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(54) Title: ENZYMATIC TABLETS FOR WEARING OUT TEXTILE PRODUCTS COMPRISING CELLULOSE FIBERS AND METHOD FOR THEIR USE

(57) Abstract: A method for using a kit consisting of two compounds preset for textile product aging treatment. These compounds operate in water, either simultaneously or in succession, and contain all chemical substances useful for the process.

WO 2020/255177 A1

DESCRIPTION

Title

ENZYMATIC TABLETS FOR WEARING OUT TEXTILE PRODUCTS COMPRISING CELLULOSE FIBERS AND METHOD FOR THEIR USE

* * * * * *

Technical field

The present invention belongs to the sector of products and methods for treating textile products by way of enzymes, both in the industrial and domestic sectors. Such treatments are especially useful for customizing articles of clothing. In particular, this invention concerns a kit comprising enzymatic compounds and its respective method of use, in the context of ready-made article washing, in order to impart a well-worn appearance to articles of clothing at least partially made from cellulose fibers; such treatment is generally referred to as aging.

Amongst other applications, this invention is particularly suitable for washings jeans, i.e. articles made from a Denim fabric, where an aging effect is obtained similar to that commonly referred to as stone-wash.

Present status of the art

Fabric aging is a widespread and by now unquestionable practice because in some clothes a well-worn appearance imparts a greater appeal and aesthetic value thereto.

Even though enzymes are also used in domestic washing, enzymatic washings are usually reserved for professional operators, who perform controlled treatments at an industrial level in order to obtain fashion effects. Such treatments usually require precise dosages and an addition of different chemical components, often in sequential steps and successive baths.

Textile article aging processes initially used a bath with abrasive bodies mixed therein, for example pumice stone

bodies, as described for instance in US4575887(A). Subsequently the action of abrasive bodies was replaced, or supplemented, by a chemical one. Patent US5460966(A) illustrates an aging method wherein abrasive bodies comprise a soluble chemical composition; patent US2017159235(A1) discloses a treatment that uses cellulases together with a mechanical abrasion action.

Reference will be usually made in the present patent text to a generic nomenclature of enzyme families, as if each of them represented one component. However, by this term we mean enzymatic compounds present on the market, which contain even several types of enzymes, all of them falling in the same family and, very often, further ingredients which can be taken advantage of for enzyme formation and preservation. Different strains of enzymes might present different effectivenesses; in order to take account of all of these variabilities resulting from the use of commercial products, a range of values for the dry residual of enzymes is specified for the compositions mentioned in the following text. Just as an example, some commercial products contain 10% to 25% of cellulases, 5% to 16% of amylases, and 0.5% to 4% of peroxidases respectively.

The methods according to the present status of the art usually comprise a first desizing step taking place in a first bath, which is emptied, followed by a second rinsing bath. Then an enzyme-based treatment follows, also referred to as enzyming, in a third bath. A treatment aiming at imparting a stone-washed appearance to textile products is disclosed in US5213581(A).

Enzyming is followed by a number of clean rinsings or a cleaning-up bath using oxidizing agents. In some methods known in specialized circles, the desizing and enzyming steps

are performed in one bath, but the subsequent cleaning-up steps require at least one further bath in any case.

In the reference sector, a need is particular felt for simplifying the operations necessary to obtain an aged and cleaned up product.

Objects and summary of the invention

A first object of the present invention is to provide a simple and easy-to-use solution for treating textile products comprising cellulose fibers, whose application does not require specific knowledges or particular recipes in order for it to be applicable both by a professional and/or industrial user and by a domestic user.

A further object is to provide an effective formulation while operating inside a relatively simple method of use.

Another non-negligible object is to reduce the number of baths and the power consumption necessary for a treatment as compared to those required in the known methods.

These objects and others, which will be apparent to those skilled in the art upon reading the following text, are achieved by a method based on the use of a kit consisting of two compounds comprising all of the chemical elements necessary for treating a textile product, but water. These compounds can be aggregated in the form of tablets or be contained in water-soluble bags or capsules or the like.

Note that, in the present patent text, water does not only mean the specific chemical compound, but a liquid in a broad sense, including water charged with normal components as used for purification in the frame of domestic or industrial processes.

The kit according to the present invention comprises a plurality of enzymatic components suitable for operating inside one bath, inside a machine suitable for washing clothes, i.e. a machine that determines a mechanical action

that supplements the chemical action exerted by compounds. In the present text, this machine, generally a rotating one, is sometimes referred to as a washing machine for the sake of the synthesis, whereas the term drum identifies its working volume.

