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Detergent compositions.

Machine dishwashing is carried out using a main wash composition assembled by the user (or by dispensers fitted to the machine) from two or more compositions, so that the composition is better suited to the degree of soiling of the wash load. Each composition contains a substantial proportion of salts. This avoids requiring the user to dispense small quantities or to handle small unit doses of "additive". The compositions differ in proportions of enzyme and/or bleach. On some occasions one composition may be used alone.

This invention relates to the washing of cooking and eating utensils by use of a mechanical dishwasher. The conventional procedure with such a machine is that the machine load is washed with a wash liquor formed from water and a cleaning composition. Subsequently the wash load is rinsed, and for this it is very desirable that a rinse aid is added to the water used for rinsing. For both the washing and rinsing steps the water coming into the machine customarily contacts ion exchange resin in order to soften the water. This is very desirable in order to obtain good cleaning performance.

The present invention is concerned with the washing step and the cleaning composition used in that step.

The cleaning composition which is used in the washing step customarily contains a substantial quantity of salts including detergency builder and an inorganic silicate. The cleaning composition may well include some synthetic detergent although the amount of this detergent may be quite small. The cleaning composition may also include enzyme(s) to assist in the removal of soil and/or a bleaching system. Enzyme and/or bleach included in the composition may well represent only a small percentage of the composition. Compositions are generally marketed as all purpose compositions suitable for all wash loads.

We have now appreciated that it could be desirable to provide some way for the user to vary the cleaning composition according to the degree of soiling of the load in the machine. This could avoid using unnecessary quantities of components of the composition when not required (for example for cleaning a lightly soiled load). Moreover by avoiding unnecessary usage on lightly soiled and moderately soiled loads, it becomes more acceptable to provide a more powerful and correspondingly more expensive composition for use on heavily soiled loads because the user can see that the extra expense is being used only when required.

However, we have also appreciated that it is not attractive to provide a basic composition for use on all occasions together with additives for use only when required. The additives for use only when required would be enzymes and/or bleaches. These are only a relatively small amount of a detergent composition, and a user cannot easily dispense small quantities with sufficient accuracy. Formulating the additive materials as some kind of unit dose is also not attractive. Small unit doses are unfamiliar in the context of washing dishes and crockery and therefore run the risk of being perceived as valueless or on the other hand dangerously concentrated.

Accordingly, the present invention provides a plurality of compositions which each contain a substantial proportion of organic and/or inorganic salts but nevertheless differ, probably in their proportions of enzyme and/or bleaching components. The user would then choose one composition, a different composition or a mixture of compositions for washing loads of different characteristics, such as different degrees of soiling.

It is an inherent characteristic of this invention that on at least some occasions the user will make use of a mixture formed from a plurality of compositions.

A first aspect of the present invention provides a method for washing culinary utensils, eg. cooking utensils and also eating utensils such as table crockery and glassware, in a mechanical dishwasher characterised by dispensing into the dishwasher quantities of a plurality of cleaning compositions out of respective separate containers, and operating the dishwasher so that the dispensed quantities of compositions are all mixed into the same wash liquor, the compositions being different from each other but each composition containing at least 50% by weight of organic and/or inorganic salt(s).

Thus in the above method, a mixture of at least two compositions is employed to form a single wash liquor. For some occasions of use one or other of the individual compositions may be employed alone. Mixtures in different proportions may be employed on other occasions.

For instance, one composition might be designed to be suitable for lightly soiled loads, another composition might be suitable for particularly heavily soiled loads while a mixture of the two compositions is suitable for washing a load with an intermediate level of soiling.

Alternatively two compositions may be designed with the intention that the compositions are always used in admixture but the proportions which are mixed are deliberately varied according to the degree of soiling of the load. For instance quantities of two compositions might be utilised in 3:1, 2:2 and 1:3 weight ratios for lightly soiled, intermediate and heavily soiled loads respectively.

When two compositions are used to form a mixture in a single wash liquor it is preferred that the proportions of the compositions which are dispensed lie in a weight ratio within a range from 5:1 to 1:5.

