

Development of a Novel Granular Detergent with an Interspersion Particle Comprising an Anionic Surfactant and a Polymeric Polycarboxalate

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This paper discusses a process for making a novel granular detergent with an interspersion particle comprising an anionic surfactant and a polymeric polycarboxalate. This process contains three steps to develop the interspersion particles with anionic surfactant and polymeric polycarboxalate. The first step was to form a detergent particle by spray drying of an aqueous detergent slurry comprising anionic surfactant (liner alkyl benzene sulfonate) with the different levels of polymeric polycarboxalate. In the second step, the spray-dried granules were densified and ground by a roll compacter and a grinder. The ground particles were coated by nonionic and zeolite in a vertical batch type high shear mixer (third step). In this study, the feasibility to make better performance of granular detergent was discussed.

Key words detergent; free flow; high bulk density; polymer; zeolite

There is a current trend for commercially available cleaning compositions, such as granular detergent compositions, to have higher bulk densities as well as higher active ingredient content. Such detergent compositions offer greater convenience to the consumer and at the same time, reduce the amount of packing materials that is easily disposed.^{1–4)}

However, such cleaning compositions sometimes tend to encounter problems of poor dissolution. To address this problem, builder, filler and/or surfactant, which have good water-solubility, have been used so far. Unfortunately, although these materials improve overall dissolution property of such compositions, they also readily absorb moisture content. Such affinity for moisture absorption trends to results in undesirable caking during manufacturing and storage of such compositions. Anionic surfactant, especially high-level anionic surfactant, is a major facility of such caking. This caking is very undesirable for consumers. Consequently, there is a need for high active materials used in cleaning compositions to have better physical properties, such as good dissolution, as well as low moisture absorption to minimize caking. It is believed that potential caking of particles with certain high active materials can be reduced or eliminated by interspersing polymeric polycarboxalate within individual particles containing the high active material. Such interspersion improves the overall physical property, such as anti-caking property of such interspersion particles.

In this study, a new granular detergent has been developed by the formulated anionic and polymeric polycarboxalate to provide a better physical (high bulk density and free flow) properties.

Experimental

Figure 1 shows an experimental set-up for granule compacting system. A compacter (Shinto Industrial Company–BCS 25 types) was used for porosity reduction of spray-dried granules. The maximum pressing pressure was 25 tons. The diameter and width of the compacter roll were 228 mm and 76.2 mm, respectively. The rotation speed of the roll and the screw shaft were 2.6–10.4 rpm and 8.4–33.0 rpm, respectively. The granules were loaded in to the hopper, then compressed by the tapered screw and delivered to the high-pressure roller. After being compressed by the roller, the compacted chips were produced. After that, the chips from the compacter were ground by a grinder (Fitz mill, Hosokawa Industrial Co. Ltd.) and then

screened to have the desired particle size. The coarse fraction larger than 850 μm was 4–10 wt% and fine fraction smaller than 150 μm was 19–22 wt%.

A vertical batch type high-shear mixer (FKM-50D Matsubo Company, Ltd.) having its vessel volume of $5 \times 10^{-2} \text{ m}^3$ and the effective capacity of $1.5\text{--}3.5 \times 10^{-2} \text{ m}^3$ was used for coating of detergent granules (Fig. 2). The ratio of the mixer diameter/length was 0.7. Two distinct types of internal tools were exposed in the mixer. Four plows were attached to arms placed in a spiral pattern on 120 centers around a shaft. These plows provided the “hurling and whirling” action, sometimes called a mechanical fluidized bed, and achieve almost—random mixing. A chopper was placed between each pair of plows near the wall. The chopper entered the mixer in the upward path of the plow about 45° up the circumference from the bottom of the mixer. The purpose of the chopper was to give high impact and shear stresses onto grinding large particles made by dispersing minor ingredients or atomizing liquid injections.

