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(54) A process for preparing granules for detergent compositions.

(57) There is disclosed a process for preparing granules for incorporation into a detergent composition, which process comprises the steps of

(a) tumbling together in a rotating pan a finely divided particulate smectite clay and a finely divided particulate zeolite in a weight ratio of from 1:9 to 9:1 whilst sufficient water is added slowly to the resultant mixture to bind the particles together to form granules of a mixture of said smectite clay and said zeolite; and

(b) drying the resultant granules to reduce the water content to not more than 20% of the total weight of the granules.

Also disclosed is a detergent composition including said granules.

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This invention relates to a process for preparing granules for incorporation into detergent compositions and, in particular, to a process for preparing granules comprising a zeolite; and to detergent composition containing the granules produced by the process.

It is generally desirable that the components of a detergent composition should be in granule form because the bulk density of a granular composition is higher than that of a finely divided powder with the result that a given weight of the composition can be packed into a smaller container when the composition is granular than when it is in powder form.

The granules should be free flowing and should also be resistant to crushing and abrasion so that they can withstand handling and transportation in containers. They should also be relatively white in colour and will typically have sizes in the range of from 0.75mm to 2.0mm.

Zeolite, which is a cation exchanging aluminosilicate, is included in detergent compositions to perform the function of a builder or scavenger of metal cations, especially calcium and magnesium ions, from the washing water, which ions, if allowed to remain in solution, would impair the effectiveness of the surfactant present in the detergent composition. Until recent years the function of the builder in detergent compositions has been fulfilled by water-soluble condensed phosphate salts, for example sodium hexametaphosphates, but the discharge of large quantities of phosphates into rivers is damaging to the environment, and there is therefore a growing trend towards replacing phosphate builders with zeolites. A zeolite with a particularly advantageous cation exchange capacity is known as zeolite A or zeolite 4A, and it is this zeolite which is commonly found in detergent compositions. Zeolite A is generally available in commerce in the form of a fine powder having particles of size of the order of a few microns, but it has hitherto been found difficult to form the zeolite into granules of suitable size which are sufficiently coherent to withstand handling and transportation in packages without the use of additional binding materials which add to the expense and are undesirable in the complete detergent compositions. An attempt to overcome this problem is disclosed in EP-A-0279040 in which the detergent builder is prepared by spray-drying a feed suspension containing the zeolite and bentonite.

A smectite clay, for example bentonite, is often included in a detergent composition. The clay may be included on account of its fabric softening properties, or as a stabilising agent for the detergent composition, but the use of the clay brings with it the disadvantage that the clay is somewhat dark in colour and therefore imparts an undesirable dark colouration to the composition containing it.

According to one aspect of the invention there is provided a process for preparing a granule for incorporation in a detergent composition, which process comprises the steps of

(a) tumbling together in a rotating pan a finely divided, particulate smectite clay and a finely divided, particulate zeolite in a weight ratio of from 1:9 to 9:1 whilst sufficient water is added slowly to the resultant mixture to bind the particles together to form granules of a mixture of said smectite clay and said zeolite; and

(b) drying the resultant granules to reduce the water content to not more than 20% of the total weight of the granules.

The process of the invention preferably includes the further step of:

(c) sieving the dried granules to yield a product comprising granules having sizes in the range from 0.75mm to 2.0mm.

Granules prepared in accordance with the invention are solid and will comprise the smectite clay and zeolite in a weight ratio of from 1:9 to 9:1.

The present invention also provides a detergent composition which includes granules prepared in accordance with the process of the present invention. The detergent composition will contain a surfactant and optionally other ingredients in amounts known per se.

The smectite clay may be montmorillonite, saponite, hectorite, nontronite or beidellite, but is most conveniently a bentonite clay which is a naturally occurring clay of volcanic origins consisting predominantly of montmorillonite.

The weight ratio of zeolite to smectite clay charged to the pan may preferably be in the range of from 2:8 to 8:2, more preferably 3:7 to 7:3, especially 7:3.

The smectite clay in its initial, finely divided, particulate form preferably has a particle size distribution such that substantially all of it passes through a No. 60 mesh British Standard sieve (nominal aperture 0.250mm).

The zeolite is most advantageously a synthetic zeolite such as zeolite A which has a chemical composition corresponding to the approximate formula:-

$1.0\pm0.2\ Na_2O.\ Al_2O_3.\ 1.9\pm0.1\ SiO_2,\ 0\text{-}5.1H_2O$

and an apparent pore width of approximately 4 Angstrom units. Preferably, the initial, finely divided, particulate zeolite has a particle size distribution such that it consists predominantly of particles having sizes in the range from 1 to 10 microns.

The granules of the present invention may also incorporate minor proportions of other ingredients, and up

to 20% of the total weight of the granules may be water (as residual water derived either from the water binder added or moisture present in either of the clay or zeolite components). No additional liquid binder material is essential to form the granules; i.e. the water used in step (a) is preferably the sole liquid binder employed. The size of the final granules, after sieving is preferably from 0.75mm to 2.0mm, most preferably from 0.85mm to 1.7mm. Also, the granules preferably should not contain more than about 7% by weight of particles smaller than 0.15mm.

In the process of the invention, the smectite clay and the zeolite are preferably tumbled together in a pan granulator. The Eirich pan granulator is an example of a particularly suitable device. The quantities of zeolite and smectite clay added to the pan are those necessary to give the desired proportions of each component in the resultant granules and this will normally mean that from 10 to 90% by weight of smectite clay and from 90 to 10% by weight of zeolite are added, based on the total weight of smectite clay and zeolite. The water is preferably added in the form of a fine spray and in an amount such that it constitutes preferably at least 10% and preferably no more than 40% of the total weight of the granules formed in step (a) of the process. Thus, for every 100 parts by weight of smectite clay/zeolite mixture charged to the pan, from about 11 to about 67 parts by weight of water may be employed.

