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(54) **CLEANING IN THE FOOD INDUSTRY**

REINIGUNGSVERFAHREN FÜR DIE NAHRUNGSMITTELINDUSTRIE

PROCEDE DE NETTOYAGE DESTINE A L'INDUSTRIE ALIMENTAIRE

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**DE-A- 19 630 615**

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## Description

**[0001]** Packing machines with many details made of aluminium and/or brass are common. When such machines are used for food products of different kinds their cleaning and disinfection is a very complicated problem, which, so far, has no satisfying solution. The same is true for very large parts of other food handling equipment, especially when the operators come in direct skin contact with surfaces where food is handled.

**[0002]** Construction material, of the aluminium and brass type, excludes strong alkaline cleaning agents. Usually additives, containing silicates, are used to increase the resistance of aluminium against alkali. Silicates may cause precipitates that may endanger the mechanical function of the machine, and also create growth beds for micro organisms. Such growth points are usually very difficult to get to. Chlorine and other usual disinfection agents give unacceptable discharges and may influence the food negatively. Strong acid, which is used for instance in the so-called "alternating dish washing system", is excluded due to corrosion risks, which does not just damage the equipment, but also means an environmental danger as heavy metals are set free.

**[0003]** Strong alkali and strong acids must be eliminated completely by careful rinsing, as any remains may influence the food negatively. In unlucky cases the rinse operation may be a source of new contamination with micro organisms.

**[0004]** Even surfaces that can stand strong alkali and strong acids may with advantage be treated with weaker agents, as skin contact with surfaces treated with strong agents may give burning pain and even cause worse damage. This can happen with for instance benches, cutting boards etc.

**[0005]** According to EP 0 751 211 A1 one uses a two component system for cleaning and disinfection. The first component is an alkaline composition. The second one is an acid composition. In the alkaline or the acid composition an active per-oxygen species is present. This expression is very vague in its character. It could comprise many different chemical compounds. However, the description, (column 3, line 5 to 9) confirms that a reaction product of a water containing per-oxygen solution and an inorganic carboxylic acid with 1 to 6 carbon atoms, for instance acetic acid, is meant. This shows that the wording "active per-oxygen species" should be interpreted as what is usually called activated hydrogen peroxide, in contrast to non-activated hydrogen peroxide.

**[0006]** The main component of the alkaline composition is an alkali metal hydroxide. This gives a pH far above what is acceptable for use on aluminium. The main component in the acid composition is phosphoric acid. The concentration is high. This gives a pH level far below what is acceptable for aluminium and brass. In passing, it is said that the phosphoric acid may be replaced by another acid compatible with the hydrogen peroxide compound. No examples at all are given of such an acid. The wording seems to imply that another strong, inorganic acid is intended. Nothing in this publication can be interpreted to give guidance at the recipe work for compositions that could be used for the cleaning of mechanical constructions with aluminium and/or brass components.

**[0007]** PCT/EP96/05783 concerns an alkaline composition without disinfection agent and an acid composition with a disinfection agent chosen among anionic surface active agents, fatty acids, alkyl phosphonic acids and ether-carboxylic acids or their mixtures. Special emphasis is given to the fact that the compositions should be free from halogens, halogen compound, alkyl amines, quaternary ammonium compounds, quaternary phosphonium compounds, per acids and aldehydes.

**[0008]** As often happens with patent applications from large enterprises in the chemico-technical branch, the description has an almost standardised list of all imaginable components in the alkaline and the acid compositions. The listed substances have very different qualities and hardly anything in common, beside the fact that they react alkaline respectively acidic in water solution.

**[0009]** The examples refer to compositions, in which the main components are sodium hydroxide respectively phosphoric acid. The compositions of the examples are completely unfit for the purpose of this application. No guidance for adaptation in this direction can be found.

**[0010]** Aluminium is a base metal and is easily corroded by acids and alkali. Nevertheless, it is a highly favoured construction material, due to its excellent qualities regarding shaping etc. and the fact that it may be made passive by the formation of a tight adhering layer of aluminium oxide. This layer is automatically formed in environments rich in oxygen. It may be further reinforced by for instance anodizing. The stability of the protection layer depends on the reactivity of the aluminium oxide in the surroundings, where aluminium is used. If aluminium oxide can react with the surrounding media under formation of soluble compounds, the protection layer is destroyed. If the new compounds that may be formed are even less soluble than the aluminium oxide and have equal or better qualities regarding the formation of a tight layer with good adherence the protection layer is reinforced.

