Quality Control. A raw material manufacturer has integrated a standardized online "Closed Loop" concept within their polymer plants. Using this the offline laboratories within the facilities were directly linked into the production process allowing continuous online monitoring. This permanent control helps the manufacturer to optimize their processes and react immediately where necessary. At the same time the quality of the raw material can be determined.

Online "Closed Loop" concept for the monitoring of production lines (figure: OCS)

Online Control of Polymer Manufacturing

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The "Closed Loop" concept is based on the idea of directly linking classic offline laboratories online to the production process in order to control both quality and the process itself **(Title figure)**. In doing so small samples of raw materials are regularly taken so that a representative overview of the production as a whole can be obtained. In the past as much as 10 hours were sometimes needed in order to characterize the level of quality and the process itself. Today, however, the enormous costs of out-of-specification production can no longer be tolerated. With a return on investment (ROI) of far less than a year this concept has now become standard.

Fundamentally the online quality control can be divided into two areas: testing of the pellets and testing of extruded cast or blown film. Testing of the pellets delivers information about the LAB color values, MFI (rheometer), size distribution (pellet size and shape distribution systems) as well as dust/abrasion and contamination (pellet scan systems).

Online measurements from the extruded film on the other hand give information about the gel count, additive levels and physical properties (IR spectroscopy), gloss (gloss meter) and/or opacity (haze meter).

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Online Pellet Analysis

During online pellet analysis pellets are continuously drawn from the production flow by a sampler and transported via a fully automated system to the measurement cell where they are tested and sorted. Pellets that are within specification are then returned to the process.

For color measurement pellets are transported to a cuvette where the LAB values, yellowness index and other diverse color properties are determined.

In the online pellet scan system transparent or opaque pellets are passed under a color matrix camera on a vibrating plate or turntable, checked for color contamination or foreign bodies and where necessary separated from the pellet stream.

The pellet size and shape distribution analyzer monitors pellets in free fall as they pass by a line scan camera and delivers information on size distribution, roundness, angel hair, dust/abrasion as well as doubles and triples.

All the systems mentioned here can be connected one after the other and controlled via integrated software. The systems are networked via an interface, for example Modbus, to the plant's data acquisition system (DAS). When deviations occur an alarm is automatically triggered.

"At Line" Rheology

An intermediate level between measurement taken from pellets and extruded film is given by the OP5 rheometer which uses patented technology from OCS Optical Control Systems GmbH **(Fig. 1)**. This allows highly accurate offline laboratory measurements according to ASTM 1283 and ISO 1133 to be conducted directly "at line" in parallel to the production delivering important parameters for controlling the reactor. Samples are continuously taken from the pellet stream, heated and carefully pressed under very high pressure. This process leads to relatively little change in the molecular structure and at 0.5 % similar reproducible accuracies to an offline laboratory are achieved. At the same time measurements without screw shear considerably reduce maintenance costs.

Fig. 2. Online "Closed Loop" concept for blown film lines

Online Measurement of Extruded Film

The measurement techniques listed above do not, however, give any information regarding gel count and the quality of the pellet. These measurements are performed on a narrow film tape extruded in parallel to the production which is continuously assessed for gel counts. In doing so it is very important to draw small samples as often as possible in order to provide continuous monitoring of the process. For example, a so called "gel shower" can last 5 to 10 minutes and then simply disappear. This means a rate of at least 50 samples per hour needs to be achieved.

After extrusion using cast or blown film technology the film is passed beneath a line scan camera and wound onto a coreless pneumatic spigot (**Fig. 2**). The winding unit has a single sided support and comprises several servo drives which guarantee an absolutely stable and crease-free film feed. The film feed as well as a clean environment – up to cleanroom standards – is necessary since, for example, pellets for high voltage cables are inspected with a resolution of 5 µm. For commodity raw materials such as PE standard resolutions are 50 µm or 25 µm.

Fig. 1. "At Line" OP5 rheometer (figs. 1 to 3: OCS)

The process is optimized for raw material control and works almost autonomously. Other measurements such as haze according to ASTM 1003, additive concentration and composition (infrared spectroscopy), gloss or thickness can be integrated into the winding unit and software without difficulties. Total remote control and maintenance of the system are enabled via a web browser. The system is also linked internally to the OPC server and externally to the customer DAS. →

Fig. 3. Online "Closed Loop" measurement cell for two LDPE lines

Quality Assurance Concept for a Polymer Plant

In order to minimize problems from pellet transport such as dust or abrasion it is recommended that a cleanroom measurement cell be positioned directly next to the extruders. Analysis software that is integrated via a Modbus interface into the plant DAS means that production can operate all of the equipment from the central control room. Raw material samples can be automatically distributed from here to the various measurement stations **(Fig. 3)**.

Pellet analysis systems sort out contaminated pellets, camera systems assess gel counts, IR spectroscopes determine additive concentration and composition. When problems are found production is immediately notified and the contaminated material diverted to a separate silo. At the same time set-up processes are substantially shortened and maintenance intervals predicted and optimized.