In particular, a first compound comprises cellulases and, preferably, amylases, whereas a second compound comprises laccases and/or peroxidases and/or at least one oxidizing agent. Peroxidases operates in the presence of an oxidizing agent.

The invention makes thus it possible to perform two processes that are usually made separately, in one context, namely: the fabric aging one, possibly including desizing, and the cleaning-up one.

In some circumstances, this might be fostered by sequential and controlled activations of the compounds, hence of the different ingredients that the compounds are made of.

According to the method of use of this kit, said two compounds are put inside a drum of a washing machine together with the textile products to be treated, dipped in a water bath or, at the most, soaked in water.

Each of the components of the compounds basically performs a specific action as specified below. Some components, such as the enzymatic compounds, the oxidizing agents, the surfactants, and the bath-pH control agents, perform an active action in the treatment, whereas other are inactive and are functional to the formation of the compound, such as sorbitol and polyethylene glycol, mixing agents or dyes, if any, and/or to the release of active components, such as disintegrating agents, in the case of tablets, or to a water-soluble coating, such as bags.

Amylases are a family of enzymes which perform the function of hydrolyzing and eliminating desizings and are a kind of

starches sometimes present in fabrics. However, such sizes are not always present and consequently amylases might be removed from a compound, both for economic reasons and whenever unnecessary, such as in the case of a kit for domestic use, hence dedicated to fabrics that have already been widely washed up and treated.

Cellulases are a family of enzymatic compounds that perform the function of hydrolyzing and degrading cellulose in a fabric, in order to impart a well-worn and aged appearance to an article of clothing, which is the primary object of the treatment.

Peroxidases are a family of enzymatic compounds that primarily perform a synergetic action to that of sodium percarbonate, or other oxidizing agents, in cleaning-up actions. In particular, wherever there is an indigo-based dye, this one would be partially degraded by peroxidases, thus making it possible to clear indigo out of the fabric as well as to remove the dye possibly re-deposited on the fabric during previous treatment(s).

Sodium percarbonate is an oxidizing agent and operates as a cleaning agent at the end of a treatment, in order to remove residues of dye having contaminated the fabric. Sodium percarbonate is generally not used in the case that peroxidases are replaced by laccases; or it might be replaced by another oxidizing or reducing element.

Laccases are a family of enzymatic compounds that perform an action alternative to that of peroxidases and oxidizing agents.

In general, the compounds also comprise components aiming at maintaining ideal conditions in the bath, such as, for instance surfactants, bath pH control agents, and oxidizing agents, etc.

Surfactants are used in order to keep the dye dispersed in

the bath in suspension and to reduce fabric contamination; usually non-ionic, ethoxylate alcohol-based surfactants are used.

Adipic acid and monosodium phosphate (dihydrogen phosphate) perform a bath pH control function. Other examples of substances usable for this function include citric acid and ascorbic acid.

Cellulose is used as a disintegrating or explosive agent, in that it determines water absorption in a compound, which consequently swells out and opens. Cellulose might even be replaced by another element capable of performing a similar action.

In some embodiments, the compounds used for implementing the present invention are enclosed in water-soluble containers which allow for a release of the chemical substances contained therein.

Polyethylene glycol, or PEG, is a formulation agent, generally used for implementation of tablets, but it does not appear to be essential nor does it present special functional aspects in the course of the chemical treatment of a textile product. Therefore, its use is optional. The same applies to sorbitol.

Sodium sulphate is used as a mixing or filling agent, i.e. it supplements the formulation but it does not present any special functional aspects in the course of the chemical treatment of a textile product.

The method of use of the kit comprises at least the following steps:

a) putting at least one textile product, comprising cellulose fibers, and a bath consisting of water inside a machine suitable for exerting a mechanical action on textile products;

c) putting at least the first compound of the kit in the

machine;

d) activating the mechanical action of the machine on the textile products;

e) keeping said bath with at least one textile product and at least the first compound of the kit at a temperature in the range from 25°C to 60°C for at least 15 minutes.

The order of execution of the first steps a), c, and d) is little important and might vary.

The first compound comprises surfactants and at least one enzymatic compound, i.e. at least cellulases or at least cellulases and amylases. Preferably the first compound also comprises a mixing agent and, in one embodiment, at least one pH control agent.