**[0011]** The passivity range of pure aluminium is comparative small and lays in principle between pH 4 and 8.5 at the redox potentials one normally expects in water solutions. Aluminium for construction details is usually alloyed. One criterion for choosing the alloy metals is its tendency to form a stronger protective layer. For practical purposes the passivity range has wider limits than mentioned and usually comprises the pH range 3 to 10.

**[0012]** Brass is a collective name for many alloys with copper and zinc as main components. Minor additions of most of the other frequently used alloy substances are common. When pure, copper and zinc are easily corroded. However,

the alloys show better resistance and one may, as a thumb-rule, assume that if the chemical environment is such that it does not corrode aluminium, the same applies to brass details that may be present.

5 [0013] The publications mentioned contain several examples of cleaning agents, which are commonly used for milk handling. As can be seen in the description of the corrosion resistance of aluminium and brass those agents are unfit for use for cleaning of construction material of those metals and alloys. At the alkaline side the pH becomes too high if the agents are used at normal use concentrations. If one instead reduces the concentration to an acceptable pH level the lack of buffer effect causes the alkalinity to sink so that the cleaning power disappears. A corresponding effect will occur on the acid side. Thus, known compositions, without chlorine or other common disinfection agents, cannot be used to solve the problem of getting both acceptable cleaning effect and acceptable disinfection, without a de-  
10 vating corrosion effect.

[0014] Instead one has tried to solve the problem by using alkaline pH-adapted cleaning compositions combined with chlorine disinfection. In spite of large efforts the results have usually been unsatisfying from the bacteriologic point of view.

15 [0015] The inventor has studied the cleaning problem with a knowledge of cleaning processes and metal corrosion as starting point. He has found that the cleaning can be optimised by using a low to moderately alkaline cleaning agent. The agent must have high buffering capacity and not contain silicates or other components that may form precipitates. The agent should have a pH in use solution in the range 8.5 to 11, preferably 9 to 10.5. The solution pH should not sink more than 1 pH unit during the cleaning operation.

20 [0016] The disinfection problem has shown to be more complicated and has required very thorough studies and trials. The thoughts behind the so-called "alternate washing system" cannot be applied directly. Weakly acid cleaning agents are not able to acidify or remove fat and proteins and by that eliminate the growth points for bacteria soil that remains are.

25 [0017] He has now surprisingly found that a not activated hydrogen peroxide solution acidified by an acid with high buffering capacity in the pH range 2.5 to 6 can be formulated as a rinsing agent. In combination with the mentioned alkaline cleaning agent this new rinsing agent gives results, which are acceptable from every point of view. This new rinsing agent does not corrode aluminium and brass. In fact, hydrogen peroxide improves the passivating oxide layer that protects aluminium against corrosion.

30 [0018] Non-activated acidified hydrogen peroxide is a slow disinfection agent compared to, for instance, chlorine and activated hydrogen peroxide. Therefore the agent usually requires modification of earlier rinsing routines, which have not included disinfection, which has been done at the cleaning stage already, or comprised disinfection agents with much faster effect. If the process includes automatic operations, some reprogramming or other modifications may be necessary to ensure that the part of the rinsing done by acidified hydrogen peroxide is prolonged. Even a small prolongation (2 to 3 minutes) increases the effect appreciably, but the effect will increase still more if the prolongation is made longer for instance 10 to 20 minutes. If reprogramming is difficult to accomplish, similar effects can be obtained  
35 by letting the rinsing agent remain at the surfaces and not, immediately after the rinsing, be removed by a rinsing with clean water. Also other measures that prolong the agent's action time, such as adding the hydrogen peroxide agent early, i.e. during the filling up of rinsing water instead of after completed filling up, have shown positive effects.

