Technical Benefits of Encapsulation and Granulation for Powdered Detergents

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For many years, Dow Corning has successfully used granulation through agglomeration to deliver and protect active ingredients of powdered laundry detergents. This paper describes Dow Corning's granulation facilities and illustrates the benefits of the granulation technique with two examples.

Dow Corning Agglomeration Units

The Dow Corning plant in Seneffe, Belgium has two granulation units that are complementary in terms of production capacity and manufacturing capability.

- The Encapsulated Antifoam Unit (EAF) unit is the main production plant, with a capacity of several tons per hour and a single granulation technology using a Flexomix[a] mixer.
- The Small Scale Agglomeration Unit (SSAU) was built for process development purposes and smaller production volumes (a few hundred kg per hour). It features three alternative agglomeration technologies: a Flexomix mixer, a Ploughshare[b] mixer and a fluid bed granulator and coater.

Because the SSAU offers multiple granulation technologies, this paper describes only that unit. Figure 1 shows a simplified flowchart of the powder plant.

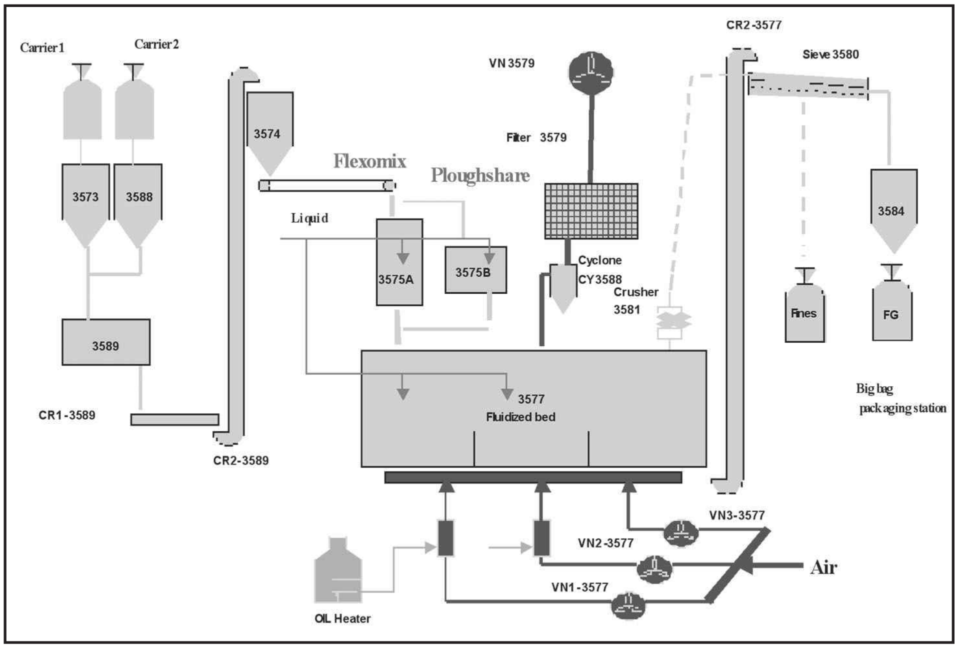

Figure 1. Simplified flowchart of the powder plant of the SSAU.

The SSAU Powder Plant

Powder preparation. Two powders, which may be either fresh or recycled, can be mixed as a batch in a ribbon blender (3589) mounted on load cells. The dry mix is discharged to a buffer silo (3574) via a bucket elevator. A loss-in-weight conveyor belt continuously doses the dry mix from the buffer hopper into the Flexomix mixer or the Ploughshare mixer, which is installed in parallel.

Granulation. The Flexomix is a continuous, high shear mixer with a residence time less than 1 s. The powder and the liquid, usually sprayed via nozzles onto the particles, are mixed in a vertical cylindrical chamber fitted with a flexible wall made of elastomer. A vertical shaft with knives rotating in the range of 1500 to 4500 rpm ensures the mechanical mixing.

