



(72) GASSENMEIER, THOMAS, DE
(72) MILLHOFF, JUERGEN, DE
(72) SCHMIEDEL, PETER, DE
(72) VON RYBINSKI, WOLFGANG, DE
(72) KRUPP, UTE, DE
(71) HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN, DE
(51) Int.Cl.⁷ C11D 7/26, C11D 7/34, C11D 7/18, C11D 7/10
(30) 1999/11/26 (199 57 036.1) DE
(54) **PROCEDE POUR LA FABRICATION DE DETERGENTS
PARTICULAIRES**
(54) **PROCESS FOR THE PRODUCTION OF PARTICULATE
DETERGENTS**

(57) The invention relates to a process for the production of particulate detergents or premixes suitable for their production by application of a flowable acidic component to a particle consisting at least partly of an alkaline detergent ingredient, the percentage of acidic component applied being governed by the formula $m_c / (m_c + m_p) = c \cdot l/r$, where m_c is the weight of the acidic component, m_p is the weight of the particle, r is the radius of the particle and c is a factor of 0.5 length units to 20 length units.

Abstract

The invention relates to a process for the production of particulate detergents or premixes suitable for their production by application of a flowable acidic component to a particle consisting at least partly of an alkaline detergent ingredient, the percentage of acidic component applied being governed by the formula $m_a/(m_c + m_p) = c \cdot l/r$, where m_c is the weight of the acidic component, m_p is the weight of the particle, r is the radius of the particle and c is a factor of 0.5 length units to 20 length units.

PROCESS FOR THE PRODUCTION OF PARTICULATE DETERGENTS

Field of the Invention

This invention relates to a process for the production of particulate detergents.

Background of the Invention

Particulate detergents normally consist of a plurality of ingredients
5 which, owing to mutual incompatibilities, are normally distributed over
several particulate components and incorporated thus in the detergent as a
whole. Examples of such mutually incompatible ingredients are bleaching
agent and bleach activator which, although intended to react with one
another under in-use conditions, i.e. in aqueous solution, to develop the
10 high bleaching performance required, must not react with one another
during storage because, otherwise, they would no longer be available for
the intended purpose.

A less graphic example of the non-optimal co-operation of individual
detergent ingredients arises out of their pH-dependent performance.
15 Whereas enzymes and certain bleaching systems, for example, have their
optimum pH in the neutral or mildly acidic pH range, anionic surfactants
and builders, for example, require an alkaline pH value in order fully to
develop their effect. Speaking quite generally, both soil particles and most
textile fibers develop an increasing number of negative charges with
20 increasing pH value which results in increasing repulsion among them and
hence contributes to the desired washing result. For this reason, the
washing of textiles has for ages been carried out with more or less alkaline
wash liquors. The same applies to aqueous solutions for cleaning hard
surfaces, for example in machine dishwashing.

25 A way out of this dilemma of different pH optima for different
ingredients is available via the time dimension of the washing/dishwashing
process and consists in initially establishing a pH value at which certain
ingredients develop their effect and then changing the pH so that other

ingredients can develop theirs. In this way, each active ingredient finds its own optimal conditions which can differ from the optimal conditions of another active ingredient. A corresponding washing/dishwashing process in which the aqueous system used initially has a relatively low pH value
5 which changes to a higher value after a certain time is the subject of German patent application **DE 199 57 038**.

Summary of the Invention

In order to carry out this process, alkaline ingredients of the detergent used, which, as mentioned above, are essential to the favorable
10 overall result of the process, have to be made up in such a way that they pass into the aqueous system at a later stage of the process and not at the beginning.

In the course of research work on this theme, it was found that this can be achieved by applying a reactive component to the alkaline particle,
15 the quantity (to be applied) of component reacting with the alkaline particle on the surface being determined in a certain way by the diameter of the alkaline particle.

