

# Spray Drying and Dry Neutralization of Powdered Detergents

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# ABSTRACT

Spray tower designs for soaps and detergents, practical operating conditions and practices in slurry preparation, and spray tower operation are described and illustrated. The specific problems of exhaust air purification, handling of fines, and the behavior of various builders and surfactants during spray drying are discussed. Dry neutralization systems and the combination of spray drying with dry neutralization to introduce heat sensitive and difficult to atomize materials, to use practically all types of surfactants, to increase capacity, and to save energy are considered also.

## INTRODUCTION

Presently about 9 million tons of powdered detergents are produced annually. Powders solely based on soap are still produced, but their quantity is only about 360 thousand tons. A relatively large quantity of the detergent powders is based on ternary mixtures of anionic surfactants, nonionics, and soap as the active material. In this respect soap had, for about 20 years, a small comeback, being now a constituent of many detergent powders, especially where foam control in washing machines is essential. It is difficult to estimate the exact percentage of spraydried powders of the total sum of powders produced in the world, but conservatively estimating it is at least 75% of the total.

Historically the first spray dryer was patented as early as 1865 (1), but its large scale introduction in the soap industry began in the 1920s. The powders obtained had a fine powdery structure being similar to the soap powders obtained by older systems. The use of a spray dryer then resulted in better mechanization, labor savings, and less health hazards against the old "tennen process." In this old system a mixture of soap and builders, mainly soda ash and silicate, was produced as a kind of pasty mass in a strong mixer and then discharged onto the ground where it was left to solidify, later broken up, and ground in a hammer mill to a fine but very dusty powder.

With the rapid rise in synthetic detergent production after the Second World War, a decisive forward step was made in the field of spray-drying technology; namely the introduction of high pressure spraying to produce our modern detergent powders in the form of beads. Actually, the importance of bead structure was recognized in 1927 (2).

In spite of the relatively high capital outlay for a modern spray dryer, the advantages and results justify the investment. The advantages are:

1. Variations in formulations are within a very wide range. Relatively high amount of active matter can be incorporated. The choice of builders, too, is practically unlimited. Bulk density of the spray-dried powder beads from the tower is variable within limits too, generally between 0.3 and 0.4. Moisture content depends to a certain extent on the water absorption by the builders used and the percentage of active matter in the finished product. The range is from 4 to 10%.

2. The product has a pleasing appearance, and, being light, has more sales appeal.

3. Spray-dried powders are dustless and free flowing and do not tend to lump. For normal formulations no special inner liners are required in packaging.

4. Because the hollow beads have a large surface area, the powder dissolves instantly when added to water. This is important when powders are used in washing machines with as short as 4 minute wash cycles. If the powder takes time to dissolve, valuable washing time is lost. Also, in tub washing, where no mechanical agitation takes place, portions of powders other than spraydried powders can still remain undissolved even when the operation has been completed.

5. Choice of active matter is practically unlimited. Only very heat sensitive surfactants like alcohol ether sulfates and nonionics with a long ethylene-oxide chain and surfactants with a strong tendency of stickiness at elevated temperatures should be excluded from spray drying.

The method of introducing sensitive materials by special cold mixing processes, where the active matter is premixed with builders and the resulting mixture added to the spray-dried powder will be described later.

#### SPRAY DRYING

## **Batch Slurry Preparation**

The first step in spray drying is production of the slurry. The older batch system for slurry preparations involves considerable material handling and labor. Also at least two batch preparation mixers (crutchers) are required. From one the slurry is pumped into the tower, while in the other a fresh batch is being prepared.

The size of each crutcher should be large enough to hold sufficient slurry to spray during the time it takes for another batch to be prepared and checked. If the crutchers are comparatively small, too many batches must be prepared per shift. If the crutchers are large, the slurry must be kept warm and stirred during the waiting period which also includes the time needed to pump the slurry into the tower. Also it is rather difficult to produce very large quantities of homogeneous slurries. Thus there is a tendency in a batch system to produce a less concentrated slurry with a lower solids content. Anyway, a great deal of power is required to obtain this homogeneity and to keep the slurry stirred until it is pumped to the spray nozzles. Furthermore, due to a prolonged residence time in the slurry mixers, changes in the rheological structure of the slurry occur especially caused by hydration of STPP. Also other components in the slurry tend to change its structure.