In an initial step, the amylases, if any, desize the fabric in co-operation with the action exerted by surfactants, which help in providing cleanliness and in preventing the dye dispersed in the bath from depositing again on the fabric, and with that exerted by the bath pH control agent, if any. In the meantime, an aging action is exerted by cellulases, which take action upon finding the fabric ready and the bath in optimum conditions in terms of surfactants and pH, if any.

The aging action, i.e. the action whereby cotton and its dye is degraded thanks to a fabric abrasion, is usually supplemented by the mechanical action exerted by the washing machine.

The second compound comprises laccases or peroxidases with at least one oxidizing agent or at least one oxidizing agent. Should peroxidases be used, the second compound preferably comprises at least one bath pH control agent.

The second compound, besides performing a cleaning-up action aiming at providing a qualitatively effective result, also reduces the number of subsequent rinsings that are necessary,

which might thus be limited down to one or two.

The second compound basically acts on the dye or color of the fabric, in particular on indigo.

In the following detailed description, a number of variants of the method are described, which aim at improving its effectiveness or at further reducing water consumption.

The use of the kit according to the teachings of the present patent text makes it possible to implement several steps while using one bath only.

If amylases are used, the treatment comprises a first desizing step, which will then be helped by cellulases to impart an aged appearance. The textile product treatment ends with an oxidizing or enzymatic process which performs a cleaning-up action, useful in increasing the wearing effect, also thanks to the removal of the dye previously re-deposited on the fabric. In one preferred embodiment, the oxidizing process makes use of peroxidases.

The compounds usually comprise further ingredients, including at least one disintegrating agent, should the compound be realized in the form of a tablet.

Detailed description of embodiments of the invention

The kit according to the invention consists of a first compound and a second compound. The first compound comprises at least one enzymatic compound and surfactants, in addition to preferably at least one mixing agent and at least one disintegrating agent, in the case of tablets. The enzymatic compounds comprise cellulases and, in one embodiment, amylases.

In one preferred embodiment, the first compound comprises the following components, in the by-mass proportions (percentage ratio of a component mass to the overall compound mass, %m/m or %w/w) indicated aside:

cellulases (9012-54-8): 0.2% - 10%;

amylases (9000-90-2): 0.05% - 2%;

surfactants : 5% - 20%.

The figures indicated above within brackets, as well as those specified below, identify the chemical substance in a unique manner via their CAS number, which is an identifier assigned by the Chemical Abstracts Service (CAS) division of the American Chemical Society.

In one particularly preferred embodiment, surfactants are used in an amount ranging from 10% to 20% of the first compound (% m/m).

If no amylases are used, the proportions indicated above remain unchanged and a greater amount of a mixing agent is generally added.

In a further embodiment, the first compound also comprises a bath pH control agent, preferably in an amount ranging from 8% to 20% (% m/m), and one preferred embodiment uses adipic acid (124-04-9).

In the case of an aggregation in the form of a tablet, good results have been achieved with a first layer comprising (%m/m, as referred to the overall mass of the tablet):

amylases: 0.05% - 2%;

surfactants: 5% - 20%;

and a second layer comprising (%m/m, as related to the overall mass of the tablet):

cellulases: 0.2% - 10%.

In a first version, the second compound comprises laccases and, preferably, at least one bath pH control agent.

In a second version, the second compound comprises peroxidases, as an enzymatic compound, sodium percarbonate, as an oxidizing agent and, preferably, a pH control agent.

In one preferred embodiment of the second version, whenever the second compound is aggregated in the form of tablets,

peroxidases are separate from sodium percarbonate, so as to prevent the component parts from interacting during storage. Therefore, a first layer with sodium percarbonate and a second layer with peroxidases are provided. If necessary, such separation might also be adopted for water-soluble bags; in this event, the term layer identifies a different portion. In one preferred embodiment, the first layer is present in an amount ranging from 10% to 30% by mass of the complete compound (%m/m) and a second layer from 70% to 90% of the complete compound (%m/m).

In the embodiment here described, the first layer comprises 40% to 95% of an oxidizing agent, preferably sodium percarbonate (15630-89-4) (ratio of the component mass to the overall layer mass, %m/m or %w/w).

Preferably the second layer comprises, in a by-mass proportion (ratio of the component mass to the overall layer mass, %m/m or %w/w) as indicated aside:

peroxidases (9003-99-0): 0.05% - 2%;

at least one pH control agent: 60% - 90%.