Accordingly, the present invention relates to a process for the production of particulate detergents or premixes suitable for their
20 production by application of a flowable acidic component to a particle consisting at least partly of an alkaline detergent ingredient, the percentage of acidic component applied being governed by the formula $m_c/(m_c + m_p) = c \cdot l/r$, where m_c is the weight of the acidic component, m_p is the weight of the particle, r is the radius of the particle and c is a factor of 0.5 length units
25 to 20 length units and more particularly 5 length units to 10 length units.

An acidic component or an alkaline detergent ingredient is understood to be a substance of which additions to water with a pH of 7 produces an acidic or alkaline pH value.

In the above formula, it is important to note that m_c and m_p or r and
30 the unit of length used for c should share the same unit of size, for example

kg and μm , respectively.

If several particles rather than just one particle are to be treated at the same time, as is generally the case, m_p is understood to be the total weight of the particles to be treated and r their mean radius.

5 A particle to be treated in accordance with the invention may optionally contain all the ingredients of a detergent, i.e. it is possible by the process according to the invention to coat a preformed detergent. However, only at least some or all of the alkaline ingredients of such
10 detergents are preferably treated in accordance with the invention, in which case the alkaline ingredients may be present as particulate individual substances or several of the alkaline ingredients may be present in a single particle to be treated. The alkaline ingredients in question are preferably alkali metal silicates, alkali metal aluminosilicates, alkali metal phosphates, alkali metal carbonates, alkali metal perborates and alkali metal
15 percarbonates and mixtures thereof, sodium being the preferred alkali metal.

 The process according to the invention is preferably carried out by applying the liquid or paste-form, optionally molten acidic component to the optionally heated particle consisting at least partly of an alkaline detergent
20 ingredient in a mixer or granulator. Particularly good coating is achieved when the acidic component is applied to the particle over a period of 5 to 20 minutes.

 Although only a theoretical assumption to which applicants do not wish to be bound, the process according to the invention presumably
25 creates a particularly dense and comparatively sparingly or slowly water-soluble coating, which remains reliably impermeable after introduction into an aqueous system and hence allows the production of detergents with a stepped pH profile, through the neutralization reaction of the acidic coating material with the alkaline particle on the surface thereof. The particles
30 obtainable in accordance with the invention almost exclusively contain the

ingredients of the particle originally used in their core. This core is surrounded by a layer in which increasingly higher concentrations of acidic coating material or the salt thereof formed with the alkaline component occur from inside outwards, the outer surface optionally being formed
5 entirely by acidic coating material. This layered structure provides the coated particle with stepped dissolving behavior. On contact with water, hardly any alkaline material is dissolved from the coated particle for a certain time to begin with although, as soon as the coating has at least partly dissolved and no longer completely surrounds the particle, the pH
10 value of the aqueous system increases so to speak suddenly through the release of the alkaline material from the core.

Besides the alkali metal component, constituents of the particle of alkaline material may be any typical detergent ingredients compatible with that component providing they are solid or can be made up in solid form.
15 Such ingredients include, in particular, other builders, surfactants, other peroxygen compounds, peroxygen activators, sequestering agents, electrolytes and other auxiliaries, such as dye transfer inhibitors, silver corrosion inhibitors, foam regulators and dyes and perfumes, the presence of peroxygen activators in the particle of alkaline material being less
20 preferred where that particle also contains peroxygen compound.

The particles produced by the process according to the invention are used as detergents for manual or machine washing or dishwashing, preferably after mixing with at least one other particulate component. In one preferred embodiment, the at least one other component contains at
25 least one active ingredient of which the washing/cleaning effect is greater at a lower pH value than that established after dissolution of the alkali metal component present in the particle produced by the process according to the invention than it is at the pH value established during dissolution of the said particle. This active ingredient is preferably selected from enzymes or
30 enzyme mixtures. In the case of the mixtures, the individual active

ingredients may also be present in several particulate components differing in their composition. The at least one other component used may also have such a solubility that, under the initially lower pH conditions, it releases as much as possible of the active ingredient present which, however, only develops its full effect after an increase in pH through the then higher alkalinity of the aqueous system surrounding it or which otherwise reacts with the alkali metal component then released. One example of this particular variant is a bleach activator which is soluble or made up to dissolve at a relatively low pH and which reacts with a bleaching agent released from the particle coated in accordance with the invention and, because it is already dissolved, is capable of developing a strong bleaching effect extremely quickly.

