

1

3,247,118

METHOD FOR PREPARING DETERGENT
COMPOSITIONSRaymond G. Mattheaei, Fair Lawn, N.J., assignor to Lever
Brothers Company, New York, N.Y., a corporation of
MaineNo Drawing. Filed Feb. 25, 1963, Ser. No. 260,907
6 Claims. (Cl. 252-99)

This invention relates to a method of preparing detergent compositions and more particularly to the manufacture of chlorinated detergents eminently suited for dishwashing purposes.

In U.S. Patent No. 2,689,225 there is disclosed detergent compositions containing as essential ingredients chlorinated trisodium phosphate, an alkaline condensed phosphate such as sodium tripolyphosphate and an "alkaline detergent salt" such as sodium silicate. These compositions were said to be particularly efficacious in cleansing stained dinnerware.

According to U.S. Patent No. 2,895,916, compositions of the aforementioned type are well-suited for use in automatic dishwashing machines. The inventive feature in this patent was in a process for preparing these detergent compositions which yielded products free of the undesirable characteristics of caking during storage and forming a gel or gum in use. According to the patentees, these undesirable characteristics can only be eliminated by a critical procedure which involves adding water and a water-soluble alkali metal silicate to an anhydrous, water-soluble alkali metal condensed phosphate to substantially hydrate the phosphate before the addition of any chlorinated trisodium phosphate to the mixture. The resulting agglomerates are then aged by agitation in a mixer for periods ranging from about 15 minutes to about 2 hours.

It has still not been possible, however, to manufacture a product of this composition exhibiting the following advantages: (1) sufficient moisture to avoid the high and possibly hazardous heat of hydration of anhydrous products; (2) the ability to retain its available chlorine at a high level; (3) an acceptable odor; and (4) the ability to remain free-flowing during shipping, during storage and after being opened. A composition having these attributes would be an extremely desirable commodity.

An object of this invention is to provide detergent compositions containing chlorinated trisodium phosphate which are free from caking during storage and which do not form a gel or gum in use.

Another object is to provide a method for manufacturing chlorinated dishwashing detergents which contain sufficient moisture to avoid the high heat of hydration of anhydrous products, retain available chlorine at a high level, have an acceptable odor and remain free-flowing during shipping and storage and after being opened.

These and other objects and advantages of the present invention are attained by a process which consists primarily of spraying the liquid ingredients of the composition on the dry ingredients followed by a critical aging step to be discussed in detail later on. The agglomerated detergent product at the completion of the mixing operation is moist and will cake if packaged immediately, and four to five hours or possibly longer is generally necessary to age this material in a rotating drum exposed to ambient air conditions before the caking tendencies of the product are such that it can be packaged without caking. This lengthy aging period requires the installation of additional processing equipment.

It has now been determined that the aging period can be reduced from an estimated four to five hours to approximately one hour by the aging step of the invention which comprises passing heated air over the agglomer-

2

ated granules while they are mixed in a rotating drum to expose maximum surface area of the granules. This aging feature insures that the product will not cake after packaging.

For a detailed description of the components which can be utilized in preparing the detergent compositions of this invention, attention is directed to the U.S. patents discussed above whose disclosures are incorporated herein by reference. In general, the chlorinated trisodium phosphate ingredient ($4(\text{Na}_2\text{PO}_4 \cdot 11\text{H}_2\text{O}) \cdot \text{NaOCl}$) is preferably utilized in proportions of about 6% to about 25% based on the weight of the final product. The alkali metal silicates including sodium and potassium silicate, are preferably employed within a range of about 8% to about 20% based on the weight of the final dry product. The alkaline condensed phosphate which is preferably sodium or potassium tripolyphosphate is utilized in proportions of about 40% to about 65% based on the content of the final dry product. Although anhydrous tripolyphosphate can be used, it is preferred to employ a mixture of anhydrous and hydrated tripolyphosphates.

