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(54) METHODS FOR CLEANING INDUSTRIAL EQUIPMENT WITH PRE-TREATMENT

VERFAHREN ZUR REINIGUNG VON INDUSTRIEANLAGEN MIT VORBEHANDLUNG

PROCEDES DE NETTOYAGE D UN EQUIPEMENT INDUSTRIEL AVEC PRE-TRAITEMENT

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Description

FIELD

[0001] The invention relates to cleaning of industrial equipment such as evaporators, heat exchangers and other such equipment that is conventionally cleaned using a CIP (clean-in-place) process.

BACKGROUND

[0002] In many industrial applications, such as the manufacture of foods and beverages, hard surfaces commonly become contaminated with soils such as carbohydrate, proteinaceous, and hardness soils, food oil soils and other soils. Such soils can arise from the manufacture of both liquid and solid foodstuffs. Carbohydrate soils, such as cellulosics, monosaccharides, disaccharides, oligosaccharides, starches, gums and other complex materials, when dried, can form tough, hard to remove soils, particularly when combined with other soil components such as proteins, fats, oils and others. The removal of such carbohydrate soils can be a significant problem. Similarly, other materials such as proteins, fats and oils can also form hard to remove soil and residues.

[0003] Food and beverage soils are particularly tenacious when they are heated during processing. Foods and beverages are heated for a variety of reasons during processing. For example, in dairy plants, dairy products are heated on a pasteurizer (e.g. HTST - high temperature short time pasteurizer or UHT - ultra high temperature pasteurizer) in order to pasteurize the dairy product. Also, many food and beverage products are concentrated or created as a result of evaporation.

[0004] Specific examples of food and beverage products that are concentrated using evaporators include dairy products such as whole and skimmed milk, condensed milk, whey and whey derivatives, buttermilk, proteins, lactose solutions, and lactic acid; protein solutions such as soya whey, nutrient yeast and fodder yeast, and whole egg; fruit juices such as orange and other citrus juices, apple juice and other pomaceous juices, red berry juice, coconut milk, and tropical fruit juices; vegetable juices such as tomato juice, beetroot juice, carrot juice, and grass juice; starch products such as glucose, dextrose, fructose, isomerose, maltose, starch syrup, and dextrine; sugars such as liquid sugar, white refined sugar, sweetwater, and inulin; extracts such as coffee and tea extracts, hop extract, malt extract, yeast extract, pectin, and meat and bone extracts; hydrolyzates such as whey hydrolyzate, soup seasonings, milk hydrolyzate, and protein hydrolyzate; beer such as de-alcoholized beer and wort; and baby food, egg whites, bean oils, and fermented liquors.

[0005] There are generally at least two sides to an evaporator. One side holds the steam or vapor heat source (typically 100°C (212° F) to 177°C (350° F)). The other side holds the process liquid to be concentrated. During the evaporation process, the liquid to be concentrated is introduced into the evaporator. The heat exchange across the tubes or plates evaporates water off the process stream concentrating the liquid solids. The liquid to be concentrated may be run through an evaporator several times until it is sufficiently concentrated.

[0006] There are many different types of evaporators including falling film evaporators, forced circulation evaporated evaporators, plate evaporators, circulation evaporators, fluidized bed evaporators, falling film short path evaporators, rising film evaporators, counterflow-trickle evaporators, stirrer evaporators, and spiral tube evaporators. In addition to the evaporators, there are several other pieces of equipment in an evaporation plant including preheaters and heaters, separators, condensers, deaeration/vacuum systems, pumps, cleaning systems, vapor scrubbers, vapor recompression systems, and condensate polishing systems. All of the evaporation plant equipment should be cleaned, however, the actual evaporator typically has the most difficult soiling problems.

[0007] When a food or beverage product contacts any surface, soiling occurs where some part of the food or beverage product is left behind on that surface. When that surface is a heat exchange surface, the soil becomes thermally degraded rendering it even more difficult to remove. Over time, the layer of soil increases in thickness as more food or beverage product is passed over the heat exchange surface. The layer of soil acts as an insulator between the heat and the product being heated, thereby reducing the efficiency of the heat exchange surface and requiring more energy to create the same effect if the heat exchange surface were clean. When the heat exchange surface is an evaporator, the difference between a clean heat exchange surface and a soiled heat exchange surface can mean the difference in millions of dollars in energy costs for an evaporator plant. With the cost of energy increasing significantly, as well as an increased awareness of protecting the environment by preserving natural resources, there remains a need for cleaning programs that can clean heat exchange surfaces and create an efficient transfer a heat.

[0008] Clean-in-place cleaning techniques are a specific cleaning regimen adapted for removing soils from the internal components of tanks, lines, pumps and other process equipment used for processing typically liquid product streams such as beverages, milk, juices, etc. Clean-in-place cleaning involves passing cleaning solutions through the system without dismantling any system components. The minimum clean-in-place technique involves passing the cleaning solution through the equipment and then resuming normal processing. Any product contaminated by cleaner residue can be discarded. Often clean-in-place methods involve a first rinse, the application of the cleaning solutions, a second rinse with potable water followed by resumed operations. The process can also include any other contacting step in which a rinse, acidic or basic functional fluid, solvent or other cleaning component such as hot water, cold water, etc. can be contacted with the equipment at any step during the process. Often the final potable water rinse is skipped in order to prevent contamination of the equipment with bacteria following the cleaning and/or sanitizing step. Clean-in-place processing requires a complete or partial shutdown of the equipment being cleaned, which results in lost production time. Many times, the equipment is not thoroughly cleaned, due to the large downtime needed. What is needed is an improved method for cleaning this equipment, using the clean-in-place process, which uses less time to thoroughly remove the soils. US 2002/0177220 A1 describes a method of cleaning a dairy pipeline using enzyme pretreatment.

[0009] US 2005/0183744 A1 describes a method for treating CIP equipment with a multiple phase treating composition comprising a treating liquid phase and a treating gaseous phase.

[0010] It is against this background that the present invention has been made.