40 [0019] It is important that pH during the rinsing phase does not rise above 7, as the disinfection effect declines quickly with increasing pH. Preferably the pH should be below 6 and still more preferred below 5. The buffering capacity should be such that pH, during the whole rinsing phase with the acidified hydrogen peroxide containing solution, does not rise more than one pH unit, maximum, and under no circumstances above 7. Alkaline substances from the cleaning agent probably remain in clefts, etc. As the alkaline cleaning agent is buffered, the rinsing agent must have high buffering capacity.

45 [0020] The rinsing agent contains environmentally acceptable components only and does not leave behind remains that may influence food negatively from the taste or health points of view. This implies the very large advantage that no after-rinse is needed.

[0021] The small quantity of acid, which may remain after that the rinse solution has been drained off, cannot influence the taste of the food. The hydrogen peroxide decomposes into oxygen and water and leaves no remains. If skin contact occurs no burning pain is experienced even if the skin is already irritated.

50 [0022] However, from a principal point of view all additions to the final rinse water are unwanted. Usually a final rinse with clean water may be done just before the equipment is put in operation after cleaning and standstill. If so it is recommended to let the rinse with acidified hydrogen peroxide be the last operation before a standstill. This will give the advantage of a remaining growth inhibition for micro organisms during time of rest, without sacrificing the principle of a final rinse with clean water.

55 [0023] Suitable acids for the acidification can be found among weak inorganic and weak organic acids. Strong acids are not suitable, as they, at the dilution to needed level for maintaining pH in the wanted range, lack acceptable buffer capacity. Further the acid should have acceptable quality from the food point of view. A very suitable acid for the purpose, especially in dairies, but also otherwise, is lactic acid.

[0024] This invention concerns a method for cleaning and disinfection of equipment containing construction details of aluminium and/or brass. The process is begun by a washing with an alkaline washing solution with pH in the range 8.5 to 11, preferably 9 to 10.5 and ended with a rinsing with an acid hydrogen peroxide solution with pH in the range 2.5 to 6, preferably 3 to 5.

5 [0025] The acidified hydrogen peroxide solution of the invention is used with an alkaline washing agent. To avoid too much acid consumption from the contact with the alkaline cleaning agent an intermediate rinsing with water may be suitable.

10 [0026] When aluminium is used as construction material, high pH must be avoided to prevent corrosion. Different silicate compounds are often used to protect aluminium against corrosion by alkaline solutions. However, silicates have a nasty tendency to precipitate at low pH and are therefore unfit for cleaning, where risk for contact between the cleaning solution and acid exists. This may be the case in narrow clefts, where an intermediate water rinse has not eliminated the silicates completely.

15 [0027] To make a low-alkaline cleaning agent efficient a complexing agent must be present. Many usual complexing agents as NTA, EDTA, phosphonates and similar reinforce the corrosive effect of alkali on aluminium and reduce the tolerance for pH increases.

20 [0028] Therefore, tripoly- or pyrophosphates are used as complexing agents instead. Tripoly- and pyrophosphate contain, for reasons of equilibrium and production technique, small amounts of orthophosphate that react with aluminium to form water insoluble aluminium phosphate. In preferred compositions of the cleaning agent the corrosion protection is reinforced by further addition of orthophosphate. The reaction between orthophosphate and aluminium makes the metal passive and reinforces the layer of aluminium oxide that serves as corrosion barrier and increases in this way the tolerance for high pH. Potassium salts are preferred due to their better solubility.

[0029] A large advantage of a phosphate based washing agent in combination with an acid rinsing agent is that no water insoluble precipitates can be formed from the contact between the washing agent and the rinsing agent.

25 [0030] Suitable surface active agents to be combined with potassium triphosphate and/or potassium pyrophosphate are the so-called sugar tensides i.e. sugar surface active agents. Their effect may, with advantage, be reinforced with the aid of an easily biologically decomposed ampholyte surface active agent such as for instance AMPHOLAC® YJH 40 or LAKELAND® 70, which are so-called betaines, i.e. ampholyte surface active agents where the anionic group is derived from a carboxylic acid. These surface active agents are considered unobjectionable from the environmental point of view. Agents with this composition become very low foaming even in presence of the fatty acid soaps formed by saponification of fats in the soil. This is a very important point for older machines, which are cleaned by intensive pump circulation of the wash solution.