In comparison, the Ploughshare mixer is a less intensive mixer, equipped with a horizontal cylindrical product chamber. The mixing speed of the horizontal shaft is in the range of 50 to 280 rpm. Powder is gently projected upwards, into a zone where the binding solution may be sprayed. A chopper rotating at 3000 rpm breaks oversized, lumpy material before it leaves the mixing chamber. The residence time may be adjusted from approximately 30 s to several minutes by altering the height of an exit weir.

Because of the differences in shear rate exerted on the solid and liquid mixture, as well as the differences in residence time, both mixers produce granules with different particle sizes, porosities and bulk densities.

Drying and cooling steps. The granules from either the Flexomix or the Ploughshare mixer enter the continuous fluid bed (3577), where they may be dried or cooled, depending on the type of binder used (aqueous or waxy emulsion). The fluid bed consists of three cells having their own airflow rate and temperature control loop. The fluid bed may also be vibrated, in the case of materials that cannot be fluidized easily, or to better control the circulation of the product from one cell to another. Exhaust air is taken in through a large fan (VN 3579), and entrained particles are recovered in a cyclone (CY 3588) and a bag filter (3579). Two spraying lances are installed in the first two cells to perform fluid bed granulation or coating. The fluid bed granulation facility may be used alone or in series with one of the two mechanical mixers (two-step agglomeration process).

Sieving and packaging of the finished product. Granules are separated in a two-stage sieve (3580). Oversized material is crushed in a hammer mill (3581) and reinjected in the last cell of the fluid bed. The finer material may be collected or reprocessed

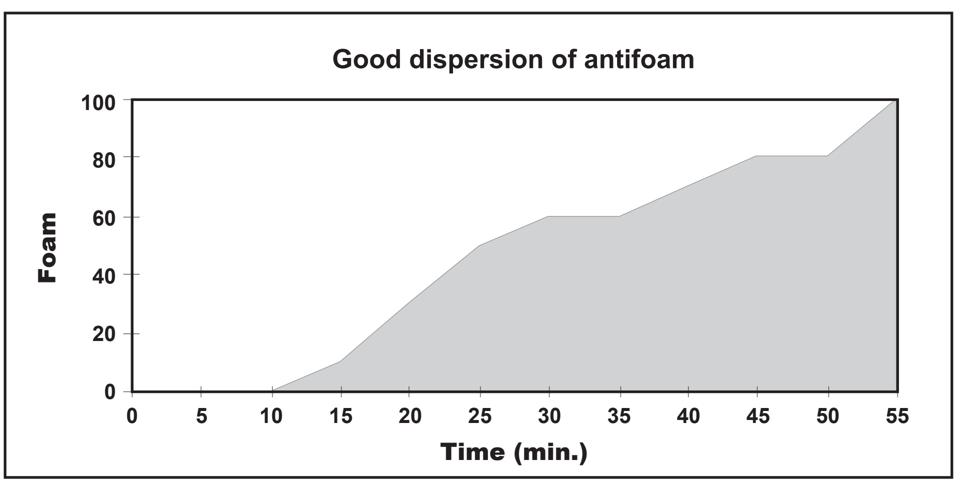

Figure 2. Good dispersion of silicone antifoam.

[a] Hosokawa Micron Ltd., Cheshire UK
[b] Morton Machine Co. Ltd., Motherwell, Scotland UK

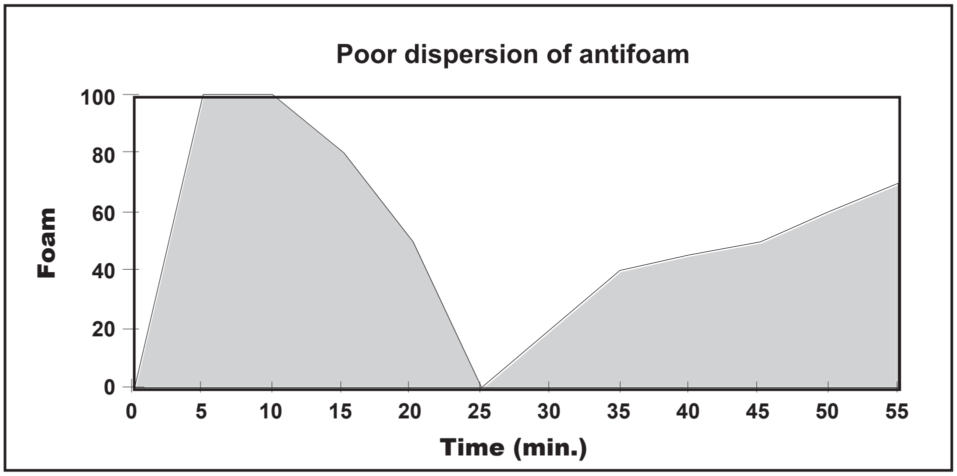

Figure 3. Poor dispersion of silicone antifoam.

