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Something Special in Separation Technology

By Andreas Diener and Helmut Schildknecht

The synthesis of most elastomers is carried out either by solution or emulsion polymerization. After the polymerization step, the polymer is separated from the solvent or emulsifying agents. This separation requires several process steps including coagulation, stripping, various mechanical separation stages, and finally drying. Beyond that, the existing technologies are energy consuming, waste solvent must be incinerated, and the installation of main and ancillary equipment occupies large spaces. The disadvantages of the solution and emulsion polymerization processes can be solved with the

direct devolatilization process of elastomers contained in polymer solutions or polymer emulsion. The process was developed in a partnership between LIST AG and DOW Olefinverbund GmbH, a manufacturer of elastomers.

Figure 1 on the next page shows the block diagrams of the existing conventional technologies and compares them with the new process technologies. The simplification is obvious. The new process solutions are more efficient, consume less energy, require less space, and minimize waste. All these advantages are beneficial to the installation and operating costs. *continued on page 52*

Direct Devolatilization

Most elastomers are produced using the solution polymerization processes with solvents of aliphatic or aromatic hydrocarbons. According to the existing processes, the separation of the elastomers from the solvent requires a water-based coagulation of the elastomers, a separation of the solvent by steam stripping, and finally the drying of the wet elastomers. The new process solution does not require water coagulation and steam stripping separation. Instead, the solvent is directly separated from the elastomers with simultaneous solvent recovery. The final elastomers have the same properties and fulfill the required product quality specifications.

The direct devolatilization process is based on two steps. First is the continuous main evaporation of the solvent and unreacted monomer. Second is the continuous final devolatilization of the elastomer.

The process can separate the solvent and non-converted

monomer to the expected residual content, which is usually less than 100 ppm. The main evaporation takes place under vacuum while the elastomer solution is kept at the pasty/viscous phase. The energy input happens mainly mechanically. The polymer solution temperature is controlled by evaporative cooling.

The devolatilization step is very sensitive regarding overheating or self-ignition of the elastomers. During the process development, it was determined that, if during the devolatilization process the elastomers can be transformed to a granular or preferably to a crumbly form, then the elastomers can be treated carefully and protected against overheating.

Direct Dewatering and Drying

Some elastomers are still manufactured by the emulsion polymerization process. The emulsifying agents are mostly water-based products. After polymerization, the elastomers are coagulated and separated from the agents in a series of mechanical separation

steps before being dried. This technology has reached its limits when processing new products. It was observed that the mechanical separation requires high work, and even then, it does not function properly. These limitations are overcome with the new processing technology in which the drying is also less energy consuming. The final elastomers have the same properties and fulfill the required product quality specifications.

The direct dewatering and drying process is based on two main steps. The first step is the continuous mechanical dewatering of the elastomer slurry. The second step is the continuous drying of the mechanically dewatered elastomers.

The process can separate the water to the expected residual content, which is usually less than 100 ppm. For the continuous dewatering, a new specialized low shear dewatering screw is applied. The screw dewateres the elastomer slurry with the least possible work. The dewatered elastomer is then fed to the continuous dryer. The initial moisture content is in the

range of 8 percent to 20 percent by weight. The continuous drying operates under vacuum.

The drying step operates under conditions that prevent overheating and self-ignition of the elastomers. During the process development, it was determined that, if during the drying process the elastomers can be transformed to a crumbly form, then the elastomers are treated carefully and are protected against overheating.

Controlled Finishing

Both finishing processes, devolatilization and drying, treat the elastomers under such conditions that the risk of overheating and self-ignition are prevented. The prevention of overheating is realized without the application of sweep media enhancing the evaporative cooling. The quality resulting thereof is excellent. The direct devolatilization and the direct drying processes are based on elastomers' finishing in particulate granular consistency.

