

Sequip Application Report # 003

Different strategies of a **polymerisation process** control by real time sizing sensor system working under insitu and in line conditions.

Key words:

- insitu Particle measurement in polymer high pressure application,
- In line sizing of precipitation under polymerisation conditions under explosion proof conditions
- PAT Live Particle Sizer
- Process integrated environmental control of resources

Benefits of an insitu size sensor as a control system of a disperse phases distribution

- Improvement of process stability prediction during R&D projects
- Faster development of new process kinetics
- Scale up scale down information
- Optimisation of process equipment and energy
- Continuous control of size and number depending on reaction

Application:

1. in any viscosity or concentration where material can flow in a pipeline or reactor under flow conditions
2. for solids in liquid systems
3. for liquids in liquid systems
4. for „clear“ particles in „clear“ solutions with a different refractive index
5. at elevated temperature from minus 120°C up to 300°C
6. at elevated pressure vacuum up to 300 bar
7. within process flow up to 30 m/ sec
8. detecting ranges 500 nm up to 3000 µm

Summery of Objectives:

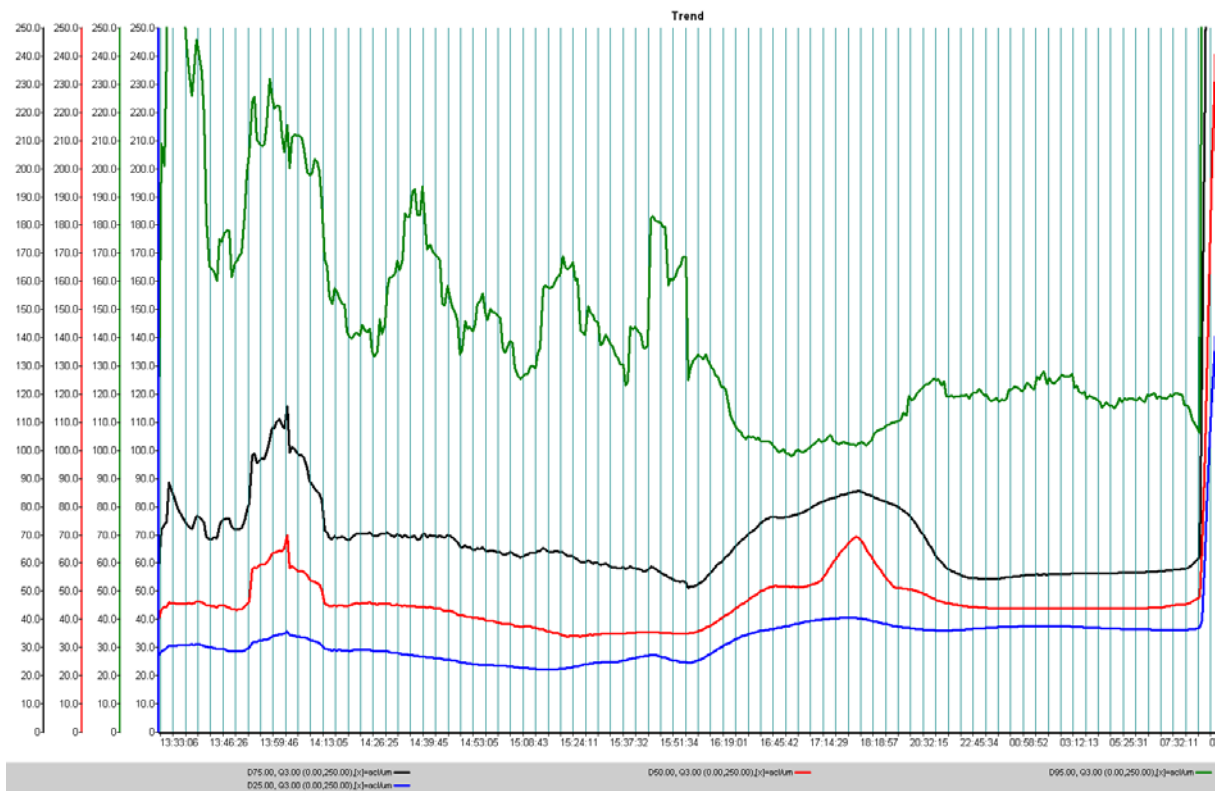
- The application department, founded in 1989, measured more than 6000 products, a lot of them were confidential, and acquire a long term knowledge of polymerization application.
- This experience generated a lot of efforts for the partners of the combined technologies.
- We have met several polymerizations, where the knowledge of the particle, crystal and droplet sizes was essential for understanding of the process and its control as well.