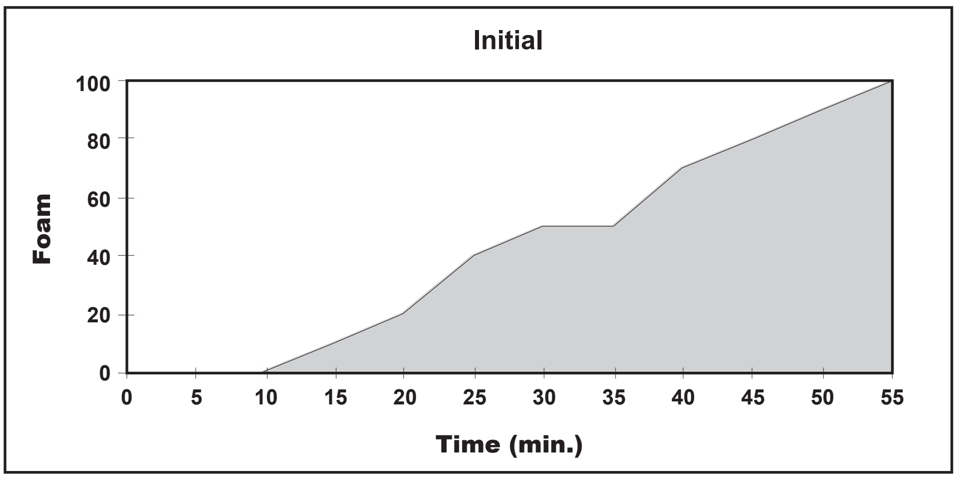

Figure 4. Initial foam profile.

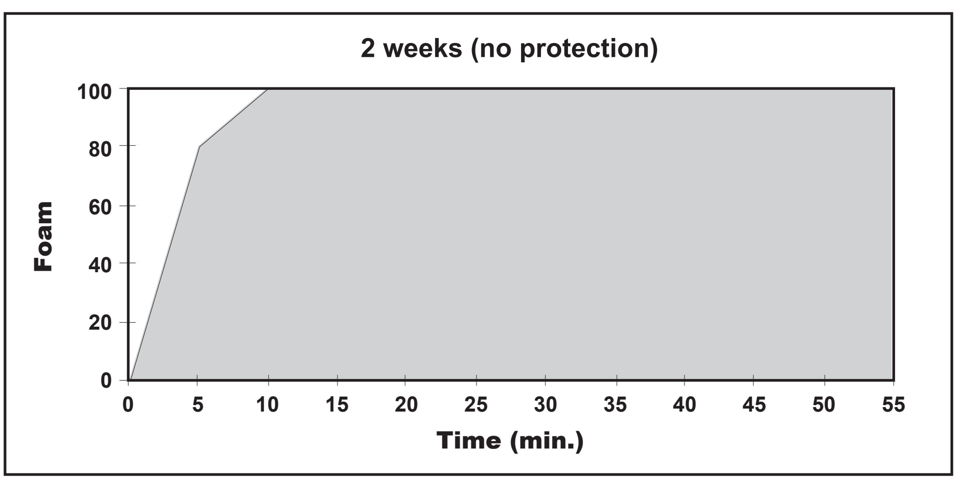

Figure 5. Foam profile after aging; no protection.

in the fluid bed, together with the fines separated in the cyclone (CY 3588). The finished granules are sent to an automatic loading station.

The SSAU Liquid Plant

There are two ways to handle the liquid feed. In the first, the mixing and emulsification of liquids can be done in a premix tank that feeds a process tank for continuous dosing. Alternatively, the mixing and emulsification can be performed in-line in a dynamic mixer. Liquids with viscosities from 10 to 30000 mm^2/s can be handled as can waxes with melting points up to 100°C.