SUMMARY OF THE DISCLOSURE

[0011] Surprisingly, it has been discovered that food and beverage soils, and especially baked-on food and beverage soils can be removed from surfaces using a two-step method where the soil is contacted with a pre-treatment composition in a pre-treatment step, followed by a conventional clean-in-place process. The invention relates to a method of cleaning according to claims 1 to 12. The method relates to cleaning processes for removing carbohydrate and proteinaceous soils from food and beverage manufacturing locations using a clean-in-place method.

BRIEF DESCRIPTION OF THE DRAWINGS

[0012] FIG. 1 is a schematic diagram of an industrial process that includes equipment to be cleaned, CIP process equipment, and pre-treatment equipment.

DETAILED DESCRIPTION OF THE INVENTION

[0013] The present invention is directed to cleaning of industrial equipment using a pre-treatment step in combination with clean-in-place procedures. Use of a pre-treatment step, in combination with conventional clean-in-place solutions and processes, provides increased soil removal than the conventional process alone. Additionally, use of a pre-treatment step, followed by a water rinse, provided unexpected amounts of soil removal. Use of a pre-treatment step allows the use of traditionally incompatible chemistries and at higher concentrations then applied in conventional cleaning programs.

[0014] As used herein, "weight percent", "wt-%", "percent by weight", "% by weight", and variations thereof refer to the concentration of a substance as the weight of that substance divided by the total weight of the composition and multiplied by 100. It is understood that, as used here, "percent", "%" are intended to be synonymous with "weight percent", "wt-%", etc.

[0015] It should be noted that, as used in this specification and the appended claims, the singular forms "a," "an" and "the" include plural referents unless the content clearly dictates otherwise. Thus, for example, reference to a composition containing "a compound" includes having two or more compounds. It should also be noted that the term "or" is generally employed in its sense including "and/or" unless the content clearly dictates otherwise.

[0016] The method of the present invention applies to equipment generally cleaned using clean-in-place (i.e., CIP) cleaning procedures. Examples of such equipment include evaporators, heat exchangers (including tube-in-tube exchangers, direct steam injection, and plate-in-frame exchangers), heating coils (including steam, flame or heat transfer fluid heated) re-crystallizers, pan crystallizers, spray dryers, drum dryers, and tanks. This method can be used in generally any application where caked on soil or burned on soil, such as proteins or carbohydrates, needs to be removed; applications include the food and beverage industry (especially dairy), and brewing.

[0017] CIP processing is generally well-known. The process includes applying a dilute solution (typically about 0.5-3%) onto the surface to be cleaned. The solution flows across the surface (91,4 cm/s to 182,88 cm/s) ((3 to 6 feet/second)), slowly removing the soil. Either new solution is re-applied to the surface, or the same solution is recirculated and re-applied to the surface.

[0018] A typical CIP process to remove a soil (including organic, inorganic or a mixture of the two components) includes at least three steps: an alkaline solution wash, an acid solution wash, and then a fresh water rinse. The alkaline solution softens the soils and removes the organic alkaline soluble soils. The subsequent acid solution removes mineral soils left behind by the alkaline cleaning step. The strength of the alkaline and acid solutions and the duration of the cleaning steps are typically dependent on the durability of the soil. The water rinse removes any residual solution and soils, and cleans the surface prior to the equipment being returned on-line. The present invention provides a pre-treatment step, prior to the CIP process, which penetrates into the soil. The penetrating materials soften the soil, act as a catalyst, or otherwise enhance the activity of the conventional CIP solution when it contacts the soil. Thus, the pre-treatment facilitates the soil removal.

[0019] Referring now to FIG. 1, a schematic diagram of process equipment is illustrated at reference numeral 10. Process 10 includes a tank 20, which is the equipment to be cleaned. A feed line 25 supplies the various cleaning solutions to tank 20, and a drain line 27 removes solution from tank 20. Operably connected via appropriate pipes, valves, pumps, etc. is equipment for a CIP process, designated as reference numeral 30. CIP process 30 includes a tank 35 for retaining the dilute CIP chemistry. Drain line 27 from tank 20 is used to recirculate solution from tank 20 back to CIP process 30 and tank 35. Process 10 also includes equipment for the pre-treatment process, designated as reference numeral 40. Pre-treatment equipment 40 includes a first tank 42 and a second tank 44. When two tanks are used, generally one tank, e.g., tank 42, will contain an alkaline pre-treatment and the other tank, e.g., tank 44, will contain an acidic pre-treatment. The appropriate pipes, valves, pumps, etc. are in place for operably connecting tanks 42, 44 with feed line 25 into tank 20. This set-up of process 10 allows a pre-treatment to be applied to tank 20 without the use of large amounts of additional equipment, such as piping. Additional details regarding the method of cleaning tank 20 is described below.

The Pre-Treatment Solution

[0020] As described above, the pre-treatment solution or pre-treatment step is applied to the soil prior to the application of conventional CIP chemistries. The chemistry of the pre-treatment solution is selected to facilitate removal of the soils on the surfaces to be cleaned. The pre-treatment solution pre-coats and penetrates into the soil, softening the soil. The specific chemistry used can be selected based on the soil to be removed. The chemistry used can be compatible with the CIP chemistry. In some embodiments, it is desired to have a pre-treatment that is incompatible with the CIP chemistry; in such instances, the pre-treatment reacts with the CIP chemistry. It has been found that using incompatible chemistries further increases the soil-removal effectiveness.

[0021] The pre-treatment solution comprises 0.25% of active ingredients, in some cases at least 0.5%, preferably at least 2% and more preferably at least 3%. By use of the term "active ingredients" what is intended is the non-inert ingredients that facilitate the softening, dissolving and removal of soil. These active ingredients include penetrant and oxidant. In most embodiments, water is the remainder of the solution. Typically, the solution has no more than about 15% active ingredients, preferably no more than about 10%. For most applications, a concentration of about 1-10% is preferred; a concentration of about 1-3% is suitable for most applications.

Alkaline or Acidic Ingredients

[0022] The pre-treatment solution optionally and preferably includes alkaline or acidic ingredients. Examples of suitable alkaline sources include basic salts, amines, alkanol amines, carbonates and silicates. Particularly preferred alkaline sources include NaOH (sodium hydroxide), KOH (potassium hydroxide), TEA (triethanol amine), DEA (diethanol amine), MEA (monoethanolamine), sodium carbonate, and morpholine, sodium metasilicate and potassium silicate.