CMC may form a gel structure, active matter, especially soap, causes increase in viscosity owing to the effect of electrolytes, i.e., the builders. Very often the slurry turns

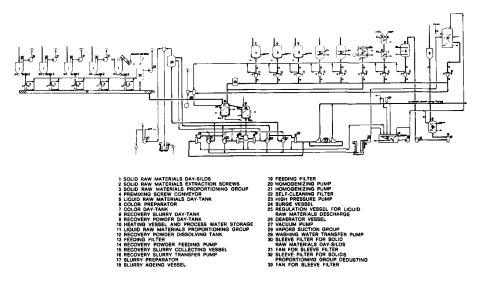


FIG. 1. Continuous slurry preparation.

into a stiff mass, which is very difficult to liquify again. The addition of water not only causes a reduction in the output of the tower, thereby increasing fuel consumption, but also irregularities in the slurry structure and fluidity, leading to lack of uniformity in the quality and structure of the powder coming from the spray tower. Thus, generally, there is a tendency in a batch system to work with a more dilute slurry, which results in a decreased output and increased fuel consumption.

Discontinuous slurry preparation is still used, however, especially for spray towers with a relatively small output, 1000 to 2000 kg/hr output and when labor costs are relatively low. Some systems combine automated dosing and weighing with a batch slurry preparation.

The shortcomings of the batch process, however, are avoided in an efficient continuous slurry preparation system. The crutchers are smaller, heat control is easy and efficient, residence time is shorter and well defined. This assures that changes in slurry structure are well under control, and homogeneity is improved. A high solids concentration in the slurry is thus obtained, and less water has to be evaporated resulting in a higher output with less fuel consumption.

#### **Continuous Slurry Preparation**

The system for continuous slurry preparation was first reported in 1964 (3). Since then great progress has been made in the field of automatic dosing with load cell scales, improvements in the solid premixing system, and many other details of the basic system.

Actually the improvements are based on the practical experience of the older system based on more conventional weighing scales. With the application of the modern load cells, weighing is carried out without rotating parts. There is an analog or digital weight indication, complete protection of the dosing system against outside influence and moisture, temperature, and dust. There is an automated selfcorrecting tare control.

The new continuous system is based on a large number of discontinuous weighing operations for active matter in liquid or paste form, solutions of optical brighteners and for solids, alkaline builders, sodium sulfate, and CMC. The system has the precision of batch weighing and the advantage of continuous material flow. Setting of the proportion of each component of the detergent formula on the control panel can be easily adjusted for new formulas.

Figure 1 depicts the basic features of the system. On the left side the silos for solids, which are static material containers instead of the older rotating drum, are connected to a strain gage transducer with tare control and compensation. The solid proportioning group is fitted with a sleeve filter for dust-free operation. At the end of the solid proportioning line there is a screw conveyor premixing the proportioned solid before transfer into the slurry crutcher.

On the right side there are the liquid material storage tanks and the strain gage weighing system, similar to the solid weighing system. The electrical output signal from the strain gages is amplified and utilized to effect the opening and closing of the feeding and discharge operations.

Also depicted in this figure is a system for preparing color solutions and/or optical brighteners. All those liquid components which may be premixed provided they are not reacting with each other are fed into the liquid discharge vessel.

Besides the advantages of being insensitive to the ambient temperature, dust and moisture, accurate and simple, the strain gage system allows for self-correcting automatic tare setting. This system is especially well adapted for handling viscous liquids or pastes, which may leave residues on the weighing container, or solids which stick to the walls. A memory records the residual weight left on the container and cancels it before beginning the next weighing. Therefore, the actual value of weight is by difference between two weighings and not by absolute value.

The premixed solids and the liquids and/or pastes are transferred into the slurry mixers. This is a high-speed crutcher designed to break up agglomerates. No air is entrapped during the slurry preparation step.

