

From Laboratory Scale to Pilot Production

A Continuous Process Automation Concept

Complex chemical syntheses require the application of expert laboratory skills and technologies. "FlexyConcept" – the process automation system from Swiss manufacturer SYSTAG – fulfills these requirements through the integration of both conventional laboratory and modern automation technologies. With the example of a suspension-polymerisation pilot plant, the advantages of FlexyALR are documented as follows.

FlexyALR is a FlexyConcept-based automated laboratory reactor unit performing all the basic operations for the organic-synthetic industry and combining the advantages of laboratory automation with the required flexibility. The main applications consist of the exact and reproducible execution of repetitive functions such as temperature regulation, dosing, pH, pressure control, and many more options.

FlexyALR is successfully bridging the gap between process development in the laboratory using only a few hundred millilitres and pilot production on a kilogram scale. It also shows functional versatility and adapts to almost any process conditions with only minor reconfigurations. The high degree of functionality and the

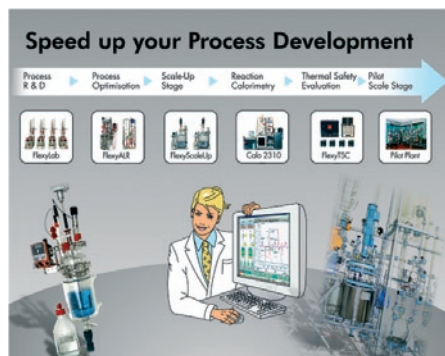
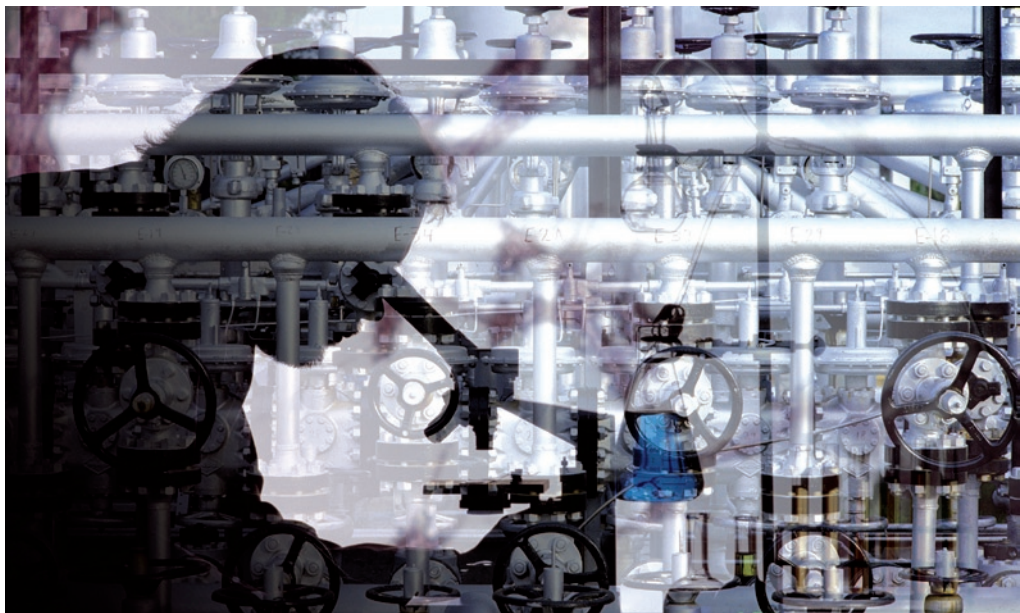


Fig. 1: FlexyConcept was developed as a modular, continuous automation concept for the chemical-pharmaceutical industry. In addition to the laboratory and pilot plant reactor units from this manufacturer (FlexyLab, FlexyALR, FlexyScale-up) and its process safety evaluation devices (Colo2310, FlexyTSC-Serie), it can also be combined with equipment and systems of numerous third party suppliers.



added customised options allow this continuous automation concept to meet the different demands from early process development to scale-up, and small batch production to kilogram manufacturing.

Fully Automated Pilot Plant

Based on FlexyALR from SYSTAG, and in cooperation with the Swiss chemical process equipment manufacturer Büchi, a customised and fully automated pilot plant for suspension-polymerisation has been designed. The technological requirements for such specialised pilot plants are especially high, while at the same time, each one retains unique characteristics. Special emphasis on reliability and performance is already placed early in the development phase. The process automation system FlexyALR could impressively prove its strength therein. Moreover, close cooperation with our partner significantly contributed to the success of this project.

Process and Instrumentation

During suspension-polymerisation in the manufacture of polystyrol- or PVC suspensions, for example, the monomer – usually in the water phase – is dispersed by vigorous stirring to form small droplets. Added protective colloids inhibit the coalescence of the droplets, and monomer-soluble reaction initiators start the polymerisation.