In general a method according to the invention can be additionally defined as comprising:

- i) placing a first load of utensils in the dishwasher, supplying a first cleaning formulation to the dishwasher and operating the dishwasher to mix the said formulation into water to form a wash liquor used during the washing phase;
- ii) subsequently to washing (and probably also rinsing and removing) the first load, placing a second load of utensils in the dishwasher, supplying a second cleaning formulation to the dishwasher and operating

the dishwasher to mix the said second formulation into water to form a wash liquor used during the washing phase;

each of the formulations being provided by dispensing from at least one of a plurality of cleaning compositions held in separate containers, and at least one of the formulations being provided by dispensing quantities of a plurality of the said compositions, with the said quantities being mixed into the same wash liquor,

the compositions being different from each other but each composition containing at least 50% by weight of organic and/or inorganic salt(s) which are other than bleaching components, enzymes and detergent surfactants.

The compositions used in this invention must, as stated, differ from each other. It is envisaged that the differences between them will generally concern enzyme(s) and/or bleaching components or both. For instance enzyme may be present in one composition but absent from another, or may be present in both but in different amounts. As for the salt(s) which provide the majority proportion of each composition, it is not essential that these be the same in all compositions although this may be the case. It is possible that one composition does not contain any enzyme or bleach so that at least one composition could consist of 100% by weight of salt(s) other than enzymes, bleaches and detergent actives.

The salts, included to an extent of at least 50% by weight of each composition, will generally include water-soluble salts and indeed will often be wholly water-soluble. However it is conceivable that water-soluble salts could be present as part of the required quantity, or in addition thereto.

It is preferred that the salt(s) in a composition are, or include, 20-50% of a sodium silicate and from 30-60% by weight of a detergency builder other than silicate.

In a further aspect this invention provides a plurality of compositions for machine dishwashing, in separate containers associated together, the compositions being different from each other, but each composition containing at least 50% by weight of organic and/or inorganic salt(s) other than bleaching components, enzymes and detergent surfactants.

Association of the containers could be by a package or wrapper which surrounds them and holds them together. Another possibility is that the containers are separate storage compartments of apparatus for dispensing the compositions. Such apparatus could itself be a part of a dishwasher. It may be noted that in both of these possibilities the containers are attached together.

Various features and preferred features of the invention will now be discussed by turn.

Water-soluble salts

As already mentioned, the compositions of the present invention preferably contain 20-50% by weight of an alkali metal silicate. This material is employed as a cleaning ingredient, source of alkalinity, metal corrosion inhibitor and protector of glaze on china tableware. Especially effective is sodium silicate having a ratio of $\text{SiO}_2:\text{Na}_2\text{O}$ of from about 1.0 to 3.3, preferably from about 2 to 3.2. Useful is sodium silicate having a ratio of $\text{SiO}_2:\text{Na}_2\text{O}$ of 2.0, also referred to as sodium disilicate (i.e. $\text{Na}_2\text{Si}_2\text{O}_5$).

It is also preferred that a water-soluble detergency builder is present in an amount from 30-60% by weight of the composition or more preferably from 35-50% by weight. Alkali metal phosphates and condensed phosphates, especially sodium tripolyphosphate are suitable. However, phosphates are now frequently avoided for environmental reasons. A preferred water-soluble builder is alkali metal citrate. Other water-soluble detergency builders which may be used alone or in combination, include the alkali metal, ammonium and substituted ammonium salts of carbonates, bicarbonates, sesquicarbonates and organic polyacetates, carboxylates, polycarboxylates, polyacetyl carboxylates and polyhydroxysulphonates. Specific examples of organic builders include sodium, potassium, lithium, ammonium and substituted ammonium salts of ethylenediaminetetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, mellitic acid and benzene polycarboxylic acids as well as citric acid which has already been mentioned. Further possibilities are tartrate monosuccinates, tartrate disuccinates, dipicolinic acid, chelidamic acid, carboxy methyloxysuccinate and hydroxyethyliminodiacetic acid.

Non-phosphate detergency builder may be accompanied by polymer containing carboxylic or sulphonic acid groups in acid form or wholly or partially neutralised, the sodium salts being preferred. Preferred polymers are homopolymers and copolymers of acrylic acid and/or maleic acid or maleic anhydride. The molecular weights of such homopolymers and copolymers are generally 1000 to 150,000, preferably 1500 to 100,000. A suitable amount is from 0.5 to 10% by weight of the composition. In particular, if citrate is employed as builder it is preferred that the composition also contains 2 to 10% by weight of a polycarboxylate polymer such as sodium polyacrylate.

