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PROCESS FOR PREPARING DETERGENT TABLETS

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The present invention relates to detergent products, and more particularly to an improved process for producing detergent tablets or briquettes.

In copending Black and Gray application Ser. No. 122,641, filed July 10, 1961, there are disclosed detergent tablets and a process for producing the same. The present invention is an improvement in that process whereby the strengthening of the tablets is greatly accelerated so that the handling and packaging of the tablets is facilitated and the production rate thereof is increased considerably. In order to give a clear presentation of the improved process of the present invention there is set forth below a description of the Black and Gray detergent tablets and process therefor.

BLACK AND GRAY DETERGENT TABLETS

The detergent tablets contain two classes of essential components, namely nonionic detergent and phosphate, which are used in certain critical amounts and have a certain critical nature as set forth hereinafter.

The detergent briquettes contain from about 4% to about 13% by weight, and preferably from about 10% to about 12% by weight, of one or more synthetic organic nonionic nonsoap detergents. The nonionic detergents are a well-known class of low sudsing detergents and are produced by the condensation of several moles of a hydrophilic alkylene oxide, such as ethylene oxide or propylene oxide, with a hydrophobic base, such as an alkylated phenol, a fatty alcohol, a fatty amine, a fatty amide, or the hydrophobic base formed by the condensation of propylene oxide with propylene glycol. The alkyl phenol-alkylene oxide condensates are generally prepared by condensing one mole of an alkyl phenol having from about 9 to about 15 carbon atoms in the alkyl radical with from about 8 to about 20 moles of an alkylene oxide, such as ethylene oxide or propylene oxide. Specific examples thereof are the condensation product of one mole of dodecyl phenol with an average of 10 moles of ethylene oxide sold commercially under the name "Sterox DJ," the condensation product of 1 mole of nonyl phenol with an average of 9 moles of ethylene oxide sold commercially under the name "Igepal CO–630," the condensation product of 1 mole of nonyl phenol with an average of 10 moles of ethylene oxide sold commercially under the name "Tergitol NPX," and the condensation product of 1 mole of nonyl phenol with an average of 20 moles of ethylene oxide sold commercially under the name "Igepal CO–850." Typical of the nonionic detergents which are condensation products of a fatty alcohol with an alkylene oxide is the condensate of 1 mole of tridecyl alcohol with an average of 10 moles of ethylene oxide sold commercially under the name "Stereox AJ–100." Condensates of tall oil with alkylene oxides also form nonionic detergents. Typical of these is the condensate of 1 mole of tall oil with an average of 10 moles of ethylene oxide sold commercially under the name "Sterox CD."

The polyoxyalkylene alkanols are also nonionic detergents and are made by the condensation of an alkylene oxide with a hydrophobic base formed by the condensation of propylene oxide with propylene glycol. These nonionic detergents, which are described in U.S. Patent No. 2,674,619 and are sold under the name "Pluronic," have the following empirical formula

$$HO(C_2H_4O)_a(C_3H_6O)_b(C_2H_4O)_cH$$

The hydrophobic base portion of this nonionic detergent, i.e., the $(C_3H_6O)_b$ portion, generally has a molecular weight from 801 to 2100. The amount of ethylene oxide in these compounds is represented by the sum of a plus *c* which is an integer such that the molecule contains from 0 to 90% of ethylene oxide. Thus, in "Pluronic L–60" *b* represents a molecular weight of 1501 to 1800 and a plus *c* is an integer such that the molecule contains 0–10% ethylene oxide. In "Pluronic L–61" *b* represents a molecular weight of 1501–1800 and a plus *c* is an integer such that the molecule contains 10 to 20% ethylene oxide. In "Pluronic L–64" *b* represents a molecular weight of 1501–1800 and a plus *c* is an integer such that the molecule contains 40–50% ethylene oxide. In "Pluronic F–38" *b* represents a molecular weight of 801–1000 and a plus *c* is an integer such that the molecule contains 80–90% ethylene oxide. In "Pluronic F–68" *b* represents a molecular weight of 1501–1800 and a plus *c* is an integer such that the molecule contains 80–90% ethylene oxide. In "Pluronic P–75" *b* represents a molecular weight of 1801–2100 and a plus *c* is an integer such that the molecule contains 50–60% ethylene oxide.