Installation of the in line measuring sensor devices

- The PAT insitu sensor for polymer application has been directly installed inside the process reactor.
- The sensor follows the kinetic process of polymerisation during a 220 bar and 120°C process operation
- The system consists of a high speed electronic and an insitu sensor, which measures the number of particles/droplets during the polymerization.
- During the measurement several process parameters have been changed and could be observed by the sensor system. The system was capable of following the different reactions of droplets and/ or particles as well.
- The installation of the sensor is in a reactor. Therefore the sensor dip was directly in contact with the steam of regenerated particles/droplets.

Results out of experiences in plant and operation control

Fig.01 shows the distribution of the measured results during the variation of the process. The measured sizes are related to process modifications.



The insitu PAT-Sensor system measured the total number of droplet and particles during polymerization process. Inside the reactor, the sensor system measured the changes based on the P/T and stirring ratios. Therefore we could always follow the habit of the droplet sizes during the different reaction kinetics.

All this kinetic could be observed in reversed mode of the process.

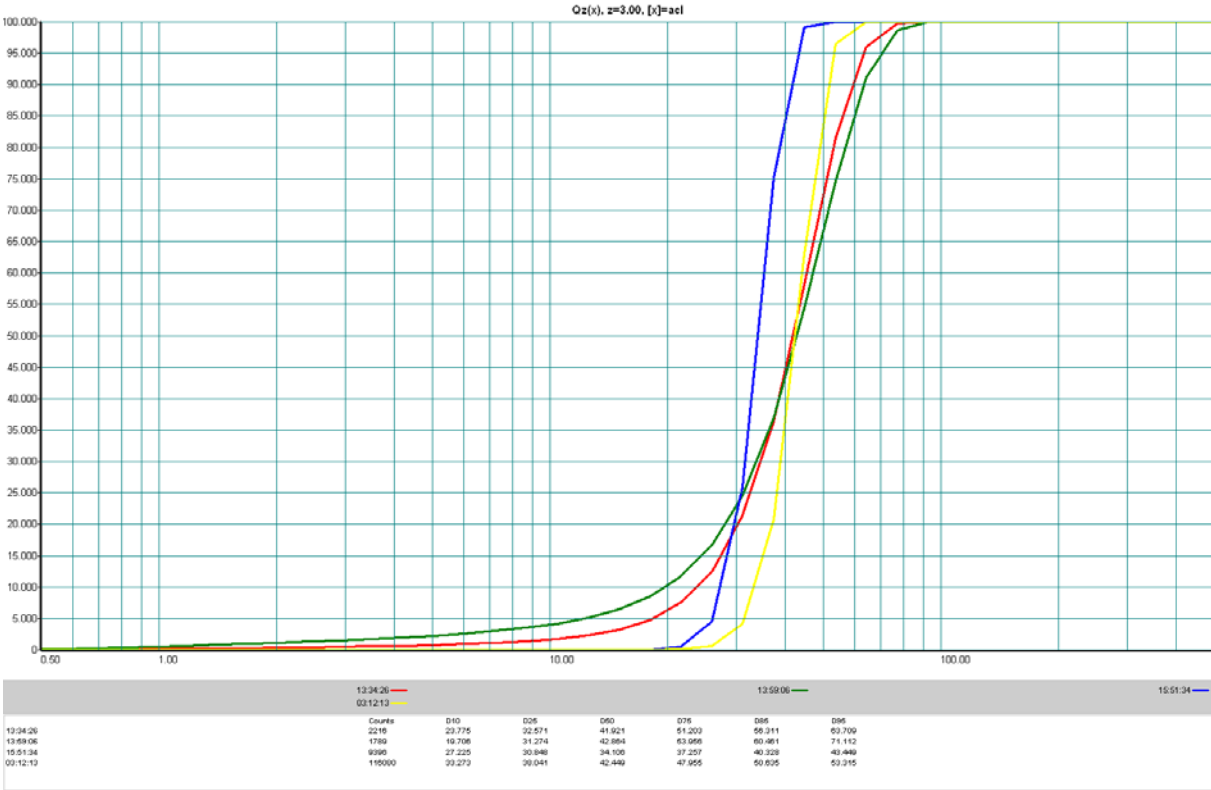


Fig.02 shows the distribution of counted particles of d10, 25, 50, 75, 95 of the volume distribution measured droplets during different kinetic phases of the reaction

It was also useful to see the different fractions over time of the operation mode in Fig.03