# **Slurry Aging**

From the crutcher, the slurry passes by overflow into the slurry aging vessel. Aging of the slurry is very important for the homogeneity and structure of the slurry and subsequently for the quality and uniformity of the beads coming from the spray-drying tower. Rheological structure of the slurry may eventually be checked continuously with a dynamic viscosimeter, e.g., a modified Brookfield type viscosimeter, which may be inserted at the overflow from the aging vessel.

It was found that varying the residence time, normally between 20 and 30 minutes, in the aging vessel may give for each specific slurry composition optimum structure of the slurry.

A level transmitter based on measuring the liquid pressure-head transmits a signal to the weighing system changing the frequency of the weighing system. If the level is too low, frequency increases; if the level is too high, frequency decreases. By adjusting the position of the level transmitter, the level in the aging vessel, and thus the resi-

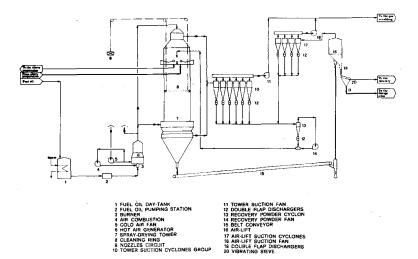


FIG. 2. Plant for spray drying of detergents and soaps.

dence time may be fixed. On the other hand, the output from the nozzles of the spray tower decisively determines the flow rate of the slurry preparation system.

Sometimes it may be desirable to neutralize and/or saponify alkylbenzene sulfonic acid and distilled fatty acids in a continuous system instead of using neutralized AB-sulfonic and/or curd soap. In this case, the neutralization vessel is set up ahead of the slurry crutchers. AB-sulfonic acid and/or fatty acids are proportioned into this neutralization vessel with sufficient caustic soda solution to complete neutralization and saponification. The pH is maintained at ca. 8.5 to 9.0. The neutralization system is, of course, coordinated and integrated into the proportioning system as a whole.

This might be an advantage when AB-sulfonic acid is available from a nearby SO<sub>3</sub>-sulfonation plant or arrives by tankcar. Especially the less viscous linear ABS is easily pumped into the slurry preparation system and requires less power for pumping and relatively smaller diameter pipes than needed for pumping a neutral ABS paste.

Soap too might easily be prepared at the same time from distilled fatty acids, particularly in cases in which a soap production plant is not available near the spray-drying installation. Actually, all kinds of variations in production can be accommodated by the automatic slurry preparation system.

In this connection we want to mention a new development; in order to overcome problems with sodium tripolyphosphate in the slurry, a new type of STPP has been developed by the German firm Hoechst-Knapsack. This special STPP is prehydrated with a certain percentage of water. This prehydrated STPP permits smoother running of the slurry preparation system and permits a very high solids content of up to 67%. Unfortunately this rather recent development comes at a time when there is a tendency to replace phosphates by other compounds, such as zeolites, organic sequestering agents, special organic polymers, and citrates.

From the aging vessel the slurry now passes through a system of filters. The first filter retains rather coarse and very hard material. From there the slurry passes through a homogenizing pump, which acts as a homogenizer and medium pressure pump. The slurry leaves the pump at about 7 atm pressure. From there the homogenized slurry is deaerated under reduced pressure and forced through another self-cleaning filter with a finer mesh. Other slurry refining systems use different kinds of so-called delumping devices and various types of filters and a series of pumps before the high pressure pumps.

From the last self-cleaning filter the now refined slurry passes to the high pressure pump. The construction of the high pressure pump, which in a modern plant should reach up to 100 atm pressure, is very important.

It was found that a short stroke is very important for such a high pressure and also accounts for the long life and smooth running of the pump. To permit variations in pressure for diverse slurry compositions and, as we will see, also for regulating bulk density, the pump is provided with a variable speed motor.

