

1

3,703,470

STORAGE STABLE DETERGENT COMPOSITION

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2 Claims

ABSTRACT OF THE DISCLOSURE

A chlorinated machine dishwashing detergent composition and process of preparing same. The process includes spraying a mixture of a poly(oxyalkylene) glycol defoamer and a low foaming organic, non-ionic surfactant onto an agitated mass of anhydrous detergent salt. The mixture is then sprayed with a solution of a water-soluble resin. The remaining ingredients of the composition typically including detergent salts and a chlorinating agent are then blended with the encapsulated combination to comprise a storage stable detergent composition which retains both available chlorine and low foaming characteristics.

This invention relates to a detergent composition including the combination of a poly(oxyalkylene) glycol defoamer and a low foaming organic, non-ionic surfactant absorbed onto a detergent salt carrier and encapsulated with a water soluble resin; and to a method for preparing the said composition. The present composition and method provide improved storage stability with respect to available chlorine and low foaming properties. The composition is particularly suited for machine dishwashing in the presence of proteinaceous matter.

In the past, when both chlorinating agents and low foaming surfactants were incorporated into a dishwashing detergent composition, considerable difficulties resulted. The surfactants used in these detergent formulations are readily susceptible to attack by chlorine and particularly the hypochlorite ion. As a consequence of such reactions, the surfactants break down and in a relatively short period lose desirable detergency properties including the ability to suppress the foam of proteinaceous soil. The chloride is also dissipated as a result of the reaction and the overall effectiveness of the dishwashing composition is greatly reduced.

The desirability of a non-deteriorating detergent composition including an organic chlorinating agent and a lower foaming surfactant has been long felt in the art.

U.S. Pat. 3,306,858 to Oberle describes one method of providing storage stability of desired defoaming characteristics. However, the compositions and method of Oberle fail to provide substantial protection of the chlorinating agent. During extended storage, compositions prepared by the Oberle process are subject to a loss of chlorinating power.

The present invention provides a means for preparing a highly effective, storage stable, machine dishwashing detergent composition containing both a surface active agent and a chlorine releasing agent.

This composition effectively removes food soils from glassware, dishes, eating utensils and the like while providing great storage stability for the chlorine releasing agent and also retaining desired low foaming characteristics during use.

2

The chlorine releasing agents are added last after the defoamer and non-ionic surfactant have been protected by encapsulation. Generally speaking, the machine dishwashing composition of this invention is prepared by encapsulating a poly(oxyalkylene) glycol defoamer and a low foaming organic, non-ionic surfactant by spraying a mixture of the two onto an agitated body or mass of an anhydrous alkali metal detergent salt such as anhydrous sodium carbonate, then coating the mixture by spraying it with a water soluble resin. The resin encapsulating coating may be a solution of a resin dissolved in an organic solvent, an aqueous solution of an alkali metal salt of a resin containing an acid group, or an emulsion of a resin. The encapsulated defoamer and surfactant combination is then blended with a chlorine releasing agent and other detergent salts normally used in formulating dishwashing type detergents.

These dishwashing compositions generally include various detergent salts including alkali salts, condensed alkali metal phosphate salts (also known as "alkali metal polyphosphate") and a chlorine releasing agent. Examples of alkalis include sodium and/or potassium hydroxide, mono-, di- and tri-sodium or potassium orthophosphates, sodium or potassium bicarbonate, and sodium or potassium carbonates; or (mixtures of any two) or mixtures of any two or more of the foregoing. Useful alkali metal polyphosphates are tetrasodium pyrophosphate, tetrapotassium pyrophosphate, sodium tripolyphosphate, potassium pyrophosphate, potassium tripolyphosphate and the like; and mixtures thereof.

Suitable chlorine releasing agents include chlorinated trisodium phosphate; trichloromelamine; sodium, calcium and lithium hypochlorites; dichloroisocyanuric acid and its water soluble salts such as potassium dichloroisocyanurate and the like.

The surface active agents useful in the process of this invention are low foaming organic non-ionic surfactants obtained by condensing alkylene oxide with water insoluble organic compounds, that is alcohols, phenols, thiols, primary and secondary amines, carboxylic and sulfonic acids and their amides. Surfactants of this type are well known in the art. For example, U.S. Pat. 3,308,067 to Diehl et al. describes a group of non-ionic surfactants formed by condensing ethylene oxide with alkyl phenols, aliphatic alcohols, the reaction product of propylene oxide and ethylenediamine, and various other water insoluble organic compounds.