30 [0031] For newer machines cleaning by foam may often be a better method. The composition may easily be changed in that direction by modification of the mixture of surface active agents such as, for instance, by adding lauryl ethersulphate and/or replacing the low foaming sugar surface active agent with a high foaming one. The ampholyte surface active agent may also be, partly or totally, replaced by amine oxide and/or alkyl fatty acid diethanolamide.

35 [0032] The cleaning effect is improved if the dosing of the washing agent is divided into three parts, with one part for the pre-wash, one part for the main wash and a third part for the first rinsing.

40 [0033] The older cleaning procedure of a pre-wash with clean water without surface active agents and complexing agents removes the water soluble soil. This gives the opportunity for fat and other less water soluble substances to spread out on surfaces in the machine. The increased contact interface between fat and the surfaces makes the fatty soil more difficult to remove. Hardness in the water contributes to making the binding to the surfaces even harder.

45 [0034] In presence of surface active agents, alkali and complexing agents the capacity of the fat to bind to the surfaces is reduced. Surface active agents and alkali contribute to keeping the fat emulsified. Hardness ions are eliminated and cannot contribute to soil binding. Pre-wash with water containing washing agents decreases substantially the soil quantity that must be removed at the main wash.

[0035] As the soil loading becomes lower, no disadvantage will be caused by the fact that the main wash will be done with lower concentration of the washing agent.

50 [0036] Shock dilution of a wash solution containing soil may break emulsions and dispersions to let the soil fall back on the surfaces. As the soil earlier has been emulsified respectively dispersed, the precipitated soil will be extremely fine distributed. Such soil is very difficult to remove. A small addition of surface active agents and complexing agents reduces the risk for breaking emulsions and dispersions and eliminates the danger of re-soiling.

[0037] A suitable distribution of the total amount of the washing agent may be 10 to 30% in the pre-wash, 40 to 80% in the main wash and 10 to 30% in the first rinse water.

[0038] Concentrate for dilution to use concentration is, with advantage, produced without water addition.

55 [0039] To ease penetration even into very narrow clefts, the rinsing agent may contain a suitable wetting agent. For applications in the food industry the wetting agent should be special. Suitable wetting agents can be found in the group of surface active agents that popularly are called sugar surface active agents, i.e. surface active agents where the hydrophilic group is derived from sugar. An alternative to a wetting agent may be a suitable short-chained alcohol.

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**[0040]** Using a foaming composition as acid rinsing agent may be preferable, too. Such an agent can be easily achieved by a suitable choice of surface active agents.

Example 1

**[0041]** A packing machine of the type TETRA BRIK® was used for the filling of 3 dl packages of cream. At the end of each packing day the machine was cleaned as follows:

1. Douching with hot water from above and from underneath during 2.5 minutes. Open bottom valve.
2. Pre-wash with 150 litres hot water during 4 minutes. (Pump circulation from the wash tank).
3. Wash with 150 litres hot water during 4 minutes then adding further 75 litres hot water. To the first water quantity 1.5 litres washing agent is added. To the second water quantity still 0.5 litres washing agent is added. Total time for washing by pump circulation of wash solution from the wash tank 12 minutes.
4. Douching with hot water from above and from underneath during 2.5 minutes. Open bottom valve.
5. Rinsing with 150 litre cold water for 4 minutes. Pump circulation.
6. Douching with hot water from above and from underneath during 2.5 minutes. Open bottom valve.
7. Final rinsing with 150 litres cold water for 4 minutes.
8. Greasing stage. Bottom valve is closed.

**[0042]** During a several weeks long trial period the earlier used washing agent was replaced with a composition, which, at the delivery, contained:

Ampholyte surface active agent	8%
Sugar surface active agent	8%
Potassium hydroxide (50% solution)	8%
Polyacrylate (molecular weight 4500, 50% solution)	4%
Trisodiumorthophosphate	2%
Potassiumtripolyphosphate (50% solution)	70%

10 litres of this concentrate were mixed with 15 litres water to 25 litres stock solution. Of this stock solution between 2 and 2.5 litres were added to 150 litres wash solution.

