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[54] SYNTHETIC DETERGENT IN BAR OR
CAKE FORM AND THE METHOD TO
MANUFACTURE SAME

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1965, abandoned.

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- [51] Int. Cl.C11d 9/10
- [58] Field of Search.....252/135, 137, 138, 109, 110,
252/368, 370, DIG. 16

[56]

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[57]

ABSTRACT

The invention concerns a solid detergent product in bar or
cake form prepared by mixing an organic detergent material
with an alkali salt of trimetaphosphate, water and an alkali
metal hydrate.

2 Claims, No Drawings

SYNTHETIC DETERGENT IN BAR OR CAKE FORM AND THE METHOD TO MANUFACTURE SAME

This application is a continuation-in-part of our copending application Ser. No. 487,328, filed Sept. 14, 1965 and now abandoned.

BACKGROUND OF THE INVENTION

The present invention relates to a solid detergent product in bar or cake form and the method to manufacture the same.

This method involves the use of a trimetaphosphate of an alkali metal having the formula $\text{Me}_3\text{P}_3\text{O}_9$, wherein Me is an alkali metal, as the main component in the preparation of the solid detergent.

The use of a trimetaphosphate of an alkali metal in preparing detergents, wherein the trimetaphosphate salt is changed by means of a hydrate of an alkali metal, into a tripolyphosphate has been disclosed in Italian Pat. applications No. 26922, of Sept. 5, 1962, and Italian Pat. No. 35066 of May 15, 1963, which corresponds to U.S. Pat. No. 3,390,093 to which reference is made in the present specification.

SUMMARY OF THE INVENTION

An object of the present invention is to provide in bar and cake form a detergent prepared from an alkali metal trimetaphosphate as a principal component.

A further object of the present invention is to provide a simple and economical method for obtaining a detergent in bar and cake form using an alkali metal trimetaphosphate as a principal ingredient.

The present invention is an improvement of the method disclosed in the above-mentioned applications and involves carrying out the reaction of the trimetaphosphate salt and alkali metal hydroxide in a manner that allows the reaction mixture to be converted into detergent bars of solid homogeneous structure and perfect cohesion, without the necessity of drying. It has been found that solid detergent may be obtained in the form of bars and cakes without a drying step, directly from the reaction mixture of alkali metal hydroxide, trimetaphosphate salt and surface active organic detergent, wherein alkali metal tripolyphosphate is formed in situ in the reaction mixture, by adding a low percentage of water to this base paste, for example 3 to 30 percent dry weight of the detergent product and preferably also with the addition of an antifoaming agent.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

According to the invention the physical state of the detergent reaction mixture is transformed so that conventional methods for example, rolling and drawing may be used to form a detergent into solid pieces, such as cake or bars. The transformation of the physical state of reaction mixture may be effected by means of the following treatments which may be applied singularly or in combination.

1. Addition of one or more antifoaming substances like paraffin, lanolin, lecithin, higher fatty alcohols with six to 22 carbon atoms and particularly some higher fatty alcohols of high molecular weight mixed with nonionic surface active products, in percentages varying from 0.1 to 10 percent by weight of the finished product in order to control or eliminate the formation of foam which occurs as a result of the reaction between trimetaphosphate and caustic soda. Further, such products improve the plasticity of the paste and offer a protective action against the possible allergies caused by the surface active substances.

2. By adjusting the percent of water within the base mixture in order to obtain better mixing of the ingredients in the paste which is to be subjected to rolling and drawing.

3. By adjusting the percent of active substance within the mixture so as to obtain a higher cohesion in the paste which is to be subjected to rolling and drawing.

4. By adjusting the temperature in the step of mixing of the main ingredients, in the step of reaction of trimetaphosphate with caustic soda and in the step of rolling and drawing, so as to control the rate of the formation of crystallization of tripolyphosphate.

The detergent in solid pieces such as bars and cakes which is the object of the present invention comprises essentially the following ingredients:

1. A surface active substance of the anionic type. The detergent product may contain from 15 to 65 percent by weight of this active substance.

The anionic active substance may be of any type and particularly:

- alkali salts of fatty acids with a carbon chain from C8 to C20;
- alkali or alkaline earth salts of alkylbenzenesulfonic acid, particularly wherein the alkyl group contains from eight to 22 carbon atoms;
- alkali or alkaline earth salts of sulphuric esters of fatty alcohols of eight to 22 carbon atoms.

The nonionic active substance may be of any type and particularly:

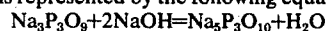
- alkylphenol ethoxylates and aminophenol ethoxylates
- ethoxylates and propoxylates of fatty alcohols of eight to 22 carbon atoms.

By the above ethoxylates and propoxylates are meant the condensation products of alcohols, acids and phenols with ethylene oxides. The starting alcohols, fatty acids and alkylphenols have a carbon chain of from eight to 22 carbon atoms. Such condensation products have a molecular weight, which is determined on the basis of the number of ethylene oxide molecules present per molecule of the starting alcohol, acid or phenol, varying from 500 to 6,000, preferably in the present case from 2,000 to 4,000.

