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3,598,743

PREPARATION OF GRANULAR DETERGENT COMPOSITIONS FOR AUTOMATIC DISH-WASHERS

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No Drawing. Filed July 23, 1969, Ser. No. 844,191
Claims priority, application Great Britain, July 25, 1968, 35,497/68

Int. Cl. C11d 7/56

U.S. Cl. 252-99

5 Claims

ABSTRACT OF THE DISCLOSURE

Granular dishwashing detergent compositions are prepared by dry-mixing sodium tripolyphosphate and chlorinated trisodium phosphate, spraying aqueous sodium silicate thereon to form agglomerated granules, and cooling by passing cold air at a temperature not exceeding 75° F. through a bed of the granules.

BACKGROUND OF THE INVENTION

This invention relates to the processing of detergent agglomerates comprising sodium tripolyphosphate, chlorinated trisodium phosphate, and sodium silicate. Such agglomerates find wide application in dishwashing, especially in automatic dishwashing machines.

General disclosures relating to the composition and the agglomeration of automatic dishwashing products can be found in U. S. Patents 2,480,579 and 3,248,330. U.S. Patent 2,895,916, granted on July 21, 1959 to Milenkevich et al., mentions a cooling and aging step after agglomeration is completed to improve resistance to caking on storage. U.S. 3,247,118, granted on Apr. 19, 1966 to Matthaei, discloses a treatment of agglomerates with hot air to partially dry them.

Despite these contributions, the products made by prior art processes have continued to show a need for improvement in chlorine stability (that is, resistance to loss of available chlorine in storage), flow properties, and resistance to caking in storage.

SUMMARY OF THE INVENTION

According to this invention there is provided a process for preparing a granular detergent composition wherein a particulate mixture of substantially anhydrous sodium tripolyphosphate and chlorinated trisodium phosphate is sprayed with an aqueous solution of sodium silicate whereby a bed of agglomerated granules is formed, and thereafter cold air at a temperature not exceeding 75° F. is passed through a bed of the agglomerated granules.

DETAILED DESCRIPTION

The mixture of sodium tripolyphosphate and chlorinated trisodium phosphate may contain one or more other compatible alkaline-reacting water soluble salts, and preferably it contains from 5% to 40% particulate sodium silicate, consisting of one or more forms having an average weight ratio of SiO_2 to Na_2O of 1:1 to 3.6:1. Particularly satisfactory results are obtained when the silicate has a ratio of about 2:1 or consists of a mixture having an average ratio of about 2:1.

Preferably the anhydrous sodium tripolyphosphate is of the so-called "granular" grade, that is having particle size such that not more than 20% passes a 100 mesh U.S. test sieve. Normally at least 15% of sodium tripolyphosphate by weight of the composition is used. If less is used it becomes increasingly difficult to obtain satisfactory agglomerates though in some cases as little as 10% may

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give an acceptable product. At the other extreme, as much as 60% tripolyphosphate may be used to constitute the balance of the composition apart from the chlorinated trisodium phosphate and the aqueous silicate used in the process. The preferred compositions contain about 25-50% by weight of sodium tripolyphosphate (anhydrous).

The term "chlorinated trisodium phosphate" is used to designate a composition consisting of trisodium phosphate and sodium hypochlorite in intimate association in a crystalline form. The proportion in the composition of the invention can vary quite widely according to the intended use of the product, for instance from 1% to about 75% by weight, but for most purposes a content of 5 to 50% by weight is preferable.

Preferably, a minor proportion of an organic surface active agent is also present in the composition. This can be of any type compatible with the chlorinated phosphate, but usually a low foaming surface active agent, especially a nonionic detergent, is preferred. Polypropylenoxy-polyethylene oxide condensates, such as the Pluronic (trade mark, Wyandotte Chemical Corporation) are very suitable. Also useful are the condensation products of ethylene oxide with long chain primary or secondary alcohols. Usually such liquid or pasty nonionics are employed at up to about 3% by weight, but a higher proportion of surface active agent may be used if it does not make the product too sticky.

