A Spray-Dried α-Olefin Sulfonate from Concept to Marketplace[1]

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ABSTRACT

This paper examines the spray drying process, particularly the spray drying of α-olefin sulfonates. Topics covered center on the spray drying process for α-olefin sulfonates, important parameters and considerations in that spray drying process, and an examination of the product from the spray tower. Product development from laboratory to pilot plant is described with attendant translation to full-scale facilities. At least 4 different cocurrent spray-drier units are illustrated and key parameters are discussed.

INTRODUCTION

Spray drying is, by definition, the transformation of feed from a fluid state into a dried form by atomizing the feed into a hot drying medium. It is a multistage continuous suspended particle processing operation. The feed can consist of either a solution, suspension, slurry, paste or wet powder. The resulting products fall into the categories of macrobeads, powders, agglomerates or granules, the form of which depends on (a) the chemical and physical properties of the feed, (b) drier design and (c) drier operation. Spray drying is a process which, in many industries, meets dried product specifications most desirable for subsequent processing or direct consumer usage (1).

We have chosen to segment the process into 5 principal stages: stage 1—atomization of feed via either high-pressure nozzle or rotating disc; stage 2—atomized feed/air contact or, in a cocurrent system where the hot air and feed enter at the top of the drying chamber, the intimate contact provided by the stream of swirling turbulent air entering through the vanes of the hot air plenum (rotating either clockwise or counterclockwise) with the product embryos issuing from the atomizer; stage 3—moisture evaporating from atomized feed or contact between the ultra-fine droplets of feed and the hot air drying medium resulting in extremely rapid evaporation of moisture from the droplets; stage 4—dried product recovery or separation of the product from the gases exiting the drying chamber, generally by deployment of 2 or more cyclones in parallel for centrifugation of particulate matter from the gas stream. Stages 1-4 are depicted in Figure 1; stage 5—recovery of residual product from exhaust gases or removal of particulate matter prior to discharge of exhaust gases into the atmosphere using either a wet scrubber or baghouse (bag filters) (Fig. 2).

In the development of a highly active spray-dried AOS, a number of preliminary considerations must be met before commercialization or market introduction can be launched. The major considerations include practicality of the process, key property requirements, maximal AOS actives attainable, predominance of favorable key process indicators, and relative ultimate competitive status, principally on a cost/performance basis.

The obvious key to total development rests with the first consideration, the practicality of the process. The ancillary

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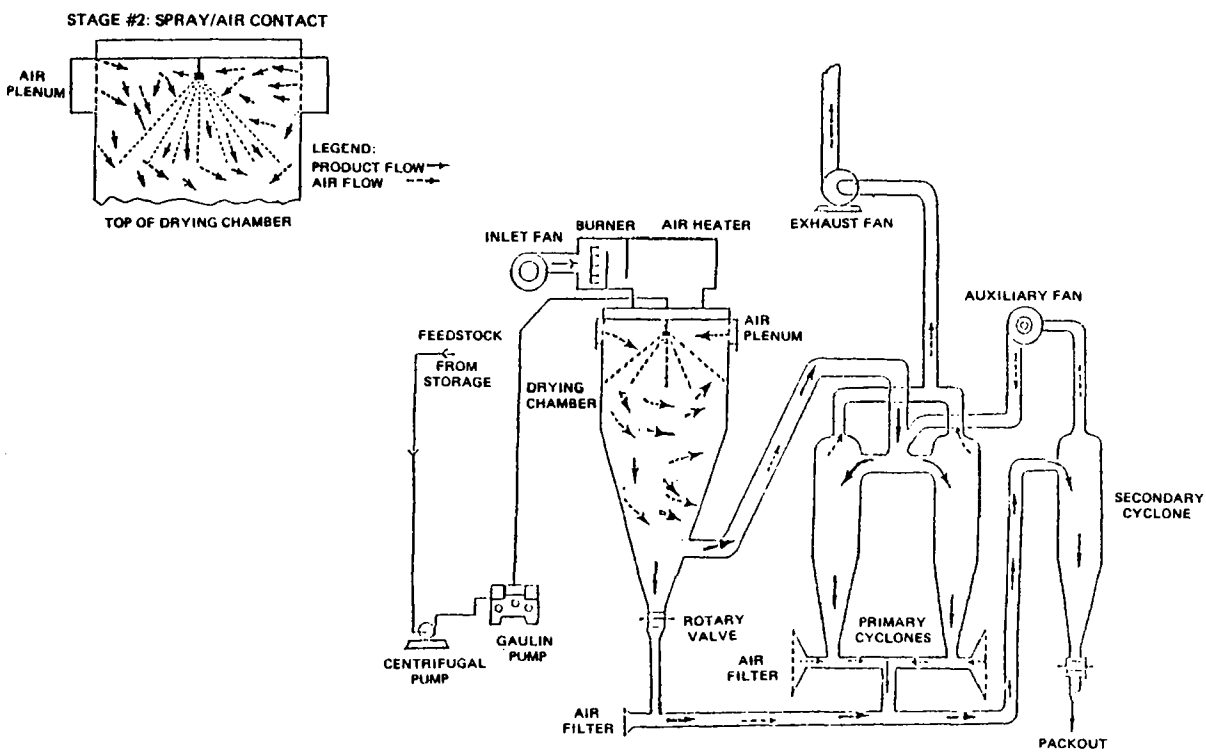

