase effect can be observed, and at a concentration above 5 mg pure lipase protein per liter of rinse liquor the lipase cost will be unreasonably high.

DETAILED DESCRIPTION OF THE INVENTION

The lipases which can be used in the invention are lipases producible by means of *Humicola lanuginosa* (US 4,810,414) and *Pseudomonas cepacia* (WO 89/04361).

The lipase activity unit, LU, is defined in AF 95/5, which is available on request from Novo Nordisk A/S, Novo allé, DK-2880 Bagsvaerd, Denmark, this publication being published before March 1, 1990.

Cationic surfactants

According to a preferred embodiment of the invention a cationic surfactant effective for fabric after-treat-

ment is used together with the lipase in the process according to the invention. Such cationic surfactants are known in the art. One example is quaternary ammonium salts such as dialkyl (C_{16}-C_{18}) dimethyl ammonium chloride, alkyl (C_{32}-C_{36}) trimethyl ammonium chloride or alkyl (C_8-C_{18}) dimethyl benzyl ammonium chloride (ABDAC); and alkyl imidazolinium methyl sulphate.

Textile treatment process

The invention uses a lipase in the rinsing stage of a conventional treatment process for soiled or unsoiled fabrics comprising washing, rinsing and drying. The rinsing stage generally comprises 2-5 subsequent steps of rinsing with water, usually 3 or 4 steps. In a preferred embodiment the lipase is added together with a cationic surfactant (e.g. 0.1-0.5 g/l). The lipase is preferably added in the second or a later rinsing step, and most preferably in the last rinsing step, where the detergent concentration is lowest (thus minimizing any incompatibility problems between detergent and lipase) and where it can conveniently be added together with the cationic surfactant, if such cationic surfactant is used in the process according to the invention. Conventional rinsing conditions can be used, e.g. 10-30°C water, 1-20 minutes at near-neutral pH (e.g. 5-9) depending on the use of cationic surfactant.

Rinsing agent

The rinsing agent used in the process according to the invention is stabilized against microbial infection, preferably by incorporation of a stabilizing agent known in the art. Examples are inorganic salts (such as NaCl), sugars (such as sucrose and glucose), polyols (such as glycerol, propylene glycol and sorbitol) and alcohols (such as ethanol and iso-propanol). These are generally effective in amounts above 10%, especially above 20%. Another example is organic acids (such as benzoic, sorbic, propionic, lactic and formic), which are generally effective in amounts of 0.01-2% at low pH (below 5). Other examples of stabilizing agents are antioxidants (such as sulphur dioxide), 1,2-benz-iso-thiazolin-3-on (BIT) and parabens. Some of these may also serve to improve enzyme stability.

Further additives may be incorporated to improve enzyme stability or physical stability of the rinsing agent. Examples are $CaCl_2$ (e.g. 0.1-0.5%) or 0.5-5% of lower alcohol (such as ethanol or iso-propanol, preferably used together with a larger amount of a polyol).

The rinsing agent ordinarily is preferably essentially devoid of anionic and nonionic surfactant (total amount of these below 5%, most preferably below 2%).

The rinsing agent may be in solid (e.g. as non-dusting granules) or liquid form (e.g. aqueous). It may be formulated by incorporating lipase into known softening/antistatic compositions based on cationic surfactants. The content of cationic surfactant is preferably 1-50%, especially 5-50% (% as active material).

EXAMPLES

The lipase used in the examples is from *Humicola lanuginosa* produced according to US 4,810,414 or from *Pseudomonas cepacia* produced according to WO 89/04361.

EXAMPLE 1

Effect of different lipases under the rinse process

Washing and rinsing tests are made as follows:

Fabric:	Polyester/cotton (50/50) or cotton. Swatch size: 9x9 cm.
Soiling:	Lard/Sudan red (1000/0.75 w/w) is heated to 70°C, and 50 µl is spotted onto each swatch by a pipette. After application of stain, the swatches are heated in an oven for 30 minutes at 75°C.
Washing:	Terg-o-tometer (100 rpm). Commercial European detergent powder, 8 g/l in 18°dH water. pH not adjusted (approx. 10). 30°C, 20 minutes. 7 swatches per beaker containing 1 liter detergent solution.
Rinsing:	4-step rinse in Terg-o-tometer. 1st-3rd rinse: 6 minutes in 1 l cold tap water. 4th rinse: 10 minutes in 1 l tap water (22°C).

