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(54) **A method for washing a polycarbonate article**

(57) The present invention relates to a method for washing a polycarbonate (PC) article, wherein the PC article is washed with an acidic concentrate containing the following components:

a) at least one acid and

b) at least one compound selected from glycolic acid, benzoic acid, bromoacetic acid, sorbic acid, a peroxy acid, hydrogen peroxide, lactic acid, salicylic acid and caprylic acid.

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Description

[0001] The present invention relates to a method for washing a polycarbonate (PC) article by employing an acidic concentrate containing at least one acid and at least one compound having biocidal properties. The present invention further relates to the use of such an acidic concentrate or to a use solution thereof for washing a PC article.

[0002] Articles completely or partially made of PC have gained significant relevance as packaging material. PC articles are widely used as bottles, in particular 5-gallon water bottles, which bottles can be reused. Such PC bottles serve both as a supply for drinking water in households and as a water dispenser or so-called cooler. The latter application is not just another way of drinking water, in particular still drinking water, but rather more a question of life-style. Originating in the USA, but also well-known in continental Europe and Great Britain, coolers equipped with PC water bottles are widely spread in offices, fitness studios, department stores and similar service facilities to offer cooled water to consumers, clients or guests. Since such PC bottles are reusable, special methods have been developed for cleaning those bottles or any other PC article. In this respect it has to be considered that the detergent employed for cleaning such PC articles has to be compatible with PC. In addition, the cleaning has to be effective in respect of the removal of residuals left in the used PC article or any other containments, which can promote growth of micro-organisms.

[0003] US-A 4,505,836 discloses a process of cleaning PC surfaces of drinking water bottles comprising the step of washing the surface with a composition consisting essentially of an aqueous basic solution containing an alkali metal carbonate, an alkali metal bi-carbonate and a mixture of mono- and diesters of phosphoric acid. The aqueous basic solution is free of chlorine and may also be employed as a use solution further diluted with water. The washing step employing the aqueous basic solution is carried out at temperatures in the range of approx. 50 to 65°C (approx. 120-150°F). US-A 4,505,836 is a continuation-in-part of US-A 4,434,069, wherein an analogous aqueous basic solution is disclosed.

[0004] US-A 5,223,162 relates to the compositions for inhibiting stress cracking in articles made of poly (alkylene terephthalate) (PET) or PC during the washing of such articles, which are in particular PET bottles. The stress crack inhibitor is preferably a sodium salt of a hydrophilic-substituted aromatic hydrocarbon having either an alkyl or aryl side chain. The stress crack inhibitor is one component of an aqueous-based bottle washing concentrate further comprising an alkali metal hydroxide, a chelant or sequestrant and water. The bottle wash concentrate is prepared by mixing together the individual components at 120°F (approx. 50°C). The concentrate may be employed as a caustic detergent use solution by further dilution with water for washing the PET or PC articles. The tests for determining the effectiveness of the stress cracking inhibitors are carried out at approx. 25°C. However, US-A 5,223,162 does not explicitly disclose at which temperature the actual washing of the PET or PC articles is performed. Instead, it is generally known in the state of the art that for performing an effective cleaning and disinfecting of such articles in the alkaline range, elevated temperatures in the range of at least 50°C have to be employed.

[0005] Furthermore, the applicant is aware about a further prior art method for cleaning PC water bottles, which is widely used by companies performing the filling or re-filling of such bottles. According to this method, a first washing step is carried out at 60-65°C for 90-120 seconds by employing alkaline detergent compositions, which are optionally chlorinated. After such first washing step a disinfection or intermediate rinse step is carried out at 30°C for about 30 seconds followed by a final rinse. The disinfection step is carried out by employing a different detergent composition containing peracetic acid. The final rinse is usually performed by employing water optionally containing ozone (ozone-treated water).

[0006] However, the use of alkaline detergent compositions, in particular those additionally being chlorinated, is connected with several disadvantages. As indicated above, the washing has to be carried out at elevated temperatures to effect the cleaning, i.e. the removal of residuals or soils in the used bottles. Depending on the employed detergent composition, an additional composition for disinfecting (or sanitizing) the PC bottles has to be performed to guaranty the required reduction of micro-organisms to a value prescribed by the authorities. Usually, those two steps cannot be combined, since the respective compositions are often not compatible with each other, especially in respect of the intended usage due to different pH ranges.

[0007] Another major disadvantage of employing alkaline detergent compositions optionally being chlorinated is - as already indicated above - the stress cracking problem also known as stress-crack corrosion. Some of the components of the alkaline compositions penetrate the surface of the PC article causing corrosion by, for example, partially cleaving monomeric units of the PC article. PC bottles showing stress cracking cannot be reused anymore due to their deteriorated mechanical properties.

