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PRODUCTION OF GRANULAR DETERGENT MIXTURES

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Our invention relates to a method of producing granular, non-caking, free-flowing alkaline compositions adapted for use in cleansing, disinfecting and other operations in which alkaline detergents are employed in aqueous solution.

It is, of course, old to prepare mixtures of detergent materials in granular form in order to simplify their handling, storage and transport. However, in many of such mixtures, the particles or granules vary greatly in size which is highly disadvantageous, among other reasons, because of the resultant varying rates of solubility. Mixtures currently available, in which the particles are sufficiently uniform in size are, in general, produced by processes requiring the re-working or further working of particles which are either too large or too small as determined by selected limits. Thus, it is the common practice to subject the large particles, after separation by screening, to a grinding operation to reduce them to a size within the permissible limits and to return the fine particles to the agglomerating device.

An advantage of the present invention resides in the fact that it greatly reduces the quantity of material which must be subjected to this additional or further processing. Our invention is specifically directed toward providing granular mixtures of detergent materials characterized in the respect of particle size in that not more than about 20% of the mixture when screened with screens of the Tyler series is retained on the 20 mesh screen and in that not substantially more than 10% passes through the 65 mesh screen.

According to the conventional method of preparing granular mixtures of the type here involved, the comminuted material representing the charge stock, after being wetted with water or while being wetted with water or steam is agitated in a mixing device such as a horizontal rotating drum for a period of time sufficient to achieve agglomeration. Subsequent processing involves screening of the agglomerated mass to the desired particle sizing with return of the undersized material to the mixing device and crushing of the oversized agglomerates.

We have now discovered that the amount of water employed in the agglomeration of mixtures of powdered or comminuted detergent materials is a critical factor. More particularly, we have found that if the quantity of water used in the agglomeration, as carried out with addition of the water during the agitation period, is restricted within the limits 70–95% of that just sufficient to convert the free-flowing mass to a paste, a minimum of 70% of the agglomerated product

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will have a particle size within the range 20–65 mesh (Tyler series). In most instances, but not always, the maximum conversion to the desired particle size is achieved by using 80–93% of the amount of water needed to form a paste. The optimum proportion of water with respect to that just sufficient to convert the mass to a pasty consistency depends largely upon the composition of the mixture and to a minor extent upon the type of mixing employed. It may be advantageous to add the water at an elevated temperature particularly where materials that are difficult to hydrate are present in the mixture. After the agglomeration, which in any event proceeds with the evolution of heat, the mass should be cooled to a temperature of 30° C. or less in order to prevent a subsequent tendency to "cake". Surface active agents where used may advantageously be dissolved in the water. In such cases the dry material subjected to the simultaneous agitation and wetting may consist of only one compound.

Our invention is particularly applicable to the production of granular mixtures comprising soda ash or mixtures of soda ash and sodium bicarbonate and phosphates such as tetrasodium pyrophosphate, sodium tripolyphosphate, trisodium phosphate, etc., but is not limited to such mixtures. Thus, it may be applied, for example, in the production of a granular product consisting of borax and a suitable wetting agent. In all cases, the material wetted should be of such particle size that a major portion will pass through a 65 mesh Tyler screen.

Our invention may be practiced using a conventional pug mill or any type of mixing device applying a mild mechanical action. Mixers of the horizontal type are preferred. In general, it is best not to add the water until the dry materials have become intimately mixed. The water should be added slowly in all cases. Spray devices may be used, with advantage, particularly in large scale operations. After the last of the water has been added, the mixing should be continued for several minutes before the granulated product is cooled.

In the following examples, submitted in further illustration of our invention, all parts are by weight and all materials are anhydrous unless otherwise specified:

Example 1

90 parts of water was slowly added to an agitated mixture consisting of 180 parts of sodium carbonate, 60 parts of sodium silicate, and 60

parts of tetrasodium pyrophosphate, each of these being present in a state of fine subdivision. The 90 parts of water represent 90% of that required to form a paste. The wetted material after a few minutes' additional stirring was air-dried. Upon screening of the granular product, 18.1% was retained on a 20 mesh screen, 57.8% on a 48 mesh screen, and 18.3% on a 65 mesh screen, while 5.8% passed through the 65 mesh screen.

Example 2

140 parts of water was slowly added to an agitated mixture consisting of 240 parts of sodium carbonate and 60 parts of tetrasodium pyrophosphate. The 140 parts of water was equivalent to 93.5% of that just required to form a paste from the dry materials. Following a few minutes' additional agitation, the wetted material was air-cooled and screened. 19.7% was retained on the 20 mesh screen, 59.0% on the 48 mesh and 11.9% on the 65 mesh with 9.4% passing through the 65 mesh. The screened material was found to be of substantially uniform chemical composition. That passing through the 20 mesh screen and retained on the 48 and 65 mesh screens analyzed 64.6% sodium carbonate.

