very good processability and control of in-use physical properties such as bar wear rate and sogginess or mushiness. In low-priced bars, a small amount of STPP is sometimes used for processability reasons. Performance-enhancing actives, such as chelants, polymers, fluorescent whritening agent (FWA), and recently, enzymes and bleach are often included as well, as are sodium carbonate, sodium sulfate, inorganic fillers such as calcite, talc, and clay. Carbonate and sometimes sodium silicate provide the necessary alkalinity, whereas the inorganic fillers give “body” to the bar and also act as process aids.

Surfactant level varies from 10–15% in low-priced bars to 20–30% in premium bars. Where a combination of surfactants is used, as in the Philippines, typically a mixture of alcohol sulfate and LAS is used in a ratio of 50 : 50–85 : 15. Bars containing only AS as the surfactant are very brittle and need a high amount of humectants such as glycerine or addition of hydrotropes to reduce the brittleness. STPP/TSPP level ranges from 0–3% in low-priced bars to 15–30% in premium bars. The moisture level in the bars varies from 3 to 12%, carbonates typically from 10 to 25%, and fillers such as calcite, talc, and clay fill the rest of the formulation. Because the presence of free water in the bars can lead to mushiness during production or use, a variety of desiccants and adsorbent materials have also been added to bars to control the moisture. Examples include phosphorous pentoxide, sulfuric acid, boric acid, and calcium oxide as well as a variety of clays.

19.1.2.7 Other Product Forms

Various novel forms such as gels, foams, and sheets have been tried in the past, each presenting unique challenges. However, none of these has resonated with consumers to date, and therefore, these remain niche products of low volume and will not be considered further in this chapter.

19.2 DETERGENT POWDER PROCESSING

Detergent powder processing, as mentioned earlier, is composed of a number of basic operations such as spray drying, agglomeration, and finished product making and handling. Each operation is considered in the following in text.

19.2.1 Processes for Making Detergents

19.2.1.1 Spray Drying

19.2.1.1.1 Introduction

Spray drying is the most important process used in the manufacture of detergent granules. It is the process route by which the main component of the vast majority of granular products is produced and the spray dried powder properties dominate the physical characteristics of the product. In the developed world, it survived the rise of the compact, agglomerate-based products, in the 1990s, the consumer preferring the lower-density product offered by spray drying. In the developing world, granular, spray dried products are the detergents of choice, and increasing prosperity in these regions has driven increased production volumes. Spray drying processes are capital intensive and typically quite large as shown in Figure 19.7.

The detergent spray drying process itself is well established. It was introduced about 60 years ago. Over the years, the process has been optimized considerably. In particular, major improvements have been made to production reliability, whereby plant utilization has increased from ~45 to over 90% in some locations due to the application of reliability engineering tools to minimize downtimes in production. The production rate of individual units has also increased quite dramatically as limits are understood and overcome by interventions such as airflow modifications and multilevel spraying. Rates of over 80 t/h are now achieved in single spray drying towers today, although smaller tower rates can be as low as 1 t/h. These two factors, along with radically improved transportation networks, have led to some significant consolidation of production units in the developed regions of the globe.

FIGURE 19.7 A typical manufacturing unit for spray dried detergent powders.

Despite its maturity, the spray drying process still has many challenges ahead. Formulations continue to change at an ever-increasing pace and frequently push the boundaries of the known operating envelope. Additionally, as the market becomes more segmented, the number of formulas increase. This, together with the drive to just-in-time production schedules (to minimize inventory) drives down production run length. Thus, start-up and shutdown times on these large-scale production units need to be significantly reduced to maintain plant utilization as well as good product quality.

19.2.1.1.2 Blown Powder Formulation

The spray dried granules are often known as blown powder. The components of blown powder are those of the detergent formulation that are robust to the operating temperatures within the tower. The core components are as follows:

Anionic surfactant—most often LAS
Builder—zeolite or STPP
Inorganic salts—sodium sulfate, sodium carbonate, sodium silicate
Polymers—polyacrylate, carboxy methyl cellulose

The properties of the slurry and the subsequent blown powder are dominated by the interaction of the LAS, which forms a liquid crystalline phase with the other ingredients within the slurry. Consequently, small quantities of some minor ingredients such as polymers, hydrotropes, and cosurfactants can have a significant effect on the slurry properties such as its rheology, and therefore, the amount of water required in the crutcher mix.