FIG. 1. Spray drying process, stages 1-4.

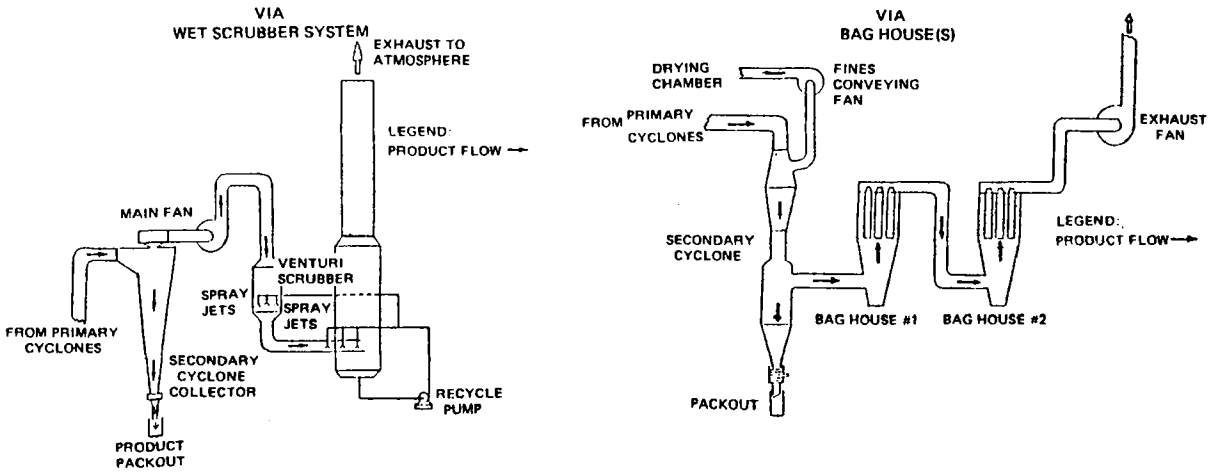

FIG. 2. Recovery of residual product from exhaust gases.

factors are yield and/or nonrecoverable losses. A once-through product yield of at least 90% favors commercialization. Excessive or cumulative adhesion of product inside process equipment, particularly within the drying chamber, presents a potential fire and explosion hazard and results in product loss and costly periodic downtime for equipment cleanout. Excessive rework generated due to malfunction of equipment and/or utility services erodes manufacturing costs and product profits. Finally, excessive stack losses, above EPA limits, would render the process technically inoperable and would mandate deployment of auxiliary continuous units such as wet scrubbers and baghouses to render the exhaust gases suitable for discharge into the atmosphere.

The second major consideration is directed toward the target property requirements for the product. Generally, these properties are typified by quality commercial surfactants offered in dry form, and include free-flowing capability, uniform discrete particle size, reasonable moisture tolerance, heat-stability, and finally, density commensurate with realistic product package costs and applications.