Other suitable nonionic detergents are the diethanolamides of long chain fatty acids, such as lauric diethanolamide.

The phosphate component of the detergent briquettes is present therein in an amount from about 20% to about 95% by weight, and preferably in an amount of about 60% by weight. This amount of phosphate substantially prevents bleeding or oiling out or separation from the tablets of any normally liquid or oily nonionic detergent present therein. The lower the level of normally liquid or oily nonionic detergent present, the less phosphate is needed to prevent bleeding.

The phosphate can be tetrapotassium pyrophosphate, or pentasodium or pentapotassium tripolyphosphate. The tripolyphosphate as used can be completely anhydrous or the commercial anhydrous variety containing small amounts of water, either uncombined or in the form of small amounts of tripolyphosphate hexahydrate, up to, for example, about 1%.

When sodium tripolyphosphate is used, it may be type II (wherein the tripolyphosphate content contains at least 95% Form II), or if rapid disintegration as well as rapid solubility is desired, it must be a mixture of Form I and Form II in which one form may vary from 10% to 90%, and the other from 90% to 10% respectively, in order for the resulting detergent tablets to have a high rate of disintegration, even in unagitated water, as well as rapid solubility in water. Commercial type I sodium tripolyphosphate containing about 20% Form I and 80% Form II is suitable. Detergent tablets prepared from completely anhydrous or commercial anhydrous type II sodium tripolyphosphate have extremely slow disintegration rates but acceptable solubility rates.

Detergent tablets prepared by using a tripolyphosphate which is entirely in the hexahydrate form are unsatisfactory, because such tablets have an extremely poor rate of disintegration and solubilization. If desired, however, a portion of the tetrapotassium pyrophosphate or pentasodium or pentapotassium tripolyphosphates can be substituted by other phosphates. Thus there can be substituted an amount up to about 50% of trisodium orthophosphate, an amount up to about 20% tetrasodium pyrophosphate, or an amount up to about 30% pentasodium tripolyphosphate hexahydrate.

The relative amounts of phosphate and nonionic detergent are also critical. Thus the weight ratio of phosphate to nonionic detergent lies in the range from about 2:1 to about 20:1, and preferably is about 6:1. These relative amounts are necessary in order for the detergent tablets to have sufficient strength to withstand packing, handling,

distribution and use without undue abrasion and fracture and yet have rapid solubilization rates.

The detergent tablets, if desired, can contain other components in addition to the essential phosphate and nonionic detergent components. Thus, it is preferred that the detergent briquettes contain one or more inorganic builder salts such as alkali metal, e.g., potassium or sodium, sulfate, carbonate and silicate. The builder component can be present in an amount from about 0.25% to about 70% by weight, although it is preferred to use from about 19% to about 30% by weight thereof. It is particularly preferred to use about 6% by weight of alkali metal silicate and about 15% by weight of alkali metal sulfate as the builder. A further optional component in the detergent tablets is water which can be present in an amount up to about 10% by weight in order to facilitate the preparation of the tablets. Other optional components include fluorescent dyes or optical brighteners, soil suspending agents, perfumes, water dispersible colorants or pigments or dyes, and the like which are generally present in a small amount up to about 1% by weight.

BLACK AND GRAY PROCESS

The nonionic detergent and phosphate, together with any optional components, such as builders, water, fluorescent dyes or optical brighteners, soil suspending agents, perfumes, water dispersible colorants or pigments or dyes, and the like, are blended together until a uniform mixture is obtained. When a silicate is used to inhibit bleeding, the blending is performed by adding the nonionic detergent to the phosphate and then adding the silicate thereto. When mottled tablets rather than uniformly colored tablets are desired, the water dispersible colorant or pigment or dye is added to the silicate (rather than to the nonionic detergent) prior to adding the silicate to the nonionic detergent and the phosphate. This blending can be readily achieved in a rotary mixer. It is preferred that one or more of the components in the mixture be in a liquid or paste state in order to facilitate agglomeration.

When the mixture is agglomerated in accordance with the preferred procedure, the resulting granules are screened by passing the granules through an 8–10 mesh screen.