A non-ionic detergent is advantageously included in the composition to provide detergent properties. Suitable compounds include the "Pluronic" which are condensates of ethylene oxide with a hydrophobic base formed by condensing propylene oxide with propylene glycol in which the proportion of ethylene oxide ranges from about 0% to 10% (Pluronic L-60), 10% to 20% (Pluronic L-61), 20% to 30% (Pluronic L-62), etc. Of course, other non-ionic detergents which are well-known in the art can be incorporated and the invention is not limited to any particular detergent so long as it provides detergent properties to the compositions. This component is preferably used in proportions ranging up to about 10% by weight of the final product.

It is within the scope of the present invention to utilize our conventional components which do not affect the basic characteristics of the composition. Thus, ingredients such as colorants, fluorescent dyes, perfumes, soil-suspending agents like sodium carboxymethylcellulose, inert diluents, etc. may be present.

In general, the process of the invention involves mixing an anhydrous condensed phosphate with a small amount of water and thereafter adding the chlorinated tripolyphosphate to the mixture. An aqueous solution of the silicate ingredient is then sprayed onto the aforementioned mixture. If non-ionic surface-active agents are to be included, these materials are also added preferably in the form of an aqueous solution. Other ingredients such as dyes and perfumes in aqueous mixture are added at this point. This procedure yields a hydrated product. Mixing is continued for a short period of time. The granulated material is then screened, added to an aging drum and conditioned by passing heated air through the drum at a temperature of about 90° F. to 185° F. It is preferred that the granules be treated for approximately one hour with air at about 140° F. to 150° F. The granules are also preferably rotated while being aged so that the maximum surface area is exposed. It is also preferred that the granules be reduced to a particle size of at least 10 mesh before aging for optimum results.

It has been found that in order to obtain a product having an acceptable density and hardness and the other advantages described above, certain limitations must be observed during processing of the formulations. For example, the maximum permissible temperature during the agglomeration process is about 120° F. The larger the batch, the longer is the mixing time during agglomeration.

Moreover, the moisture level of the final product must be in the range of about 22-35%. If the moisture level is below about 22%, the density of the product will be

too low, the appearance of the particles will be jagged rather than spherical, and the particles will be more friable than desired. At moisture levels above about 35%, the agglomerates will become too wet during formulation and the material will "ball-up" into larger lumps requiring further processing to reduce the lumps to the desired size. Consequently, proper aging conditions must be maintained to insure that the moisture level of the product is within the desired range.

It has also been determined that from about 5 to about 13% of the free moisture content of the unconditioned particles must be removed during the aging procedure if a satisfactory free-flowing product is to be obtained. Thus, even unaged products with a moisture level approximately within the 22-35% range disclosed above should be properly conditioned in accordance with the principles of this invention to yield an acceptable material.

The silicate ratio ($\text{Na}_2\text{O}:\text{SiO}_2$) utilized is also a pertinent factor in determining the characteristics of the product. It has been observed that the use of the so-called dry or powdered type of silicate may cause "balling" when liquids are added to the mixture during agglomeration. This results in the production of large amorphous lumps which require further processing to reduce the size of the particles and thereby obtain the desired product. When liquid silicates (water solutions of sodium silicates having $\text{Na}_2\text{O}:\text{SiO}_2$ ratios ranging from about 1:1.65 to about 1:3.75) are used, it is desirable to control the water content within certain limits to minimize or avoid the formation of large amorphous masses.

The aging step of this process is vitally important if a product is to be obtained which is free-flowing and has the other desirable attributes mentioned. By the aging treatment with heated air, the moisture content of the product is maintained at an acceptable level. The aging step of the invention gives a greater control over the moisture content of the product since the amount of moisture removed during aging must be controlled within the limits discussed. An unduly large loss of moisture will result in a reduction of the density of the product, the particles will become friable and break down into fines and the heat of solution of the product will increase. If sufficient moisture is not removed, the caking characteristics of the product will not be changed appreciably and the product will not be free-flowing.

Another precaution must be observed during processing. Temperatures in excess of about 120° F. while agglomerating may result in decomposition of the chlorinated trisodium phosphate.