[0023] Examples of suitable acidic sources include mineral acids (such as phosphoric acid, nitric acid, sulfuric acid), and organic acids (such as lactic acid, acetic acid, hydroxyacetic acid, citric acid, glutamic acid, glutaric acid, and gluconic acid).

[0024] The amount of alkaline or acid in the pre-treatment solution in some cases is at least 0.25 wt-% and no greater than 10 wt-%. Suitable levels of alkaline or acid include 2 to 5 wt-% and 0.5 to 1.5 wt-%.

Penetrants

[0025] A penetrant may be present in the pre-treatment solution. The penetrant may be combined with an alkaline or acid source in the solution, or, the penetrant may be used without an alkaline or acid source. Preferably, the penetrant is water miscible.

[0026] Examples of suitable penetrants include alcohols, short chain ethoxylated alcohols and phenol (having 1-6 ethoxylate groups). Organic solvents are also suitable penetrants. Examples of suitable organic solvents, for use as a penetrant, include esters, ethers, ketones, amines, and nitrated and chlorinated hydrocarbons.

[0027] Another preferred class of penetrants is ethoxylated alcohols. Examples of ethoxylated alcohols include alky, aryl, and alkylaryl alkloxylates. These alkloxylates can be further modified by capping with chlorine-, bromine-, benzyl-, methyl-, ethyl-, propyl-, butyl- and alkyl-groups. A preferred level of ethoxylated alcohols in the solution is 1 to 20 wt-%.

[0028] Another class of penetrants is fatty acids. Some non-limiting examples of fatty acids are C_6 to C_{12} straight or branched fatty acids. Preferred fatty acids are liquid at room temperature.

[0029] Another class of preferred solvents for use as penetrants is glycol ethers, which are water soluble. Examples of glycol ethers include dipropylene glycol methyl ether (available under the trade designation DOWANOL DPM from Dow Chemical Co.), diethylene glycol methyl ether (available under the trade designation DOWANOL DM from Dow

Chemical Co.), propylene glycol methyl ether (available under the trade designation DOWANOL PM from Dow Chemical Co.), and ethylene glycol monobutyl ether (available under the trade designation DOWANOL EB from Dow Chemical Co.). A preferred level of glycol ether in the solution is 0.5 to 20 wt-%.

[0030] Surfactants also are a suitable penetrant for use in the pre-treatment solution. Examples of suitable surfactants include nonionic, cationic, and anionic surfactants. Nonionic surfactants are preferred. Nonionic surfactants improve soil removal and can reduce the contact angle of the solution on the surface being treated. Examples of suitable nonionic surfactants include alkyl-, aryl-, and arylalkyl-, alkoxylates, alkylpolyglycosides and their derivatives, amines and their derivatives, and amides and their derivatives. Additional useful nonionic surfactants include those having a polyalkylene oxide polymer as a portion of the surfactant molecule. Such nonionic surfactants include, for example, chlorine-, benzyl-, methyl-, ethyl-, propyl-, butyl- and other like alkyl-capped polyoxyethylene and/or polyoxypropylene glycol ethers of fatty alcohols; polyalkylene oxide free nonionics such as alkyl polyglycosides; sorbitan and sucrose esters and their ethoxylates; alkoxylated ethylene diamine; carboxylic acid esters such as glycerol esters, polyoxyethylene esters, ethoxylated and glycol esters of fatty acids; carboxylic amides such as diethanolamine condensates, monoalkanolamine condensates, polyoxyethylene fatty acid amidese; and ethoxylated amines and ether amines and other like nonionic compounds. Silicone surfactants can also be used.

[0031] Additional suitable nonionic surfactants having a polyalkylene oxide polymer portion include nonionic surfactants of C_6-C_{24} alcohol ethoxylates having 1 to about 20 ethylene oxide groups; C_6-C_{24} alkylphenol ethoxylates having 1 to about 100 ethylene oxide groups; C_6-C_{24} alkylpolyglycosides having 1 to about 20 glycoside groups; C_6-C_{24} fatty acid ester ethoxylates, propoxylates or glycerides; and C_4-C_{24} mono or dialkanolamides.

[0032] If a surfactant is used as a penetrant, the amount of surfactant in the pre-treatment solution is typically at least 0.25%. Acceptable levels of surfactant include 0.4 to 8 wt-%, and 1 to 4 wt-%.

[0033] Overall, when an alkaline or acid source is present, the amount of penetrant in the pre-treatment solution is at least 0.2 wt-% and no greater than 2.5 wt-%. Acceptable levels of penetrant, when an alkaline or acid source is present, include 0.4-2 wt-%; 1-2 wt-% is preferred. The amount of penetrant, in relation to any alkaline or acid source when present, is generally 1:1 to 1:5.

[0034] For pre-treatment solutions without an alkaline or acid source, the amount of penetrant in the solution is at least 0.05 wt-% and no greater than 50%. Generally, the level is 0.1 to 25 wt-%. Acceptable levels of penetrant include 0.5 to 10 wt-%, and 1 to 5 wt-%.

Oxidants

[0035] Pre-treatment solutions include an oxidant, such as a peroxide or peroxyacid.

[0036] Suitable ingredients are oxidants such as peroxygen compounds, which include peroxides and various percarboxylic acids, including percarbonates.

[0037] Peroxycarboxylic (or percarboxylic) acids generally have the formula $R(CO_3H)_n$, where, for example, R is an alkyl, arylalkyl, cycloalkyl, aromatic, or heterocyclic group, and n is one, two, or three, and named by prefixing the parent acid with peroxy. The R group can be saturated or unsaturated as well as substituted or unsubstituted. Medium chain peroxycarboxylic (or percarboxylic) acids can have the formula $R(CO_3H)_n$, where R is a C_5-C_{11} alkyl group, a C_5-C11 cycloalkyl, a C_5-C_{11} arylalkyl group, C_5-C_{11} aryl group, or a C_5-C_{11} heterocyclic group; and n is one, two, or three. Short chain fatty acids can have the formula $R(CO_3H)_n$ where R is C_1-C_4 and n is one, two, or three.