In one particularly preferred version, some of the above-indicated components are present in the following proportions:

peroxidases: 0.2% - 0.6%;

adipic acid, used as a pH control agent: 60% - 80%.

The formulation of the kit takes account of the need for peroxidases to operate in a more acid environment, tendentially at a pH in the range from 3 to 6; this is the reason why at least one bath pH control agent is used, preferably adipic acid or monosodium phosphate (10049-21-5).

A further advantage given by an article of clothing being cleaned up by the second compound here described consists in that it increases the light and shade contrast in the

abrasion points, by uniformly lighting all the article of clothing and emphasizing the white points. This effect makes it possible, if necessary, to eliminate the use of pumice stone from washing, which results in improved operating levels, being it not necessary any longer to handle such material, which entails not a few handling, logistic, treatment, and disposal problems. A lower environmental impact also follows, both because no pumice stone is used any longer, and because of its consequent impact on sewage depuration processes.

In a third version, the second compound comprises an oxidizing agent, preferably sodium percarbonate.

The compounds and their layers might comprise further elements in addition to those listed above, in particular possible additives for dying the tablets or other ingredients useful for the operation, formation, or preservation thereof. Also remember that some compounds used might already commercially include other ingredients useful for the same purposes.

Every compound is aggregated to form tablets or is contained in water-soluble bags or the like.

It is worth pointing out that tablets also comprise a plurality of ingredients of a known type, such as for instance compactant and disintegrating agents; good results are achieved by using sorbitol (50-70-4) or polyethylene glycol (25322-68-3) and cellulose (9004-34-6) respectively.

In an embodiment of the tablet of the first compound which fosters the succession of the action exerted by cellulases downstream of that exerted by amylases, the first compound might comprise a first layer comprising amylases and a second layer comprising cellulases, the second layer featuring a smaller proportion of disintegrating agents.

For both tablets and water-soluble bags, the compounds might

comprise at least one mixing agent, preferably sodium sulphate (7757-82-6), up to completing the composition.

In a first embodiment, the method comprises the following steps:

a) putting at least one textile product, comprising cellulose fibers, and one bath consisting of water inside a drum of a machine suitable for exerting a mechanical action on textile products;

c1) putting the first compound of the kit in the drum of the washing machine;

d) activating the mechanical action of said machine on the textile products;

e1) keeping said bath with at least one textile product and the first compound of the kit at a temperature in the range from 25 °C to 55 °C for at least 15 minutes;

f) putting the second compound of the kit in the drum of the washing machine;

g) keeping said bath with at least one textile product and the second compound of the kit at a temperature in the range from 25 °C to 65 °C for at least 5 minutes.

Usually, step g) is followed by step:

h) emptying said bath from the drum.

In a second embodiment of the method, step c1) and step f) are combined together before step d), but step g) is omitted, consequently the method comprises, after step a):

c2) putting the first and second compounds of the kit in the drum of the washing machine;

d) activating the mechanical action of said washing machine on the textile products;

e2) keeping said bath with at least one textile product and the first and second compounds of the kit at a temperature in the range from 25 °C to 60 °C for at least 15 minutes.

According to one preferred version of the invention, a bath

ratio, i.e. a ratio of textile products to water, is provided in a range from 1:1 to 1:20, very preferably in a range from 1:3 to 1:10.

In a further implementation of the method, the bath is emptied into a container after step a), then re-used in the first rinsing following step g), should the first embodiment be applied, or step e), should the second embodiment be applied. In the latter implementation of the method, the treatment takes place on articles of clothing soaked in water and the above specified temperatures do not refer to the bath, but rather to the environment of the drum. Therefore, the following steps will take place:

b) emptying the bath into a container;

i) performing a first rinsing with said bath emptied into the container.

For all embodiments, at the end of the method it is possible to provide further rinsings or further actions on the textile product so as to provide a finished product.

Preferably the duration of said step e1) is in a range from 20 to 80 minutes. However, when the second compound is introduced together with the first compound in step c), the duration of said step e2) preferably ranges from 20 to 80 minutes; in both cases, the duration affects the degree of abrasion obtained.

Preferably the duration of said step g) is in a range from 5 to 20 minutes.

If necessary, abradant bodies, such as pumice stones, might be added inside the drum during the application of the above-described method.