Example 3

To 150 parts each of sodium carbonate and sodium silicate was slowly added 94.5 parts of water, 70% of that just required to convert the dry materials to a paste. When the resulting material was subjected to screening, 9.4% remained on the 20 mesh screen, 73.5% on the 48 mesh, and 15% on the 65 mesh. Only 1.8% passed through the 65 mesh screen.

Example 4

60 parts of water, 80% of that necessary to form a paste, was slowly added to a dry mixture consisting of 225 parts of sodium carbonate, 56.3 parts of tetrasodium pyrophosphate, and 18.7 parts of a sodium alkyl aryl sulfonate used as a wetting agent. Upon screening of the product, 14.0% was retained on the 20 mesh screen, 69.5% on the 48 mesh, 9.6% on the 65 mesh and 6.8% passed through the 65 mesh.

Example 5

To 225 parts of sodium carbonate, 56.3 parts of tetrasodium pyrophosphate and 18.7 parts of cetyl trimethyl ammonium bromide was slowly added, with agitation, 64 parts of water, 80% of that required to form a paste. Upon screening of the granular product, 18.7% was retained on the 20 mesh screen, 47.8% on the 48 mesh, 23.9% on the 65 mesh and 9.6% passed through the 65 mesh.

Example 6

To 80 parts of borax was slowly added, with agitation, 5 parts of a long chain alkyl dimethyl benzyl ammonium chloride dissolved in 15 parts of water, about 80–85% of that required to form a paste. In excess of 70% of the granular product was of a particle size within the range 20–65 mesh.

Example 7

2.5 parts of diisobutyl phenoxy-ethyl dimethyl benzyl ammonium chloride in 14.5 parts of water was slowly added, with agitation, to a dry mixture consisting of 45 parts of sodium carbonate, 18 parts of tetrasodium pyrophosphate and 20 parts of trisodium phosphate dodecahydrate. In excess of 70% of the resulting product was of a particle size within the range 20–65 mesh. The amount of water added to the dry materials was equivalent to about 80–90% of that required to form a paste.

Example 8

A similar product was obtained by the slow addition of 10.40 parts of water, approximately 90% of that required to form a paste, to a dry agitated mixture consisting of 8.70 parts of trisodium phosphate dodecahydrate, 17.80 parts of sodium carbonate, 28.30 parts of sodium bicarbonate, 4.35 parts of sodium silicate pentahydrate, 8.70 parts of tetrasodium pyrophosphate and 21.75 parts of a high molecular weight fatty alcohol sulfate.

Example 9

The addition of 100 parts of water to a dry mixture of 200 parts of sodium carbonate and 100 parts of sodium tripolyphosphate, $Na_5P_3O_{10}$, gave a paste. However, when 80 parts by weight of water instead of 100 was used a granular product was produced, 21% of which was retained on a 20 mesh screen, 54% on 48 mesh and 13.2% on 65 mesh.

Example 10

24.4 parts of water was added slowly, with agitation to a mixture consisting of 50 parts of sodium carbonate, 14 parts of tetrasodium pyrophosphate, 7 parts of a higher fatty alcohol sulphate and 7 parts of sodium-p-toluene-sulphochloramide. In excess of 70% of the resulting product which analyzed 2.10% available chlorine was of a particle size within the range 20–65 mesh. The amount of water added to the dry materials was equivalent to about 80–90% of that required to form a paste.

Example 11

In each of the foregoing experiments, the amount of water employed was within the range 70–95% of that just required to form a paste from the dry ingredients. In further experiments with the same detergent materials in which smaller and larger amounts of water were used, it was found that as the proportion of water is increased above the optimum amount the particle sizing becomes increasingly coarse and that as the proportion of water is decreased below the optimum amount the particle sizing progressively decreases until finally there is no agglomeration whatsoever.

We claim:

1. A process for granulating dry powdered detergent mixtures of such particle size that a major portion passes through 65-mesh (Tyler) and which consist of soda ash and at least one material selected from the class consisting of sodium silicate, sodium bicarbonate and sodium phosphates which comprises mechanically agitating the dry powdered mass in the presence of water which is slowly added in an amount exceeding about 10 per cent by weight of the mass but between about 70 to 95 per cent of the water required to convert the dry powdered mass to a paste wherein there is just sufficient water to form the continuous phase and convert the free-flowing mass to a unitary mass, and screening the granulated mass to recover the fraction within the size range of 20 to 65 mesh (Tyler).

2. The process of claim 1 wherein the dry powdered mixture contains a major proportion of soda ash.

3. The process of claim 1 wherein a surface active agent is dissolved in the water employed.

4. The process of claim 1 wherein the amount of water is about 80 to 93 per cent of that required to convert the dry powdered mass to a paste wherein there is just sufficient water to form the continuous phase and to convert the free-flowing mass to a unitary mass.

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