After the components have been blended together, either with or without agglomeration and screening, the resulting uniform mixture is compressed into the shape of tablets. The ultimate shape can be any suitable one, such as cylindrical, hexagonal, square, cylindrical with truncated faces, etc. The amount of pressure necessary to compress the mixture into tablet form will, of course, vary with the nature, physical state, total amounts and relative amounts of the components present in the mixture. In general, however, the mixture is compressed into tablets at a pressure ranging from 200 to 5000 pounds per square inch, and usually at a pressure from 500 to 1500 pounds per square inch.

The resulting tablets or briquettes can be further treated, if desired, to improve the properties thereof. Thus, the surface of the tablets can be moistened with from 0.1% to 0.4% by weight of water. The water can be in the form of steam, water spray, or humid atmosphere. The moistening of the tablet surface does not appreciably change the fracture strength of the tablets, but it does increase the surface hardness, abrasion resistance and surface smoothness of the tablets. The amount of water used in moistening the surface of the tablets is critical, because the use of 0.5% or more by weight of water on the tablet surfaces causes the surface to swell and pull away from the body of the tablets and thereby form a very fragile shell. This shell is very easily cracked and flakes away from the tablet leaving a core of untreated tablet which generally lacks sufficient abrasion resistance. This does not lessen friability but rather increases it. Moreover, the use of 0.5% or more by weight of water in the surface moistening treatment may also cause unsightly protuberances to appear on the surface of the tablets.

When surface moistening is employed, the pellets are aged for a time up to 8 hours or longer after the moistening treatment to harden the surface of the pellets. This ageing can be performed at room temperature or higher, although the ageing temperature should not be so high as to have a deleterious effect on any of the components in the tablet, such as any perfume which might be present therein.

The formulations set forth below in Table I are representative of those which were used in the above described Black and Gray process to produce detergent tablets.

TABLE I

Example No.	1	2	3	4	5	6	7	8	9	10	11	12
Sterox AJ–100												12.6
Sterox CD												
Sterox DJ	11.2	11.2	11.2	11.2	8.4	5.6		5.6	4.44		4.75	
Igepal CO–850												
Pluronic L–60	1.4	1.4	1.4	1.4	1.05	0.7		0.7	0.56			
Pluronic L–64												
Pluronic F–68					3.15	6.3			5.0	10		
Pluronic P–75							12.6	6.3				
Pentasodium Tripolyphosphate (at least 95% Form II)												
Pentasodium Tripolyphosphate (10% Form I, 90% Form II)	60		40		60		60					
Pentasodium Tripolyphosphate (20% Form I, 80% Form II)										20	95	60
Pentasodium Tripolyphosphate (90% Form I, 10% Form II)		60		60		60		60	60			
Pentapotassium Tripolyphosphate												
Tetrapotassium Pyrophosphate												
Pentasodium Tripolyphosphate Hexahydrate												
Trisodium Orthophosphate												
Tetrasodium Pyrophosphate												
Sodium Silicate Solids ($Na_2O:SiO_2=1:2.4$)	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5	6.5			
Potassium Silicate Solids												
Sodium Sulfate (Anhydrous)	12.57	12.57	32.76			12.59	13.16	12.66	16.84	70	0.25	26.69
Sodium Carbonate				12.82	12.59							
Water plus Miscellaneous	7.52	7.52	7.52	7.52	7.52	7.52	7.52	7.52	5.82			
Water plus Miscellaneous, dyes and perfume												
Sodium Carboxymethylcellulose, active	0.56	0.56	0.56	0.56	0.56	0.56	0.56	0.56	0.56			0.56
Fluorescent Dyes	0.15	0.15	0.06		0.13	0.13	0.06	0.06	0.13			0.15
Perfume	0.10	0.10			0.10	0.10	0.10	0.10	0.15			