Detailed Description of the Invention

Brief Description of the Drawings

The accompanying Figure 1 is used to illustrate the invention.

Figure 1 is a pH curve after incorporation in water of a detergent product produced by the application of 1.5g of stearic acid.

Examples

Example 1

1 kg of spheronized sodium percarbonate with a mean particle diameter of 400 μm were premixed cold with 25 g of stearic acid (flakes). The resulting premix was transferred to a commercially available plowshare mixer with horseshoe blades preheated to 90°C (casing temperature) in which it was reactively compounded for 20 mins. at speed stage 2 and at a measured product temperature of 80°C. The hot product was removed and, after cooling in the usual way, was used, for example, for the production of detergents.

Example 2

Quantities of 2.5 g and 1.5 g of stearic acid were added to quantities

of 100 g of spheronized sodium percarbonate (mean particle diameter 1400 μm) in a glass beaker, followed by heating with stirring to around 80°C. After the molten stearic acid had been absorbed into the alkaline particles, the whole was reactively compounded for another 20 mins. at
5 around 80°C.

The pH curve after incorporation in water of the product produced by application of 1.5 g of stearic acid is shown in Fig. 1. For comparison, Fig. 1 also shows the pH curve of the uncoated percarbonate.

The invention may be varied in any number of ways as would be
10 apparent to a person skilled in the art and all obvious equivalents and the like are meant to fall within the scope of this description and claims. The description is meant to serve as a guide to interpret the claims and not to limit them unnecessarily.

Claims:

1. A process for the production of particulate detergents or premixes suitable for their production by application of a flowable acidic component to a particle consisting at least partly of an alkaline detergent ingredient,
5 the percentage of acidic component applied being governed by the formula $m_a/(m_c + m_p) = c \cdot 1/r$, where m_c is the weight of the acidic component, m_p is the weight of the particle, r is the radius of the particle and c is a factor of 0.5 length units to 20 length units.
2. A process as claimed in claim 1, wherein the particle consisting at
10 least partly of an alkaline detergent ingredient has a radius r of 100 μm to 1,000 μm .
3. A process as claimed in claim 1 or 2, wherein c is a factor of 5 length units to 10 length units.
4. A process as claimed in any of claims 1 to 3, the acidic component
15 is solid at room temperature and the application of the flowable acidic component is carried out at a process temperature above room temperature.
5. A process as claimed in any of claims 1 to 4, wherein the acidic
20 component is applied to the particle over a period of 5 minutes to 20 minutes.
6. A process as claimed in any of claims 1 to 5, wherein the alkaline detergent ingredient is selected from the alkali metal silicates, alkali metal aluminosilicates, alkali metal phosphates, alkali metal carbonates, alkali metal perborates and alkali metal percarbonates and mixtures thereof.
- 25 7. A process as claimed in any of claims 1 to 6, the acidic component is selected from mono- or dicarboxylic acids containing 10 to 22 carbon atoms, sulfuric acid monoalk(en)yl esters containing 10 to 20 carbon atoms, alk(en)yl or alkylaryl sulfonic acids containing 10 to 20 carbon atoms, polymeric polycarboxylic acids obtainable by polymerization of
30 ethylenically unsaturated mono- and/or dicarboxylic acids, such as acrylic

acid, methacrylic acid and/or maleic acid, and mixtures thereof.

8. The use of the premix obtained by the process claimed in any of claims 1 to 7 as a detergent after mixing with at least one other particulate component.

5 9. The use claimed in claim 8, wherein the at least one other component contains at least one active ingredient of which the washing/cleaning effect is greater at a lower pH value than that established after dissolution of the alkali metal component present in the particle produced by the process claimed in any of claims 1 to 7 than it is at the pH
10 value established during dissolution of the said particle.

Figure 1