The preferred organic, low foaming non-ionic surfactant for the purpose of this invention is a linear alcohol alkoxyate, such as Olin's Poly-Tergent S 305-LF, a liquid polyalkoxylated aliphatic based detergent, 97-100%, non-ionic and non-foaming.

The defoaming agents used in the practice of the invention are monobutoxy ethers of random oxyethylene-oxypropylene glycols known as poly(oxyalkylene) glycols. Innumerable examples of suitable glycols are disclosed in Lissant et al. U.S. Pat. 3,382,178 issued May 7, 1968, especially column 4, line 55 through column 11, line 3. The entire disclosure of the said Lissant U.S. Pat. 3,382,178 is hereby incorporated herein by reference thereto.

A preferred poly(oxyalkylene) glycol has a molecular weight of about 4000 and is prepared by polymerizing 50% ethylene oxide and 50% propylene oxide.

The anhydrous salts useful as carriers in the process of this invention include anhydrous sodium carbonate, tri-

sodium phosphate, sodium tripolyphosphate, tetra sodium pyrophosphate, sodium bicarbonate, sodium sesquicarbonate, sodium borate, and possibly sodium metasilicate and sodium orthosilicate.

The general types of water soluble synthetic resins which are useful in the process of this invention are:

(1) Cellulose derivatives such as carboxymethylcellulose, carboxyethylcellulose, methylcellulose, ethylcellulose, hydroxy ethylcellulose and water soluble salts of such polymers;

(2) Water soluble vinyl polymers such as poly(vinyl alcohol), poly(vinyl acetate) partially hydrolyzed poly(vinyl acetate), poly(vinyl methyl ether), polyvinyl pyrrolidone, and copolymers thereof.

(3) Acrylic polymers such as poly(acrylic acid), poly(methacrylic acid), acrylic acid-methacrylic acid copolymers, copolymers of acrylic acid with other monomers, polyacrylamide, hydrolyzed polyacrylamide, polymethacrylamide, hydrolyzed polymethacrylamide, acrylamide-methacrylamide copolymers, copolymers of acrylamide with other vinyl monomers, polyacrylonitrile, hydrolyzed polyacrylonitrile, polymethacrylonitrile, hydrolyzed polymethacrylonitrile, acrylonitrile-methacrylonitrile copolymers, and hydrolyzed acrylonitrile-methacrylonitrile copolymers, or the water soluble, (e.g. the alkali metal, ammonium or amine) salts of such polymers, or mixtures of any two or more of said polymers and/or water soluble salts.

(4) Maleic copolymers prepared by the copolymerization of maleic acid or maleic anhydride with a second monomer, such as styrene-maleic anhydride copolymers.

(5) Other operable synthetic resins are polyethylene, polyethylene oxide, polypropylene, polypropylene oxide, polythyleneimine and the like.

The preferred synthetic resins for the purpose of this invention include methyl cellulose, ethyl cellulose, ethyl hydroxyethylcellulose, styrene-maleic anhydride copolymers, poly(acrylic acid) polymers, polyethylene and polypropylene.

The preferred styrene-maleic anhydride copolymer useful in the practice of this invention is one commercially available from Monsanto Company under the name "Lytron." This copolymer has an equivalent weight of 295 and an acid number of about 190. The copolymer has an average molecular weight within the range of from about 1,500 to 50,000 and preferably from about 5,000 to about 15,000; e.g. 10,000 ("Lytron 822").

The polyethylene resin useful in the practice of this invention has a molecular weight within the range of from about 10,000 to about 32,000 and preferably from about 16,000 to 28,000. One exemplary suitable product is a 55% solids polyethylene emulsion, 16,000 average molecular weight, sold by Gulf Oil Company, U.S. Chemicals Department, under the trade name "Poly-Em 12."

Suitable acrylic polymers are commercially available from Rohm & Haas Company under the trade name "Acrysol" e.g. Acrysol ASE 75 and Acrysol ASE 108.

The present composition may be used for automatic machine dishwashing purposes by forming an aqueous solution containing from about 0.05 to about 1.0 parts by weight and preferably from about 0.05 to about 0.5 parts by weight. The solution is desirably used at a temperature of from about 140° F. to about 200° F. and preferably from about 140° F. to about 160° F.

The encapsulated particles of the present machine dishwashing composition contain, as essential ingredients, the following components in amounts indicated:

Components	Approximate parts by weight	
	Operable	Preferred
Coating: Synthetic resin.....	0.2 to 10...	0.5 to 5.
Encapsulated components:		
Defoaming agent.....	0.1 to 5....	0.1 to 3.
Non-ionic surfactant.....	0.1 to 5....	0.1 to 3.
Anhydrous detergent salt.....	1 to 60....	20 to 50.