The aging step conditions the product without appreciably affecting its active chlorine content. Therefore, it is a distinct advantage of the invention that a sufficient level of available chlorine is maintained in the product to insure the effective use thereof. A further advantage of the aging procedure is that the surfaces of the particles are polished and the product is therefore more uniform in appearance.

Other means may be employed to effect conditioning of the particles. The use of drying ovens may cause the particles which are not on the surface to retain more moisture than the particles on the top layer. This could result in an inferior conditioning of the material and subsequent caking of the product. However, equipment can be used to keep the granules in motion while they are drying in the oven. The use of a fluidized bed to keep the particles in motion while they are being heated is an alternative embodiment.

The following examples illustrate the use of the method according to the present invention. It will be understood, however, that these examples are included merely for purposes of illustration and are not intended to limit the scope of the invention as described herein unless otherwise specifically indicated.

EXAMPLE I

A formulation processed in accordance with the method

of the invention is listed below in order of addition of the ingredients. The designation TPP refers to pentasodium tripolyphosphate. The "Pluronic" were identified above.

Component (as used):		Lbs. per 100 lbs. of product
TPP, granular, medium density, anhydrous, type I	-----	21.50
TPP, powdered, anhydrous, type I	-----	20.00
Water	-----	3.125
TPP hexahydrate	-----	3.25
Chlorinated trisodium phosphate	-----	20.00
Premix No. 2 ¹	-----	12.8649
Premix No. 3 ²	-----	13.1141
Premix No. 4 ³	-----	10.1260
Perfume	-----	0.02
		104.0000
¹ RU-silicate (1:2.40 ratio of $\text{Na}_2\text{O}:\text{SiO}_2$)	-----	Lbs. 12.6123
Water	-----	0.2521
		12.8649
² N-silicate (1:3.25 ratio)	-----	9.6130
Pluronic L-62	-----	2.25
Pluronic L-61	-----	0.75
Water	-----	0.5011
		13.1141
³ N-silicate	-----	10.0000
Water	-----	0.1260
		10.1260

In the procedure, the powdered and granular pentasodium tripolyphosphate components were added to the mixing drum and mixing was commenced. The water was then sprayed onto the particles in motion. The pentasodium tripolyphosphate hexahydrate and the chlorinated trisodium phosphate were added to the drum and mixing was continued for about a three-minute period. Premix No. 2, No. 3 and No. 4 were successively sprayed onto the particles and mixing was continued for about 10 minutes. The agglomerated material was passed over a 10 mesh shaker screen. The over-sized particles were processed through a Stokes Granulator equipped with a 10 mesh screen.

The two screened fractions were combined in a rotatable drum and conditioned by passing hot air through the drum for approximately one hour at a temperature of about 140°-150° F. and at a rate of about 40 cubic feet per minute. The granules were continuously tumbled during the operation. The hot air was shut off and the perfume added to the particles. Mixing was continued for about five minutes after the addition of the perfume and the granules were screened through a 10 mesh shaker screen and packaged. The product was free-flowing, had an acceptable odor and had a chlorine content within the desired level.

EXAMPLE II

A composition was prepared containing essentially the same ingredients as in Example I with the following differences in proportions expressed in terms of weight of the final product: the granular TPP was used in a proportion of 41.50%; the TPP hexahydrate in a proportion of about 2.50%; the chlorinated trisodium phosphate in an amount of about 10.56%; and the water in proportions of about 29.1246%. The first three values are expressed on an anhydrous basis. The balance of the formula is the same as that described in Example I.

The processing was essentially the same as that utilized in Example I. The granular TPP was added to the mixing drum and about 1.5% water was sprayed on the particles. The maximum temperature during agglomeration was about 110° F. with a somewhat longer mixing time being used than the three-minute period of Example I. The granules were conditioned by passing hot air through the mixing drum at 140°-150° F. and at the rate of about 40 cubic feet per minute for about one hour. The maxi-

5 mum temperature of the batch during the addition of the perfume was 105° F. and 95° F. after screening. Bulk density of the free-flowing product was 0.91-0.95.