An Illustration of Granulation Benefits

Encapsulated Antifoam

Foam control in laundry washing machines is critical because too much foam reduces cleaning performance, is detrimental for the washing machine and leads to customer complaints. Today the majority of West European powdered detergent manufacturers use an encapsulated antifoam. A granulation process produces this particular foam control agent, made of silicone antifoam compound entrapped in a carrier such as an alumino-silicate/polycarboxylate matrix.

Silicone antifoam compounds are mixtures of polydimethylsiloxane polymers and finely dispersed silica particles. Due to its low surface tension, the silicone fluid spreads at the air-solution interface and exposes the hydrophobic silica particles that act as foam breakers.

Delivery and protection of the silicone antifoam compound. To obtain an appropriate foam profile, the silicone compound must be dispersed in the wash liquor in the proper form and, in particular, with the right particle size. To accomplish this, the antifoam compound must be properly incorporated into the powdered detergent. Figures 2 and 3 show the initial foam profile obtained when the antifoam compound is properly incorporated (Figure 2) and is not properly incorporated (Figure 3) into the powdered detergent. The test was performed with 0.1% silicone active at 95°C.

Antifoam stability upon storage is another important factor to consider. Performance of the antifoam can be significantly reduced as a result of two phenomena. The first is degradation of the antifoam by aggressive components present in the detergent. The second relates to spreading of the silicone on the detergent powder, which leads to a smaller silicone droplet size. In this case, the droplet becomes too small to adequately control the foam. Figure 4 represents the initial foam profile. Figures 5 and 6 show foam profiles obtained after accelerated aging when the antifoam is incorrectly protected (Figure 5) and correctly protected (Figure 6). The tests were performed with 0.1% silicone active at 95°C.

Antifoam/optical brightener co-granule
Another example of benefits brought by the granulation technology is the antifoam/optical brightener co-granule currently under joint development by Dow Corning and Ciba Specialty Chemicals. This granule, which contains only detergent active ingredients, was shown to be a suitable candidate for providing new fluorescent whitening agent post-addition technology. It improves the storage stability of the optical brightener in the presence of activated bleach systems (Figure 7) while maintaining the initial whiteness performance (Figure 8). In addition to better performance of the optical brightener after aging, the granulation should help maintain the shade consistency of powder detergents upon storage.

The co-granule also reduced or eliminated the risk of undesirable brightener staining on fabrics. Antifoam performance of the co-granule before and after aging is similar to performance obtained with the encapsulated antifoam. In addition to these benefits, the co-granule offers supply chain benefits because two active ingredients can be dosed at the same time by post addition or dry mixing.

Conclusion

The encapsulated antifoam and co-granule are two examples that illustrate the benefits brought by granulation technology, both in terms of delivery and protection of active ingredients.

Dow Corning currently offers contract manufacturing for the detergent industry or other industrial applications. As part of these manufacturing capabilities, a full-scale production unit and small scale or pilot granulation facilities are available. Dow Corning provides a wide range of additional services and solutions for fast commercialization of granulated products, requiring little or no capital investment from our partners. These include:

- Laboratory development of formulations for granulation applications, based on specific customer requirements and marketing objectives.
- Feasibility trials on a continuous production unit.
- Quality control, supported by experienced analytical and characterization departments.
- Flexible packaging and logistics solutions.

Dow Corning's new contract manufacturing activity covers significant and successful applications such as granulation of surfactants, de-dusting and re-agglomeration of powders, and processing of raw materials for tablet-forming processes. In the future, Dow Corning will continue to use its granulation expertise and facilities to offer new solutions to detergent manufacturers.