The granules may conveniently be thermally dried in step (b) in, for example, a fluidised bed dryer.

The process of the present invention provides granules which are sufficiently coherent to withstand handling and transportation in containers without the use of an additional binding material. It is believed that the smectite clay itself acts as a binder which makes it possible to form coherent granules containing a zeolite. The granules of the invention also have the advantages of being relatively light in colour compared with the smectite clay on its own.

A typical, conventional detergent composition contains the following ingredients normally in amounts in the ranges given below:-

Ingredient	% by weight
Anionic surfactant	1 - 30
Nonionic surfactant	1 - 17
Suds controlling agent	0 - 0.6
Zeolite A	1 - 45
Sodium carbonate	10 - 35
Bleaching agent	0 - 5
Smectite clay	1 - 5
Cellulose ether	0 - 0.5
Enzymes	0 - 2.5
Optical brightening agent	0.05 - 0.25
Sodium silicate	0 -25
Formulation aid	0 - 1
Water	to 100

With the present invention, at least part of the Zeolite A and at least part of the smectite clay in the foregoing composition can be replaced by granules in accordance with the present invention.

The anionic surfactant may be, for example, an alkyl benzene sulphonate or a fatty alcohol ether sulphate.

The nonionic surfactant may be, for example, an alkyl polyethyleneglycol ether or a nonylphenol polyethyleneglycol ether.

The suds controlling agent may be, for example, a soap, a silicone oil or a paraffin.

The bleaching agent may be, for example, sodium perborate or sodium percarbonate.

The invention is illustrated by the following Example.

EXAMPLE

A bentonite clay having a particle size distribution such that 99% by weight passed a No. 60 mesh British

Standard sieve (nominal aperture 0.250mm) and 85% by weight passed a No. 200 mesh British Standard sieve (nominal aperture 0.076mm) and a zeolite A powder having a particle size distribution such that 70% by weight consisted of particles in the range from 2 to 6 microns were loaded into the pan of an Eirich granulator in the proportions 70% by weight of zeolite and 30% by weight of bentonite. The pan was set in motion at a speed of 46 rpm and the agitator at a speed of 1429m.min $^{-1}$ and the clay and zeolite were mixed in a substantially dry state for 15 seconds. During the next 10 seconds, with the pan and agitator rotating at the same speeds, sufficient water was sprayed on to the mixture of clay and zeolite to give a final water content of the granules of 33% by weight. Finally the moist granules were tumbled in the pan of the granulator for a further 75 seconds at the same speed settings for the pan and for the agitator.

The moist granules were then dried in a fluidised bed dryer at a temperature of 80°C for a time sufficient to reduce the water content of the granules to 14% by weight. The granules were then screened on a sieve having a nominal aperture size of 1.2mm and the granules passing through the sieve were retained as the product. The starting materials and the product granules were tested for bulk density and for reflectance to violet light of wavelength 457nm and the results obtained are set forth in the following Table:

TABLE

	Bulk density ($g.cm^{-3}$)	% reflectance to light of 457nm wavelenth	
granule		powder	
Bentonite	0.70	76.0	53.0
Zeolite A	0.50	94.5	-
Bentonite/zeolite A granule	0.73	-	77.1

The granules were found to be sufficiently coherent to be able to withstand bulk handling and transportation in containers, and the reflectance to violet light of 457nm wavelength of 77.1% was sufficiently high to give the granules an acceptably light colour such that they would not impair the white appearance of a detergent composition in which they were incorporated.

Claims

1. A process for preparing granules for incorporation into a detergent composition, which process comprises the steps of
   (a) tumbling together in a rotating pan a finely divided particulate smectite clay and a finely divided particulate zeolite in a weight ratio of from 1:9 to 9:1 whilst sufficient water is added slowly to the resultant mixture to bind the particles together to form granules of a mixture of said smectite clay and said zeolite; and
   (b) drying the resultant granules to reduce the water content to not more than 20% of the total weight of the granules.

2. A process according to claim 1 wherein the smectite clay is a bentonite clay.

3. A process according to claim 1 or 2 wherein the smectite clay in its initial, finely divided, particulate form has a particle size distribution such that substantially all of it passes through a No. 60 mesh British Standard sieve (nominal aperture 0.250mm).

4. A process according to any one of claims 1 to 3, wherein the zeolite is zeolite A.

5. A process according to claim 3, wherein the zeolite A has a chemical composition corresponding to the approximate formula:-
   $1.0\pm0.2\ Na_2O.\ Al_2O_3.\ 1.9\pm0.1\ SiO_2,\ 0\text{-}5.1H_2O$
   and an apparent pore width of approximately 4 Angstrom units.

6. A process according to any preceding claim, wherein the initial, finely divided, particulate zeolite has a particle size distribution such that it consists predominantly of particles having sizes in the range from 1 to 10 microns.

7. A process according to any one of the preceding claims, wherein the water added in step (a) is the sole liquid binder.

8. A process according to any one of the preceding claims, comprising the following further step:
(c) sieving the dried granules to yield a product comprising granules having sizes in the range from 0-75mm to 2-0mm.

9. A detergent composition including granules prepared by the following process:
(a) tumbling together in a rotating pan a finely divided particulate smectite clay and a finely divided particulate zeolite in a weight ratio of from 1:9 to 9:1 whilst sufficient water is added slowly to the resultant mixture to bind the particles together to form granules of a mixture of said smectite clay and said zeolite; and
(b) drying the resultant granules to reduce the water content to not more than 20% of the total weight of the granules.