[0038] Some peroxycarboxylic acids include peroxypentanoic, peroxyhexanoic, peroxyheptanoic, peroxyoctanoic, peroxynonanoic, peroxyisononanoic, peroxydecanoic, peroxyundecanoic, peroxydodecanoic, peroxyascorbic, peroxyadipic, peroxycitric, peroxypimelic, or peroxysuberic acid, mixtures thereof.

[0039] Branched chain peroxycarboxylic acid include peroxyisopentanoic, peroxyisononanoic, peroxyisohexanoic, peroxyisoheptanoic, peroxyisooctanoic, peroxyisonananoic, peroxyisodecanoic, peroxyisoundecanoic, peroxyisododecanoic, peroxyneopentanoic, peroxyneohexanoic, peroxyneoheptanoic, peroxyneooctanoic, peroxyneononanoic, peroxyneodecanoic, peroxyneoundecanoic, peroxyneododecanoic, mixtures thereof.

[0040] Typical peroxygen compounds include hydrogen peroxide (H_2O_2), peracetic acid, peroctanoic acid, a persulphate, a perborate, or a percarbonate.

[0041] The amount of oxidant in the pre-treatment solution is at least 0.01 wt-% and no greater than 1 wt-%. Acceptable levels of oxidant are 0.01 to 0.50 wt-%; 0.3wt-% is a particularly suitable level. Suitable levels of oxidant, in relation to any acid source, are generally 1:1 to 1:10, 1:3 to 1:7, or 1:20 to 1:50. Solutions of 0.25 wt-% to 10 wt-% phosphoric acid with 50-5000 ppm (0.005 wt-% to 0.5 wt-%) hydrogen peroxide are particularly suitable. An example pre-treatment solution includes 0.75 wt-% phosphoric acid and 500 ppm (0.05 wt-%) hydrogen peroxide, which is a 1:15 ratio of oxidant:acid.

Builders

[0042] The pre-treatment solution preferably includes a builder. Builders include chelating agents (chelators), sequestering agents (sequestrants), detergent builders. The builder often stabilizes the composition or solution. Examples of builders include phosphonic acids and phosphonates, phosphates, aminocarboxylates and their derivatives, pyrophosphates, polyphosphates, ethylenediamene and ethylenetriamene derivatives, hydroxyacids, and mono-, di-, and tricarboxylates and their corresponding acids. Other builders include aluminosilicates, nitroloacetates and their derivatives, and mixtures thereof. Still other builders include aminocarboxylates, including salts of ethylenediaminetetraacetic acid (EDTA), hydroxyethylenediaminetetraacetic acid (HEDTA), and diethylenetriaminepentaacetic acid. Preferred builders are water soluble.

[0043] Particularly preferred builders include EDTA (including tetra sodium EDTA), TKPP (tripotassium polyphosphate), PAA (polyacrylic acid) and its salts, phosphonobutane carboxylic acid, and sodium gluconate.

[0044] The amount of builder in the pre-treatment solution, if present, is typically at least 0.25 wt-% and no greater than 5 wt-%. Acceptable levels of builder include 0.5 to 1.0 wt-% and 1 wt-% to 2.5 wt-%.

Methods of Pre-Treating

[0045] The method of the present invention is directed to applying the pre-treatment solution to the surface to be cleaned, prior to a conventional CIP process. The resulting CIP process requires less steps and/or less time for each step. For example, a conventional CIP process includes five steps after an initial water rinse: a conventional alkaline (NaOH) wash to remove soil, an interim rinse, a conventional acid wash to remove minerals and scale, a water rinse, and a conventional sanitizing step. This process can be replaced with a three-step process after the initial water rinse: A pre-treatment step, a conventional wash, and a water rinse.

[0046] By using either of the two pre-treatment processes described immediately above, the amount of water used in the overall cleaning process with pre-treatment is reduced by about 30% or more compared to the conventional five-step process. The amount of time for the overall process with pre-treatment is reduced by about 30% or more compared to the conventional five-step process. The specific number of steps, the water usage, or the processing time reduced will depend on the concentration and chemistry of the pre-treatment solution.

[0047] Referring again to FIG. 1, pre-treatment solution is stored at the equipment designated as 40. In this process 10, tank 42 holds an alkaline pre-treatment solution and tank 44 holds an acidic pre-treatment solution that includes peroxide.

[0048] To clean 20, tank 20 and its connection lines are drained of any product that may be present. A water rinse may be included to remove any residual product. In one embodiment, alkaline pre-treatment solution from tank 42 is pumped via piping and feed line 25 into tank 20. Conventional CIP application equipment, such as a spray head, applies the pre-treatment solution onto the interior surface of tank 20. The pre-treatment solution cascades or otherwise flows down the surface of tank 20, softening the soil. A second application of pre-treatment solution may be applied, although this is not generally needed.

[0049] After application and draining of the pre-treatment solution, a conventional CIP process, using the detergent from process 30 and tank 35, is performed. The CIP detergent may be acidic or alkaline. Detergent from tank 35 is recirculated through tank 20 via feed line 25, return line 27, and other appropriate piping.

[0050] In another embodiment, a pre-treatment solution containing hydrogen peroxide from tank 44 is pumped via piping and feed line 25 into tank 20. After application and draining of the peroxide pre-treatment solution, a conventional CIP process, using an alkaline detergent such as sodium hydroxide, from process 30 and tank 35, is performed. The sodium hydroxide activates any residual peroxide on the walls of tank 20.

[0051] When introducing the pre-treatment solution into the CIP process, it may be beneficial to add the pre-treatment solution at specific places depending on the piece of equipment. For example, when treating an HTST pasteurizer, it is preferable to introduce the pre-treatment solution at the feed balance tank. Alternatively, the pre-treatment solution can be introduced on the suction side of the booster pump or at the T or valve location just prior to plate assembly. When treating a UHT pasteurizer, it is preferable to introduce the pre-treatment solution at the water balance tank. Alternatively, the pre-treatment solution can be introduced at the product balance or feed tank, or the suction side of the booster pump. When treating an evaporator, it is preferable to introduce the pre-treatment solution on the suction side of the effect recirculation pump. Alternatively, the pre-treatment solution can be introduced at the CIP balance tank. Finally, when treating a beer distillation re-boiler, it is preferable to introduce the pre-treatment solution on the suction side of the boiler recirculation pump. Alternatively, the pre-treatment solution can be introduced in the valves between recirculation pump and the distillation column, or the CIP balance tank. The pre-treatment solution is preferably injected closer to the surface to be cleaned. This allows for higher chemical concentrations by avoiding dilution of the pre-treatment chemistry by the entire volume of the CIP supply tank and distribution lines.