The next important factor is the chemical composition of the product, for which the objective is to produce material containing a maximum of functional, active organic AOS with a bare minimum of diluents, fillers and crisping agents. Processing capability of both the feedstock slurry and spray-dried product is the key criterion.

Another facet that serves a major part in assessing the potential for commercialization of the product concerns a strong, favorable response from nearly all of the key processing variables. These key variables include ease of product density control, minimal generation of rework, minimal adhesion of product-to-process equipment, no charring or discoloration of product, minimal stack loss, and minimal product dusting.

EXPERIMENTAL DEVELOPMENT

The initial stage of the laboratory study involves development of a workable composition for the feedstock slurry. A C_{14}/C_{16} aqueous blend of sodium α-olefin sulfonate having a composition of roughly 2:1 C_{14}/C_{16} was selected as the surfactant base. The active organic (AOS) composition was greater than 90% on a bone dry solids (BDS) basis. The drum drying performance or response of virgin AOS feed was determined in conjunction with those at progressively lower active organic levels effected by dilution with inert fillers such as sodium sulfate and/or sodium chloride.

We found that objectionable thermoplasticity and hygroscopicity prevailed at high-active organic levels and that dramatic reductions occurred with the addition of minor amounts of fillers. Thus, optimal drum drying performance was achieved at an anionic active ingredient level of over 80%. The ideal solids content of the slurry was optimized so that the relatively low-viscosity slurry offered little resistance to feedstock make-up and subsequent atomization via either high pressure nozzle or spinning disc.

To confirm the qualitative drying features of the experimental NaAOS, a series of experiments was conducted in a cocurrent, lab-scale Stork-Bowen spray drier at Stepan Chemical's facility in Northbrook, IL, using air inlet/outlet temperatures of about 380/240 F, feedstock temperature of 110-120 F, and a drying rate of 1,100-1,300 g/hr. The air flow through the drier at the outlet was about 250 cfm. Atomization was effected by either nozzle (2) or spray disc. In the first instance, an air atomizing nozzle, equipped to deliver 100-150 g feed/min, was used with 100-110 psig air as the driving force. Alternatively, the Stork-Bowen centrifugal-disc atomizer was used in the operating range of 40,000-50,000 rpm, with gravity feed to the unit. The viscosity of the feed was kept at a minimum at 100 F. A majority of the runs showed significant adhesion of product to the chamber walls and dryer cone; however, it was readily removed by periodic opening of strategic air-sweep ports located on the outer wall of the drying chamber. Product recoveries generally ranged over 75% and particle size was characteristically extremely small (ca. $< 50\ \mu$).

Pilot Plant Scale-Up

The next phase of our development work was conducted using a Stork-Bowen drier (3), shown in Figure 3.

The spray-drier is a cocurrent unit equipped with a variable speed Bowen spray-disc for atomization of the liquid product feed. The drier and accessory units are constructed of Type 304 stainless steel. The drier is 7 feet in diameter and can be operated at 2 different heights.

The product feed to the drier is prepared in a 45-gal, stainless steel jacketed tank mounted on a portable scale. The tank has a bowl-shaped bottom and is equipped with an air-driven Lightnin mixer. The feed is transferred to the spray-disc via centrifugal pump.

The drier can also be operated as a pseudo-countercurrent unit by implementing a nozzle, located near the bottom third of the drier, to spray the feed upward into the drying chamber.