The structure of the blown powder is also dictated by the crystallization of the inorganic phases. In this respect, silicate and polyacrylate both improve granule crispiness and toughness.

19.2.1.1.3 Process Description

The spray drying process enables efficient, countercurrent, contact of an atomized detergent slurry with hot air, producing a detergent granule. The process itself can be split into five sequential operations as follows (Figure 19.8):

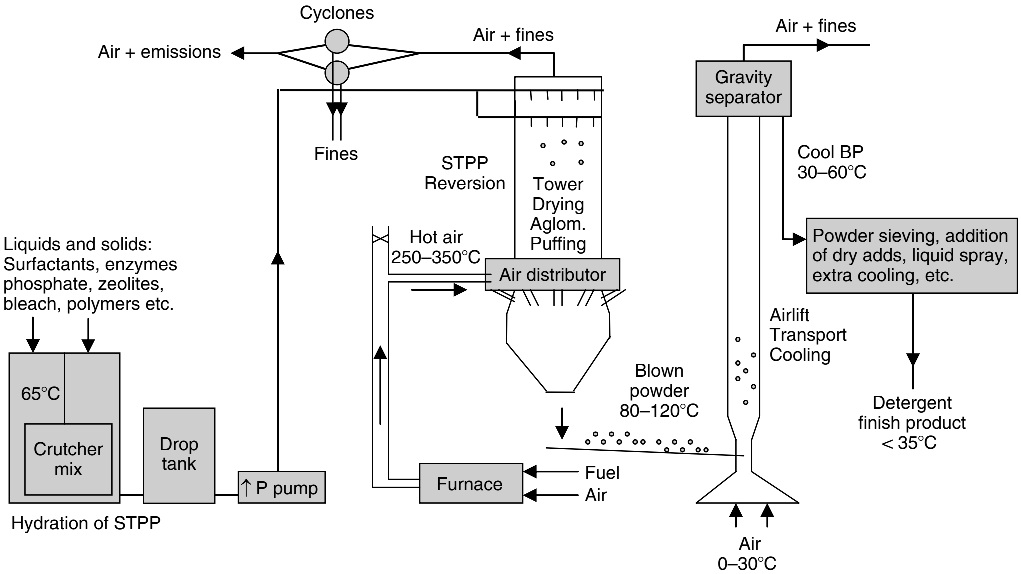

FIGURE 19.8 Schematic drawing of a spray dried detergent unit.

Slurry making
Pumping
Atomization
Drying
Cooling and classification

These operations are considered in the following text. Raw material storage, handling and dosing are common to many industries are not considered in this chapter.

19.2.1.1.3.1 Slurry Making

The objective of this process is to make a homogeneous slurry of consistent composition and aeration, with minimum water content, that is, the lowest possible drying load. The first challenge of incorporating powder and liquid raw materials is achieved by a continuous or batch mixer, known in the industry as a crutcher. Both of these types of mixer have been optimized by experience, and advocates for both methods will argue their benefits, for example, in minimizing aeration or in their ability to handle lower water content slurries. In reality, there are rarely two mixers that are the same and it is unclear whether either has a fundamental advantage. Over recent years, as production scales have increased and control technology improved, the systems for dosing these mixers have become highly automated and more accurate. In addition, the increase in production rates in the drying tower has meant considerable work to reduce the dosing and mixing times, and therefore avoid large capital investment and plant shutdowns during upgradation. From the crutcher, the slurry is transferred to a holding tank, sometimes known as a drop tank; here, further mixing occurs and the slurry ages allowing various phase formations and crystallization processes to take place. Filtration is carried out to achieve further homogenization and ensure that the spray nozzles do not block the final stage of slurry making. Typically, magnetic filters are used followed by disintegrators, which incorporate a filter with a smaller orifice size than the nozzles. Systems for adjusting slurry temperature and aeration, by injecting stream or air into the slurry, can be incorporated at any point in the process.

19.2.1.1.3.2 Sodium Tripolyphosphate Products

STTP was, for a long time a significant part of detergent formulations. Not only is it good at controlling water hardness, it also has good sequestering properties and provides buffering capacity and a crisp, robust granule. It is still used in many countries and can present processing challenges.