General spray dried synthetic detergent was used for this study which had bulk density of 200–300 kg/m^3 , mean particle size of 300–700 μm , and the cake strength value (shown in the next session) of less than 1 kg. The formulation of the spray-dried particles was 34–38 wt% of anionic surfactant, 10–19 wt% of polymer, 40–46 wt% of inorganic and 3 wt% of free water (Table 1). Nonionic surfactant was used for a binder. Nonionic surfactant (C12-15 alcohol 9.0 ethylene oxide ethoxylate) was supplied by Shell Chemical Company.

After spraying the nonionic surfactant binder, zeolite A (Cosmo Co.) as a shell powder was used to cover the surface of the core particles to improve the physical properties. The typical chemical formulation of zeolite A is $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 4.5\text{H}_2\text{O}$.

The coated particles were produced by the following steps using powder samples shown in Table 2.

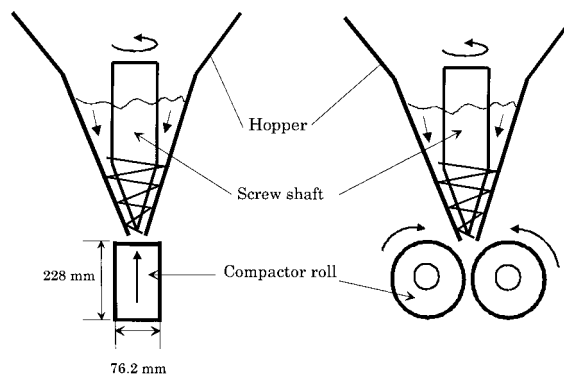


Fig. 1. Experimental Set-Up of Roller Compacter

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(i) The spray dried detergent granule was continuously fed into the roller compactor through a force feeder that was located at the top of the rollers to produce the compacted chips. The operating condition was that the roller rotational speed of 3.6 rpm, the roll pressure of 1.7–2.1 tons to obtain the chip density of $1.3\text{--}1.6 \times 10^3 \text{ kg/m}^3$.

(ii) The compacted chips were constantly fed into a grinder (Fitz mill). The operating condition was that 4726 rpm of the rotational speed of the shaft with 2.0 mm punch out size of screen. After the grinding, the coarse fraction larger than $850 \mu\text{m}$ was 4–10 wt% and fine fraction smaller than

$150 \mu\text{m}$ was 19–22 wt%. The bulk density of the ground chip was about 660 kg/m^3 .

(iii) To produce the coated agglomerates by a high shear mixer (FKM-50D), Froude number (Fr) was used for the process parameter. Here, Fr was defined as follows.

$$Fr = (Np)^2 D_{(b \text{ or } c)} / g \quad (1)$$

where, Np , $D_{(b \text{ or } c)}$ and g represent rotational speed of the plow, diameter of the blade (D_b) or chopper (D_c) and acceleration of gravity, respectively. 88 wt% of the densified ground granule was fed into the high shear mixer and mixed for 70 s at $Fr = 1.29 \times 10^{-2}$ of the blade and $Fr = 40.1$ of the chopper speeds, respectively. Volume occupation level was set at 37% based on the assumption that the bulk density of finished product should be 700 kg/m^3 .

(iv) A hot nonionic surfactant heated up at 343 K was sprayed onto the well mixed powder prepared by Step (iii) in the same mixer for 60 s at $Fr = 1.29 \times 10^{-2}$ of the blade and $Fr = 40.1$ of the chopper. Feed ratio of hot nonionic was 4 wt%.

(v) 7 wt% of zeolite A was added/mixed in the high shear mixer at same blade and chopper speeds for 200 s.