# Spray Tower

The actual spray-drying process is shown in Figure 2. The nozzles are arranged to assure that the sprayed slurry does not touch the wall of the tower. The orifice of the nozzles ranges from 2.5 to 3.5 mm. There are enough extra nozzles in the top of the tower in case they must be cleaned during the running of the plant. For this eventuality a steam cleaning system is provided, which in a modern plant may be operated from the control panel. Drying is accomplished by a current of hot air. The direction, speed, and temperature of this air current determine to a very large extent the properties of the powder beads. Hot air is generated in a special direct fired furnace. Complete combustion of the fuel is essential because incompletely burned fuel, which gives off carbon, darkens the finished product. Warm air is circulated by three blowers. The first two are for carrying the air through the furnace and the hot air fuel gas mixture to the spray tower. The third blower, situated after the cyclones, produces the required pressure drop and suction effect in the drying chamber. Some outside air is sucked into the bottom cone of the tower cooling the dry beads. Flaps in each air stream regulate the flow of the diverse air currents. The quality of the hollow beads depends, to a large extent, on the direction of the air circuits. Other factors, too, influence structure, density, and quality of the beads: structure and solids content of the slurry, degree of refining of the slurry reaching the nozzles, and the pressure in the high pressure pump.

Basically, there are three different air flow systems: (a) parallel or co-current, (b) countercurrent, and (c) mixed. With parallel air flow, beads of very low density may be obtained, i.e., 80-150 g/l and with a maximum moisture content of 3 to 8%. Bulk density may be automatically determined and recorded with a special instrument based on weighing a constant volume flow of powder from the tower.

Concurrent air flow produces beads with a density of 150-450 g/l and a moisture content of 6-15%.

With the mixed air flow, density is variable. As may be seen from the diagram, air is liberated from the dried beads, which later fall on the belt conveyor at the bottom of the tower.

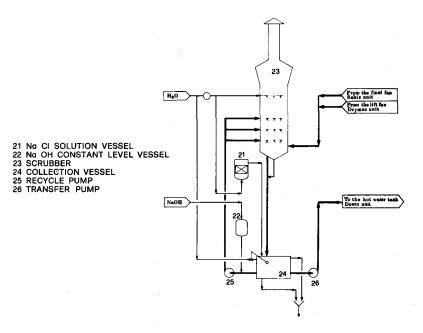


FIG. 3. Discharge gas scrubbing.

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Utility Data for Spray Dryers<sup>a</sup>

Nominal Con capacity si	Installed g	Installed power (kw)			Compressed air	
	Continuous slurry preparation	Batch slurry preparation	Gas fuel consumption (kg/hr)	Steam consumption (kg/hr@6 bar)	consumption (nm <sup>3</sup> /hr@ 10 bar) **	Water consumption (m <sup>3</sup> /hr)
250		70	25	100		
500	125	120	40	200	18	0.3
750	140	135	60	225	25	0.4
1,000	155	150	80	250	27	0.6
1,500	185	175	120	250	27	0.7
2,000	210	195	160	300	30	1.2
3,000	250		240	300	33	1.3
4,000	275		320	350	33	1.9
5,000	300		400	350	36	2.3
6,000	405		480	380	38	2.7
7,000	460		560	420	38	3.1
8,000	485		640	450	40	3.8
9,000	505		720	475	42	4.0
10,000	530		800	500	45	5.0
15,000	895		1200	700	54	6.5
20,000	1120		1600	1000	66	9.0

<sup>a</sup>Facilities are for plants with de-aeration system and recycle of fines. (\*) For SABIZ 8000 kg/hr, and more, including airlift. (\*\*) For plants with continuous slurry preparation only.

#### Handling of Fines

Fines are carried with the air to the cyclones. The number of cyclones depends on the size and output of the drying tower. In the spray-drying plant as shown in Figure 2, the fines from the cyclones are reintroduced into the upper part of the tower where they come into intimate contact with the still wet spray of slurry falling down from the nozzles.

Each cyclone is provided with a double-flap discharge device which opens and closes automatically. Thus, the whole recirculation system for the fines is completely and automatically integrated into the system. This new system eliminates any fines from the spray-drying plant. Besides this obvious advantage of cutting out fines and increasing the output of beads, this arrangement permits a dust-free operation and eliminates the rather unpleasant handling of separated fines. If no system for reintroduction of fines into the spraying chamber is available, the fines are to be introduced back into the slurry. Sometimes they find use as the detergent base for scouring powders.