Swatches squeezed by hand between each rinse.
0 or 100 LU/l of lipase added in 4th rinse.
0 or 0.20 g/l of Arquad 2HT-75 (dialkyl dimethyl ammonium chloride, 75% active matter, Akzo-Chemicals) added in 4th rinse.

Drying: Line-drying (16 hours) at room temperature.

Repeats: Washing, rinsing and drying of swatches repeated 3 times.

Evaluation: After the 3rd wash.
Reflectance is measured at 460 nm.
Content of fatty matter is determined by Soxhlet extraction with chloroform for 5 hours.

It appears from the following tables that addition of lipase during the last rinse improves the removal of fatty matter, in relation to both lipases, both with and without cationic.

Lipase	Arquad 2HT-75 (g/l)	Reflectance R	Reflectance ΔR	Fatty matter mg	Fatty matter Residual fat %
-		63.2	-	175	50.0
Humicola lanuginosa	0.00	86.8	23.6	65	18.6
Pseudomonas cepacia		70.7	7.5	118	33.7
-		73.3	-	138	39.4
Humicola lanuginosa	0.20	81.2	7.9	110	31.4
Pseudomonas cepacia		80.8	7.5	109	31.1

Effect of *Humicola lanuginosa* and of *Pseudomonas cepacia* lipase on lard/cotton swatches. The lipases were added initally under the 4th rinse step; no lipase was present during the washing process.

Lipase	Arquad 2HT-75 (g/l)	Reflectance R	Reflectance ΔR	Fatty matter mg	Fatty matter Residual fat %
-		57.4	-	188	53.7
Humicola lanuginosa	0.00	86.7	29.3	41	11.7
Pseudomonas cepacia		76.9	19.5	79	22.6
-		77.9	-	100	28.6
Humicola lanuginosa	0.20	84.6	6.6	69	19.7
Pseudomonas cepacia		83.1	5.2	67	19.1

Effect of *Humicola lanuginosa* and of *Pseudomonas cepacia* lipase on lard/polyester-cotton swatches. The lipases were added initially under the 4th rinse step; no lipase was present during the washing process.

EXAMPLE 2

Lipase performance when used in combination with different cationic surfactants

The performance evaluation was carried out as described in Example 1, the only difference being that other kinds of cationics were used in combination with the *Humicola lanuginosa* lipase and that the lipase dosage was varied between 0 and 3000 LU per litre.

The cationics used were:

1) Dodigen 1490 from Hoechst (dialkyl dimethyl ammonium chloride, 75% active matter)

2) Empigen FRC75/S from Albright & Wilson (alkyl imidazoline methosulfate, 75% active matter)

Cationic surfactant	Lipase dosage (LU/l)	Reflectance R	Reflectance ΔR	Fatty matter mg	Fatty matter Residual fat %
	0	67.6	-	174	49.7
	30	85.8	18.3	74	21.1
	100	86.9	19.3	66	18.9
	300	87.6	20.0	60	17.1
	1000	86.9	19.3	57	16.3
	3000	89.2	21.6	44	12.6
Dodigen 1490 (0.2 g/l)	0	77.5	-	168	48.0
	30	83.2	5.7	139	39.7
	100	83.2	5.7	134	38.3
	300	83.4	5.9	128	36.6
	1000	85.2	7.7	120	34.3
	3000	86.5	9.1	107	30.6
Empigen FRC75/S (0.2 g/l)	0	72.8	-	141	40.3
	30	81.5	8.7	114	32.6
	100	82.8	10.0	107	30.6
	300	82.8	10.0	97	27.7
	1000	82.6	9.8	95	27.1
	3000	82.3	9.5	92	26.3

EXAMPLE 3

Lipase performance - comparison with prior art

Performance evaluation was carried out as described in Example 1. In this example the use of *Humicola lanuginosa* lipase in the rinse liquor (this invention) was compared to the use of the same enzyme in the main wash liquor (prior art). Lipase dosage was 100 LU/l.