Online Polymer Analysis in Practice

The rise in production rates of polyolefin plants over the years **(Fig. 4)** has increased the necessity for online analysis. With production rates that have in the interim reached around 60 t/h the original methods used have proved to be unsuitable **(Table 1)** because the analysis and reaction times are simply too long.

The first online polymer analysis instruments were introduced at Borealis in the early 80s and the number of online instruments has increased over the years to around 100 in Europe. As a result the amount of quality related data has grown exponentially.

In order to optimize these online installations Borealis has assembled a team of experts at corporate level in order to support individual technologies within the company as a whole.

An online classification module (OLC) with an integrated online SPC module and outlier recognition software was developed in order to automatically calculate weighted averages and provide the results to the LIMS for COA (Certificate of Analysis) purposes.

Agreements were concluded with the following suppliers in order to guarantee continuity and high levels of availability for the installed equipment:

- Göffert GmbH: Rheometer for MFR

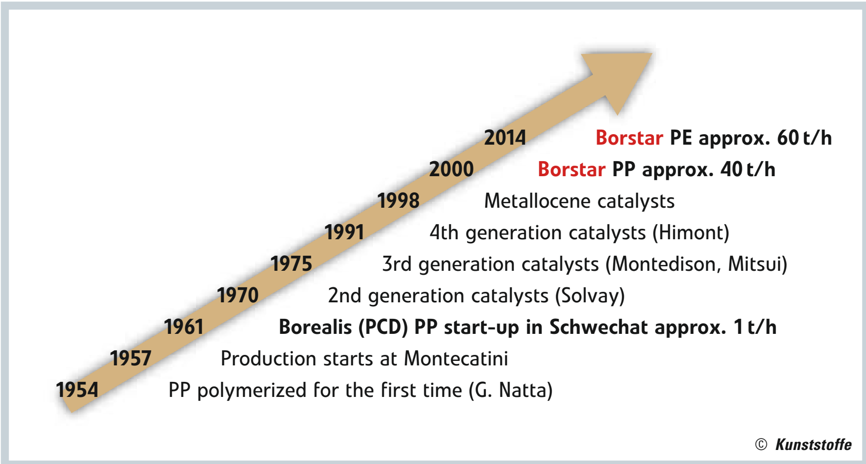

Fig. 4. Growth in production rates in the polyolefin sector (figs. 4 to 6: Borealis)

- OCS GmbH: PA66 for pellet geometry and contamination, FTIR for density and additives, FSA 100A for gels and contamination
- Progression Inc.: Low Resolution NMR for density, XS, DS, ethylene content, flexural modulus

OLPA instruments have contributed to an improvement in process monitoring as well as product consistency via timely and continuous adaptation of the process. These continuous data analyses have also led to new insights into production and product quality.

The success of these online installations is also emphasized by the fact that

Fig. 5. Smoothness analyzer for cable grades

Borealis has implemented full OLPA resources for the product mix being manufactured at all of its new polyolefin plants.

Speeding Up

A typical OCS installation for the analysis and classification of products from two LDPE lines is shown in **Figure 3**. Each line comprises a PA66 pellet analyzer, an ME20 extruder, a CR9 Chill Roll unit and an Aplairs FTIR. Both lines are fed from a common PTS (Pellet Transport System). The arrangement also offers the possibility of taking a sample for quality control or external uses from either line. Data is sent to the Borealis IP21 process historian from an ACS (analysis control system) PC.

With increasing production rates the quantities of material for special product groups (e.g. wire & cables) that needs to be analyzed has also increased. For this reason larger extruders with higher throughputs are needed, which in turn leads to a greater need for high speed cameras, cutting units, marking units and processing software. OCS and Borealis are working closely together to find optimal solutions for these products.

	Time
Density	2 h
Xylene solubility	6 h
Decalin solubility	6 h
Heptane insolubility	1 h
Mechanical properties	24 h – 1 week
Gels and contamination	1.5 h
Melt flow	15 min
Pellet geometry	30 min
Additive content	15 min – 2 h

Tab. 1. Approximate historical analysis times

Fig. 6. Die scraper for an extrusion die

An example of this partnership can be seen in the retro-fitting of a die scraper **(Fig. 6)** to a recently manufactured surface smoothness analyzer (SSA) **(Fig. 5)** used to measure surface defects and contamination of a black tape in order to remove the build-up of material due to die drool.

By using the PA66 to identify contamination on pellets it is possible to segregate out of specification products by diverting these to an alternative silo. The geometric data on the pellets is used to optimize blade changes in the plant. All parameters are made available to staff via the process history so that they can follow trends and where necessary make changes to the settings.

OLPA instrumentation also has several advantages and practical benefits for Borealis customers. The accumulated OLC data offers comprehensive information and delivers more accurate records of the quantities of materials than conventional QC analysis. OLC also offers the ability to more quickly characterize and release products, which in the final analysis leads to better customer service. ■

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