The development and preservation of the elastomers' crumbs was

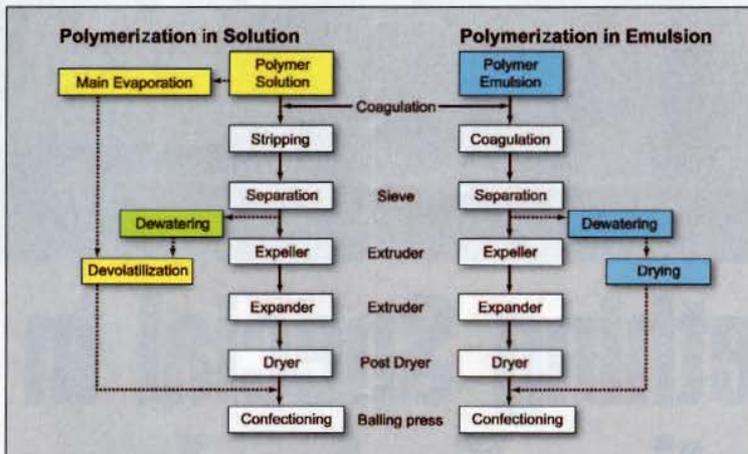


Figure 1: Differences between old and new technologies

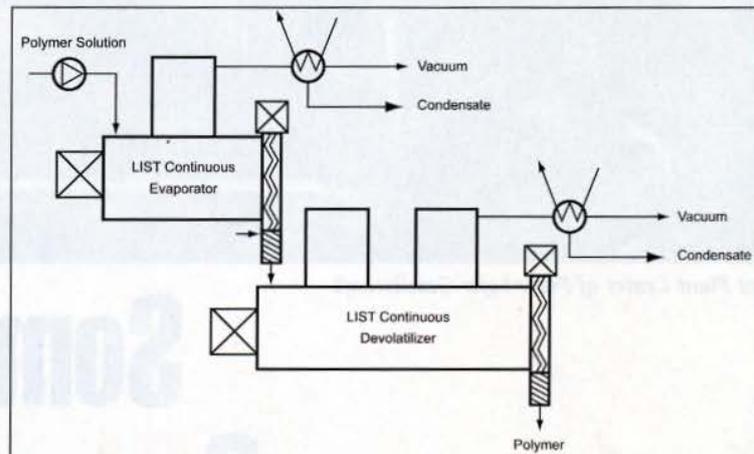


Figure 2: Process flow of direct devolatilization process

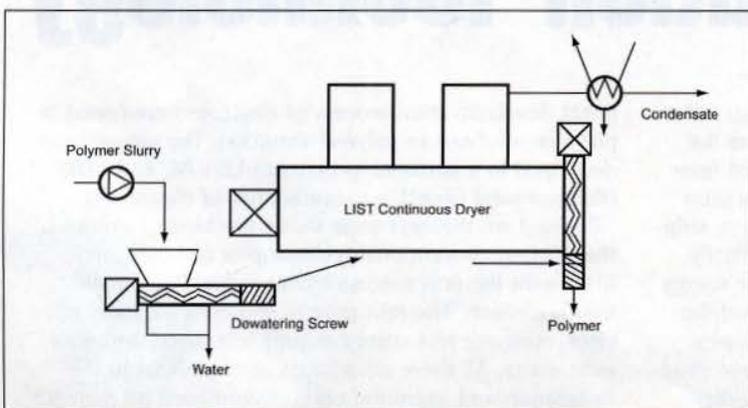


Figure 3: Process flow of direct dewatering and drying

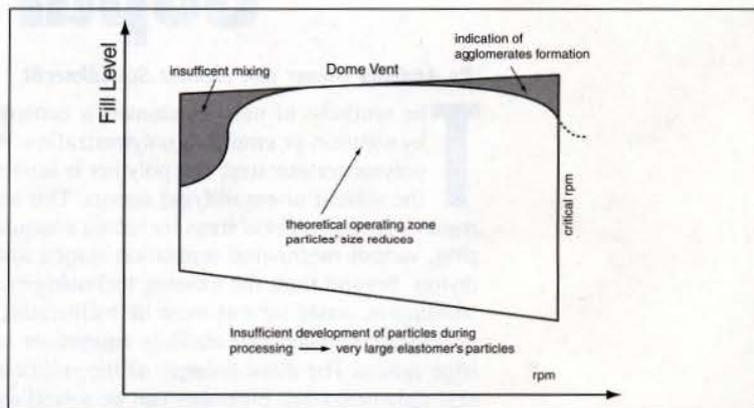


Figure 4: Operating zone of the finishing process with crumb elastomers



Figure 5: Elastomer crumbs after completion of finishing step

achieved through the design of a specialized LIST Polymer-Finisher (finisher = devolatilizer and/or dryer). The specific characteristic of that processing unit is that when operated at a specific range of product fill level and speed of revolution, it transforms the elastomers' mass into particulate crumbs and preserves them until the completion of the process.

The operating range of the specialized LIST Polymer-Finisher is defined as operating zone. The operating zone is shown in Figure 4. It depends on the elastomer type and the processing capacity.

Either finishing processes are diffusion controlled operations. The driving force for this diffusion controlled process is the difference of volatile concentration between the gas phase and the elastomers' phase. This diffusion step is a slow, time-consuming process, whereby the size of the granules is the limiting factor.

Figure 5 shows an elastomer at the end of its finishing process. The elastomer is free of volatiles and its color is clear white, as a first visual quality control. The perfect self-cleaning of the metallic surfaces, as seen in Figure 5, is required for high quality and low volatile content product.

Process Efficiency

Process efficiency is judged according to the criteria listed below:

1. Thermal energy and cooling water consumption
2. Space requirement for the installation
3. Environmental aspects concerning amount of water effluent
4. Investment and operating costs for the simplification of the

comparison, the accumulation of energy and energy losses are not considered.

