All in one

Large volume kneaders combine unit operations and multiple phase processing in a single unit

Conventionally processes, which are characterized by multiple phase changes, are realized in several distinct steps, and the related space requirement, investment and operating costs are high. This article shows, how specialized large volume kneader processing equipment combine unit operations and multi-phase processing in a single unit – a solution that reduces capital and operating expenditure, improves the competitive edge and maximizes revenues.

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Fig. 1: DISCOTHERM B 12500 CONTI Kneader/Crystallizer/Dryer

Crystallization by evaporation followed by drying and devolatilization, concentration by evaporation with subsequent dissolution polymerization, and solid-solid reactions are common unit operations in the process industries, which are characterized by multiple phase changes. They often create fouling of heat exchange surfaces, and they are heat transfer controlled, but also diffusion limited unit operations. Evolution of large volume of vapors is possible. Highly viscous phases develop that demand considerable power input for being processed. Conventionally, such processes are realized in several distinct steps. The following case studies show how specialized large volume kneaders combine unit operations and multiple-phase processing in a single unit.

Continuous crystallization

The process of crystallization by flash evaporation is found in almost every branch of process industries. A typical example from the food industries is the crystallization of sugar alcohols (sugar substitutes). Representative applications from the polymer industries are the crystallization of speciality elastomers and polymers. In such applications the feed stream is a low viscosity pumpable solution. It consists of at least two components. Under the prevailing operating conditions one of the components is volatile. Crystallization takes place as soon as the solution has become super-saturated in the dissolved solid material. The solid crystalline material maintains this stable phase, if the operating temperature would not exceed its melting point.

At the operating pressure flash evaporation takes place when the feed temperature is higher than the boiling temperature of the contained volatile component. Crystallization is initiated when supersaturation of the solution is achieved, i.e. the excess amount of the volatile component is flash-evaporated. Consequently, it must be ensured that the accumulated heat in the feed stream would suffice to evaporate the volatile component till super-saturation is reached. Provided that the prerequisites are fulfilled and taking also into account the evaporative cooling effect, the liquid feedstock would turn into a particulate crystalline solid. Nevertheless, the solid is still wet with the volatile component.

According to the target-residual volatile content the further separation of the volatile component from the solid is usually realized by means of drying, but in some cases devolatilization as the final drying stage is inevitable. It is a time demanding operation controlled by the diffusion of the volatile component from the solid matrix to its surface and finally to the surrounding environment. Within operational limits, devolatilization can be accelerated. One measure is to increase the operating temperature. Care must be taken not to exceed the melting temperature of the solid material. If the solid material is a polymer, the glass transition temperature must not be exceeded. The second measure is to increase the concentration gradient of the volatile component between the solid and the surrounding environment. This is accomplished by means of a sweep gas. The application of sweep gas demands additional investment and operating costs. That disadvantage is accentuated when the devolatilization is realized under vacuum. Even so residence time must be provided.

The scale of operation of specialized single or twin shaft kneader processing equipment is 16 m^3 respectively 10 m^3. Operating at a fill level typically in the range of 50% to 70% the residence time would lie in the range of 3 to 5 hours. Figure 1 shows a single shaft DISCOTHERM B CONTI kneader/crystallizer/dryer of 12.5 m^3 installed volume. It is applied for the continuous crystallization by flash evaporation and drying of a sugar alcohol.

Large cross-sectional area

Flash evaporation generates large volume of vapors, which increases proportional to the vapor temperature and indirectly proportional to the molecular weight of the volatile component and the operating pressure. A large volume of vapors would lead to high vapor velocity,

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which in turn could result to drag over of liquid droplets and fine crystalline particles. This is prevented when a large cross-sectional area is available for the disengagement of the vapors. Large cross-sectional area is yet another design feature of the specialized kneader processing equipment. It matches with the requirements of the crystallization by flash evaporation even under vacuum. The table summarizes design features and functions of specialized kneader processing equipment.

Crystallization by flash evaporation experiences also limitations due to insufficient accumulated heat in the feed stream. In such a case the status of super-saturation is not reached and the formation of a particulate crystalline solid is not achieved. Instead a suspension is formed. Crust formation and excessive fouling of the heat exchange surface is inevitable. Conventional processing equipment would recycle solid material in order to induce super-saturation, thus preventing crust formation, fouling and the risk of blocking the crystallizer/dryer. Specialized kneader/crystallizer/dryer features self-cleaning effect and intensive mixing, thus allowing the uninterrupted efficient processing of all product phases.