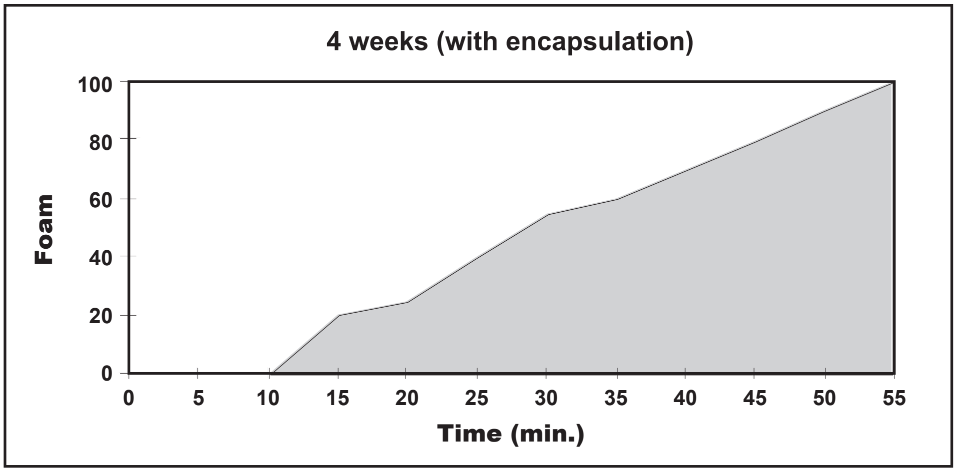

Figure 6. Foam profile after aging; good protection.

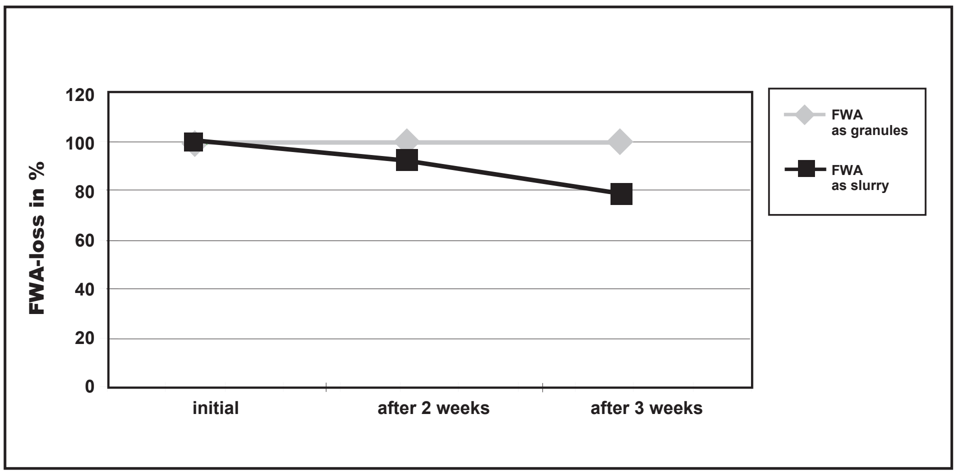

Figure 7. Optical brightener stability (accelerated aging).

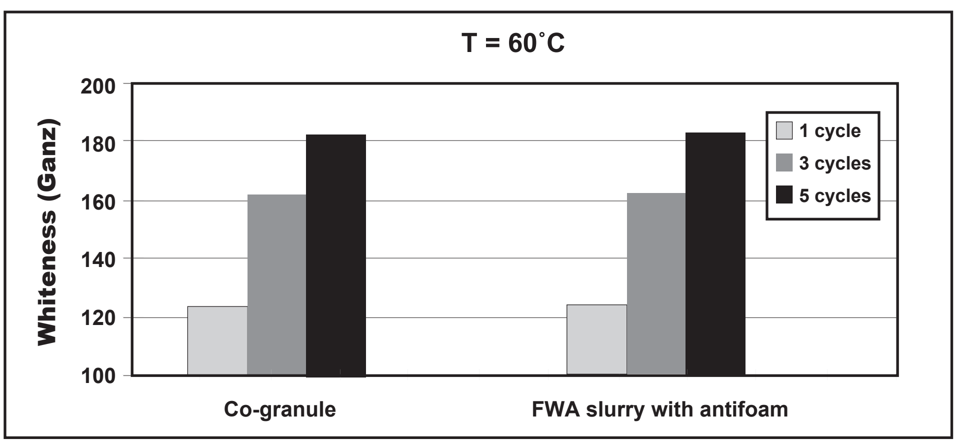

Figure 8. Initial whiteness performance (0.030 g FWA AS/kg fabric).

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