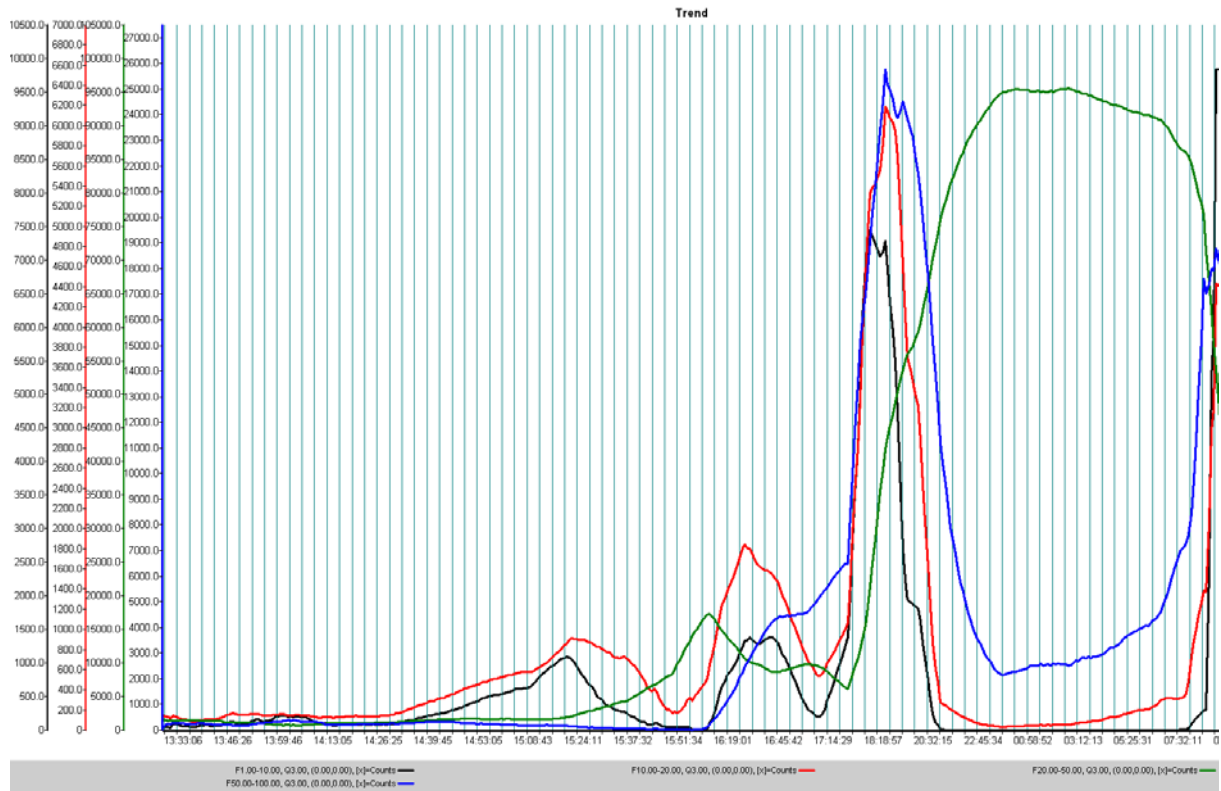


Fig.03 Fraction

1 to 10 μm	Black line
10 to 20 μm	Red line
20 to 50 μm	Green line
50 to 100 μm	Blue line

as a function of changes of the reaction conditions

During the same time we have also got access to the total number of measurement related to the amount of droplets or particles or and droplets in Fig.04



Fig.04 shows the change of number of droplets or particles during the process

During the reaction time it was questionable, what is the habit of the amount of fine particles. Based on fact that the insitu measuring sensor system takes all measured and counted signals as row data in store, these were easily to give as reports shown in Fig.05.



Fig.05 d10/2 over time of total reaction during the polymerisation

Summaries Control of a selective process system

- 1. All steps of the polymerisation process operation could be confirmed as reproducible.**
- 2. The measurements could be done under insitu and real time conditions.**
- 3. The sensor system could be installed under in line condition directly inside the process reactor.**
- 4. The operated sensor can be installed in several places of reactor vessels and autoclaves and will show the same results during the scaling up actions.**
- 5. The sensor system can be operated in a large various range of temperatures and pressures based on customer requirements.**
- 6. The sensor did not show any problems, even if particles or polymers arising on the surface of the sensor dip in numbers of months of operation.**
- 7. Technical specification under www.sequip.de/en/1/pat-particle-analysis.html**