It is possible that insoluble detergency builder could be included, notably insoluble aluminosilicate builder.

Bleach system

Compositions according to the present invention may contain bleaching components. If present the amount will preferably lie in a range from 1 to 30% by weight. Alkali metal hypochlorite may be incorporated in liquid compositions. Other chlorine bleaches which may be incorporated are alkali metal salts of di- and tri-chloro and di- and tri-bromo cyanuric acids.

Another type of bleach which may be employed is the peroxygen bleaches, notably sodium perborate, persulphate and percarbonate can serve as hydrogen peroxide sources. Such peroxygen bleaches are preferably accompanied by a bleach activator which allows the liberation of active oxygen species at a lower temperature. A preferred bleach activator is tetraacetyl ethylene diamine (TAED) but other activators for perborate are known and can be used. The amounts of sodium perborate and bleach activator in an individual composition preferably do not exceed 20% and 15% by weight respectively.

Further peroxygen bleaches which may be used are alkyl, alkenyl and aryl peroxy organic acids and their metal salts. Typical peroxy acids include

- (i) peroxybenzoic acid and ring-substituted peroxybenzoic acids, e.g. peroxy- α -naphthoic acid
- (ii) aliphatic and substituted aliphatic monoperoxy acids, e.g. peroxy lauric acid and peroxy stearic acid
- (iii) 1,12-diperoxydodecanedioic acid
- (iv) 1,9-diperoxyazelaic acid
- (v) diperoxybrassylic acid; diperoxysebacic acid and diperoxyisophthalic acid
- (vi) 2-decyldiperoxybutane-1,4-dioic acid
- (vii) phthaloyl amino peroxy caproic acid (PAP).

A further development of this invention, applicable particularly when a bleach is used, is to arrange that two compositions which are to be used as a mixture do not enter the wash water simultaneously, and take advantage of conditions which occur transiently before the second composition enters the wash water.

For instance, a first composition could contain perborate bleach and the activator TAED, and generate a pH of about 11 when it enters the water. This is optimum pH for the reaction to form peracetic acid. The second composition could reduce the pH to about 9, which is optimum for the bleaching action of the peracid.

Sequential entry to wash water could be achieved by dispensing in succession from separate storage compartments, or putting the compositions into a dispenser compartment in a specific order.

Enzymes

Enzymes which are known to be suitable for use in machine dishwashing compositions include both proteolytic and amylolytic enzymes. These may be used in combination.

Proteolytic enzyme (also termed protease) can, for example, be used in an amount ranging from about the order of 0.0002 to about the order of 0.05 Anson units per gram of the detergent composition. Expressed in other units, the protease can also be included in the compositions in amounts of the order of from about 500 up to 10^5 Glycine units (GU)/gram of the composition. Preferably, the amount ranges from 10^3 up to 5×10^4 and particularly preferably from 2000 or even 5000 to 15000 or 20000 GU/gram of composition.

A GU is a Glycine Unit, defined as the proteolytic enzyme activity which, under standard conditions, during a 15-minute incubation at 40 °C with N-acetyl casein as substrate, produces an amount of NH₂-group equivalent to 1 micromole of glycine.

Enzyme activities are sometimes also measured in kilo Novo units (KNPU) : a measurement depending on the type of protease and assay used. We have found that the KNPU/AU ratio is in the range of about 3:1 to 5:1 for Alcalase, Esperase and Savinase and for the purpose of these formulations it is not necessary to be more precise.

Preferred examples of protease enzyme to be used in the present composition are the subtilisin varieties sold as Savinase (TM of Novo-Nordisk A/S) or Maxacal (TM of Gist-Brocades/IBIS) or as Opticlean (ex MKC) or AP122 (ex Showa Denko), which has pI approximately 10. Other useful examples of proteases include Maxatase, Esperase, Alcalase (Trade Marks), and subtilisin BPN'. Protinase K can also be used.