[0008] Besides alkalis such as sodium hydroxide further chemicals are known to promote stress cracking, for example, amines or surfactants. To avoid or at least to reduce stress cracking, alkaline detergent compositions usually contain corrosion inhibitors such as silicates. However, those corrosion inhibitors tend to precipitate on the surface of the PC bottles causing additional cleaning efforts in subsequent washing steps to eliminate said precipitate. Thus, it becomes clear that many of the commercially available detergent compositions are not really suited to be employed in the cleaning of PC articles, since they contain considerable amounts of one or more components causing or promoting stress cracking.

A further disadvantage of (chlorinated) alkaline products is the possible risk of leaving objectionable odor or taste inside the PC article during the respective washing step.

[0009] EP-B 1 444 316 discloses acidic sanitizing and cleaning compositions, which are useful in food harvesting and food and beverage processing operations. In particular, the compositions can be employed for hard surface cleaning and/or sanitizing of in-place or clean-in-place (CIP) or steam-in-place (SIP) assemblies. This means that said compositions are employed for cleaning and/or sanitizing machines or other equipment which are used in the food and beverage industry to fill containers with food or beverages, respectively. However, EP-B 1 444 316 does not disclose that said compositions may also be employed for the cleaning of the containers to be filled with food or beverages, respectively. The disclosed compositions comprise at least one aliphatic short chain antimicrobially effective C_5 - C_{14} fatty acid, at least one weak C_1 - C_4 carboxylic acid and a strong acid which is nitric acid or a mixture of nitric and phosphoric acid. Glycolic acid is an example for a weak C_1 - C_4 carboxylic acid. Furthermore, the composition may optionally contain additional components such as surfactants as demonstrated in most of the examples.

[0010] Further examples of acidic sanitizing and/or cleaning compositions for being employed in CIP applications are, for example, disclosed in WO 2005/073359 or EP-B 245 928. In both cases there are compositions disclosed comprising among other components a strong acid such as nitric acid or phosphoric acid and a further (weak) acid such as glycolic acid.

[0011] Furthermore, those compositions may contain additional components such as detergents as exemplified within the experimental section.

[0012] However, such acidic compositions useful for CIP applications as described above have until now not been connected with the cleaning of PC articles.

[0013] The object of the present invention is to provide a new method for washing PC articles more efficiently and economically, especially in respect of removing unwanted residues or soil, providing the required reduction of microorganisms, and avoiding or reducing stress cracking during the washing procedure.

[0014] The object is achieved by a method for washing a polycarbonate (PC) article, wherein the PC article is washed with an acidic concentrate containing the following components:

- a) at least one acid and
- b) at least one compound selected from glycolic acid, benzoic acid, bromoacetic acid, sorbic acid, a peroxy acid, hydrogen peroxide, lactic acid, salicylic acid and caprylic acid.

[0015] A major advantage of the method according to the present invention is that time and/or costs can be saved, since it is a single stage process in respect of the cleaning (removal of residues) and disinfecting (sanitizing; causing the antimicrobially effect). By employing (chlorinated) alkaline detergent composition, usually a two stage process has to be carried out to achieve a comparable efficiency (efficacy) in terms of cleaning and disinfecting. Furthermore, a typical alkaline washing of PC articles has to be performed at elevated temperatures, whereas the process according to the present invention can be carried out at ambient temperatures, for example, in a range between 15 and 25°C.

[0016] Another advantage compared to the alkaline detergent compositions known from the state of the art is that the stress cracking problem can be avoided or at least significantly reduced due to the acidic conditions and/or the preferably employed lower temperatures. There is no need to add some stress cracking inhibitors to the detergent composition, which cause additional washing steps to remove any precipitate therefrom. The acidic concentrate employed in the process according to the present invention is compatible with PC bottles, in addition no objectionable odor or taste inside the bottles is caused by it. The acidic concentrate employed in the process of the present invention has a strong microbiological efficiency, especially in respect of the cleaning of PC bottles which are low to moderately soiled, whereby the soil is preferably of inorganic origin.

[0017] Subsequently, the method for washing a PC article according to the present invention is explained in detail.

[0018] The polycarbonate (PC) article to be washed may be any article completely or at least partially made of PC. Articles which are only partially made of PC comprise both articles, where some individual parts are made of PC and the remaining parts are made of different materials, and articles, which are covered or coated by a PC. Preferably, the PC article is a container, more preferably a bottle, in particular a 5-gallon bottle which can be reused or refilled, respectively. Such bottles are preferably filled with drinking water, in particular with still (non-gaseous) drinking water. These bottles preferably serve as a water dispenser or so-called cooler. A preferred PC is Makrolon® which is the trade name for a commercially available PC from Bayer (Leverkusen, Germany).