#### Exhaust Gas Scrubbing

Rules against air pollution have become very stringent all over the world. To achieve the discharge of very pure exhaust air, the spray-drying plant is provided with an efficient wet-scrubbing system. Scrubbing with brine solution permits efficient scrubbing without undue formation of foam in the scrubbing solution. In case of fuel-containing sulfur, NaOH is used for scrubbing out  $SO_2$ .

As may be seen in Figure 3, the scrubbing solution, when it has reached its saturation point, is recycled to the water tank for the slurry preparation unit. The amount of material to be recycled is not significant enough to change the composition of the powder formulation.

The spray tower is provided with a cleaning ring, and at regular intervals the ring is slowly moved up and down the straight sides of the tower. The ring brushes off excess powder before it can build up. It also makes periodic cleaning of the tower superfluous.

In connection with the pollution problem in spray dryers, we would like to mention the very detailed work

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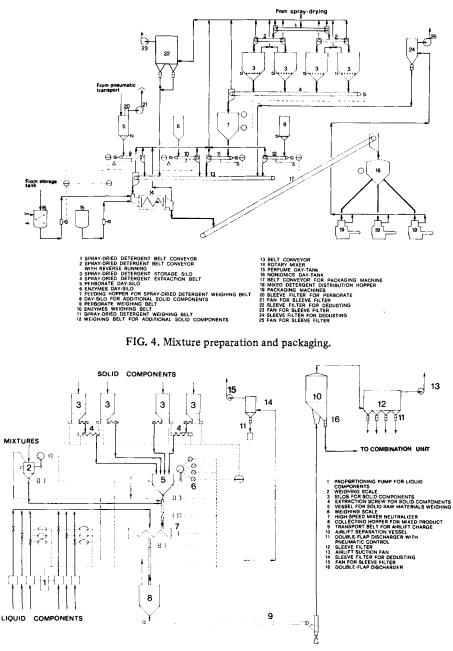


FIG. 5. Dry neutralization of alkylbenzene sulfonic acid and fatty acids.

dealing with the formation of organic aerosols in the exhaust gas from spray towers (4). Organic aerosols, according to this research group, are formed especially from the unsulfonated matter of sulfonates or sulfated anionics and also from nonionics with relatively low vapor pressures. It was found that the aerosols are mainly caused by a kind of vapor distillation of organic material. Cracking and/or oxidation, according to this research, is generally not, or less, responsible for this aerosol formation. Our experience with an efficient scrubbing system indicates that these kinds of aerosols are retained in the scrubbing liquid as described. Of course, it must be conceded that high amounts of "volatile" organic matter may eventually pass through. Naturally, perfectly sulfonated anionic components should be used, and nonionics liable to vapor distillation in the spray-drying system should best be excluded from the formulation in the slurry. Those components, if considered essential in a formulation, should be introduced outside the spray dryer, namely in a kind of premix system. This subject will be discussed later.

The powder from the tower passes via a belt conveyor to an airlift (Fig. 2). The airlift has the additional beneficial effect of further cooling the powder and facilitating crystallization.

Utility data for spray towers of different outputs are shown in Table I. Labor requirements for smaller plants with continuous slurry preparation are about four skilled and unskilled workers, and up to seven workmen for the very large plants with 10 to 20 ton/hr output. In actual practice, especially with slurries produced with solid content of 60% and more, and with active matter permitting operation at the higher temperature level for the hot air/ fuel gas mixture entering the tower, the consumption of electrical power, fuel, and steam will be considerably below the data given in the table.

The following parameters give indications for the actual running of a spray-drying plant:

1. The temperature of the hot air mixture entering the tower is around 250-350 C. Generally with powders having a high active matter content the temperature is maintained at the lower level, whereas with less active matter content at the higher level.