After considerable experimentation with the Stork-

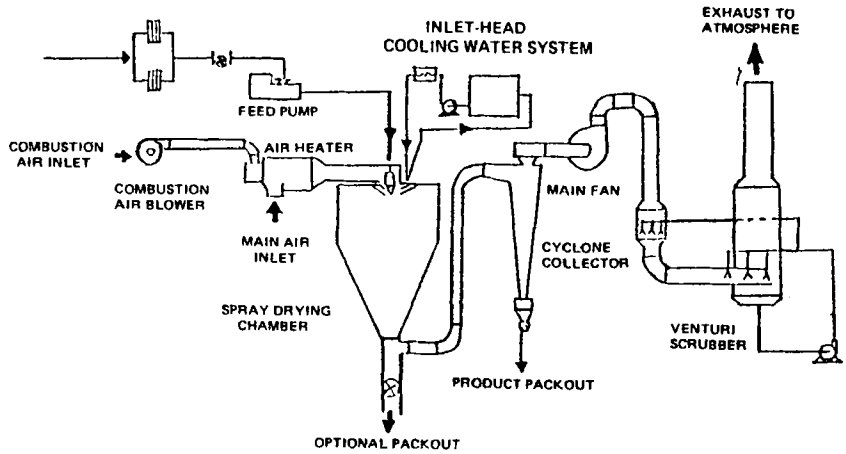

FIG. 3. Stork-Bowen cocurrent spray drier (pilot plant).

Bowen unit, operating conditions were established to give a once-through product recovery in the range of 93-96% of theory. This "good news" was achieved by centrifugal-disc atomization using inlet/outlet air temperatures greater than 325/200 F and an air flow at the rated capacity for the drier. The product feed was kept heated to ensure a minimal viscosity at the optimal solids strength. Drying was at a rate of about 95-120 lb/hr. As expected with spray-disc atomization, the product density was low, roughly 0.19 g/mL (i.e., $\approx$ 17.6 lb/ft^3). The moisture of the spray-dried AOS ranged from 0.4 to 0.8%.

It was noted that higher inlet temperatures increased thermoplasticity of the product with attendant substantial adhesion to the chamber walls. A decrease in inlet air temperature promotes agglomeration because of higher residual moisture levels in the dried product.

In subsequent pilot plant work, a DeLaval (4) drier was used with a baghouse being deployed for treatment of the exhaust gases. The DeLaval drier and accessory units are depicted in Figure 4.

The pilot plant drier is a cocurrent DeLaval design, Model DE-72-12B, that operates at an air flow of 1,100-1,600 SCFM. It is 6 ft in diameter by 12 ft on the straight side equipped with a 60° cone or bustle. Construction is Type 304 stainless steel. A high-pressure nozzle is used for atomization of the feed.

The drier is equipped with an air ring to shroud the hot inlet air with ambient air and an air curtain to keep the uppermost (4-6 ft) chamber wall clean. The air ring serves to keep the ceiling of the chamber clean. In addition, a