[0019] The acidic concentrate employed in the washing step of the PC article contains the components indicated in the following. It has to be indicated that the below listed components are listed with their chemical structure/name before mixing the individual components with each other to prepare the concentrate or any other composition derived thereof, such as a use solution. Due to the presence of further components, an individual component may, for example, be partially or completely transferred into a corresponding salt, base or acid, respectively. This also relates to the indicated proportions of the individual components, which refer to the individual components as starting materials (educts) before

mixing them with each other.

The component a):

[0020] The acidic concentrate contains as component a) at least one acid. The acid is preferably a strong acid, preferably having a $\text{pH} \leq 3$, more preferably ≤ 2 , which strong acid may be an organic or an inorganic acid. The definition of component a) does not include any compounds as defined for component b). This means that component a) differs from component b). More preferably, the acid is selected from formic acid, acetic acid, citric acid, an alkyl sulfonic acid, phosphoric acid, sulfuric acid, nitric acid, hydrochloric acid and sulfamic acid.

[0021] Methane sulfonic acid is an example of a preferred alkyl sulfonic acid. Nitric acid is preferably employed in combination with a small amount (preferably about 1 wt.-%) of urea to prevent NO_x formation.

[0022] Most preferably, the acid is nitric acid and/or phosphoric acid, in particular nitric acid.

[0023] The acidic concentrate contains component a) generally in an amount between 0.1 and 99 wt.-% (percent by weight). Said amount of component a) relates to its weight as contained in the respective concentrate. Any further compounds contained in the respective acid employed as educt of the concentrate, such as additional water in case of diluted acids, are not comprised by these numbers. All numbers are based on the total amount (100 wt.-%) of the respective concentrate. The same applies for all other components of the acidic concentrate or any other composition within the context of the present invention unless indicated otherwise. Preferably, component a) is present in an amount of 0.5 to 75 wt.-%, more preferably in an amount of 15 to 50 wt.-%.

The component b):

[0024] The acidic concentrate contains as component b) at least one compound selected from glycolic acid, benzoic acid, bromoacetic acid, sorbic acid, a peroxy acid, hydrogen peroxide, lactic acid, salicylic acid and caprylic acid.

[0025] It is assumed that component b) is biocidal active, which means that it is stable under pH-conditions less than 7, preferably ≤ 2 , and causes the microbiological efficacy during the sanitizing or disinfecting step (antimicrobially activity). Besides the before-listed compounds any other compound which is biocidal active and stable under pH-conditions less than 7, preferably ≤ 2 can also be employed as component b) or in addition to the above-listed components.

[0026] A peroxy acid may be any peroxy acid known by a skilled person, for example, a percarboxy acid. Preferred peroxy acids are peroxyoctanoic acid, percitric acid and peracetic acid. In one embodiment of the present invention, the peroxy acid is employed together with hydrogen peroxide.

[0027] Component b) is more preferably selected from glycolic acid (also known as hydroxy acetic acid), lactic acid, salicylic acid and caprylic acid.

[0028] Component b) is most preferably glycolic acid.

[0029] The acidic concentrate contains component b) generally in an amount of 0.1 to 50 wt.-%, preferably in an amount of 0.5 to 25 wt.-%, more preferably in an amount of 2 to 10 wt.-%.

The component c):

[0030] The acidic concentrate optionally contains as component c) water. Preferably, component c) is softened water.

[0031] The acidic concentrate contains component c) in an amount of 0 to 90 wt.-%. If present, component c) is contained in an amount of at least 0.1 wt.-%. A value of 1 to 70 wt.-% is preferred, 4 to 68 wt.-% are more preferred and 20 to 60 wt.-% are most preferred.

[0032] In a preferred embodiment of the present invention, the acidic concentrate contains component c). In this embodiment, component c) may completely or partially be separately added to the acidic concentrate, preferably as softened water, or the water may completely or partially be already contained in the educts employed as components a) and/or b). The latter may be the case when, for example, diluted acids are employed.

The component d):

[0033] The acidic concentrate optionally contains as component d) at least one compound selected from urea, a surfactant, an oxidiser, a solubilizer, and a defoamer. Surfactants, oxidisers, solubilizers or defoamers are known to a person skilled in the art. A solubilizer may be, for example, an organic solvent.

[0034] Component d) is more preferably urea.

[0035] The acidic concentrate contains component d) generally in an amount of 0 to 20 wt.-%. If present, component d) is contained in an amount of at least 0.1 wt.-%. A value of 0.1 to 10 wt.-% is preferred, a value of 0.2 to 5 wt.-% is more preferred.