2. The exhaust from the tower is at around 90-110 C.

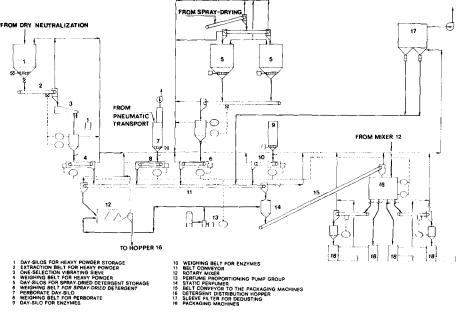


FIG. 6. Combination unit for spray-dried and dry-mixed powders.

TABLE II

Particle	Sizes

Particles retained on (ASTM sieve number)	Heavy powder (%)	Spray-dried powder (%)	Mixed powder (1:1 ratio)
14	2	2	2
25	16	25	24
50	37.5	54	46
70	40	15	26
100	4	4	2
Below 100	0.5		

3. The jet of slurry at the exit of the nozzles is 80-100 C.

4. The temperature of the powder coming from the lower cone of the tower is about 60-70 C.

5. When leaving the airlift, the temperature of the powder is around 35 C.

6. The quantity of air introduced is 10,000-15,000 m<sup>3</sup> for each ton of powder, when the slurry has a solids content not less than 60%.

Figure 4 depicts the finishing operation for powders in which there are special additives like enzymes, perborate, or percarbonate, eventually the new zeolite type builders, and the new phosphate replacements now being widely discussed (5).

### DRY NEUTRALIZATION

This brings us now to powder production by dry neutralization and describes the eventual combination of the two powders in a coordinated completely automated system.

Ternary mixtures of anionic surfactants, such as linear alkylbenzene sulfonates and/or higher fatty alcohol sulfates, soap, and nonionics are now used in most of the high grade detergent powders on the market. The trend is to increase the ratio of nonionics in these ternary mixtures. These combinations of surfactants, with a high ratio of nonionics, make it possible to obtain good detergency and also good foam control in modern household washing machines.

However, an increase in the percentage of nonionics above a certain level may cause difficulties in the spraydrying process. Stickiness, fragibility of powder coming from the tower, stickiness, and the volatility problem, are sometimes difficult problems to overcome. Qualitatively, many of the more efficient nonionic surfactants, from the point of view of detergency and biodegradability, are heat sensitive and therefore difficult to atomize.

A further trend is to increase the bulk density of the final powder above the usual level of 0.3-0.35. This again, combined with the trend to use nonionics in high percentages, has added another headache for the plant engineer. Indeed, to produce powder in bead form and high percentage of active matter with a density above 0.4 is rather difficult. The process to be described (Fig. 5) serves to overcome these difficulties and makes it possible to work out optimum detergent formulations without causing difficulties in the production process (6).

With all the new developments in the detergent industry, flexibility in processing plants has become a primary consideration in the trade now. To obtain this flexibility is the purpose of the dry neutralization process. This process consists of a dispersion system which distributes the active matter components onto the detergent builders. This very homogeneous dispersion of the surfactants onto the builders is accomplished in a mixer which has especially designed plough type horizontal mixing blades rotating at high speed and passing very close to the mixer walls. This design guarantees very efficient blending of all the components. In addition, ultra high speed rotating disintegrators are inserted to prevent any lump formation.

In contrast to "spray-mixing," "fluid bed," and "agglomeration" processes, fixation of the active matter components is accomplished by mechanical disintegration of the solid builder material onto which the active matter components are added by means of a rather simple dispersion system. In most spray-mixing systems special high