In the preferred compositions of the present invention the following components in the amounts indicated are admixed with the encapsulated particles.

Components	Approximate parts by weight	
	Operable	Preferred
Sodium tripolyphosphate.....	5 to 60....	15 to 45.
Tetrasodium pyrophosphate.....	1 to 20....	5 to 15.
Sodium hydroxide.....	5 to 30....	5 to 20.
Sodium dichloroisocyanurate.....	0.5 to 8....	1 to 5.

The invention is further illustrated by the following non-limiting examples.

EXAMPLE 1

A detergent composition was prepared from the following ingredients in the amounts indicated:

Components:	Parts by wt.
Sodium carbonate, anhydrous	45.0
Poly(oxyalkylene) glycol defoamer	1.6
Linear alcohol alkoxylate (Olin's Poly-Tergent S-305-LF)	0.4
Solution of ethyl cellulose dissolved in ethyl alcohol	3.0
Sodium tripolyphosphate	15.0
Tetrasodium pyrophosphate	15.0
Sodium hydroxide	16.5
Sodium dichloroisocyanurate	3.5
	100.0

The poly(oxyalkylene) glycol, and linear alcohol alkoxylate are mixed together and sprayed onto the sodium carbonate while being agitated in a suitable mixer. The solution of ethyl cellulose dissolved in ethyl alcohol is sprayed onto the above mixture thus forming a capsule. The other ingredients are sequentially added to the mixer in the tabulated order.

A sample of the above composition was tested according to the usual procedures and was found to be an effective machine dishwashing detergent.

EXAMPLE 2

In this example, the encapsulating coating is made of an aqueous sodium salt solution of a styrene-maleic anhydride copolymer.

A detergent composition was prepared from the following ingredients in amounts indicated.

Components:	Parts by wt.
Sodium carbonate, anhydrous	45.5
Sodium tripolyphosphate	15.0
Poly(oxyalkylene) glycol defoamer	1.6
Linear alcohol alkoxylate (as in Example 1) ..	0.4
Aqueous sodium salt solution (25% solids) of styrene-maleic anhydride resin ("Lytron 822")	2.5
Tetrasodium pyrophosphate	15.0
Sodium hydroxide	16.5
Sodium dichloroisocyanurate	3.5
	100.0

The polyoxyalkylene glycol and linear alcohol alkoxylate are mixed together and sprayed onto the anhydrous sodium carbonate while being agitated in a suitable mixer. The aqueous solution of styrene maleic anhydride resin is sprayed onto the above mixture thus forming a capsule. The other ingredients are sequentially added to the mixer in the tabulated order.

A sample of the above composition was tested according to the usual procedures and was found to be an effective machine dishwashing detergent.

5 EXAMPLE 3

In this example, the encapsulating coating is made of an emulsion of a low molecular weight polyethylene.

A detergent composition was prepared from the following ingredients in amounts indicated:

Components:	Parts by wt.
Sodium carbonate, anhydrous	45.5
Poly(oxyalkylene) glycol	1.0
Linear alcohol alkoxylate (as in Example 1)	1.0
Aqueous 55 percent emulsion of low molecular weight (16,000) polyethylene (Poly-Em 12)	2.5
Sodium tripolyphosphate	15.0
Tetrasodium pyrophosphate	15.0
Sodium hydroxide	16.5
Sodium dichloroisocyanurate	3.5
	100.0

The polyoxyalkylene glycol and linear alcohol alkoxylate are mixed together and sprayed onto the anhydrous sodium carbonate while being agitated in a suitable mixer. The aqueous polyethylene emulsion is sprayed onto the above mixture thus forming a capsule. The other ingredients are sequentially added to the mixer in the tabulated order.

A sample of the above composition was tested according to the usual procedures and was found to be an effective machine dishwashing detergent.

EXAMPLE 4

In this example, the encapsulating coating is made of an emulsion of an acrylic resin.

A detergent composition was prepared from the following ingredients in amounts indicated:

Components:	Parts by wt.
Sodium carbonate, anhydrous	45.5
Poly(oxyalkylene) glycol	1.0
Linear alcohol alkoxylate	1.0
Aqueous emulsion of a high molecular weight acrylic acid copolymer resin (Acrysol ASE 108)	2.5
Sodium tripolyphosphate	15.0
Tetrasodium pyrophosphate	15.0
Sodium hydroxide	16.5
Sodium dichloroisocyanurate	3.5
	100.0

The polyoxyalkylene glycol and linear alcohol alkoxylate are mixed together and sprayed onto the anhydrous sodium carbonate while being agitated in a suitable mixer. The aqueous emulsion of acrylic acid copolymer resin is sprayed onto the above mixture thus forming a capsule. The other ingredients are sequentially added to the mixer in the tabulated order.