Foam suppressors may be used in the composition of this invention if control of foam is desired. Especially suitable are the alkyl acid phosphates, particularly as combined with mineral oil, as disclosed by Hathaway et al. in U.S. Patent 3,399,144, granted Aug. 27, 1968. Minor amounts of other compatible components commonly included in detergent compositions may be added, such as colors, perfumes, stabilizers for the chlorinated phosphate, and tarnish inhibitors.

In carrying out the agglomerating stage of the process the dry materials, namely, sodium tripolyphosphate, solid sodium silicate if any, and chlorinated trisodium phosphate are mixed together in any suitable mixing device, such as an inclined pan granulator, a rotating drum, or a vessel with suitable agitator (an inclined pan granulator is preferred) to form a particulate mixture. With continuous mixing, the mixture is sprayed with an aqueous solution of sodium silicate having weight ratio of SiO_2 to Na_2O of from 1:1 to 3.6:1, preferably about 2:1 to 3.3:1. The optimum amount and concentration of the silicate solution depends on a number of factors such as the actual $\text{SiO}_2/\text{Na}_2\text{O}$ ratio; the nature of the dry mixture, especially its content of sodium tripolyphosphate; the type of mixing device; and the like. The amount should be such as to cause the particulate mixture to form a bed of agglomerated granules, but not so great that its particulate nature is destroyed. Usually between about 10% and 40%, by weight of the detergent composition, of a solution containing 20-60% (preferably 30-50%) of silicate solids (total of SiO_2 and Na_2O) gives satisfactory results. The solution to be sprayed on the dry mixture is usually at ambient temperature; i.e., between 50 and 100° F. If desired it may be warmed to as high as 160° F. for better atomization.

When a liquid nonionic detergent is used it may be sprayed onto the mixture before, at the same time as, or after the silicate. The above mentioned foam suppressors and minor liquid constituents, such as perfume, color dispersion in water, etc., may be sprayed on in admixture with the nonionic detergent or separately.

The process may be carried out batchwise or continuously. In the latter case the solid components are metered at the appropriate rates into the mixing device and sprayed continuously with metered amounts of the liquids from one or more atomizers.

The product consisting of agglomerated granules leaving the agglomerating stage of the process is usually warm as a result of the heat of hydration of the sodium tripolyphosphate, and usually feels moist. A stream of cold air is then passed through the bed of agglomerated granules in any suitable vessel. The air temperature must not exceed 75° F.; particularly good results have been obtained with air at 60–70° F. This treatment may be carried out batchwise or continuously, and it serves inter alia to cool the granules and to render them crisp and free-flowing; hence it is referred to as "crisping" air. The shape of the vessel is not critical provided that care is taken that the air flow is substantially evenly distributed over the cross section of the bed of product. Good results have been obtained using a vessel whose height was two to three times its diameter, the air entering through a sintered disc or a matrix of holes across the cross section of the vessel near the bottom. The volumetric flow rate of air may be sufficient to fluidize the bed or it may be lower than this. The duration of the treatment by air depends among other things upon the air temperature and rate of flow. Usually it is about ½ to 1½ hours when the air flow is sufficient to fluidize the bed of granules and about 1 to 3 hours if the flow is insufficient to fluidize the bed. To avoid an excessively long crisping treatment it is preferred to use a volumetric air flow rate that is at least one-fourth of that which would be just sufficient to fluidize the granules. If the air treatment is to be carried out continuously the desired duration can be provided by appropriate choice of the amount of material in the bed in relation to the throughput, for instance such that the mean residence time is about one hour when the bed is fluidized. In the course of the air treatment the product is cooled, for example, from about 90° F. to about 70° F., and rendered crisp to the touch and free flowing. The drying which takes place is slight, is only incidental to the process, and is not in any way critical. As little as ½% moisture loss is usual based on the weight of the composition, though up to 3% can be tolerated.