[0052] For a more complete understanding of the invention, the following examples are given to illustrate some embodiments. All parts are by weight, except where it is contrarily indicated.

EXAMPLES

EXAMPLE 1 (removed)

EXAMPLE 2 (removed)

EXAMPLE 3

[0053] Example 3 tested the effectiveness of various different pre-treatment and main wash chemistries on removing corn beer thin stillage syrup. For this test, the corn beer thin stillage syrup soil was prepared by weighing 7,62 cm by 12,7 cm (3 inch by 5 inch) stainless steel screens. A mixture of 85% corn beer thin stillage syrup and 15% deionized water was prepared and the screens were dipped in the mixture and set aside to drain the excess for 10 minutes. The screens were then baked at 125° C for 2 hours. The screens were re-dipped and baked another 2 times for a total of 3 times. The final screens were weighed again. For cleaning, 1000 mL of the chemical cleaning solutions in Table 1 were heated to 82,2 °C (180 °F). The screens were inserted into the cleaning solution. A stir bar was in the cleaning solution and set at 400 rpm for the entire test (30 minutes). After 30 minutes, the screens were removed and allowed to dry before weighing. The percent soil removal was calculated using the following formula:

$$\frac{\text{Soiled wt} - \text{After wt}}{\text{Soiled wt} - \text{virgin wt}} \times 100 = \%\ \text{Soil Removal}$$

[0054] Table 1 shows the percent soil removal of various pre-treatment and main wash chemistries.

Table 1 Pre-treatment Solution Effectiveness On Corn Beer Thin Stillage Syrup

Exp #	15 Min Pre-treatment Chemistry		Percent %	15 Min CIP Main Wash Chemistry	Percent %	% Soil Removal
	Chemistry	Tradename				
1*	NaOH		3.00	-	-	75.70
2*	Na2CO3		2.00	-	-	65.00
3*	MEA (99%)		4.00	NaOH	3.00	75.00
4*	DEA		4.00	NaOH	3.00	82.40
5*	TEA		4.00	NaOH	3.00	79.50
6*	Morpholine		4.00	NaOH	3.00	82.60
7*	Cyclohexylamine		4.00	NaOH	3.00	84.50
8*	n-Methyl Pyrolidone		4.00	NaOH	3.00	84.90
9*	Monoisopropanol amine		4.00	NaOH	3.00	94.60
10	H_2O_2		0.50	NaOH	3.00	95.00
	MEA (99%)		4.00			
11	H_2O_2		0.50	NaOH	3.00	97.40
	DEA		4.00			
12	H_2O_2		0.50	NaOH	3.00	90.00
	TEA		4.00			

(continued)

Exp #	15 Min Pre-treatment Chemistry		Percent %	15 Min CIP Main Wash Chemistry	Percent %	% Soil Removal
	Chemistry	Tradename				
13	H_2O_2		0.50	NaOH	3.00	89.80
	Morpholine		4.00			
14	H_2O_2		0.50	NaOH	3.00	94.80
	Cyclohexylamine		4.00			
15	H_2O_2		0.50	NaOH	3.00	82.10
	n-Methyl Pyrolidone		4.00			
16	H_2O_2		0.50	NaOH	3.00	92.70
	Monoisopropanol amine		4.00			
17*	H_2O_2		0.50	NaOH	3.00	94.90
18*		Dowanol EB	4.00	NaOH	3.00	96.40
19*		Dowanol DM	4.00	NaOH	3.00	84.60
20*		Dowanol PnB	4.00	NaOH	3.00	97.00
21*		Dowanol EpH	4.00	NaOH	3.00	80.20
22*		Dowanol DpnP	4.00	NaOH	3.00	87.60
23*		Dowanol PnP	4.00	NaOH	3.00	86.20
24*		Dowanol PPh	4.00	NaOH	3.00	84.80
25*	Propylene Carbonate		4.00	NaOH	3.00	71.90
		Dowanol EB	4.00			
26	H_2O_2		0.50	NaOH	3.00	92.00
		Dowanol DM	4.00			
27	H_2O_2		0.50	NaOH	3.00	96.50
		Dowanol PnB	4.00			
28	H_2O_2		0.50	NaOH	3.00	97.00
		Dowanol EpH	4.00			
29	H_2O_2		0.50	NaOH	3.00	94.20
		Dowanol DpnP	4.00			
30	H_2O_2		0.50	NaOH	3.00	99.00
		Dowanol PnP	4.00			
31	H_2O_2		0.50	NaOH	3.00	98.80
		Dowanol PPh	4.00			
32	H_2O_2		0.50	NaOH	3.00	99.20
	Propylene Carbonate		4.00			
33	removed					
34*		Dequest 2000	4.00	NaOH	3.00	89.30
35*		Dequest 2010	4.00	NaOH	3.00	86.20

(continued)

Exp #	15 Min Pre-treatment Chemistry		Percent %	15 Min CIP Main Wash Chemistry	Percent %	% Soil Removal
	Chemistry	Tradename				
36*	EDTA		4.00	NaOH	3.00	89.20
37*	STPP		4.00	NaOH	3.00	79.70
38*	TKPP		4.00	NaOH	3.00	89.10
39*	Sodium Gluconate		4.00	NaOH	3.00	89.50
		Dequest 2000	4.00			
40	H_2O_2		0.50	NaOH	3.00	95.30
		Dequest 2010	4.00			
41	H_2O_2		0.50	NaOH	3.00	94.10
	EDTA		4.00			
42	H_2O_2		0.50	NaOH	3.00	95.00
	STPP		4.00			
43	H_2O_2		0.50	NaOH	3.00	95.00
	TKPP		4.00			
44	H_2O_2		0.50	NaOH	3.00	97.00
	Sodium Gluconate		4.00			
45*	H_2O_2		0.50	NaOH	3.00	93.00
*= comparative example						

EXAMPLE 4 (comparative)

[0055] Example 4 compared the ability of various oxidants to remove corn beer thin stillage syrup. For this example, the screens were soiled with corn beer thin stillage syrup and cleaned as described in Example 3. Table 2 shows the impact of various oxidants on soil removal.