A sample of the above composition was tested according to the usual procedure and was found to be an effective machine dishwashing detergent.

The following test was employed to evaluate the defoaming stability of detergents prepared in accordance with the process of this invention.

Sample solutions for foam height measurements are prepared by dissolving 1.5 grams of the compositions of Examples 1, 2, 3 and 4 in 500 milliliters of water, heating to 70° centigrade and immediately adding the heated solution to a Waring Blendor which contains 1.0 gram of non-fat dried milk. The components are mixed for about 30 seconds in the blender, and the foam is measured at the moment that a definite line of demarcation is observed in the aqueous mixture.

6

Results are given for the compositions of Examples 1, 2, 3, and 4.

Examples:	Foam height in inches		Storage time, months
	Initial	Stored ¹	
1	3/8	3/8	6
2	1/4	1/2	6
3	1/4	1	4
4	1/4	1/2	4

¹ At 70-80° Fahrenheit.

Available chlorine retention tests were also performed. The available chlorine is determined by a conventional iodometric titration procedure known in the art. Results are given in Table II for the compositions of Examples 1, 2, 3 and 4.

TABLE II

Examples:	Percent available chlorine		Storage time, months
	Initial	Stored ¹	
1	2.28	2.17	6
2	2.38	1.94	6
3	1.94	1.98	4
4	1.67	1.77	4

¹ At 70-80° Fahrenheit.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit or scope of this invention.

What is claimed is:

1. A machine dishwashing composition which consists essentially of:

(I) on encapsulated component consisting of: approximate 0.1 to 5 parts by weight of defoaming agent, approximate 0.1 to 5 parts by weight of a low foaming non-ionic surfactant, approximate 1 to 60 parts by weight of an anhydrous detergent salt, said defoaming agent and said surfactant being absorbed into the anhydrous detergent salt, and approximate 0.2 to about 10 parts by weight of a synthetic resin coating disposed about the said salt with absorbed ingredients; said defoaming agent being poly(oxyalkylene) glycol having a molecular weight of about 4,000 and having 50% ethylene oxide polymerized with 50% propylene oxide; said low foaming non-ionic surfactant being linear alcohol alkoxylate; said anhydrous detergent salt being selected from the group consisting of the anhydrous salts of sodium carbonate, tri-sodium phosphate, sodium tripolyphosphate, tetrasodium pyrophosphate, sodium bicarbonate, sodium sesquicarbonate and sodium borate; said resin selected from the group consisting of ethyl cellulose, methyl cellulose, ethyl hydroxyethylcellulose, styrene-maleic acid copolymers, acrylic acid copolymers, and low molecular weight polyethylene; and blended with (I);

(II) approximate 0.5 to 8 parts by weight of a chlorine release agent selected from the group consisting of chlorinated trisodium phosphate, trichloromelamine, sodium hypochlorite, calcium hypochlorite, lithium hypochlorite, sodium dichloroisocyanurate and potassium dichloroisocyanurate;

(III) approximate 5 to 30 parts by weight of an alkali component selected from the group consisting of sodium hydroxide, potassium hydroxide, mono-, di-, and tri-sodium or potassium orthophosphate, sodium or potassium bicarbonate, and sodium or potassium carbonate;

(IV) approximate 1 to 20 parts by weight of alkali metal polyphosphate selected from the group consisting of tetrasodium pyrophosphate and tetrapotassium pyrophosphate; and

(V) approximate 5 to 60 parts by weight of alkali tripolyphosphate selected from the group consisting of sodium tripolyphosphate and potassium tripolyphosphate.

2. The composition of claim 1 wherein:

(I) said defoaming agent is present in approximate 0.1 to 3 parts by weight, said surfactant is present in approximate 0.1 to 3 parts by weight, said detergent salt is present in approximate 20 to 50 parts by weight, and said resin is present in approximate 0.5 to 5 parts by weight;

(II) said chlorine release agent is present in approximate 1 to 5 parts by weight;

(III) said alkali component is present in approximate 5 to 20 parts by weight;

(IV) said alkali metal polyphosphate is present in approximate 5 to 15 parts by weight; and

(V) said alkali tripolyphosphate is present in approximate 15 to 45 parts by weight.

References Cited

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