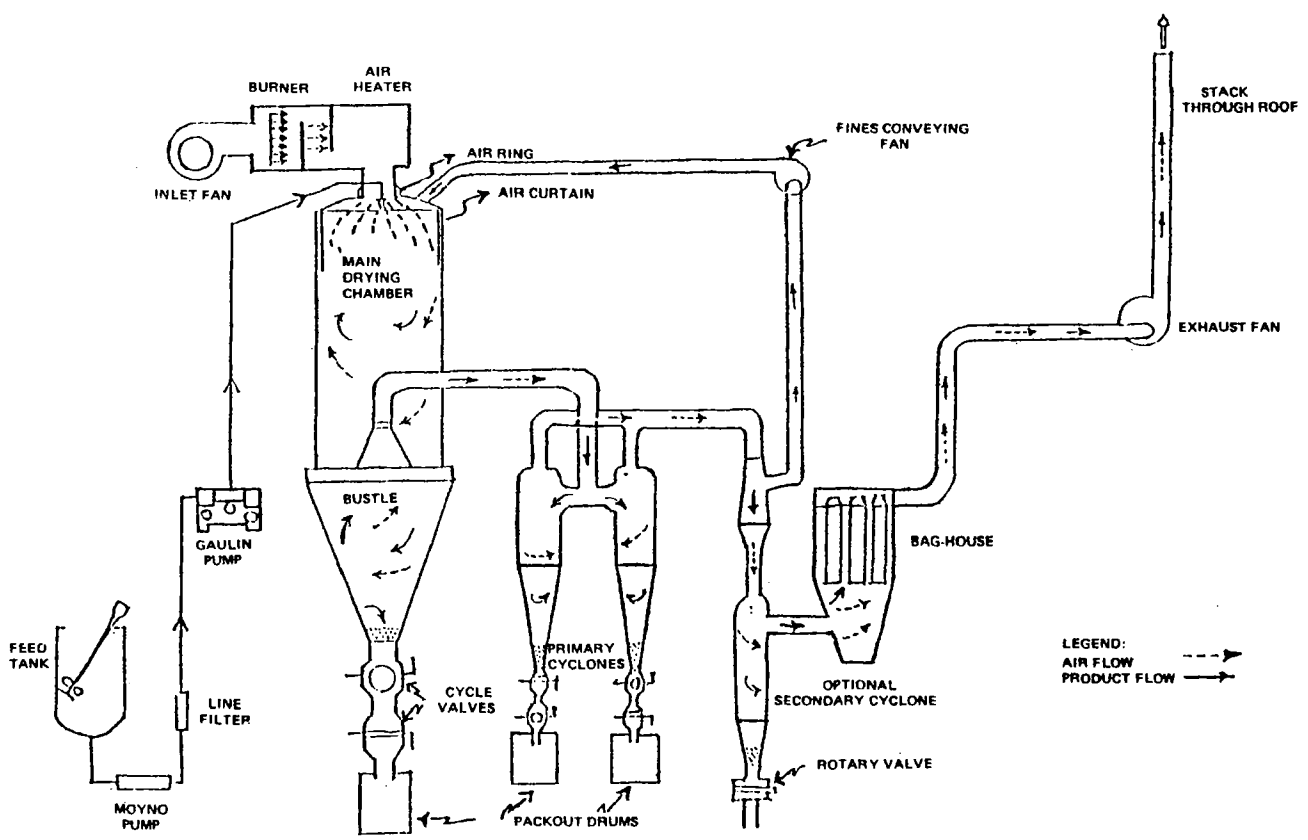

FIG. 4. Cocurrent De Laval spray drier (pilot plant unit, Niro Atomizer, Inc.).

clean-in-place (CIP) system is featured to keep drier downtime at a minimum for cleanout and for switching from one product to another.

The pilot plant data generated to this point in our development work were advantageously applied to this drier to give minimal equipment downtime, negligible rework material, and exceptionally good once-through product yield to warrant upstaging to full-scale.

Inlet and outlet temperatures were explored in a range from 350-490 F and 200-340 F, respectively. Atomization was accomplished via a high-pressure nozzle augmented by a Gaulin pump. Product densities were increased and moisture levels generally remained under 1%. The spray-dried particle size varied from 20 to 200 μ with a predominance of particles in the 70-90 μ range. Recycle of fines to the drying chamber resulted in their agglomeration. Microscopic examination of product discharge from the drying chamber revealed miniature satellites adhering to the product matrix (Fig. 5). However, our data revealed that a significant portion of the agglomerated fines were regenerated in transit to the cyclones. This was attributed to removal of the satellites from the product matrix through abrasion in the ductwork. A more instantaneous contact of the fines with the atomized product droplet probably forms a strong bond that is more resistant to failure during transit in the ductwork.

Sustained runs of 10 hr each showed minimal adhesion of product to the walls of the drying chamber and no charring at all. Although the unique air curtain in the drier contributed to this favorable performance, the evidence obtained indicated that we had nearly optimized the chemical composition of the product feedstock to make it resistant to adhesion and charring.

The effect of atomizing the feed by using a rotating disc was studied. In general, the spray-dried particle size was increased several-fold over that obtained by high-pressure nozzle but at a significant drop in product density. The decrease in density can be explained, for the most part, because the disc gave hollow beads and the high-pressure nozzle gave solid spheres.