[0036] The acidic concentrate may be prepared according to any method known in the state of the art. Preferably, the

individual components are mixed together until the obtained composition is homogenous. The individual components may be mixed in any order. In one embodiment of the present invention, the mixing process is started with water.

[0037] In one embodiment of the present invention, the acidic concentrate is free or substantially free of surfactants and/or defoamers. In this embodiment, surfactants and/or defoamers are not comprised by the definition of component d).

[0038] A preferred acidic concentrate employed in the present invention contains 30 to 50 wt-% of component a), 0.5 to 25 wt-% of component b), 4 to 68 wt-% of component c) and 0.2 to 5 wt-% of component d).

[0039] The acidic concentrate may have any pH-value < 7, preferably < 5, more preferring ≤ 3 and most preferably ≤ 2 .

[0040] The method for washing the PC article may be carried out in any apparatus known in the state of the art, for example, in commercially available washing machines. Commercial washing machines are, for example, produced by R. Bardi s.r.l. (Fidenza, Italy).

[0041] The washing of the PC article (washing step) may be carried out at any temperature and/or for any period of time. Preferably, the washing step with the acidic concentrate is carried out at a temperature in the range of 10 to 30°C, more preferably in the range of 15 to 25°C and/or for a period of 1 to 10 minutes, more preferably for a period of 3 to 6 minutes.

[0042] Preferably, the washing step with the acidic concentrate is a cleaning and/or sanitizing step. Preferably, the PC article is a container, preferably a bottle. The container may be a used container, which may be low, moderately or heavily soiled. The soil may be of any origin. Preferably, the method according to the present invention is employed for the cleaning of low to moderately soiled containers, preferably used bottles and/or the soil is of inorganic origin, for example mineral deposits.

[0043] In one embodiment of the present invention the washing step is carried out by employing a use solution. Said use solution contains the above-explained acidic concentrate and at least one solvent. Said solvent is preferably water, which may be hard or softened water. The use solution is obtained by diluting the acidic concentrate with the solvent by a dilution factor of 1 to 1000, preferably by a dilution factor of 1 to 100.

[0044] Subsequently to the washing step with the acidic concentrate a rinse step may be carried out with water optionally containing ozone (ozone-treated water). In addition, further washing and/or rinse steps may be carried out prior to or after the washing step with the acidic concentrate or the use solution containing the acidic concentrate. For example, the respective PC article may simultaneously or subsequently be washed with any other detergent composition known in the state of the art, which is compatible with the acidic concentrate according to the present invention or its use solution.

[0045] Another subject of the present invention is the use of the acidic concentrate as described above or of a use solution containing the acidic concentrate and at least one solvent, preferably water, for washing a PC article. Preferably, the PC article is a container, preferably a bottle, which container is preferably low to moderately soiled and/or the soil is of inorganic origin. The washing may be a cleaning and/or sanitizing step.

[0046] The following examples serve to present a more detailed explanation of the invention.

Examples

[0047] In the following all percent (%) -values of components of compositions are expressed as percent-by-weight (wt.-%) unless indicated otherwise.

Compositions according to the present invention

[0048]

Composition A:

Component	As supplied	As 100%
water (softened)	0.60000	0.60000
nitric acid (53%)	94.20000	49.92600
urea	0.20000	0.20000
glycolic acid (70%)	5.00000	3.50000

[0049] Mix the ingredients in the order given, mix until homogeneous.

PC stability trials:

[0050] Visual comparison after a Bayer (Leverkusen, Germany) bend strip test

Test conditions and procedure:**[0051]**

- 5 Concentration: 1,0% w/w of the respective composition diluted in deionized water (unless indicated otherwise)
 Temperature: ambient temp. (24h) or 60°C (6h)
 Strain level: 0,9%

- 10 **[0052]** The polycarbonate coupons are clamped to stainless steel multi-strain fixtures. The coupons are put into the detergent solution (1,0% w/w) at ambient temperature for 24 hours / 6 hours at 60°C

Table I:

No.	Descr.	pH	Temp.	duration	0	A	B	C	D
15	1	Composition A: (as defined above)	1,3	22°C	24h	X			
20	2	Composition B: Mildly alkaline containing sodium trisilicate (5,2%), EDTA sodium salt (9%), sodium (1,65%) and potassium hydroxide (2,5%), up to 100% softened water	12	22°C	23h			X	
25	3	Composition C: Mildly alkaline containing sodium trisilicate (5,2%), EDTA sodium salt (9%), sodium (1,65%) and potassium hydroxide (2,5%), sodium cumene sulphonate (2,2%), alcohol alkoxyate (EO/PO)(1%), up to 100% softened water	12	22°C	23h			X	
30	4	Composition D: sodium hydroxide	>13	22°C	23h				X
35	5	Composition E: Chlorinated alkaline containing sodium trisilicate (2,55%), sodium (11%) and potassium triphosphate (6,5%), potassium hydroxide (2,75%) and sodium hypochlorite (2%), up to 100% softened water	11	22°C	19h			X	
40	6	Composition F: employed at a concentration of 0,1%(w/w) Detergency booster additive based on alcohol (C13-15) alkoxyate (EO/BO)(100%)	7	22°C	24h		X		
45	7	Composition A: (as defined above)	1,3	60°C	6h	X			
	8	Composition B: (as defined above)	12	60°C	6h			X	
	9	Composition C: (as defined above)	12	60°C	6h			X	
	10	Composition D: (as defined above)	>13	60°C	6h			X	
	11	Composition E: (as defined above)	11	60°C	6h			X	
50	0: no damages A: minor, very shallow cracks B: moderate, shallow cracks C: major, moderately deep cracks D: major deep cracks EO: ethylene oxide; PO: propylene oxide; BO: butylene oxide EDTA: ethylene diamine tetraacetic acid								

- [0053]** From Table I it can be seen that PC articles (PC coupons) experience much less stress cracking by a washing

process according to the present invention, both at higher and ambient temperatures, compared to methods employing alkaline detergent compositions. Since the PC articles to be washed experience much less stress cracking, they may be refilled more often.

5 Microbiological efficacy:

[0054] The above-listed composition A is tested on its efficacy by employing the EN 1276 standard test of the European Committee for Standardization (CEN). EN1276: passed at 1.0% dilution in hard water (300ppm CaCO₃), clean conditions (0.3g/l bovine albumin) and 1 minute contact time (log 5 reduction of the respective bacteria) at 20°C. Test bacteria E.coli and P.aeruginosa.

Claims

- 15 1. A method for washing a polycarbonate (PC) article, wherein the PC article is washed with an acidic concentrate containing the following components:
 - a) at least one acid and
 - 20 b) at least one compound selected from glycolic acid, benzoic acid, bromoacetic acid, sorbic acid, a peroxy acid, hydrogen peroxide, lactic acid, salicylic acid and caprylic acid.
- 25 2. A method according to claim 1, wherein component a) is selected from formic acid, acetic acid, citric acid, an alkyl sulfonic acid, phosphoric acid, sulfuric acid, nitric acid, hydrochloric acid and sulfamic acid and/or component b) is selected from glycolic acid, lactic acid, salicylic acid and caprylic acid.
- 30 3. A method according to claim 1 or 2, wherein the acidic concentrate further contains the components
 - c) water and/or
 - d) at least one compound selected from urea, a surfactant, an oxidiser, a solubilizer, and a defoamer
- 35 4. A method according to any of claims 1 to 3, wherein the acidic concentrate is substantially free of surfactants and/or defoamers.
5. A method according to any of claims 1 to 4, wherein the washing step with the acidic concentrate is carried out at a temperature in the range of 10° to 30°C and/or for a period of 1 to 10 minutes.
6. A method according to any of claims 1 to 5, wherein subsequently to the washing step with the acidic concentrate a rinse step is carried out with water optionally containing ozone.
- 40 7. A method according to any of claims 1 to 6, wherein
 - i) the washing step with the acidic concentrate is a cleaning and/or sanitizing step,
 - ii) the PC article is a container, preferably a bottle, which container is preferably low to moderately soiled and/or the soil is of inorganic origin, and/or
 - 45 iii) the washing step is carried out with a use solution containing the acidic concentrate and at least one solvent, which solvent is preferably water, and the acidic concentrate is diluted with the solvent by a dilution factor of 1 to 1000.
- 50 8. A method according to any of claims 1 to 7, wherein component b) is at least one compound, which is biocidal active and stable under pH-conditions less than 7.
9. Use of an acidic concentrate or of a use solution containing the acidic concentrate as defined in any of claims 1 to 8 for washing a PC article.
- 55 10. Use according to claim 9 wherein the PC article is a container, preferably a bottle, which container is preferably low to moderately soiled and/or the soil is of inorganic origin, and/or the washing is a sanitizing step.



European Patent
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EUROPEAN SEARCH REPORT

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EP 06 02 6530

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Place of search The Hague		Date of completion of the search 16 May 2007	Examiner Neys, Patricia
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**ANNEX TO THE EUROPEAN SEARCH REPORT
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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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