The data in Table I below was collected during a number of conditioning runs in accordance with the principles of this invention. Conditioning temperatures and air rates were varied considerably within the limits discussed above. The air rate is not a critical factor but can be widely

4. A process for preparing a dishwashing composition containing about 40% to 65% of a water-soluble, alkaline condensed phosphate, about 8% to 20% of a water-soluble alkali metal silicate and about 6% to 25% chlorinated trisodium phosphate which comprises: (a) adding water to substantially anhydrous pentasodium tripolyphosphate, (b) adding hydrated pentasodium tripolyphosphate and chlorinated trisodium phosphate to the mix, (c) adding

Table I

Run No.	Unconditioned Product			Inlet Air				Conditioned Product			Outlet Air, Rel. Hum., percent
	Moisture, percent	Chlorine, percent	Caking	Temp., ° F.	Rel. Hum., percent	Rate (ft./min.)	Volume (ft. ³ /min.)	Moisture (1 hour), percent	Chlorine, percent	Caking	
1.....	30.4	0.65	Caked..	110	19	172	76	-----	0.63	free-flowing	54
2.....	29.8	0.67	---do---	147	15	90	39.5	27.7	0.65	---do---	52
3.....	29.3	0.65	---do---	181	10	80	35.1	25.6	0.62	---do---	75
4.....	29.2	0.68	---do---	141	17	92	40.6	27.4	0.68	---do---	42

varied depending upon a number of factors including the air temperature, size of the equipment, etc.

The relative humidity data in Table I show that moisture was removed from the product in association with the heated air passed over the particles. A free-flowing material was obtained with substantially no loss in the chlorine content of the product.

It will occur to those skilled in the art that there are many modifications to the invention as specifically described herein. It is intended to include all such modifications in the scope of the appended claims.

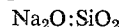
I claim:

1. A process for preparing a detergent composition containing a water-soluble, alkaline condensed phosphate, a water-soluble alkali metal silicate and chlorinated trisodium phosphate which comprises: (a) mixing a hydrated, water-soluble, alkaline condensed phosphate with chlorinated trisodium phosphate, (b) adding subsequently water and the total amount of an alkali metal silicate having Na₂O:SiO₂ ratios ranging from about 1:1.65 to about 1:3.75 to form agglomerates at a maximum temperature of 120° F. and (c) aging the agglomerates by treating with hot air while agitating the particles to remove about 5% to about 13% of free moisture content to form a moisture level of 22-35% in the final product therefrom.

2. The process of claim 1 wherein the alkaline condensed phosphate is sodium tripolyphosphate.

3. The process of claim 1 wherein the temperature of the hot air during aging is about 90° F. to 185° F.

25 subsequently an aqueous solution of the total amount of an alkali metal silicate selected from the group consisting of sodium silicate and potassium silicate having



30 ratios ranging from about 1:1.65 to about 1:3.75 containing an amount up to about 10% of a non-ionic synthetic detergent, (d) continuing agitation until agglomerated particles are produced at a maximum temperature of 120° F., (e) reducing the particles to a size of about 10 mesh and (f) passing hot air through the particles at a temperature of about 90° F. to 185° F. while tumbling them to remove about 5% to about 13% of free moisture content to form a moisture level of about 22-35% in the final non-caking product therefrom.

40 5. A process according to claim 4 in which the hot air is passed through the tumbling particles at a temperature of about 140° F. to 150° F. for about one hour.

6. A process according to claim 4 wherein a mixture of powdered and granular pentasodium tripolyphosphate is used in step (a).

References Cited by the Examiner

UNITED STATES PATENTS

2,689,225 9/1954 Anderson et al. ----- 252-99
2,895,916 7/1959 Milenkevich et al. ---- 252-99

JULIUS GREENWALD, *Primary Examiner.*

ALBERT T. MEYERS, *Examiner.*