Example I

39.3 parts by weight of granular anhydrous sodium tripolyphosphate, of particle size such that not over 20% by weight passed a 100 mesh U.S. sieve, 18.4 parts of "C soluble" silicate (crystalline sodium silicate having weight ratio of $\text{SiO}_2:\text{Na}_2\text{O}$ of 2.0:1 and containing 83% solids), and 20.3 parts of chlorinated trisodium phosphate were mixed together in an inclined pan granulator of 22 inches diameter. A solution consisting of 10 parts "C soluble" silicate and 10 parts water, at 120° F., was sprayed onto the dry mixture with continuous mixing in the rotating pan granulator by means of an air-atomized paint-spray gun. Thereafter, 2 parts of Pluronic L62 were sprayed on using similar spraying equipment. (Pluronic L62 is the trade name of Wyandotte Chemical Corporation for a propylene oxide/ethylene oxide condensate of average molecular weight 2200–2800.) This formed a bed of agglomerated granules, a portion of which was transferred to a 4 ins. diameter vertical tube to form a bed of agglomerated granules about 12 ins. deep. Air at 65° F. was passed up through this bed of agglomerated granules for 80 minutes at a velocity sufficient to fluidize the product. The resulting product was free flowing and had little tendency to cake in storage. Moisture loss was ½% by weight of the composition.

By way of comparison, 44.5 parts of the same granular sodium tripolyphosphate, 31 parts of a 50:50 mixture (by weight) of sodium metasilicate (ratio $\text{SiO}_2:\text{Na}_2\text{O}$ of 1:1, 57% solids) and Silicate M (Crystalline Sodium Silicate having weight ratio $\text{SiO}_2:\text{Na}_2\text{O}$ of 3.3:1, 83% solids) and 20.0 parts of chlorinated trisodium phosphate were mixed together in the same pan granulator. A mixture of 3 parts Pluronic L62 and 1 part of water was sprayed onto the mixture with continuous mixing in the rotating pan granulator. Cold air was not passed through the bed of

agglomerated granules that formed, and the resulting product had poor flow properties and caked on storage.

The two products were stored in polyethylene bags at 100° F. and tested after the intervals stated below for available chlorine content. The results were as follows:

	Product of invention, percent available Cl	Comparative product, percent available Cl
Time (weeks):		
0.....	0.71	0.65
1.....	0.40	0.03
2.....	0.24	0
4.....	0.16	0

Example II

Compositions having the constitution given below were made by a method analogous to that of Example I, except that the air was passed through the bed of agglomerates at about 30% of the rate needed to just fluidize the bed. All composition percentages in this table are on a weight basis.

	A	B	C	D	E	F
Sodium tripolyphosphate, percent.....	13	40	40	40	40	40
Chlorinated trisodium phosphate, percent.....	75	20	20	20	20	20
Solid "C soluble" sodium silicate, percent.....		18	18	18	18	36
Pluronic L62, percent.....	2	2	2	2	2	2
Water, percent.....						1
Spray on silicate solution:						
Weight ratio: $\text{SiO}_2:\text{Na}_2\text{O}$	2.0	2.0	3.2	1.0	2.0	Nil
Amount of solution, percent.....	10	20	20	20	20	
Concentration of solution solids, percent.....	50	20	50	50	50	
Crisping air temperature ° F.....	70	72	68	68	72	70
Crisping time (hours).....	1	1	1	1	1	1
Moisture loss, percent.....	0.4	1.0	0.2	1.5	0.9	0.9
Available chlorine, percent:						
As made.....	2.5	0.60	0.63	0.67	0.66	0.68
Stored 100° F.:						
2 weeks.....	2.5	0.60	0.56	0.41	0.27	Nil
4 weeks.....	2.2	0.58	0.58	0.16	0.16	

All the products with the exception of F (which was included for comparison) were free flowing powders, and all had, as shown, better chlorine stability than F.

Example III

Additional examples of granular detergent compositions that can be prepared by the process of Example I of the instant invention are given in the table below, the ingredients of which are defined as follows:

- STP—The same granular anhydrous sodium tripolyphosphate used in Example I.
 Cl-TSP—The same chlorinated trisodium phosphate used in Example I.
 Tergitol 15-S-3—The trade name of the Union Carbide Company for an ethylene oxide-secondary alcohol condensation product wherein the average carbon chain length is 13 and the average number of mols of ethylene oxide per mol of alcohol is 3.
 TE₉—The ethylene oxide-primary alcohol condensation product wherein the alcohol is derived from tallow and the average number of mols of ethylene oxide per mol of alcohol is 9.