		Residual fat %
Prior art	Lipase used in main wash liquor	44.9
Invention	0.2 g/l Dodigen 1490	38.3
	0.2 g/l Empigen FRC 75/S	30.6
	without cationics	18.9

EXAMPLE 4

Lipase stability in rinse preparations (after treatment preparations)

Composition % w/w	1	2	3	4
Lipolase® 100 L	0.5	0.5	0.5	0.5
Dodigen 1490	8.0	8.0		
$CaCl_2$	0.1	0.1	0.1	
Propylene glycol		20.0	25.0	25.0
Deionized water	91.4	71.4	74.4	74.5
pH	6.0	6.0	7.0	7.0

The above mentioned lipase containing rinse preparations were stored at room temperature (20-22°C) for 240 days and the lipase activity was followed. The following results expressed as % relative activity were obtained:

	Days of storage					
Formulation no.	0	1	9	14	24	240
1	100	95	82	68	72	35
2	100	84	72	80	88	42
3	100	90	92	97	98	102
4	100	92	83	90	93	84

Claims

1. Process for treatment of textiles by use of a lipase, which is used in a rinse liquor for a rinsing step after washing, wherein the lipase is producible by means of *Humicola lanuginosa* or *Pseudomonas cepacia.*

2. Process according to Claim 1, characterized by the fact that the lipase is used in the rinse liquor for the rinsing step after washing, together with a fabric softening agent and/or an antistatic agent.

3. Process according to Claim 2, characterized by the fact that the fabric softening agent and/or the antistatic agent is a cationic surfactant.

4. Process according to Claims 1 - 3, wherein the lipase is used in the rinse liquor for a second or later rinsing step, preferably the last rinsing step.

5. Process according to Claims 1 - 4, wherein the concentration of lipase in the rinse liquor is in the range 0.001 to 5, preferably 0.01 to 2 mg pure enzyme protein per liter of rinse liquor.

Patentansprüche

1. Verfahren zum Behandeln von Textilien durch Verwendung einer Lipase, die in einer Spüllauge für einen Spülschritt nach dem Waschen eingesetzt wird, wobei die Lipase mittels Humicola lanuginosa oder Pseudomonas cepacia herstellbar ist.

2. Verfahren nach Anspruch 1, gekennzeichnet durch die Tatsache, daß die Lipase in der Spüllauge für den Spülschritt nach dem Waschen zusammen mit einem gewebeerweichenden Mittel und/oder einem antistatischen Mittel eingesetzt wird.

3. Verfahren nach Anspruch 2, gekennzeichnet durch die Tatsache, daß das gewebeerweichende Mittel und/oder das antistatische Mittel ein kationisches Tensid ist.

4. Verfahren nach den Ansprüchen 1-3, wobei die Lipase in der Spüllauge für einen zweiten oder späteren Spülschritt, vorzugsweise den letzten Spülschritt, eingesetzt wird.

5. Verfahren nach den Ansprüchen 1-4, wobei die Lipasekonzentration in der Spüllauge im Bereich von 0,001 bis 5, vorzugsweise 0,01 bis 2 mg reines Enzymprotein pro Liter Spüllauge liegt.

Revendications

1. Procédé de traitement des textiles au moyen d'une lipase qui est utilisée dans un liquide de rinçage pour une étape de rinçage après le lavage, dans lequel la lipase peut être produite au moyen de Humicola lanuginosa ou de Pseudomonas cepacia.

2. Procédé selon la revendication 1, caractérisé par le fait que la lipase est utilisée dans le liquide de rinçage pour l'étape de rinçage après le lavage, en même temps qu'un agent adoucissant textile et/ou qu'un agent antistatique.

3. Procédé selon la revendication 2, caractérisé par le fait que l'agent adoucissant textile et/ou l'agent antistatique est un tensioactif cationique.

4. Procédé selon les revendications 1 à 3, dans lequel la lipase est utilisée dans le liquide de rinçage pour une seconde étape de rinçage ou pour une étape de rinçage ultérieure, de préférence dans la dernière étape de rinçage.

5. Procédé selon les revendications 1 à 4, dans lequel la concentration de la lipase dans le liquide de rinçage est située dans la plage de 0,001 à 5, de préférence de 0,01 à 2 mg de protéine enzymatique pure par litre de liquide de rinçage.