The polymerisation plant consists of two identical 20L pressure reactors and a central

pressure vessel as the starting reactor. From the starting reactor, up to seven different chemicals can be weighed and added to one or both reaction pressure chambers, in mixed form or individually. The central starting reactor is the key component of this pilot plant. It stands on a 300 kg precision industrial scale with a 1g resolution capacity. Seven automatic valves regulate the feed of the different chemicals to the reaction vessels and ensure a dosing accuracy of ± 1 g for a volume of 20 kg. Through the use of a pressure-resistant septum with syringe, a more accurate dosing is also possible, according to the requirements. A pump finally transfers the mixture in one of the two reaction vessels. The specially designed mechanical stirrers reproduce the conditions in the production plant. For easy access, all three reaction vessels have each be equipped with a lift to facilitate cleaning and servicing. The entire pilot plant is crafted from stainless steel and designed for pressures of 20 bar. The reaction vessels are equipped with sensors for the measurement of temperature, pressure, number of stirrer revolutions, pH, and torque.

Each parameter is recorded and controlled by the process control software. There are three additional liquid feeds to the reactors: two through burettes and another one through a dosing pump, controlled by an additional scale.

The pilot plant was installed in an ex-zone which requires the installation of applicable, certified building blocks and components. The electrical interface contains a suitable transducer for

galvanic isolation as well as a transmitter and the SPS controls etc. The entire plant is controlled by FlexyALR, which, in addition to the extensive control functions, also allows for recipe process controls. All events are recorded real-time. Thanks to an intuitive operation platform, FlexyALR offers a manual mode that enables the even untrained process automation operator to use the equipment easily and quickly. All parameters can be manually entered or revised respectively, based on predefined user access privileges. The user friendly functions allow for a successful equipment operation after only a few minutes of training, and thus ensuring immediate productivity.

Improved Reproducibility to Ensure Quality

Besides the above mentioned manual mode, FlexyALR also allows for fully automated controls with specific recipes. All available process functions can be freely combined using the "Drag&Drop" function in the recipe window of the software. In this way, process controls and parameters are pre-set and graphically displayed.

The automated process control checks for safety limits as well as for individual process parameters. A graphical trend is displayed with all relevant data, allowing for a constant monitoring of the process status. In order to permit variations in the process during recipe controlled process run, a "manual" mode is available at all times. Changes in set values, through formulation or manually, are recorded in a permanent log file.



Fig. 2: The automated turn-key pilot plant for suspension-polymerisation was automated based on FlexyConcept.

CFR 21 Part II

The process control software increases reproducibility and accuracy; it also allows for the standardisation of all test documentation and reasonable process automation. The MS Word-based test protocol is generated automatically

and can be adapted to the customer's needs (template). The systems can be modified to conform to the guidelines in CFR 21 Part II. The recorded test data (fix intellution) is also accepted by the FDA. Appropriate software user levels allow for supervised access privileges with different tasks.

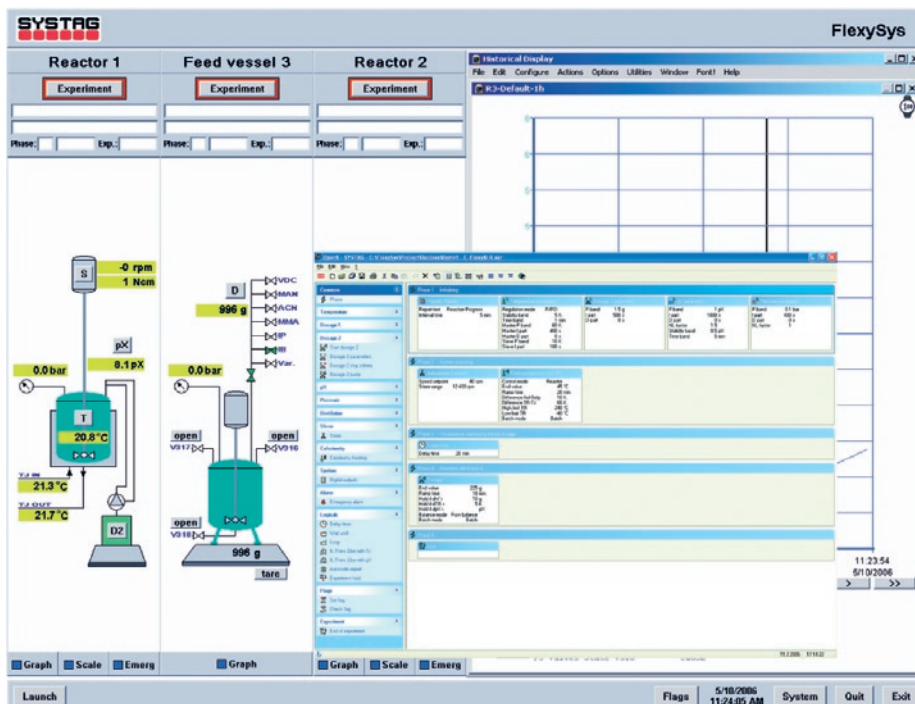


Fig. 3: The recipe control ensures a fully automated operation; in parallel, modification in the recipe process run can be made using the manual mode.

Concept and Flexibility

Solutions for automation from SYSTAG are based on FlexyConcept. As a continuous concept for process automation SYSTAG products can be integrated much in the same way as other external equipment, leading to a protection of your investment. In addition, the user is in the position to control the processes from laboratory-scale to small batch production and thus less training is required.

Our customised pilot plants as described above are generally assembled and tested before delivery. The mentioned pilot plant was skid-mounted and shipped to the customer, who was able to put it into operation within a few days after delivery.

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