(vi) After sieving through a mesh with $1180 \mu\text{m}$ opening, the coated particles were blended with the other parts of the detergent formula including other surfactant particles, additive particles, enzyme, bleach, bleach activator particles and *etc.*

The physical property of coated sample was analyzed by a pressure (cake strength value) required to break a cake of the sample and bulk density. The method and equipment were the same as the previous reported.⁵⁾

Table 1. Formulation of Spray Died Powder

Formulation	A	B
Total anionic surfactant (wt %)	38	34
Total polymer (wt %)	10	19
Total inorganic (wt %)	46	40
Miscellaneous	6	7
Total	100	100

Table 2. Powder Samples

Powder sample	Mixing ratio (wt %)
Spray dried particles	88
Nonionic binder ^{a)}	4
Zeolite A	7
Miscellaneous	1
Total	100

a) Binder temperature: 343 K.

Results and Discussion

Figure 3 indicates the physical properties of detergent particles prepared by different polymeric polycarboxylate levels in the spray-dried particles. Higher polymeric polycarboxy-

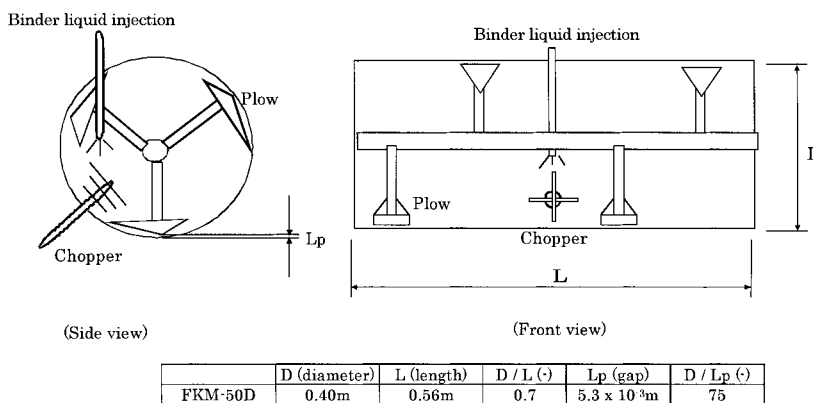


Fig. 2. Schematic Diagram of High Share Mixer

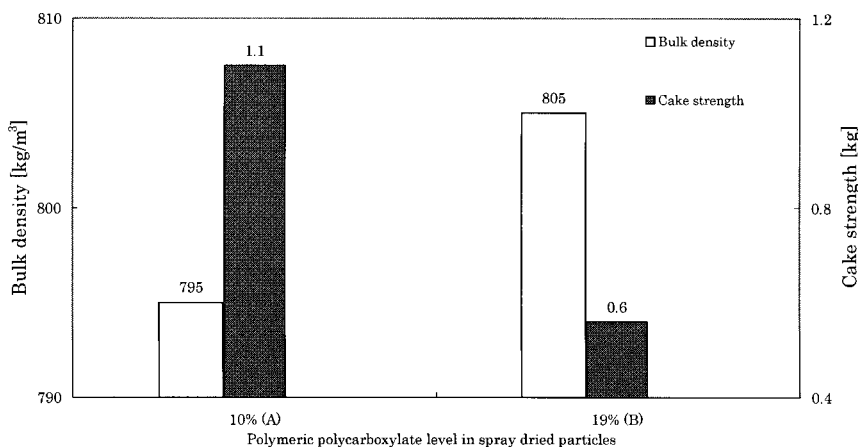


Fig. 3. Effects of Polymeric Polycarboxylate Level on the Physical Properties

late level provided significantly better physical properties as compared with 10%. Based on the past information, the higher organic level tends to show worse flowability because organic material (anionic surfactant, polymer, *etc.*) has a tendency to be hygroscopic as compared with inorganic material (sodium carbonate, sodium sulfate, zeolite, *etc.*). However, formulation B has more free flow material even though the organic material is higher. This suggests that there might be some chemical reaction/chemical structure occurred between anionic surfactant and polymeric polycarboxylate in the slurry before the spray drying. Additionally, there is a strong possibility of providing the high active granular detergent with better physical properties (high bulk density and no cak-

ing). In other words, it seems that further compacted granular detergent can be produced.

As the next step, it should contemplate the best ratio of polymeric polycarboxylate and anionic surfactant.

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