TABLE III

Formulas for Powders Produced by Dry Neutralization

Component (%)	1 All-purpose non-machine	2 All-purpose machine	3 Light duty (hand)	4 Heavy duty (hand)	5 Heavy duty machine	6 Solvent powder
AB sulfonic acid	14-15	5	15	12-15	5	5
Dist. fatty acids		4			4	3
Nonionic	2-3	6	2		4	3
TPP and/or STPP Metasilicate (5 H <sub>2</sub> O) or	30	30	20	15	20	20
spray-dried disilicate	3	3		5-6	5-6	5
Soda ash	15-20	15-20	20% max.	to make 100%	to make 100%	to make 100%
Sodium bicarbonate			30			
Sodium sulfate	to make 100%	to make 100%	to make 100%			
CMC	2	2	***	1.5	1.5	1.5
Optical brightener	0.2	0.2	0.2	0.2	0.2	0.2
Perborate	10	10		10	10	
Enzymes calc.						
300,000 DU	0.5	0.5				
Perfume	0.2	0.2	0.2	0.2	0.2	0.2
NaOH sol. 30%	2-3	2-3	2-3	2-3	2-3	2-3
Solvent deo-kerosene						4

absorption types of builders are used, whereas in a system based on mechanical dispersion and powerful disintegration, a much wider variety of builders in normal powder form may be used. The mixing disintegration unit is of a relatively small size in relation to its output. Loading, discharging, and residence times are short. While most spraymixing systems are designed to obtain powders approaching the structure of spray-dried beads with relatively low density, the system described here aims at a powder with relatively high density, but with a structure to make it adhere strongly to the surface of beads coming from a spray tower.

The mixing unit may be used either for the incorporation of high percentages of nonionics or for the incorporation of sulfonic acid and/or distilled fatty acids, with or without nonionics. In this case, the ABS and fatty acids are neutralized immediately in contact with the builders. Fatty acids are transformed in a single step operation into soap without leaving any unreacted fatty acids. Certain processing details are to be observed to guarantee the complete neutralization of ABS and the complete saponification of distilled fatty acids.

This "dry neutralization and saponification" is accomplished by using a small percentage of an alkaline aqueous phase (representing only a small fraction of the stoichiometric amount of NaOH) as a kind of accelerator or "trigger" for the reaction. Actually the aqueous phase, generally only 2 to 4% on the total mix, is added at the same time, but not premixed with the surfactant components. To give a general indication of the efficiency of the mixing system: only about 3 to 4 minutes are required for even distribution of nonreacting nonionic components onto the builder mix, and about 4 to 6 minutes for "reacting" components, i.e., AB sulfonic acid and/or distilled fatty acids. Automatic charge and discharge takes approximately an additional minute or two, depending on the size of the mixing system. The process can be run completely automatically with weighing and dosing similar to continuous slurry preparation.

Depending on the bulk density of the builders used, the amount of the surfactants added, the bulk density of this powder from the first phase of the process is in the range of 0.6 to 0.9. The product from this process may be used as such for detergent powders, e.g., washing powders for commercial laundries, metal cleaning detergents, etc. The powder is less dusty than otherwise produced "heavy powders." The process may be carried out in such a manner that powders, either with a granular or a finer structure, are produced. The process is flexible enough to produce powders in a wide range of particle size.

The powder from the dry neutralization system may be combined with powder from the spray dryer (Fig. 6). Surprisingly the bead structure of the powder from the spray dryer is not changed when adding the "heavy" powder from the dry neutralization system - even in proportion of 1:1. Also, the density of such mixtures does not change proportionally to the ratio of heavy powder to spray-dried powder. An indicative formula to calculate the bulk density of mixtures is given as:

Density of mixture = 
$$\frac{100}{\frac{a}{d'} + \frac{b}{d''}}$$

a = Percentage of heavy powder (by weight) b = Percentage of beads (by weight) d'= Density of heavy powder d"= Density of beads

The unchanged structure and the relative small change in bulk density of mixtures is explained by the fact that the hollow bead structure is not affected by the "heavy" powder, which actually covers and is fixed onto the large surface of the beads.

Typical particle size data of "heavy powder," spraydried powder, and the 1:1 mixture of the two are shown in Table II.

Attaching a dry neutralization unit to an existing spray dryer will at least double production with only minimal additional space requirements.

Under present energy conditions the obvious savings in this respect must also be considered. An increase in bulk density of powders, of course, still with a bead structure, means increase in packing machine output, saving in transport, and saving in packing material.

This combination of powder with spray-dried powder permits great freedom in powder formulation and bulk density requirement (Table III).

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