The direct devolatilization process consumes as much energy as required for the solvent evaporation. No mechanical

and thermal energy are spent for the coagulation, mechanical water separation, and stripping. Less energy is consumed for the condensation of the solvent if this condensation is at all necessary. Additional savings result from the absence of the coagulation agents and the unnecessary expensive separation of water and solvent.

Compared with the existing technology, direct devolatilization furnishes a process simplification, water-free separation, elimination of the solvent/water separation, prevention of water effluent and its subsequent treatment, no air contamination.

The initial investment for the direct devolatilization process is around the same as existing technology. However, the investment costs are recovered much quicker due to the lower operating costs (energies, space requirements for the installation, wastewater and off-gas treatment, personnel) of the new technology versus existing.

Figure 6 shows the comparison of the energy consumption between the technologies. Referenced is the operation of a plant in Western Europe.

In the last years, new special elastomers have been developed, which cannot be processed with the existing technology. Here again, the direct dewatering and drying process create the possibility to efficiently process such elastomers. The overall energy consumption between the existing and the new process is similar, but the direct dewatering and drying process requires

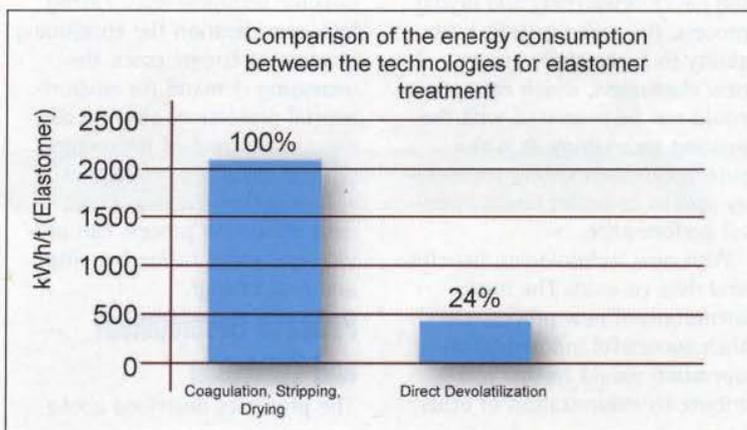


Figure 6: Energy comparison

less space, and the effluent gas is considerably less.

Benefits, Shortcomings, Risks

With the direct devolatilization process, the main benefit is the absence of water, which in turn translates into the following:

1. Lower energy consumption for evaporation, cooling, and water/solvent separation
2. Less installation space requirement

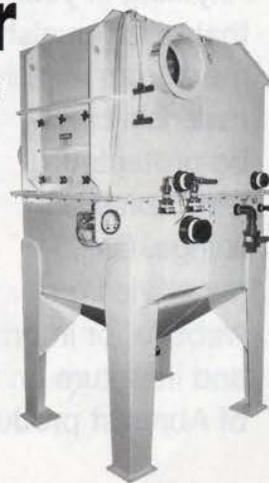
One possible shortcoming of the direct devolatilization process is

that impurities, which were removed during the steam stripping stage using the existing technology, may remain in the elastomers' mass. This shortcoming could be resolved by using clean monomers and adequate catalyst systems. Such catalytic systems would select catalysts and initiators that could evaporate during the devolatilization of the solvent or would not influence the elastomers' quality when contained in the final product. With

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the direct dewatering and drying process, the major benefit is its ability to successfully process new elastomers, which otherwise could not be processed with the existing technology. It is also superior to the existing technology due to its better environmental performance.

With new technologies, benefits and risks co-exist. The implementation of new processes and their successful industrial-scale operation would further contribute to minimization of other

possible technical risks. Taking into consideration the continuing increase of energy costs, the increasing demand for environmental protection, and the continuous demand of improving product qualities, it becomes apparent that the new direct devolatilization process can provide enormous future benefits and cost savings.

Process Development and Validation

The processes described above

were jointly developed with DOW Olefinverbund GmbH in Schkopau (D). In a first stage, parametric investigations were performed on lab and pilot scale units. After the successful completion of that stage, the processes were implemented on a large-scale pilot plant. That plant is installed at the pilot plant center of Fraunhofer Gesellschaft in Schkopau. Fraunhofer Gesellschaft is an independent German research and development institute.

The Pilot Plant Center for Polymer Synthesis and Processing in Schkopau is equipped with modern specialized LIST Polymer-Finisher technology. Beyond the implementation of the processes described above, that installation can perform new polymerization, polycondensation, and reactive compounding processes. A comparative advantage of Fraunhofer Gesellschaft is that the institute provides the necessary analytic laboratories

and the units for application testing. This facility also allows the immediate analysis of sample products.

The installed LIST processing technology is designed for continuous and batch operation. The testing capacities range from 3 to 50 kg/h final product.

The objectives of this pilot plant are the following:

1. The transfer and validation of chemical development works done in R&D laboratories to a continuous large-scale pilot process
2. Collection of process experiences
3. Production of samples for application tests
4. Validation of time-extensive processes and product stability
5. Comparison of different processing technologies
6. Training of personnel ●

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