Suitable examples of amylolytic enzymes are commercially available amylases such as Maxamyl[®] (ex Gist-Brocades) and Termamyl[®] (ex Novo Industri A/S). A route for the production of amylases is described in UK patent 1296839 (Novo). Amylases may be included in diswashing compositions in amounts ranging

from 1 to 1000 Maltose units per gram of composition, preferably 2 to 200, more preferably 5 to 100 MU/gram. Measurement of amylolytic activity is described by P. Bernfield in "Methods of Enzymology" Vol 1 page 149 (1955). Other enzymes may also be included. For instance examples of commercial lipolytic enzymes are e.g. Lipase YL, Amano CE, Wallerstein AW, Lipase My etc. and typical examples of
 5 cellulolytic enzymes are cellulases ex Humiscla insolens as described in German patent application 3,117,250.

Optional components

10 Compositions for use in the present invention will in general contain some surfactant. It is well known for machine dishwashing compositions to contain a small percentage, typically as little as 1%, of a low foaming nonionic surfactant. Such surfactants will generally contain both ethylene oxide and propylene oxide residues. There is a wide range of such surfactants. Examples of suitable and preferred low to non-foaming nonionic surfactants for use in the invention are the ethoxylated and propoxylated straight chain alcohols
 15 sold under the trade names of Plurafac^R RA 30, Plurafac^R RA 40 and Plurafac^R LF 403 by the Eurane Company, Lutensol^R LF 403 and Lutensol^R LF 1300 by the BASF Company, and Triton^R DF 112 by the Rohm & Haas Company.

Other surfactants may be employed either to inhibit foaming or to function as detergents. Surfactants are preferably chosen from the conventional classes of nonionic, anionic and amphoteric surfactants. The
 20 amount of surfactant in an individual composition preferably lies in the range from 0.1-10% by weight of the composition.

A material which is desirably included is a layered clay, for the purpose of improving the appearance of machine washed glassware, as disclosed in our European Patent Application 139,329.

25 Compositions used in accordance with the present invention may contain other materials conventionally included in machine dishwashing compositions. Possibilities include perfume, phosphonates to act as transition metal complexants and anti-scaling agents and on the other hand transition metal salts such as zinc sulphate to act as anti-corrosion agents for glassware.

Form of the Compositions

30 The compositions for this invention may be in solid or liquid form or a semi-solid such as a gel or a paste. Solid forms may be preferred. A solid may be particulate, as is customary, but could also be in the form of shaped articles such as tablets. A user might then use three tablets of one composition and one of another, or two of each, for instance, with all the tablets being substantially equal in size and weight.

35 The compositions may be coloured, such as with dye or by coating at least some particles with a coloured outer layer. The compositions may be given distinctive different colours to reduce the chance that a user makes a mistake when dispensing the compositions into the dishwasher.

Packaging and dispensing

40 Compositions according to this invention can be packed in containers suitable for retail sale which may be conventional packages made from fibre board or moulded from thermoplastic. Dispensing into a machine may consist of putting appropriate quantities of each of two compositions into a dispensing compartment of a dishwasher. If the compositions are both particulate solids, the compositions will become mixed together
 45 when they are washed into the main body of the dishwasher during the normal operation of that machine.

As briefly referred to earlier, it is also conceivable that the present invention could be implemented by means of a mechanical dishwasher fitted with compartments for the storage of bulk quantities of cleaning compositions in which case the containers from which quantities are dispensed into the machine would be these storage compartments.

50 It is envisaged that containers of compositions for use in the invention would be marketed under a common trademark and with sufficient similarity of appearance of the packaging to make clear that they could be used together. For retailing it will probably be appropriate to market packs consisting of one or more containers of each composition, enclosed in an outer package or wrapper. However, a user will rarely if ever finish all containers simultaneously, and so it will probably be appropriate to sell the compositions
 55 individually as well.

Examples

All of the compositions of the following examples contain sodium disilicate. Preparation of the compositions began by spraying the liquid constituents, which were nonionic detergent and perfume, onto sodium disilicate in a pan granulator. This premix was then mixed with the other solid ingredients using a Y cone blender.