- alkylamides of fatty acids, ethanol- and polyethanolamides of fatty acids particularly of higher fatty acids having eight to 22 carbon atoms for example monoethanolamide of laurilic acid, diethanolamide of laurilic acid and triethanolamide of laurilic acid.

2. Sodium tripolyphosphate produced in situ by the reaction of the sodium trimetaphosphate or other alkali metal hydrate of a strong base as described in Italian Pat. application No. 26,922 of Sept. 5, 1962 and Italian Pat. No. 47,066 of May 15, 1963.

By considering now that the alkali metal cation is sodium, the reaction is represented by the following equation:



The tripolyphosphate can absorb from one to 15 water molecules of water in the form of water of crystallization, tripolyphosphate with an average of six molecules of water of crystallization is very stable even at high temperatures.

3. Other organic ingredients, such as:

- carboxymethylcellulose
- optical brighteners, that is dyestuffs which absorb ultraviolet energy and emit energy in the visible range such as stilbene-triazine derivatives and the like.

4. Other inorganic ingredients, such as:

- sodium silicate
- sodium sulphate
- sodium carbonate, etc.
- bentonite, kaolins, etc.

By means of the present invention, a mixture is obtained which is sufficiently fluid to be mixed thoroughly so as to yield a homogeneous paste; this paste is then further treated according to traditional methods, in order to obtain a detergent in solid bars or cakes without the necessity of drying. The omission of the drying step is possible because the water present remains in the finished product as a result of its being absorbed, mainly by the crystallization of the sodium tripolyphosphate produced in situ, by the reaction of the metaphosphate with an alkali metal hydrate.

The solid detergent produced according to this invention contains 3-30 percent by weight of water, said water remain-

ing in the finished product as water of crystallization of the tripolyphosphate salt.

According to this invention, the transformation of the alkali salt of trimetaphosphate into alkali salt of tripolyphosphate hydrate and if necessary, the neutralization of the organic acid base of the detergent substance are effected in the mixing step or in the steps of mixing and the successive treatments of the resultant paste to obtain a solid detergent in cakes and bars which are homogeneous and of good cohesion. The temperature during each of the steps is maintained at an optimum value of from 15° to 120° C. Thus, the crystallization of the tripolyphosphate can be controlled, so that the final crystallization of the hydrate occurs during the operation of rolling or drawing i.e., milling and extruding or other traditional operations used to obtain cohesion of the paste such as is necessary to produce a detergent in the shape desired.

The method characterizing the present invention may be of four main types:

1. Mixing the cooperating substances, i.e., antifoaming agent, optional ingredients and so forth of the mixture and the active detergent substance in the neutral state with trimetaphosphate and successively adding the alkali metal hydroxide so as to change trimetaphosphate into tripolyphosphate.

2. Mixing the cooperating substances of the mixture and the active substance in the acid state, neutralizing the active substance in the acid state by a solution of alkali hydrate, adding the trimetaphosphate and the alkali hydrate necessary to change the former into tripolyphosphate.

3. Mixing the cooperating substances of the mixture, the anionic active substance in the acid state and the trimetaphosphate and adding the alkali hydrate necessary to neutralize the active substance and change the trimetaphosphate into tripolyphosphate.

4. Mixing the cooperating substances of the mixture, the active substance in the neutral state and the trimetaphosphate, all in the dry phase; successively adding water which starts the transformation reaction of trimetaphosphate into tripolyphosphate.

Additional objects, advantages and particularities of the invention will be apparent from the following description wherein some examples of the invention are illustrated:

EXAMPLE NO. 1

In a mixer provided with a stirring means and jacket and/or coils for heating and/or cooling, are placed:

- 1,500 parts of water,
- 2,000 parts of sodium dodecylbenzene sulfonate, sulfonated by SO₃,
- 1,500 parts of sodium lauryl sulfate,
- 2,840 parts of sodium trimetaphosphate,
- 1,140 parts of sodium silicate having a ratio Si O₂/ Na₂O of 2.40,
- 55 parts of carboxymethylcellulose,
- 500 parts of sodium sulfate.

The mixture is mixed for about 5 minutes at a temperature between 20° to 50° C. Then 1,480 parts of caustic soda at 50 percent are added to the mixture adjusting at the same time the mixture temperature between 40° and 110°. The mixture is immediately changed into scales or "fluffed-up" slurry and fed direct to the rolling device. During the reaction and transformation of trimetaphosphate into tripolyphosphate and during the successive hydration of the tripolyphosphate, the mixture reaches the optimum in plasticity and solidity in order to be drawn or extruded and changed into pieces of solid detergent.

EXAMPLE NO. 2

By the method of example no. 1 are treated:

- 500 parts of sodium dodecylbenzenesulfonate, sulfonated by SO₃;
- 1,900 parts of sodium sulfates of tallow fatty alcohols;
- 500 parts of monoethanolamide of lauric acid;
- 1,500 parts of water;

- 3,000 parts of sodium trimetaphosphate;
- 250 parts of sodium sulfate;
- 40 parts of carboxymethylcellulose;
- 6 parts of an optical brightener, diaminostilbenesulphonates;
- 1,560 parts of caustic soda at 50 percent.