**[0043]** At the beginning the wash solution's pH was ~10.8. It sank rapidly during the wash cycle to ~10.1. No corrosion of aluminium details could be seen.

**[0044]** To 150 litres of the rinse water (point 7 in the program) one added 2 to 2.5 litres of a rinsing agent composition were added. This composition consisted of:

- 20% acetic acid (60%),
- 70% lactic acid (80%),
- 9% ethanol denatured with 0.9 % iso-propanol
- 1 % sugar surface active agent.

**[0045]** At the end of the rinsing a pH of ~2.7 was measured.

**[0046]** The result of the cleaning was checked by a standard method comprising measurement adenosinriphosphate (ATP) at some chosen critical points in the machine. At first the ATP figures were high, but constantly lower than in a reference machine using a conventional cleaning system. When earlier soil accumulations in the machine had been removed by the new combination's better cleaning effect the values sunk dramatically and then remained at a very low level.

Example 2.

**[0047]** The trials of example 1 were repeated on another machine with the change that the alkaline washing agent was dosed with 1 dl for pre-wash, 3 dl for main wash and 1 dl for the first rinsing. Here too the wash and rinse water quantities were 150 litres. Also in other details the wash process was the same. As washing agent a super concentrate, i.e. a mixture without a water admixture (pH in the concentrate 12, in the use solution 9 to 10.5), was used. The mixture's composition was:

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Ampholyte surface active agent	10%
Sugar surface active agent	7,5%
Potassiumtripolyphosphate(50% solution)	67%
Potassium hydroxide(50% solution)	8%
Polyacrylate	4%
Trisodiumphosphate	2%
Laurylethersulphate	1,5%

I.e. very similar to the composition of example 1.

**[0048]** For the final rinsing one litre of an acid rinse agent was added. This agent had pH in concentrate 2.5 and pH in use solution 2.5 to 2.8 and the composition:

Ethanol/isopropanol	9%
Lactic acid	80%
Sugar surface active agent	1%
Acetic acid	10%

**[0049]** The cleaning result was much better compared with earlier methods without acid rinsing agent. Compared with the method of example 1 the new dosing method brings substantial advantages.

Example 3.

**[0050]** At this trial series the washing agent of example 2 was used. The Following wash program was applied:

Douching from above and from underneath 2 minutes  
Pre-wash, 1 dl washing agent per 150 litres of water, 4 minutes  
Emptying

**[0051]** Wash stage, 3 dl washing agent per 150 litres of water divided into two separate portions, 12 minutes. Emptying.

**[0052]** Douching from above and from underneath 2 minutes

**[0053]** First rinsing, 1 dl washing agent to 150 litres of water, 4 minutes

**[0054]** Second rinsing, 1 litre rinsing agent to 150 litres of water, 4 minutes.

**[0055]** The concentrate of the rinsing agent was prepared by the mixing of a hydrogen peroxide solution, containing 30 to 35% by weight hydrogen peroxide, with 2% by weight of a lactic acid mixture, containing 60% by weight acid.

**[0056]** Due to limitations in the programming system the wanted rest period in the second rinse stage could not be achieved. Instead the rinsing agent was added at the start of filling rinse water.

**[0057]** The next working day was started with a short douching of the interior of the machine with clean water of high bacteriological quality. The result of the cleaning became very good. Essentially all test points showed very low bacteria number.

### Claims

1. Method for cleaning and disinfection of mechanical constructions with aluminium and or brass components **characterised in that** one washes with an alkaline washing agent free from silicates and other components that may form precipitates and comprising pyro- and/or tripolyphosphate as complexing agent, said washing agent having a pH between 8.5 and 11 in use solution and a buffering capacity that prevents pH decrease during the washing by more than 1 unit, and then rinses with a disinfecting and passivating solution comprising hydrogen peroxide, said solution having a pH in the range 2.5 to 6 and containing an acid or acid mixture, which has sufficient buffering capacity for preventing the pH during the rinsing from rising from more than 1 pH unit, and under no circumstances above 7.
2. The method of claim 1 **characterised in that** the part of the cleaning process that comprises rinsing with hydrogen peroxide is prolonged and lasts at least 4 minutes, preferably 15 minutes or more.