The reason for this is that it hydrates to the hexahydrate during the slurry making and continues to hydrate after the making process. Such hydration reduces the free water in the mix and thus can have a big impact on viscosity. The material has two different crystalline phases—I and II. The former hydrates at a much faster rate than the second. The hydration rates in both cases are sensitive to temperature also—lower temperatures giving more hydration.

Too much hydration in the slurry can lead to extremely high viscosities, even beyond the processing capability (instances are known whereby the slurry has set solid due to overhydration). In addition, the dehydration of the hydrate during spray drying can lead to degradation of the STPP to poorer performing molecules. This is known as reversion.

In contrast, too little hydration can lead to the production of a poorly hydrated blown powder. This then tends to pick up moisture and hydrate on storage and can lead to overheating and caking. Therefore, good control of STPP hydration is crucial for steady operation and for producing a good-quality product. Thus, it is important to have a consistent quality of the raw material.

19.2.1.1.3.3 Pumping

As a result of the low moisture content of the slurry and the short mixing times, the slurry can have inorganic lumps, which would block the atomization nozzles. The typical way of resolving this is to pump the mix through a filter and provide a disintegrator to break up any such lumps before passing to the main slurry pump. To achieve the high pressures (up to 100 bar) required for atomization, one or more piston pumps are typically used. In most cases these require a booster pump, typically a positive displacement pump, to keep them efficiently filled with viscous aerated slurry.

19.2.1.1.3.4 Atomization

The objective of the atomization process is to create drops small enough to dry in the spray drying tower. This is done with a number of high-pressure nozzles known as hollow-cone pressure swirl nozzles. These nozzles are distributed at one or more levels within the spray drying tower and have to be sufficiently distant from the wall to avoid buildup caused by wet drops sticking before they have dried sufficiently. For this reason, and for reasons of residence time, smaller towers typically run with smaller nozzles.

Two problems are often encountered with spray nozzles: nozzle wear and nozzle blockages.

Nozzle wear. The abrasive slurry causes the nozzle tips to become enlarged and rounded with time, even with the hard tungsten carbide or yttrium carbide nozzles that are typically used. This wear causes bigger drops, wall buildup, and poor product properties. This is particularly the case with zeolite-based slurries where nozzles must be carefully monitored and changed every few weeks.

Nozzle blockages. Occasionally nozzles get blocked, therefore several spare nozzles are usually kept available during operation. With a suitable filter size, good operating, and clean-out practices, these occurrences can be minimized. However, most towers have inspection windows that allow operators to monitor atomization during operation. Alternatively, constant monitoring of the spray dried particle size can indicate upstream problems before their impact can get out of control.

Typically, a nozzle will produce a wide range of fine droplets, which do not have time for the surface tension to pull them into a sphere before they are dried. In addition, the different sizes tend to agglomerate in the turbulence, and thus, a spray dried granule is more like an irregular-shaped bunch of grapes. Problems can occur if the nozzles are too close to one another in the tower, leading to overagglomeration and hence too many coarse particles. Figure 19.9 shows a typical sample of granules.

On further magnification, the knobbly shape and internal porous structure can be seen as in Figure 19.10—a scanning electron microscope (SEM) picture.

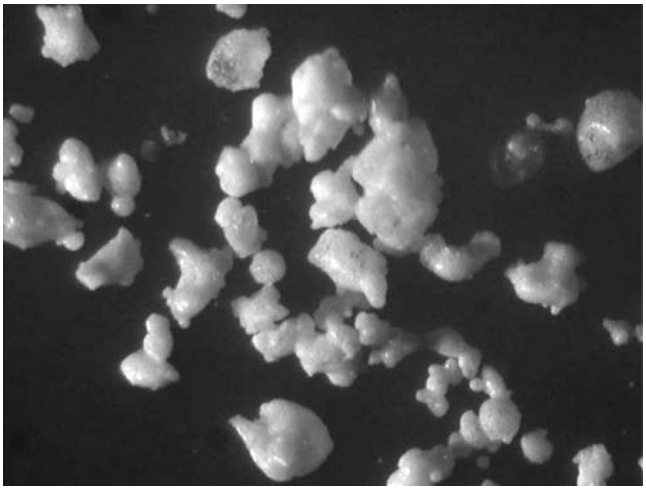

FIGURE 19.9 Typical spray dried detergent particles.