All of the compositions made in this way were particulate solids having a bulk density lying between 800 and 1,000 grams per litre. All of them were free flowing as solid powders and dissolved in water at a satisfactory rate. When added to distilled water at a concentration of 1% by weight each composition gave a pH in the range from 10.7 to 11.1. When added to distilled water at a concentration of 10% by weight each composition gave a pH in the range from 11.3 to 12.0.

Example 1

Two compositions (referred to as Example 1A and Example 1B) were prepared. They contained the materials set out in Table 1 below. As can be seen from that Table, the compositions both contained enzymes in equal amounts but the composition 1B contained a high level of sodium perborate and TAED as a bleaching system with a correspondingly reduced content of sodium citrate.

The compositions of Examples 1A and 1B are suitable for lightly soiled, intermediate and heavily soiled wash loads to be washed using the compositions in proportions of 3:1, 1:1 and 1:3 respectively. The overall formulations achieved by these proportions are also included in Table 1.

The composition 1A could be used alone for lightly soiled wash loads, if desired. The composition 1B could be used alone as a strong bleaching composition for heavily soiled loads, especially in areas where the water supply is soft, so that the fairly low level of citrate builder is not detrimental.

TABLE 1

INGREDIENT	COMPOSITION (% BY WEIGHT)		MIXTURES (OVERALL % BY WEIGHT)		
	EXAMPLE 1A	EXAMPLE 1B	1A:1B = 3:1	1A:1B = 1:1	1A:1B = 1:3
Sodium Disilicate	36.0	30.0	34.5	33.0	31.5
Nonionic (Plurafac 403)	1.5	1.1	1.4	1.3	1.2
Sodium Citrate	54.3	35.7	49.6	45.0	40.4
Sodium polyacrylate	4.0	8.0	5.0	6.0	7.0
Clay	2.0	1.0	1.75	1.5	1.25
Perfume	0.2	0.2	0.2	0.2	0.2
Sodium Perborate Monohydrate	--	15.0	3.75	7.5	11.25
TAED	--	6.0	1.5	3.0	4.5
Savinase	1.5	1.5	1.5	1.5	1.5
Amylase	1.5	1.5	1.5	1.5	1.5
	----- 100.0	----- 100.0	----- 100.0	----- 100.0	----- 100.0

Example 2

Two compositions (2A and 2B) were prepared. They were as set out in Table 2 below. Both compositions contained bleach components in equal amounts. Composition 2B contained a high level of

enzymes.

Composition 2A is suitable for washing lightly soiled loads (although a 3:1 mixture of 2A and 2B could also be used). A 1:1 mixture of both compositions would be used for normal loads. A 1:3 mixture of 2A and 2B would be used for heavily soiled loads. Alternatively composition 2B could be used alone for this. The overall formulations achieved by 1:1 and 1:3 mixtures are included in Table 2.

Since both compositions contain equal amounts of bleach components, it is ensured that all wash liquors would contain bleach to act against microorganisms.

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TABLE 2

INGREDIENT	COMPOSITION (% BY WEIGHT)		MIXTURES (OVERALL % BY WEIGHT)	
	EXAMPLE 2A	EXAMPLE 2B	2A:2B = 1:1	2A:2B = 1:3
Sodium Disilicate	38.0	32.0	35.0	33.5
Nonionic	1.6	2.0	1.8	1.9
Sodium Citrate	41.8	38.4	40.1	39.25
Sodium polyacrylate	4.0	8.0	6.0	7.0
Clay	2.5	1.5	2.0	1.75
Perfume	0.1	0.1	0.1	0.1
Sodium Perborate Monohydrate	8.0	8.0	8.0	8.0
TAED	4.0	4.0	4.0	4.0
Savinase	--	2.0	1.0	1.5
Amylase	--	4.0	2.0	3.0
	----- 100.0	----- 100.0	----- 100.0	----- 100.0

Example 3

Two compositions (3A and 3B) were prepared. The materials contained in them are listed in Table 3 below.

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Composition 3A contains neither bleach nor enzyme and would only be used alone for a very lightly soiled wash load.

Composition 3B contains bleach and enzyme in high amounts. It could be used alone for washing very heavily soiled loads, especially in areas where the water supply is soft.