	Weight ratio $\text{SiO}_2:\text{Na}_2\text{O}$	Percent silicate solids
Silicates:		
1.0 R dry.....	1.0:1	99
2.0 R dry.....	2.0:1	83
1.0 R liquid.....	1.0:1	35
2.0 R liquid.....	2.0:1	50
3.6 R liquid.....	3.6:1	40

All entries in the following table are weight percent based on the total composition.

	A	B	C	D	E	F	G	H	I
STP.....	45	34	31	55	20	43	35	35	45
Cl-TSP.....	22	50	50	33	45	10	20	20	27
1.0 R dry silicate.....					20		20	20	
2.0 R dry silicate.....						5			15
1.0 R liquid silicate.....		16	16						
2.0 R liquid silicate.....		13		10	15	20			10
3.6 R liquid silicate.....		17				20	25	24	
Pluronic L62.....			3						
Tergitol 15-S-3.....				2				1	
TE ₂	3					2			3

These compositions, when prepared by the process of Example I, have resistance to the loss of available chlorine in storage, have improved flow properties, and have resistance to caking in storage. Comparable results are obtained when the silicate solution for spray-on is at 70° F.

What is claimed is:

1. A process for preparing a granular detergent composition comprising the steps of:

- (a) mixing together from 15% to 60% by weight of the composition of substantially anhydrous sodium tripolyphosphate with from 1% to 75% by weight of the composition of chlorinated trisodium phosphate; to form a particulate mixture,
- (b) with continuous mixing, spraying onto said particulate mixture from 10% to 40% by weight of the composition of an aqueous solution of sodium silicate having a weight ratio of SiO₂ to Na₂O of from 1:1 to 3.6:1; to form agglomerated granules, and
- (c) passing cold air at a temperature not exceeding 75° F. through said agglomerated agglomerated granules in an amount sufficient to render the granules crisp and free flowing.

2. A process for preparing a granular detergent composition comprising the steps of:

- (a) mixing together (i) from 25% to 50% by weight of the composition of substantially anhydrous sodium tripolyphosphate having a particle size such that not more than 20% passes a 100 mesh U.S. test sieve, (ii) from 5% to 50% by weight of the composition of chlorinated trisodium phosphate, and (iii) from 5% to 40% by weight of the composition

of particulate sodium silicate consisting of one or more forms having an average weight ratio of SiO₂/Na₂O of 1:1 to 3.6:1, to form a particulate mixture,

- (b) with continuous mixing, spraying onto said particulate mixture from 10% to 40% by weight of the composition of an aqueous solution of sodium silicate containing from 20% to 60% by weight of the solution of silicate solids (total of SiO₂ and Na₂O) having a weight ratio of SiO₂ to Na₂O of from 1:1 to 3.6:1; to form agglomerated granules, and
- (c) passing cold air at 60°-70° F. through said agglomerated granules in an amount sufficient to render the granules crisp and free flowing.

3. The process of claim 2 wherein the aqueous solution of sodium silicate contains from 30% to 50% by weight of the composition of silicate solids (total of SiO₂ and Na₂O), and wherein the cold air is passed through the agglomerated granules for ½ to 1½ hours at a rate sufficient to fluidize the granules.

4. The process of claim 2 wherein the aqueous solution of sodium silicate contains from 30% to 50% by weight of the composition of silicate solids (total of SiO₂ and Na₂O), and wherein the cold air is passed through the agglomerated granules for 1 to 3 hours at a volumetric flow rate that is not sufficient to fluidize the granules but is at least one-fourth of that flow rate which would be just sufficient to fluidize them.

5. The process of claim 2 wherein the mixing is carried out by means of an inclined pan granulator.

References Cited

UNITED STATES PATENTS

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U.S. Cl. X.R.

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