Table 2 Impact of Oxidants on Corn Beer Thin Stillage Syrup Removal

Exp #	15 Min Pre-treatment Chemistry	Percent %	15 Min CIP Main Wash Chemistry	Percent %	% Soil Removal
1	H_2O_2	0.50	NaOH	3.00	94.90
2	Sodium Perborate	1.50	NaOH	3.00	96.40
3	Sodium Percarbonate	1.75	NaOH	3.00	82.80
4	Sodium Persulfate	3.38	NaOH	3.00	72.30
5	Potassium Permanganate	1.12	NaOH	3.00	93.90

EXAMPLE 5

[0056] Example 5 compared the amount of time it took to clean the screens using the pre-treatment solutions of the present invention compared to using only sodium hydroxide. This example tested the time to clean on corn beer thin stillage syrup and whole milk soil. The corn beer thin stillage syrup soil was prepared and cleaned as described in Example 3.

[0057] For the whole milk soil, the soil was prepared by weighing stainless steel discs to be soiled and affixing the disk to the bottom of a 45,72 cm (1.5 foot), 7,62 cm (3 inch) diameter stainless steel tube. A water bath was heated to 96°C (205° F) to 99°C (210° F) and the tubes with the discs were placed in the water bath. A 1,261 L (1/3 gallon) of whole milk was added to each tube used and allowed to sit for 4 hours. After 4 hours, the disks were removed and allowed to dry for at least 48 hours before weighing. When cleaning the whole milk soil, the disc was affixed to an overhead stirrer. The desired cleaning solution was heated to 82,2°C (180 °F) using a hot plate. The disc was inserted into a 1L beaker of cleaning solution for 10 minutes and the overhead stirrer was set to 50 rpm. After 10 minutes, the disc was removed from the cleaning solution and placed in a beaker of deionized water. The overhead stirrer was set at 200 rpm for 30 seconds. The disc was removed and allowed to dry at least 48 hours before weighing. The percent soil removal was calculated using the following formula :

$$\frac{\text{Soiled wt} - \text{After wt}}{\text{Soiled wt} - \text{virgin wt}} \times 100 = \%\ \text{Soil Removal}$$

[0058] Tables 3 and 4 show the time it took to remove 100% of the soil on the screen or disc when using sodium hydroxide alone, Stabicip Oxi pre-treatment followed by a sodium hydroxide wash, and Formula A (74% hydrogen peroxide (35%), 9.75% sodium cumene sulfonate (40%), 5.25% sodium octane sulfonate, 3.50% hydroxyethylidene diphosphonic acid (60%), 3% methane sulfonic acid, 1% n-butyl capped alcohol ethoxylate (5EO), and 3.5% pelargonic acid) followed by a sodium hydroxide wash. Stabicip Oxi is a hydrogen peroxide based composition commercially available from Ecolab Inc. (St. Paul, MN).

Table 3 Time to Clean Corn Beer Thin Stillage Syrup Soil

Exp #	15 Min Pre-treatment Chemistry	Percent %	15 Min CIP Main Wash Chemistry	Percent %	Time (Min) to 100% Soil Removal
1*	NaOH	2.00	-	-	60
2	Stabicip Oxi	1.50	NaOH	2.00	30
*=comparative example					

[0059] Table 3 shows that including a hydrogen peroxide based pre-treatment composition together with a sodium hydroxide wash cuts the time to clean corn beer thin stillage syrup in half when compared to a sodium hydroxide wash alone.

Table 4 Time to Clean Whole Milk Soil

Exp #	15 Min Pre-treatment Chemistry	Percent %	15 Min CIP Main Wash Chemistry	Percent %	Time (Min) to 100% Soil Removal
1*	NaOH	2.00	-	-	>60 min
2	Stabicip Oxi	1.50	NaOH	2.00	33
3	Formula A	1.50	NaOH	2.00	27
*=comparative example					

[0060] Table 4 shows that including a hydrogen peroxide based pre-treatment composition together with a sodium hydroxide wash cuts the time to clean whole milk soil in half when compared to a sodium hydroxide wash alone. Using Formula A together with a sodium hydroxide wash cuts the time to clean whole milk soil by more than half when compared to sodium hydroxide wash alone.

EXAMPLE 6

[0061] Example 6 tested the effectiveness of various different pre-treatment and main wash chemistries on the removal of whole milk soil. For this test, the whole milk soil was prepared and cleaned as described in Example 5. Table 5 shows the percent removal of the various combinations.

Table 5 Pre-treatment Solution Effectiveness On Whole Milk

Exp #	**5 Min Pre-treatment Chemistry**		**Percent %**	**5 Min CIP Main Wash Chemistry**	**Percent %**	**% Soil Removal**
	Chemistry	**Tradename**				
1*	NaOH		1.00	-	-	37.90
2*	MEA (99%)		0.50	-	-	23.00
3*		Dowanol EB	0.50	-	-	9.70
4	removed			-	-	
5		Stabicip Oxi	0.50	-	-	12.60
6*	HNO_3		0.50	-	-	20.00
7*		Dowanol EB	0.50	NaOH	1.00	34.50
8*		Stabicip Oxi	0.50	NaOH	1.00	57.50
9	removed					
10*	MEA (99%)		0.50	NaOH	1.00	41.80
11*	HNO_3		0.50	NaOH	1.00	49.20
12*	MEA (99%)		0.50	H2O2	0.15	19.90
*=comparative example						

Claims

1. A method of cleaning proteinaceous and/or carbohydrate soils from industrial equipment using a CIP process, wherein the soil is from food and beverage manufacturing locations, the method comprising :

   (a) applying a pre-treatment solution to the soil, the solution comprising at least 0.10 wt-% active ingredients, the active ingredients including a penetrant and an oxidant selected from the group consisting of hydrogen peroxide, peroxygen compounds and mixtures thereof;
   (b) recirculating a second solution through the equipment after the pre-treatment solution, the second solution comprising a dilute detergent; and then
   (c) rinsing the equipment.