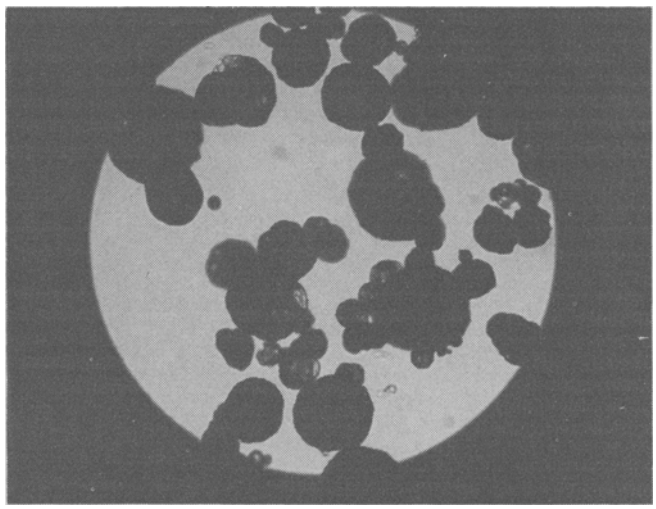

FIG. 5. Spray-dried agglomerates.

The moisture tolerance of the spray-dried product is roughly the same, irrespective of atomization of the product feed by rotating disc or high-pressure spray nozzle. No adverse differences in product storage stability have been noted; we would surmise that the solid beads could tolerate even higher moisture levels because, at equilibrium, the moisture that has migrated to the outer surface of the solid bead is still far below that of the hollow bead.

Full-Scale Translation

Two product properties—density and particle size—surfaced as primary considerations in assessing acceptability of the product. Consequently, in upstaging the process to full-scale operation, our efforts were directed toward optimizing the foregoing product properties on a sustained basis.

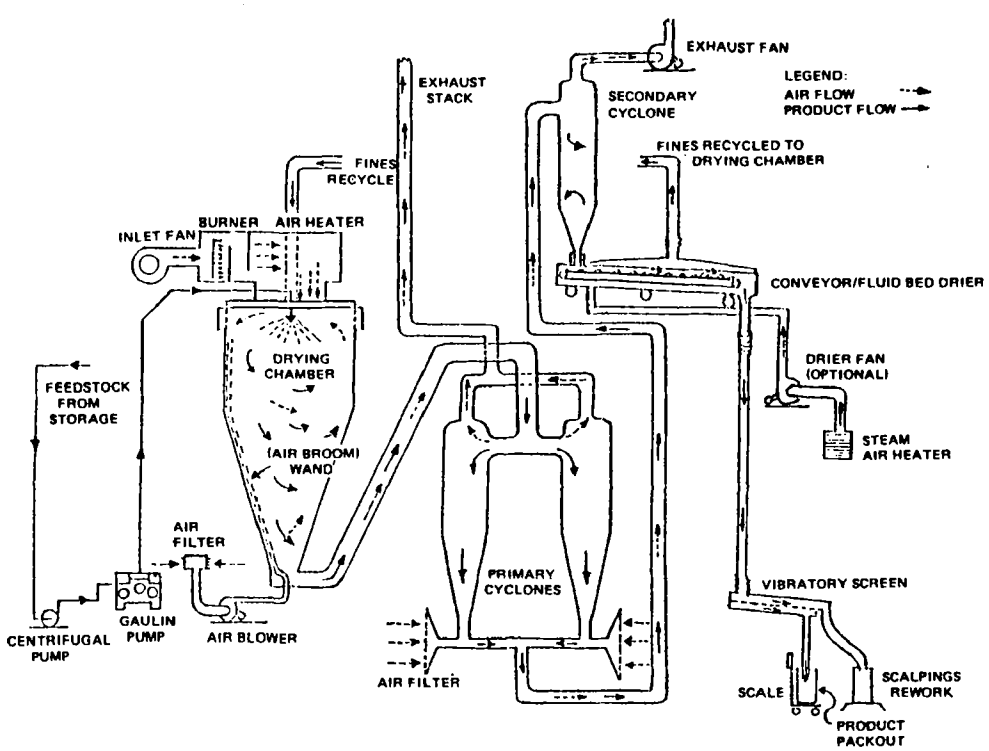

FIG. 6. Foremost spray drier.

Because the chemical composition of the feedstock slurry had been optimized, we resorted to physical means of increasing product particle size and, attendantly, product density, i.e., via agglomeration and/or change in atomization mode.