3. The method of claim 2 **characterised in that** rinsing with hydrogen peroxide is the last step of the cleaning process.
4. The method of claim 1 **characterised in that** the alkaline washing agent contains added orthophosphate as reinforcer for the passivating layer of aluminium.
5. The method of claim 1 and 2 **characterised in that** the total quantity of washing agent is divided into three parts, one small part for pre-wash, one main part for the main wash and one small part for the first rinsing.

#### Patentansprüche

1. Verfahren der Reinigung und Desinfektion mechanischer Konstruktionen mit Aluminium- und/oder Messingteilen **gekennzeichnet dadurch, dass** man mit einem alkalischen Waschmittel wäscht, das ohne Silikate und andere Komponenten ist, die Ausfällungen bilden können, und pyro- und/oder Tripolyphosphate als Komplexmierungsmittel umfasst; gesagtes Waschmittel hat ein pH zwischen 8,5 und 11 in Gebrauchslösung und eine Pufferkapazität, die pH Abnahme während des Waschens mit mehr als einer Einheit verhindert; und danach mit einer desinfizierenden und passivierenden Lösung spült, die Wasserstoffperoxyd umfasst; gesagte Lösung hat ein pH zwischen 2.5 und 6 und enthält eine Säure oder Säuremischung, die genug Pufferkapazität hat, um pH-steigerung mit mehr als einer Einheit und unter allen Umständen über 7 zu verhindern.
2. Das Verfahren des Anspruchs 1 **gekennzeichnet dadurch, dass** der Teil des Reinigungsprozesses, der Spülen mit Wasserstoffperoxyd umfasst, verlängert ist und mindestens 4 Minuten, vorzugsweise 15 Minuten oder mehr dauert.
3. Das Verfahren des Anspruchs 2 **gekennzeichnet dadurch, dass** das Spülen mit Wasserstoffperoxyd der letzte Schritt des Reinigungsprozesses ist.
4. Das Verfahren des Anspruchs 1 **gekennzeichnet dadurch, dass** das alkalische Waschmittel zusätzliche orthophosphate als Verstärker der passivierenden Schicht des Aluminiums enthält.
5. Das Verfahren der Ansprüche 1 und 2 **gekennzeichnet dadurch, dass** die gesamte Quantität des Waschmittels in drei Teilen aufgeteilt ist, ein Kleinteil für die Vorwäsche, ein Hauptteil für die Hauptwäsche und ein Kleinteil für die erste Spülung.

#### Revendications

1. Procédé pour lavage et désinfection des constructions mécaniques avec des détails de l'aluminium et/ou du laiton **caractérisé en ce qu'on** lave avec un détergent alcalin libre du silicate et d'autres composants, qui peuvent former précipités et comprenant des pyro- et/ou tripolyphosphate comme agent complexant, le dit détergent ayant un pH entre 8.5 et 11 dans la solution d'usage et une capacité de tamponnement qui prévient pH réduction pendant le lavage avec plus que 1 unité, et après rince avec une solution désinfectant et passivant comprenant du peroxyde d'hydrogène, la dite solution ayant un pH entre 2.5 et 6 et contenant une acide ou une mixture des acides, qui ont une capacité de tamponnement suffisant pour prévenant pH accroissement pendant le rinçage avec plus que 1 pH unité et en aucun cas au-dessus 7.
2. Le procédé de revendication 1 **caractérisé en ce que** la part du procédé de nettoyage qui comprend rinçage avec du peroxyde d'hydrogène est prolongé et dure au moins 4 minutes, de préférence 15 minutes ou plus.
3. Le procédé de revendication 2 **caractérisé en ce que** le rinçage avec du peroxyde d'hydrogène est le dernier pas du procédé de nettoyage.
4. Le procédé de revendication 1 **caractérisé en ce que** le détergent alcalin contient orthophosphate additionné comme renforcement de la couche passivant d'aluminium.
5. Le procédé de revendication 1 **caractérisé en ce que** la quantité totale du détergent est divisé en trois parts, une petite part pour pré-lavage, une part principale pour le lavage principal et une petite part pour le premier rinçage.