5 However the compositions are intended to be used in proportions of 3:1, 1:1 and 1:3 for lightly soiled, normal and heavily soiled loads. The overall formulations achieved by such proportions are set out in Table 3.

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TABLE 3

INGREDIENT	COMPOSITION (% BY WEIGHT)		MIXTURES (OVERALL % BY WEIGHT)		
	EXAMPLE 3A	EXAMPLE 3B	3A:3B = 3:1	3A:3B = 1:1	3A:3B = 1:3
Sodium Disilicate	40.0	30.0	37.5	35.0	32.5
Nonionic	1.2	2.0	1.4	1.6	1.8
Sodium Citrate	52.6	37.8	48.9	45.2	41.5
Sodium polyacrylate	3.0	5.0	3.5	4.0	4.5
Clay	3.0	2.0	2.75	2.5	2.25
Perfume	0.2	0.2	0.2	0.2	0.2
Sodium Perborate Monohydrate	--	12.0	3.0	6.0	9.0
TAED	--	6.0	1.5	3.0	4.5
Savinase	--	3.0	0.75	1.5	2.25
Amylase	--	2.0	0.5	1.0	1.5
	----- 100.0	----- 100.0	----- 100.0	----- 100.0	----- 100.0

In the above Examples the compositions 1B, 2B and 3B could be coloured one colour, say red, by including particles which had been previously sprayed with red dye, or given a coloured coating. The compositions 1A, 2A and 3A could be coloured differently, including using particles sprayed with a different colour dye, or could be left uncoloured.

Claims

1. A method for washing culinary utensils in a mechanical dishwasher characterised by dispensing into the dishwasher quantities of a plurality of cleaning compositions out of respective separate containers, and operating the dishwasher so that the dispensed quantities of compositions are all mixed into the same wash liquor,
the compositions being different from each other, but each composition containing at least 50% by weight of organic and/or inorganic salt(s) other than bleaching components, enzymes and detergent surfactants.
2. A method according to claim 1 further comprising dispensing only a single one of the compositions to wash other loads of culinary utensils.
3. A method for washing culinary utensils in a mechanical dishwasher providing a washing phase followed by a rinsing phase, comprising
 - i) placing a first load of utensils in the dishwasher, supplying a first cleaning formulation to the dishwasher and operating the dishwasher to mix the said formulation into water to form a wash liquor used during the washing phase;
 - ii) subsequently to washing the first load, placing a second load of utensils in the dishwasher, supplying a second cleaning formulation to the dishwasher and operating the dishwasher to mix the said second formulation into water to form a wash liquor used during the washing phase;
 each of the formulations being provided by dispensing from at least one of a plurality of cleaning compositions held in separate containers, and at least one of the formulations being provided by dispensing quantities of a plurality of the said compositions, with the said quantities being mixed into the same wash liquor,
the compositions being different from each other but each composition containing at least 50% by weight of organic and/or inorganic salt(s), other than bleaching components, enzymes and detergent surfactants.
4. A method according to claim 3 further comprising placing a further load of utensils in the dishwasher on another occasion, dispensing to the dishwasher a single one of the cleaning compositions, which single composition was not used alone with the first or the second load, and operating the dishwasher to mix the composition into water to form a wash liquor during the washing phase.
5. A method according to any one of the preceding claims wherein the compositions differ from each other in the presence or amounts of enzyme, bleaching components or both.
6. A method according to any one of the preceding claims wherein the said salt(s) comprise
 - 20 to 50% by weight of a sodium silicate
 - 30 to 60% by weight of a water-soluble detergency builder which is not a silicate.
7. A plurality of compositions for machine dishwashing, in separate containers associated together, the compositions being different from each other, but each composition containing at least 50% by weight of water-soluble organic and/or inorganic salt(s) other than bleaching components, enzymes and detergent surfactants.
8. A plurality of compositions according to claim 7 wherein the compositions differ from each other in the presence or amounts of enzyme, bleaching components or both.
9. A plurality of compositions according to claim 7 or claim 8 wherein the said water-soluble salt(s) comprise
 - 20 to 50% by weight of a sodium silicate
 - 30 to 60% by weight of a water-soluble detergency builder which is not a silicate.