EXAMPLE NO. 3

By the method of example 1;

- 3,500 parts of dodecylbenzenesulfonic acid, sulfonated by SO₃;
- 2,000 parts of water;
- 2,840 parts of sodium trimetaphosphate;
- 1,140 parts of sodium silicate having a ratio Si O₂/ Na₂O of 2.40;

55 parts of carboxymethylcellulose

are introduced and mixed in a common mixer provided with a heating and/or cooling jacket. The mixture is mixed for about 2 minutes at a temperature of about 20° C. Then 2,350 parts of caustic soda at 50 percent are added to the mixture and the temperature thereof is adjusted at about 40° C. After further 5 minutes of stirring at about 40° C., the temperature is allowed to increase up to about 110° C., and then example no. 1 is followed.

EXAMPLE NO. 4

The following ingredients are mixed together without heating:

- 3,500 parts of water;
 - 1,180 parts of solution of NaOH at 50 percent.
- Then the following ingredients are added:
- 3,740 parts of dodecylbenzene sulfonic acid
 - 2,000 parts of melted stearic acid.

As a consequence of such additions, the temperature increases up to about 50°/60° C. The following ingredients are then added:

- 300 parts of melted paraffin wax having a melting point of 70° C.;
- 3,600 parts of anhydrous sodium sulfate;
- 3,400 parts of borax in the crystal phase.

Such a mixture is heated up to 70° C. and then 5,480 parts of sodium trimetaphosphate are added thereto. The temperature is maintained at about 70° C. by continuous heating and finally 3,040 parts of solution of NaOH at 50 percent heated up to 80° are added to the mixture.

In such a way the strong exothermic reaction between sodium trimetaphosphate and caustic soda is caused with a consequent increase of the temperature over 100° C.

EXAMPLE NO. 5

By the same or similar method the following ingredients are treated:

- 1,000 parts of water;
- 2,810 parts of dodecylbenzene sulfonic acid;
- 500 parts of stearic acid;
- 500 parts of sodium pentasilicate pentahydrate;
- 3,600 parts of sodium bicarbonate;
- 125 parts of carboxymethylcellulose;
- 3,420 parts of sodium trimetaphosphate;
- 1,760 parts of a solution of NaOH at 50 percent.

EXAMPLE NO. 6

By the same or similar method the following ingredients are treated:

- 400 parts of water;
- 800 parts of a solution of NaOH at 50 percent;
- 3,270 parts of dodecylbenzenesulfonic acid;
- 300 parts of monoethanolamide of lauric acid;
- 200 parts of paraffin wax;
- 1,650 parts of anhydrous sulphate;
- 2,920 parts of sodium trimetaphosphate;
- 1,520 parts of a solution of NaOH at 50 percent.

In order to describe the present invention fully, reference has been made to several specific examples; however, the invention is not limited in any way thereby and many changes and additions may be made in practicing this invention without departing from the spirit thereof.

What we claim and desire to secure by letters patent is:

1. A process for making a solid detergent in cake or bar form which consists essentially of

- A. mixing without heating 3,500 parts by weight of water and 1,180 parts by weight of a 50 percent solution of sodium hydroxide;
- B. adding thereto 3,740 parts by weight of dodecylbenzene sulfonic acid and 2,000 parts by weight of melted stearic acid;
- C. then adding to the mixture 300 parts by weight of melted paraffin wax, 3,600 parts by weight of anhydrous sodium sulfate and 3,400 parts by weight of borax;
- D. heating the mixture to about 70° C. and adding 5,480 parts by weight of sodium trimetaphosphate thereto;
- E. adding 3,040 parts by weight of a 50 percent solution of sodium hydroxide whereby a strong exothermic reaction takes place between sodium trimetaphosphate and the sodium hydroxide causing the temperature of the mixture to increase to above 100° C. and changing the mixture to scales or an expanded slurry and

- F. then feeding the mixture to a rolling device and drawing the mixture into a solid product.
- 2. A process for making a solid detergent in cake or bar form which consists essentially of
 - A. mixing without heating 400 parts by weight of water and 800 parts by weight of a 50 percent solution of sodium hydroxide;
 - B. adding thereto 3,720 parts by weight of dodecylbenzene sulfonic acid and 300 parts by weight of the monoethanolamide of lauric acid;
 - C. then adding to the mixture 200 parts by weight of melted paraffin wax, 1,650 parts by weight of anhydrous sodium sulfate;
 - D. heating the mixture to about 70° C. and adding 2,920 parts by weight of sodium trimetaphosphate thereto;
 - E. adding 1,520 parts by weight of a 50 percent solution of sodium hydroxide whereby a strong exothermic reaction takes place between sodium trimetaphosphate and the sodium hydroxide causing the temperature of the mixture to increase to above 100° C. and changing the mixture to scales or an expanded slurry and
 - F. then feeding the mixture to a rolling device and drawing the mixture into a solid product.

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