TABLE I.—Continued

Example No.	13	14	15	16	17	18	19	20	21	22	23	24
Sterox AJ-100												
Sterox CD												
Sterox DJ	12.6											
Igepal CO-850				4.44	4.44	4.44	4.44	4.44	11.2	4.5	4.5	11.2
Pluronic L-60		10										
Pluronic L-64				0.56	0.56	0.56	0.56	0.56	1.4			1.4
Pluronic F-68			12.6									
Pluronic P-75				5.0	5.0	5.0	5.0	5.0		5.0	5.0	
Pentasodium Tripolyphosphate (at least 95% Form II)												
Pentasodium Tripolyphosphate (10% Form I, 90% Form II)											60	60
Pentasodium Tripolyphosphate (20% Form I, 80% Form II)												
Pentasodium Tripolyphosphate (90% Form I, 10% Form II)	60	60	60	30	48		42		60	60		
Pentapotassium Tripolyphosphate												
Tetrapotassium Pyrophosphate								60				
Pentasodium Tripolyphosphate Hexahydrate						60						
Trisodium Orthophosphate							18					
Tetrasodium Pyrophosphate				30								
Sodium Silicate Solids ($Na_2O:SiO_2=1:2.4$)					12							
Potassium Silicate Solids										6.5	6.5	6.5
Sodium Sulfate (Anhydrous)	26.69	29.29	26.69	29.29	29.29	29.29			6.5			
Sodium Carbonate									12.57	14.8	14.8	12.57
Water plus Miscellaneous												
Water plus Miscellaneous, dyes and perfume												
Sodium Carboxymethylcellulose, active	0.56	0.56	0.56	0.56	0.56	0.56	0.56	0.56	7.77	8.64	8.64	7.77
Fluorescent Dyes	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.15	0.56	0.56	0.56	0.56
Perfume												

The detergent tablets of the above Table I were tested for abrasion resistance by rubbing them with a plain woven cotton cloth for three minutes. After this abrasion treatment, the pellets showed no loss in weight and the surface of the pellets remained smooth.

The fracture strength of these tablets was at least 10 pounds and in some instances as high as 40 pounds. The fracture strength of these tablets was determined by a test which consisted in standing a tablet on edge on a spring scale and then pressing a lever down on the tablets until they fractured.

These tablets were tested to determine their rate of disintegration and solubilization by placing a tablet in a washing machine containing water having a temperature of 100° F. and measuring the time elapsed until the tablet had completely disintegrated (broke into small fragments) and dissolved. The detergent tablets of Examples 1–22 had an extremely rapid disintegration and solubilization rate in this test in view of the fact that they completely disintegrated and dissolved within one minute. The tablets of Examples 23 and 24 did not disintegrate in this test, although they completely dissolved in 3 minutes.

The amount of bleeding, oiling-out or separation of any normally liquid nonionic detergent component from these detergent tablets was determined by placing the tablets on brown absorbent paper and observing any dampening of the paper. These tablets showed no bleeding when left in contact with the absorbent medium for 48 hours.

IMPROVED PROCESS OF PRESENT INVENTION

The above described Black and Gray process is made more commercially practicable by the improvement therein of the present invention. This improvement comprises chilling the tablets, either with or without prior surface moistening, to accelerate the strengthening thereof so that the handling and packaging of the tablets without crumbling is facilitated and the production rate is increased considerably. The chilling can be done by exposing the tablets to substantially quiescent or moving cold air for a short period of time. In general the time of chilling can be decreased as the temperature of the cold air is lowered or as the velocity of the cold air is increased. The strengthening of the tablets can be accelerated by chilling them at a temperature not substantially above 45° F. for at least about 5 minutes. Typical conditions using substantially quiescent cold air are a temperature from about 10° F. to about 45° F. and a time from about 10 minutes to about 20 minutes. Representative conditions using moving cold air are an air velocity of from about 1 foot per second to about 15 feet per second, an air temperature from about 30° F. to about 45° F. and an exposure time from about 10 minutes to about 20 minutes.

The representative data on fracture strengths (pounds pressure) of tablets set forth in Table II below were obtained by using a typical formulation, i.e., Example 22, in the above described improved process of the invention. In Section A of Table II the tablets were surface moistened prior to chilling, while in Section B of Table II the tablets were chilled without prior surface moistening.