2. The method according to claim 1, wherein the pre-treatment solution further comprises 0.10 to 1.5 wt-% acid.

3. The method according to claim 2, wherein the pre-treatment solution comprises 0.01 to 1 wt-% oxidant.

4. The method of claim 3, wherein the oxidant is a peroxide.

5. The method according to claim 1, wherein the pre-treatment solution comprises 0.4 to 10 wt-% penetrant.

6. The method according to claim 2, wherein the pre-treatment solution comprises 0.25 to 1.5 wt-% acid and 0.4 to 8 wt-% penetrant.

7. The method according to claim 1, wherein the pre-treatment solution comprises 0.3 to 10 wt-% of an alkalinity source.

8. The method according to claim 1, wherein the pre-treatment solution comprises:

   (a) 0.25 to 1.5 wt-% acid, being at least one of phosphoric acid, nitric acid, sulfuric acid, lactic acid, acetic acid, hydroxyacetic acid and citric acid; and
   (b) 0.01 to 1 wt-% oxidant, being a peroxygen compound.

9. The method according to claim 1, wherein the oxidant is present at a level of 0.05 to 0.5 wt-%.

10. The method according to claim 1, comprising:

(a) applying a pre-treatment solution comprising 0.25 to 1.5 wt-% acid and 0.01 to 1 wt-% oxidant; and
(b) recirculating an alkaline second solution through the equipment after the pre-treatment solution.

11. The method according to claim 10, wherein:

(a) the acid is at least one of phosphoric acid, nitric acid, sulfuric acid, lactic acid, acetic acid, hydroxyacetic acid and citric acid; and
(b) the oxidant is a peroxygen compound.

12. The method of claim 1, wherein the second solution comprises sodium hydroxide.

Patentansprüche

1. Verfahren zur Reinigung industrieller Anlagen von eiweiß- und/oder kohlenhydrathaltigen Verschmutzungen unter Anwendung eines CIP-Verfahrens (*Cleaning in Place,* ortsgebundene Reinigung), wobei der Schmutz von Orten der Herstellung von Nahrungsmitteln und Getränken stammt, wobei das Verfahren umfasst:

(a) Anwenden einer Vorbehandlungslösung auf den Schmutz, wobei die Lösung wenigstens 0,10 Gew.-% an Wirkstoffen umfasst, wobei die Wirkstoffe ein Durchdringungsmittel und ein Oxidationsmittel einschließen, ausgewählt aus der Gruppe bestehend aus Wasserstoffperoxid, Persauerstoffverbindungen und Gemischen davon;
(b) das Umlaufen einer zweiten Lösung durch die Anlage im Anschluss an die Vorbehandlungslösung, wobei die zweite Lösung ein verdünntes Detergens umfasst; und anschließend
(c) Ausspülen der Anlage.

2. Verfahren nach Anspruch 1, wobei die Vorbehandlungslösung ferner 0,10 bis 1,5 Gew.-% an Säure umfasst.

3. Verfahren nach Anspruch 2, wobei die Vorbehandlungslösung 0,01 bis 1 Gew.-% an Oxidationsmittel umfasst.

4. Verfahren nach Anspruch 3, wobei das Oxidationsmittel ein Peroxid ist.

5. Verfahren nach Anspruch 1, wobei die Vorbehandlungslösung 0,4 bis 10 Gew.-% an Durchdringungsmittel umfasst.

6. Verfahren nach Anspruch 2, wobei die Vorbehandlungslösung 0,25 bis 1,5 Gew.-% an Säure und 0,4 bis 8 Gew.-% an Durchdringungsmittel umfasst.

7. Verfahren nach Anspruch 1, wobei die Vorbehandlungslösung 0,3 bis 10 Gew.-% an einer Alkalinitätsquelle umfasst.

8. Verfahren nach Anspruch 1, wobei die Vorbehandlungslösung umfasst:

(a) 0,25 bis 1,5 Gew.-% an Säure, welche wenigstens eine der folgenden ist: Phosphorsäure, Salpetersäure, Schwefelsäure, Milchsäure, Essigsäure, Hydroxyessigsäure und Citronensäure; und
(b) 0,01 bis 1 Gew.-% an Oxidationsmittel, welches eine Persauerstoffverbindung ist.

9. Verfahren nach Anspruch 1, wobei das Oxidationsmittel vorliegt in einer Konzentration von 0,05 bis 0,5 Gew.-%.

10. Verfahren nach Anspruch 1, umfassend:

(a) Anwenden einer Vorbehandlungslösung, umfassend 0,25 bis 1,5 Gew.-% an Säure und 0,01 bis 1 Gew.-% an Oxidationsmittel ; und
(b) das Umlaufen einer alkalischen zweiten Lösung durch die Anlage im Anschluss an die Vorbehandlungslösung.

11. Verfahren nach Anspruch 10, wobei:

(a) die Säure wenigstens eine der folgenden ist: Phosphorsäure, Salpetersäure, Schwefelsäure, Milchsäure, Essigsäure, Hydroxyessigsäure und Citronensäure; und
(b) das Oxidationsmittel eine Persauerstoffverbindung ist.

12. Verfahren nach Anspruch 1, wobei die zweite Lösung Natriumhydroxid umfasst.

Revendications

1. Procédé de nettoyage de salissures protéiques et/ou de carbohydrate à partir d'un équipement industriel utilisant un procédé CIP, dans lequel la salissure provient de lieux de fabrication d'aliments et de boissons, le procédé comprenant :

(a) l'application d'une solution de prétraitement à la salissure, la solution comprenant au moins 0,10 % en poids d'ingrédients actifs, les ingrédients actifs comprenant un agent pénétrant et un agent oxydant choisis dans le groupe constitué de peroxyde d'hydrogène, de composés peroxygène et de mélanges de ceux-ci ;
(b) la recirculation d'une seconde solution à travers l'équipement après la solution de prétraitement, la seconde solution comprenant un détergent dilué, puis
(c) le rinçage de l'équipement.