Example: speciality rubber

Figure 2 shows a representative process flow diagram for the continuous crystallization by flash evaporation followed by drying and devolatilization of SEBS speciality rubber. The process takes place under vacuum at moderate temperature. The feed stream contains 80 to 85% by weight solvent and 15 to 20% by weight SEBS. The final solvent content must be ≤0.1% by weight. The process is realized in the single shaft Discotherm B Conti kneader/crystallizer/dryer in one-through single stage operation without solids recycling. The feed system comprises the feed pump followed by the flash valve. The uniqueness of the flash valve is that it keeps the pressure in the feed line above the vapor pressure of the volatile solvent at its boiling temperature. Hence the presence of two-phase flow between feed pump and feed nozzle is avoided. Another design feature of the flash valve is that it is mounted in the feed nozzle of the kneader/crystallizer/dryer. As the feed stream enters the process chamber of the processor it flashes immediately. The disengaged solvent vapors flow through a vapor dome before reaching the condenser and the vacuum pump. The final crystalline particulate solid SEBS product is discharged through the lateral discharge head of the processor. Its design allows the control of the product fill level in the process chamber. The product is discharged to the product silo by means of a lock discharge vessel.

Summary of pertinent features and functions of specialized kneader processing equipment

Design features		Functions for combined operations
■ Intermeshing static and rotating mixing/kneading elements; adjustable geometry ■ High interface renewal frequency ■ Large free sections ■ Heavy mechanical design	Mechanical	■ Intensive mixing/kneading in all phases ■ Efficient mass transfer ■ Gas or vapor disengagement; narrow residence time distribution in continuous operation ■ Processing of highly viscous products
■ Large self cleaning heat exchange surface	Thermal	■ Tight control of product temperature profile ■ Significant heat transfer capacity for controlling highly endothermic or exothermic reactions ■ Effective removal of excess dissipated heat
■ Large total volumes ■ Closed design ■ Configuration for batch or continuous operation	Chemical	■ Long residence times for slow diffusion controlled processes ■ Vacuum or pressure operation ■ Integration within an existing processing line

Solid-solid reactions

Solid-solid reactions are defined as "dry processes" because they are realized in the absence of a liquid carrier phase, usually water or a solvent. Representative examples are cyanuric acid and/or copper phthalocyanine, food grade pyrophosphates, sodium salicylate, para-hydroxybenzoic acid etc. The advantage of "dry processes" over "wet processes" is the elimination of the recovery, the rectification and effluent treatment of the carrier phase. The process simplification, seen from the necessary process stages and related equipment installed, ensures lower investment expenditure and minimization of operating costs.

Dry processes are characterized by extensive crust formation and the poor flow characteristics of reactants and products. The formation of crust inhibits the heat transfer, could be a hurdle to axial conveyance, could cause abrasion and could even block the reactor. Insufficient supply of heat leads to low conversion and yield, possibly to the formation of by-products (impurities). Poor axial conveyance complicates the control of product hold-up and disturbs the residence time distribution.

Successful realization of a dry process demands for efficient mixing of the solid components, interface renewal in the bulk of the reaction mass, large self-clean heat exchange surfaces and heavy-duty mechanical design. All those process and equipment constrains are fulfilled by the specialized kneader/reactor.

Figure 3 shows the process flow diagram for the manufacture of cyanuric acid. This chemical, which is used for the production of water treatment chemicals, is produced by the thermolytic conversion of urea. The prevailing chemical reaction is

$$3\ H_2N\text{-}CO\text{-}NH_2 \rightarrow 3\ NH_3\uparrow + C_3H_3N_3O_3$$

The reaction is realized in the Discotherm B Conti kneader/reactor, which ensures an efficient and homogeneous mixing of the reactants. The temperature of the reaction mass, which is a critical parameter influencing the quality of the raw cyanuric acid, is monitored along the axis of the reactor.

The thermolytic reaction takes place at atmospheric pressure and elevated temperature. The reactor is heated with thermal oil. The NH_3 gas stream, which is generated during the course of the reaction, and the vapors of the sublimed urea pass through the integrated vapors/dust filter, before leaving the reactor. Dust is developed in the dryer due to the slight carry over of cyanuric acid fine particles. The filtrate falls back to the process room of the reactor thus preventing losses.

The thermolytic conversion of urea to cyanuric acid takes place in the presence of cyanuric acid. The two components are mixed at the specific ratio. Cyanuric acid is recycled from discharge section of the reactor back to the feed section of the same. This is effected by means of an integrated screw conveyor. This conveyor is cooled by means of cooling water. Cooling of cyanuric acid, although energetically not meaningful, prevents excessive sublimation of urea at the mixing point of the two streams.

The sublimation pipe is of pertinent importance. Its design prevents desublimation of urea avoiding plugging and interruptions of the process. Urea is desublimed in a separate desublimer and recycled to the reactor. The overall losses of urea would not exceed 2% by weight of the stochiometric urea demand.