Of the many drier units explored, our ultimate selection for full-scale demonstration was a commercial Foremost (5) unit equipped with an agglomerator and, optionally, either a single or 4-fluid bed drying train augmented with a delumper and rotary screen for product packout. The unit is shown in Figure 6 with a single fluid bed drier. Construction throughout is of Type 304 stainless steel.

The drier is a cocurrent model with a diameter of 20 ft, straight wall of 16 ft, and roughly a 45° cone that is 26 ft high. The overall tower height is about 42 ft. Average air flow in the drying chamber is about 30,000 SCFM. The drier is equipped with a rotating air broom or wand to keep the chamber walls clean.

Atomization of the feed was accomplished through a high-pressure spray nozzle. The most attractive product densities and particle sizes were obtained via the Foremost drier coupled with the single-stage fluid-bed drier. Operation temperatures used in pilot plant drying in the DeLaval drier were applied to the Foremost unit. To minimize viscosity of the feedstock, it was again heated to 140 F prior to drying. Free moisture levels were equilibrated at about 1.0%. Particle sizes were increased from earlier pilot plant work to a level of roughly 100-400 μ, and the product exhibited minimal dusting at packout. Again, as had been done in pilot plant work, fines were recycled to the drying chamber to allow agglomeration with the freshly atomized feed. The problem of attrition of these agglomerates in the duct work persisted, however, and consideration was given to the use of the 4-stage fluid bed drier as an alternate means of agglomeration and reduction of fines.

Attempts were made to agglomerate the product issuing from the spray drier in a Fenske agglomerator and subsequently drying in the 4-stage fluid bed drier. Product moisture levels of 1-12% entering the agglomerator were explored. The Fenske agglomerator relied heavily on jets of air, steam, and water to create turbulence and intimate wetting of the entering product. The agglomerated product discharged onto the first stage of the fluid bed drying train and was dried eventually to a residual moisture of 1.0-2.0% by the time it discharged from the 4th stage via a rotary screen to packout.

Particle size of the agglomerates was inconsistent, with a low yield in the desired range. Density remained roughly equal to the spray-dried product and dusting was not significantly reduced. Thus, the 4-stage drying train run was aborted, and the balance of feedstock dried via the single-stage fluid bed.

Although agglomeration attempts met only moderate success, the drying conducted with the Foremost spray drier yielded a number of valuable facts. It was determined that the feedstock would withstand sustained, full-scale drying runs with no evidence of charring and only minimal adhesion to chamber walls. Also, the finished product exhibited storage stability at high moisture levels. Armed with these new facts, we prepared to move to our final full-scale development work.

The final full-scale development was conducted using a modified Grey-Jensen (6-8) spray drier. A brief summary of the spray drier and accessories follows and the equipment is depicted in Figure 7.

The equipment includes a Sparkler Model 33x538 scalping filter used to improve the color of the feedstock. A high-pressure Gaulin feed pump was used to introduce feed via spray nozzle(s) to the modified Grey-Jensen spray drier. The drier itself is 34 ft in diameter x 10 ft straight side