TABLE II.—SECTION A

	Substantially Quiescent Air			Moving Air		
	82° F.[1]	35° F.	15° F.	39° F., 3.0 ft./sec.	33° F.–35° F., 3.0 ft./sec.	33° F.–35° F., 12 ft./sec.
Minutes:						
5	4.5	5.75	6.25	7	7	8
10	5.75	7.5	12	19	18	18.5
15	6.5	16.25	25.5	23.5	21.5	23

TABLE II.—SECTION B

	Substantially Quiescent Air			Moving Air		
	82° F.[1]	35° F.	15° F.	39° F., 3.0 ft./sec.	33° F.–35° F., 3.0 ft./sec.	33° F.–35° F., 12 ft./sec.
Minutes:						
5	1–2	5–6	7	5	5–6	10
10	2	10–11	11–12	7	9–10	15–16
15	2	14	16–17	9	13	20

[1] Comparative temperature.

The above comparative data clearly demonstrate the unexpected acceleration of the strengthening of detergent tablets by chilling the surface moistened or non-surface moistened tablets rather than ageing the tablets immediately at room temperature or higher.

The process of the present invention is also applicable to the strengthening of detergent tablets or briquettes wherein the total and relative amounts of phosphate and nonionic detergent differ from those necessary in the above described Black and Gray process. Furthermore, it can be applied to the strengthening of high sudsing detergent tablets containing the well known synthetic organic anionic nonsoap detergents, e.g., the alkylarylsulfonates. The following two formulations are representative thereof.

Components	Percent by Weight	
Alkane 60 Sulfonate (1:1 mixture of sodium dodecylbenzene sulfonate and sodium pentadecylbenzene sulfonate)	5.0	8.0
Lauric Isopropanolamide	1.5	3.0
Pentasodium Tripolyphosp ate (20% Form I, 80% Form II)	60.0	50.0
Sodium Silicate Solids ($Na_2O:SiO_2=1:2.4$)	6.0	5.0
Sodium Carboxymethylcellulose	0.3	0.3
Fluorescent Dyes	0.14	0.13
Perfume	0.15	0.15
Water	13.33	5.68
Sodium Sulfate and Miscellaneous Inert Matter	13.58	27.74
Total	100	100

It will be appreciated that various modifications and changes can be made in the improved process of the invention without departing from the spirit thereof and accordingly the invention is to be limited only within the scope of the appended claims.

What is claimed is:

1. In the process of preparing strong, abrasion resistant, fast dissolving, low sudsing, compressed detergent tablets and briquettes comprising blending together a mixture consisting essentially of (1) from about 4% to about 13% by weight of synthetic organic nonionic detergent and (2) from about 20% to about 95% by weight of a mixture of 5% to 90% by weight of Form I pentasodium tripolyphosphate plus 95% to 10% by weight respectively of Form II pentasodium tripolyphosphate, the weight ratio of phosphate to nonionic detergent being from about 2:1 to 20:1; and compressing the resulting granular mixture into tablets; the improvement which comprises chilling the compressed tablets to accelerate the strengthening thereof by exposing the compressed tablets to air having a temperature from about 10° F. to about 45° F. for from about 5 minutes to about 20 minutes.

2. The process as set forth in claim 1 wherein the surface of the compressed tablets is moistened with from 0.1% to 0.4% by weight of water prior to chilling the compressed tablets.

3. The process as set forth in claim 1 wherein the chilling is performed by exposing the compressed tablets to substantially quiescent air having a temperature from about 10° F. to about 45° F. for from about 10 minutes to about 20 minutes.

4. The process as set forth in claim 1 wherein the chilling is performed by exposing the compressed tablets to air moving at a velocity of from about 1 foot per second to about 15 feet per second and having a temperature from about 30° F. to about 45° F. for from about 10 minutes to about 20 minutes.

5. The process as set forth in claim 2 wherein the chilling is performed by exposing the surface-moistened compressed tablets to substantially quiescent air having a temperature from about 10° F. to about 45° F. for from about 10 minutes to about 20 minutes.

6. The process as set forth in claim 2 wherein the chilling is performed by exposing the surface-moistened compressed tablets to air moving at a velocity of from about 1 foot per second to about 15 feet per second and having a temperature from about 30° F. to about 45° F. for from about 10 minutes to about 20 minutes.

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