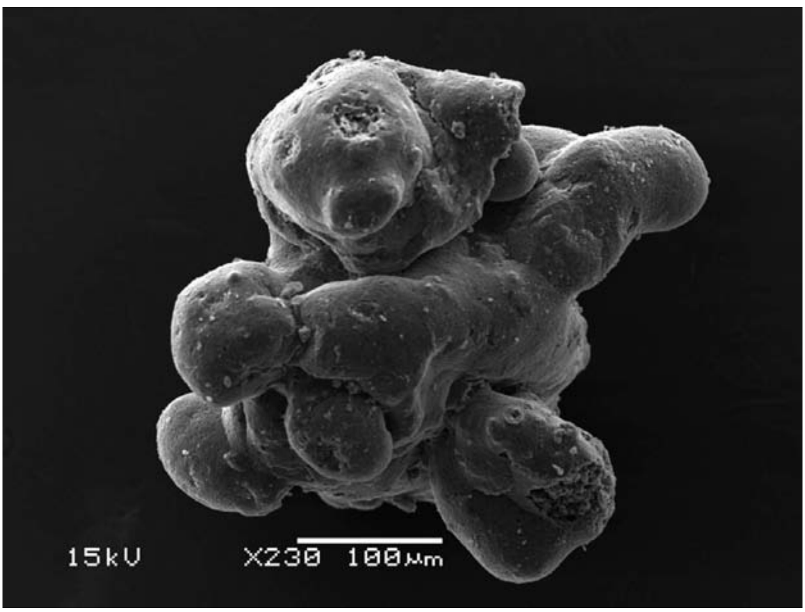

FIGURE 19.10 Spray dried particle showing typical agglomeration.

19.2.1.1.3.5 Drying

Countercurrent spray drying towers are used to dry the droplets and operate with an inlet temperature of ~300°C. The tower design has not changed much over the years; a typical tower outline is shown in the schematic Figure 19.8. The cylindrical section of the diameter is typically in the range 3–10 m. The geometry of the tower around the hot air inlets is dictated by the need to minimize any contact of the hot air with the built up product, and thereby reducing the amount of browning that occurs. This is an important area since higher air temperatures improved thermal efficiency and lead to increased production rates. However, if the distribution is poor, overheating of the powder occurs, leading to potential exothermic runaway and subsequent fires in the tower.

The hot air is generated by an oil- or gas-fired furnace and is uniformly distributed around the tower circumference using a plenum ring. Care must be taken for oil furnaces since poor combustion can lead to black specks entering the tower and causing discoloration of the blown powder. Failure to get a uniform distribution of air or temperature can lead to poor product quality, whereby some granules can be underdried and sticky and some can be overdried and brown. The inlet air can enter radially or with a swirl, that is, some tangential component of velocity. The swirl tends to stabilize the airflow and decrease the exhaust temperature a little, improving thermal efficiency. The exhaust air is ducted out of the top of the tower and any fine product carried over is removed using technologies such as cyclones, bag filters, and electrostatic precipitators.

19.2.1.1.3.6 Cooling and Classification

The powder temperature exiting the tower base is typically over 70°C. This needs to be reduced to allow temperature-sensitive additives to be mixed in. Typically, this is done in an airlift, which transports the powder to the top of the building. A gravity separator is then typically used to disengage the particles from the air. This type of system also performs an initial classification of the powder as large lumps are not transported up the airlift and fine particles do not disengage from the gravity separator. (These fine particles are subsequently removed by a bag filter.) Further classification is often required to remove coarser particles. This is typically done using mechanical screens.

19.2.1.1.4 Operation

19.2.1.1.4.1 Overview: Control and Changeovers

The detergent drying process is a large-scale process, and in modern installations digital control systems are used to control the plant. However, the control of product properties such as density and blown powder moisture tends not to be fully automated, partly due to the multivariable nature of the product properties and lack of robust measurement systems. Therefore, there tends to be a human operator responsible for the starting-up, center lining, and shutting down of the process.