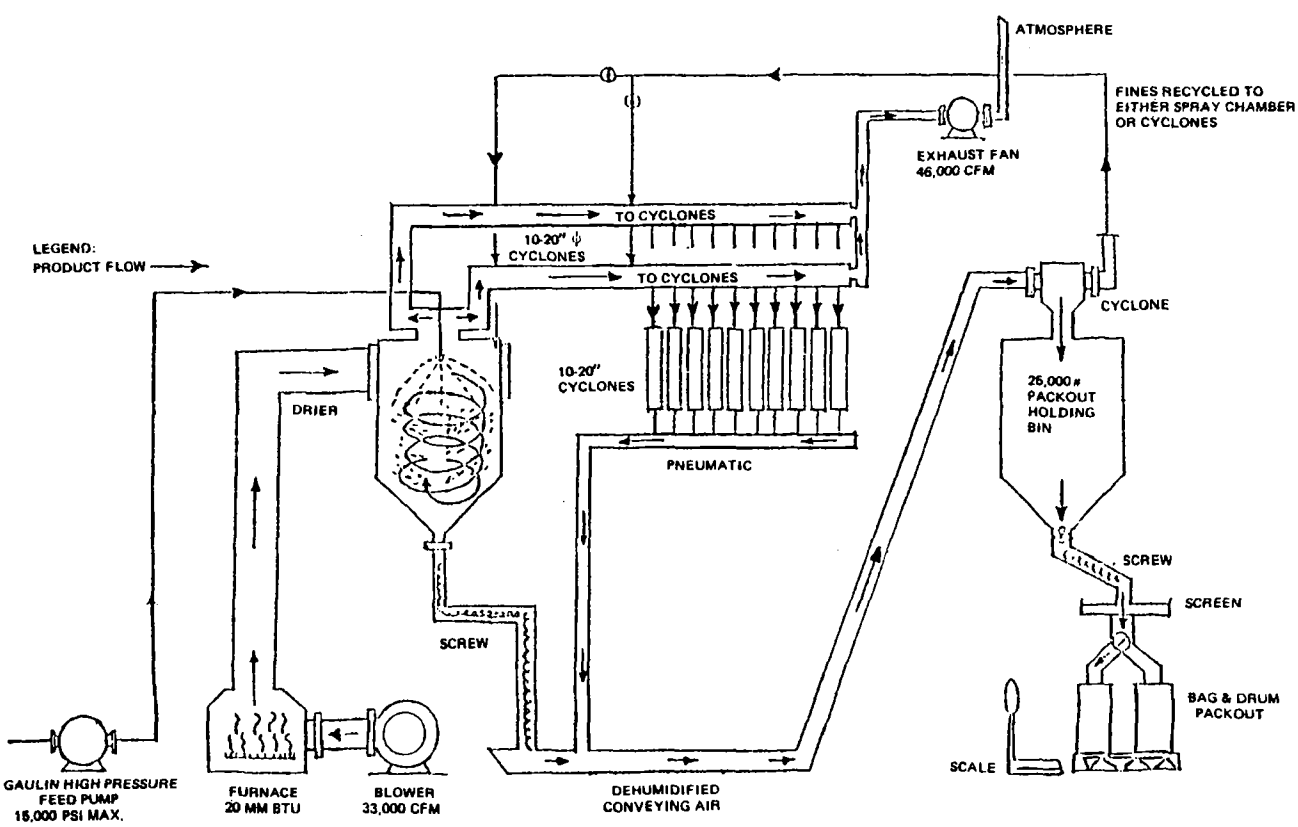

FIG. 7. Modified Grey-Jensen cocurrent spray drier (with bank of 20 cyclones).

equipped with a 28-ft cone. The inlet fan is rated at 33,000 SCFM with an outlet fan of 46,000 SCFM. The spray nozzle is located at the center of the drier, roughly 1 ft above the cone. Accessory units include a direct gas-fired furnace rated at 20 MM BTU and 2 banks of ten 20-in. diameter cyclones in parallel for fines collection and recycle to the drier. A number of high-pressure spray nozzles were used.

Because the composition of the feedstock had been optimized earlier, the primary considerations of this work were (a) sustained production of product at a yield of 90% or greater, (b) sustained production of beads of target density, (c) minimal dusting at the packout station and (d) as large a particle size as possible without sacrificing product density. Moisture was also a key consideration to the extent that it had to be kept at a level that would not lead to agglomeration of the dried product in the drum. The product had been found to tolerate high moisture levels. Therefore, moisture level did not become a limiting factor in our work.

Our full-scale work conducted with the Grey-Jensen drier was generally successful, surpassing all of the goals outlined here. The product yield exceeded 90% in all sustained drying runs, the target product density was achieved consistently, particle size was comparable to that achieved in earlier work, and product dusting was minimal.

Because of its relative mildness to the skin and uniformity in particle size, the spray-dried α-olefin sulfonate (AOS) lends itself to dry compounding of numerous concentrates, especially for personal care applications. The spray-dried beads blend instantly with other functional ingredients and inert fillers to give a homogeneous product receptive to uniform dyeing/perfuming and resistant to classification in transit or during storage.

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