CLAIMS

1. A method for using a kit for aging textile products comprising cellulose fibers, said kit consisting of a first compound and a second compound, said first compound comprising cellulase and surfactants, said second compound comprising a first and a second layers, the first layer comprising said oxidizing agent and the second layer comprising peroxidase, the first layer amounts to from 10% to 30% by mass of the whole second compound (% m/m) and the second layer amounts to from 70% to 90% of the whole second compound (% m/m) wherein said first layer comprises from 40% and 95% (% m/m) of said at least one oxidizing agent and **in that** said second layer comprises from 0,05% to 2% of peroxidase (% m/m) and from 60% and 90% of at least one pH regulator agent (% m/m), said method comprising the following steps:

a) putting at least one textile product, comprising cellulose fibers, and one bath consisting of water within to a machine suitable for applying a mechanical action to the textile products;

c) putting at least the first compound of the kit in the machine;

d) activating the mechanical action of the machine on the textile products;

e) keeping said bath with at least one textile product and at least the first compound of the kit to a temperature ranging from 25°C to 60°C for at least 15 minutes;

wherein said second compound of the kit is introduced in the machine together with said first compound in step c), or the following steps follow step e):

f) putting the second compound of the kit in the machine;

g) keeping said bath with at least one textile product and the second compound of the kit to a temperature in the range

from 25°C to 65°C for at least 5 minutes.

2. The method according to claim 1 **characterized in that** said second compound comprises peroxidase, at least one oxidizing agent, and at least one bath pH regulator agent.

3. The method according to any of the previous claims **characterized in that** said first compound comprises the following components in the specified by-mass percentages as referred to the unit mass of said first finished compound (% m/m):

cellulase	:	0,2% - 10%;
surfactants	:	5% - 20%.

4. The method according to any of the previous claims **characterized in that** said first compound also comprises from 0,05% to 2% of amylase (% m/m).

5. A kit for aging textile products comprising cellulose fibers, said kit consisting of a first compound and a second compound, said first compound comprising the following components in the specified by-mass percentages as referred to the unit mass of said first finished compound (% m/m):

cellulase	:	0,2% - 10%;
surfactants	:	5% - 20%;

said second compound comprising a first and a second layers or portions, the first layer comprising from 40% to 95% of sodium percarbonate (% m/m of said first finished layer) and the second layer comprising the following components in the specified by-mass percentages as referred to the unit mass of said second finished layer (% m/m):

peroxidase	:	0,05% - 2%;
at least one pH regulator agent	:	60% - 90%.

6. The kit according to claim 5 **characterized in that** said first compound and said second compound are aggregated in the form of tablets.

7. The kit according to claim 5 **characterized in that** said

first compound and second compound are contained within water-soluble bags.

8. A kit for aging textile products comprising cellulose fibers, said kit consisting of a first compound and a second compound, said first compound comprising the following components in the specified by-mass percentages as referred to the unit mass of said first finished compound (% m/m):

cellulase	:	0,2% - 10%;
surfactants	:	5% - 20%;

said second compound comprising lactase and wherein said first compound and second compound are aggregated in the form of tablets or contained within water-soluble bags.

9. A kit for aging textile products comprising cellulose fibers, said kit consisting of a first compound and a second compound, said first compound comprising the following components in the specified by-mass percentages as referred to unit mass of said first finished compound (% m/m):

cellulase	:	0,2% - 10%;
surfactants	:	5% - 20%;

said second compound comprising sodium percarbonate and wherein said first compound and second compound are aggregated in the form of tablets or are contained within water-soluble bags.

INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER
INV. D06M16/00 D06P5/15
ADD. D06M11/50 D06M101/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
D06M D06P

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	CN 109 652 988 A (WUHAN SUNHY BIOLOGICAL CO LTD) 19 April 2019 (2019-04-19)	8,9
A	paragraph [0025] paragraph [0027] paragraph [0125] examples 1-4	1-7

Y	US 2017/159235 A1 (MCDEVITT JASON P [US] ET AL) 8 June 2017 (2017-06-08) cited in the application	8,9
A	paragraph [0014] paragraph [0045] paragraphs [0048] - [0049] claims 8, 13 and 15	1-7

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
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4

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INTERNATIONAL SEARCH REPORT

International application No
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 02/16540 A1 (CLARIANT FINANCE BVI LTD; CLARIANT INT LTD [CH]; SIGMUND HARALD [DE]) 28 February 2002 (2002-02-28) page 7, lines 14-17 claim 4 -----	1-9

4

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/IT2020/050148

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US 2017159235 A1	08-06-2017	NONE	
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