Multiple formulations tend to be run on one tower and with increasing formula numbers, run times for a particular formulation are decreasing. Therefore, the challenge for the tower operators is to quickly bring the system to centerline and acceptable product properties, thereby minimizing the amount of material that has to be recycled back to the slurry.

19.2.1.1.5 Powder Properties

In the majority of granular detergent formulations, the physical properties of the products are determined by the blown powder properties. The key properties are as follows:

- Density
- Particle size distribution
- Cake strength—flow properties
- Solubility

Density is typically 250–550 kg/m^3 and determined by many variables such as the formulation, slurry air content, and process conditions. Density is an important quality item since most consumers dose by volume, and therefore, the bulk density is the prime variable to control. Typically, droplets tend to form a skin on the outside as they are dried, leading to the internal water diffusion out of the drop being very slow. In certain cases, this water can turn to steam and expand the droplet causing puffing, and hence, reduced density. The extent to which this can occur will depend on the water content of the slurry, air temperature, and chemical composition. Generally, lower density is caused by a higher slurry water content, higher air temperatures, and a high concentration of film-forming materials. Incorporation of air is also used at high pressures to increase this expansion. However, care must be taken not to overdo this effect since too rapid an expansion can lead to the fracture of the granule and result in an excess of fine material.

An additional factor in bulk density is the ease with which the particles pack. One practice is to spray perfumes and de-dusting agents onto the powder. If these are not absorbed into the granules the liquid can stay on the outside of the particles, leading to poor flow properties and a reduction in bulk density, which is difficult to control.

Particle size distribution. A typical volume-based median particle size is ~400–500 μm. The spread of the distribution is typically wide with some significant fine and coarse powder resulting from the agglomeration process, which takes place within the tower as wet drops collide. Acceptable ends of the spectrum typically range from <5% below 100 μm to <2% above 800 μm. The former is for dustiness of the powder and subsequent dispersion when wet. The latter is to control appearance and dissolution of the powder over time during the washing process.

Cake strength. The cake strength as measured by a uniaxial shear test is often used as a measure of flowability and granule stickiness. These are important properties for both postprocessing and consumer acceptance. The cake strength tends to be an intrinsic function of the formulation and blown powder moisture. Apart from this, the process conditions tend to have only a minor effect.

Solubility. The consumer prefers a fully soluble granule, therefore insoluble residues left by a granule are undesirable. The key factor for solubility tends to be formulation and component interactions, although process conditions can play a role as well. Some of the better-known interactions that can cause insolubles involve sodium silicate—a common ingredient. Poor mixing control with acidic ingredients can result in the production of insoluble silica, and interactions can also occur with zeolite formulations, which will also cause large insolubles. In both cases, careful control of mixing and pH is needed to avoid these problems.

19.2.1.1.5.1 Makeup

One of the common factors that can impact good operation is the attachment of wet slurry to the tower walls, where they dry and form a base for more droplets to stick. This makeup can lead to large lumps inside the tower, reducing thermal efficiency. In the worst case, the makeup near the hot air inlet temperature can result in the overheating of the dried slurry and generate brown and black speckles, which ruin the appearance of the product, leading to shutdown and cleanout. If this is not done, the powder can self-ignite. Makeup is most often caused by poor nozzle positioning and poor airflow control. It is therefore important to ensure these are resolved during the commissioning of a spray drying tower.

19.2.1.1.5.2 Future Developments

As mentioned earlier, the current spray drying process is very well established but is still undergoing constant change and optimization in the drive to reduce costs and increase formulation flexibility and product quality. This is most relevant to the large production units, which make a number of different formulations. As new tools become available, they are applied to these units. Thus, reliability engineering has increased plant utilization, CFD modeling has improved tower airflow control, and process control advances has reduced start-up and shutdown times. Further improvement in modeling capability will allow much better control of product quality and allow the operating windows to be widened in the drive for more cost-effective density and moisture control.

19.2.1.2 Agglomeration in Detergent Processing

19.2.1.2.1 Introduction

Agglomeration is a very widely used process in detergent making. Agglomerators range from high-shear, high-speed mixers to low-shear fluid beds. Processes can range from small-scale batch making to large-scale continuous production. In other words, agglomeration is simply the “sticking together” of smaller particles into some sort of combined entity. This is often not difficult—the real challenge lies in making agglomerates of the desired properties. Agglomeration has been widely used in the