The benefits of conducting the thermolytic reaction in the specialized kneader/reactor are:

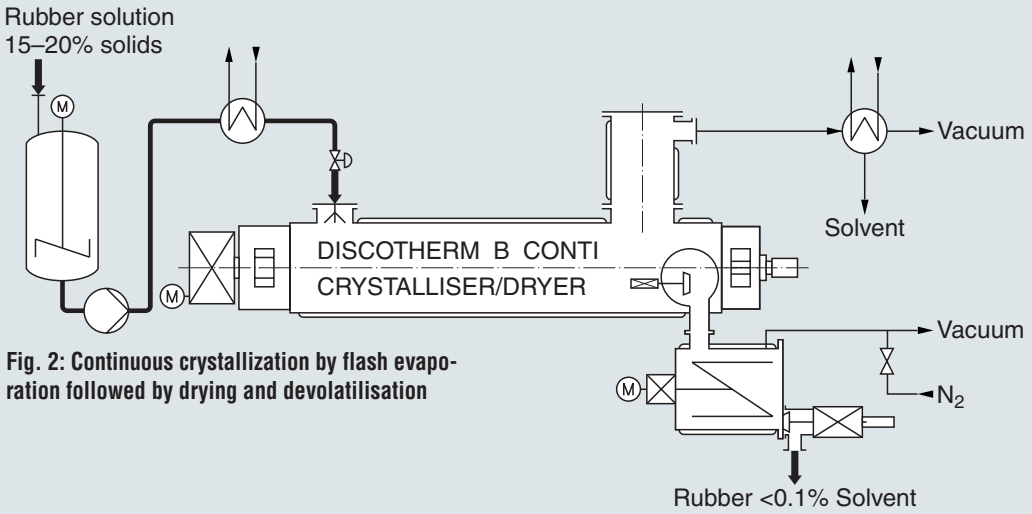

Fig. 2: Continuous crystallization by flash evaporation followed by drying and devolatilisation

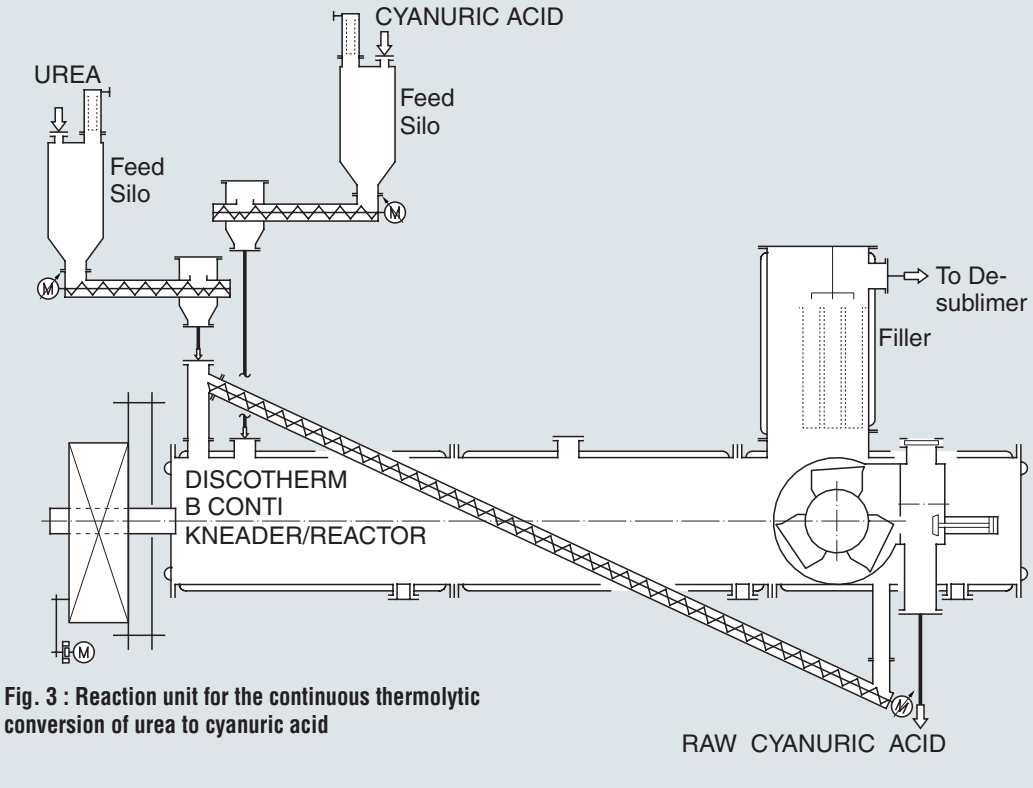

Fig. 3 : Reaction unit for the continuous thermolytic conversion of urea to cyanuric acid

■ continuous and automated process with limited materials handling;
■ mixing of reactants and the reaction itself take place in one equipment in a single one-through process;
■ the reaction mass temperature is closely controlled thus minimizing the risk of product discoloration and the amount of by-products;
■ the final cyanuric acid product is a free flowing granular material simplifying its handling where required;
■ the high conversion of urea to cyanuric acid;
■ the minimization of urea losses;
■ the elimination of solvent as carrier phase its recovery and rectification.

A major benefit, which should not be omitted but needs separate consideration and comment, is the purity of the raw cyanuric acid produced. The purity has been determined to be in the range of 85 to 95% and the color is white.

Closing remarks: The application of specialized kneader processing equipment masters combining several unit operations and multiple phases processing in a single unit. When process optimization would hardly further improve its overall economy, combined one-step processing offers new possibilities and finds increasing acceptance in the chemical and related process industries.

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