2. Procédé selon la revendication 1, dans lequel la solution de prétraitement comprend en outre 0,10 à 1,5 % en poids d'acide.

3. Procédé selon la revendication 2, dans lequel la solution de prétraitement comprend 0,01 à 1 % en poids d'oxydant.

4. Procédé selon la revendication 3, dans lequel l'oxydant est un peroxyde.

5. Procédé selon la revendication 1, dans lequel la solution de prétraitement comprend de 0,4 à 10 % en poids d'agent pénétrant.

6. Procédé selon la revendication 2, dans lequel la solution de prétraitement comprend de 0,25 à 1,5 % en poids d'acide et 0,4 à 8 % en poids d'agent pénétrant.

7. Procédé selon la revendication 1, dans lequel la solution de prétraitement comprend de 0,3 à 10 % en poids d'une source d'alcalinité.

8. Procédé selon la revendication 1, dans lequel la solution de prétraitement comprend :

(a) 0,25 à 1,5 % en poids d'acide, étant au moins un parmi l'acide phosphorique, l'acide nitrique, l'acide sulfurique, l'acide lactique, l'acide acétique, l'acide hydroxy-acétique et l'acide citrique ; et
(b) 0,01 à 1 % en poids d'oxydant, étant un composé peroxygène.

9. Procédé selon la revendication 1, dans lequel l'oxydant est présent à un niveau de 0,05 à 0,5 % en poids.

10. Procédé selon la revendication 1, comprenant :

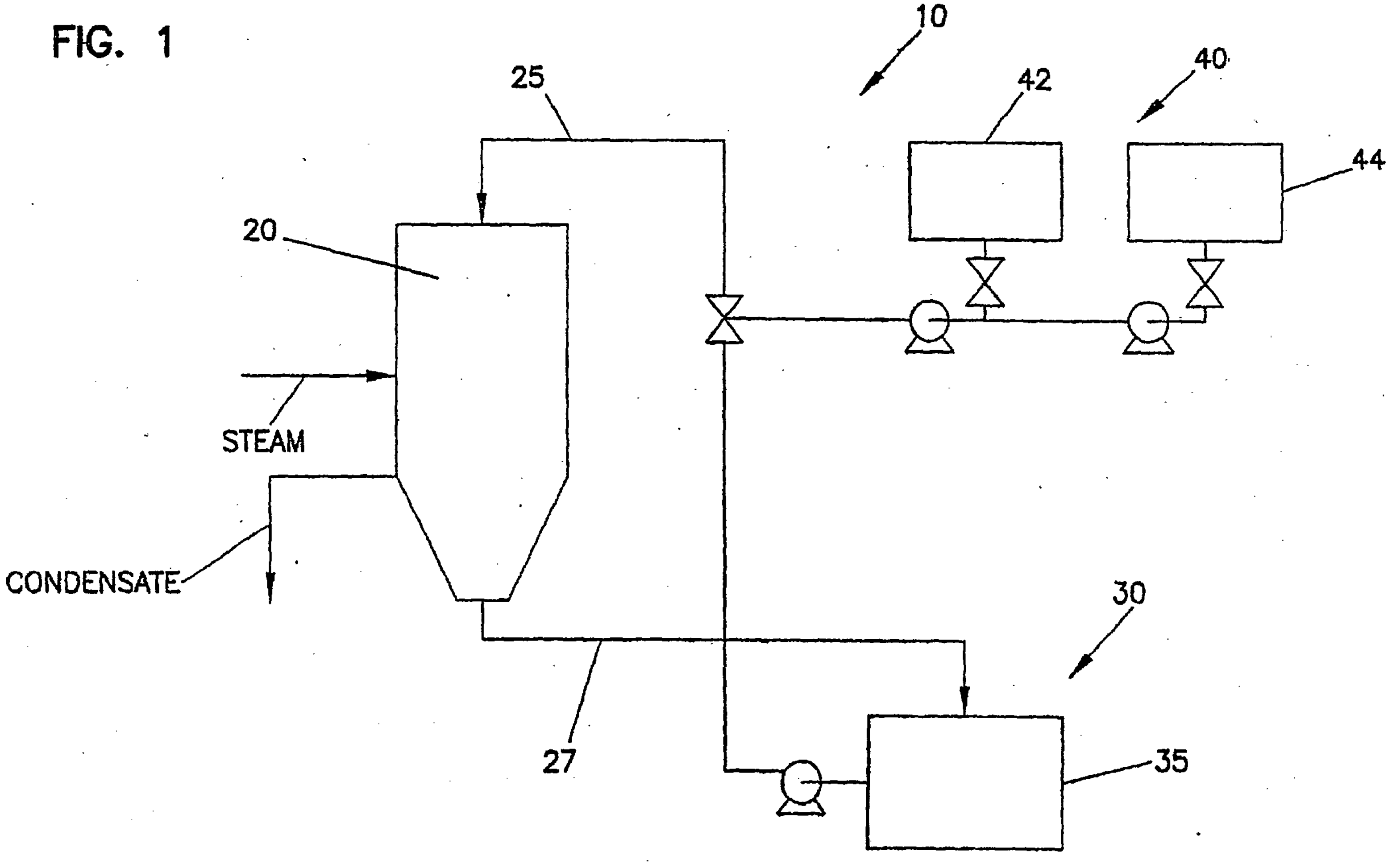
(a) l'application d'une solution de prétraitement comprenant 0,25 à 1,5 % en poids d'acide et 0,01 à 1 % en poids d'oxydant ; et
(b) la recirculation d'une seconde solution alcaline à travers l'équipement après la solution de prétraitement.

11. Procédé selon la revendication 10, dans lequel :

(a) l'acide est au moins un parmi l'acide phosphorique, l'acide nitrique, l'acide sulfurique, l'acide lactique, l'acide acétique, l'acide hydroxy-acétique et l'acide citrique ; et
(b) l'oxydant est un composé peroxygène.

12. Procédé selon la revendication 1, dans lequel la seconde solution comprend de l'hydroxyde de sodium.

FIG. 1



REFERENCES CITED IN THE DESCRIPTION

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- US 20020177220 A1 **[0008]**
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