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Model and Control Concept for an intensified Pre-reactor for the Production of high-viscous Polymer

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ACRONYMS AND DEFINITIONS

ACRONYMS	DEFINED AS
EC	European Commission
EU	European Union
CFI	Coiled Flow Inverter

THE CONSENS PROJECT

Intensified continuous processes are a key innovation of the last decade for the production of high quality, high value and customer-specific products at competitive prices in a sustainable fashion. To realize the potential of this technology, key steps must be made towards long-term stable, tightly controlled and fully automated production. The goal of the CONSENS project is to advance the continuous production of high-value products meeting high quality demands in flexible intensified continuous plants by introducing novel online sensing equipment and closed loop control of the key product parameters. CONSENS will focus on flexible continuous plants but the results will be transferable also to large-scale continuous processes. The research and development is driven by industrial case studies from three different areas, spanning the complete value chain of chemical production: complex organic synthesis, specialty polymers, and formulation of complex liquids. Innovative PAT technology will be developed for online concentration measurements (mid-resolution process NMR), for the online non-invasive measurement of rheological properties of complex fluids, and for continuous measurements of fouling in tubular reactors. New model-based adaptive control schemes based on innovative PAT technology will be developed. The project results will be validated in industrial pilot plants for all three types of processes, including validation in production containers that have been developed in the F3 Factory project. Further, methods for sensor failure monitoring, control performance monitoring and engineering support for PAT-based solutions will be developed. The exploitation of the new technologies will be facilitated by a tool for technology evaluation and economic impact assessment. A Cross sectorial Advisory Board supports the transfer of PAT technologies and adaptive control to neighbouring sectors of the European processing industry.

The CONSENS Consortium consists of:

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1 Executive summary

The report deals with the development of a control concept of the pre-reactor for a continuously operated 2-stage copolymerization process that leads to polymers of a very high viscosity. The idea behind setting up the 2-stage process is to use a tubular reactor in the first stage to produce a polymer with a certain viscosity which is still low enough such that a tubular reactor can be used but high enough to enable the use of a kneader reactor to finalize the polymerization in the second stage of the process. Starting from a batch recipe provided by SOLVAY, a suitable reactor concept has been developed and investigated experimentally. The chosen reactor design is a coiled tube (coiled flow inverter, CFI), which provides a narrow residence time distribution at the outlet without any mixing elements. To derive a dynamic model for the development of the control concept, experimental data has been used to estimate the crucial model parameters. The model is based on the equations for a tubular reactor including reaction, convection, diffusion and heat transfer through the reactor wall to the environment. Due to limitations in varying the inlet composition, the control concept is based on the variation of the total mass flow that enters the reactor – and hence the residence time – as the manipulated variable. The goal of the controller is to maintain the specified viscosity at the outlet of the tubular reactor, especially to avoid that the viscosity rises sharply and this leads to blocking of the reactor. The sensors investigated for application in the control loop is a RAMAN sensor for detecting the residual monomer at the outlet of the reactor, a viscosimeter for a measurement of the viscosity at the outlet of the reactor, and pressure sensors for the total pressure drop in the reactor. Due to the fact that a variation of the total inlet flow as the only manipulated variable changes the dynamics of the system, a controller concept was designed that adapts the controller parameters depending on the desired operation point.

The deliverable is organised as follows:

- Chapter 2 summarizes the design of the pre-reactor concept
- Chapter 3 presents the generic model description including the derivation of correlations needed to parametrize the model
- Chapter 4 shows the simulation results which are used to derive the control concept
- Chapter 5 deals with the controller design and the determination of the controller parameters
- Chapter 6 explains the future steps towards the validation of the model and the application of the controller

2 Pre-reactor concept

The recipe which is investigated for the production of high viscous polymers in a continuous plant is a batch procedure for a copolymerization that is operated in an adiabatic reactor. The molar ratios of both monomers and the added initiators are fixed as no changes in the product quality, e.g. the chain length distribution and the final viscosity should take place. The steps of the provided recipe are:

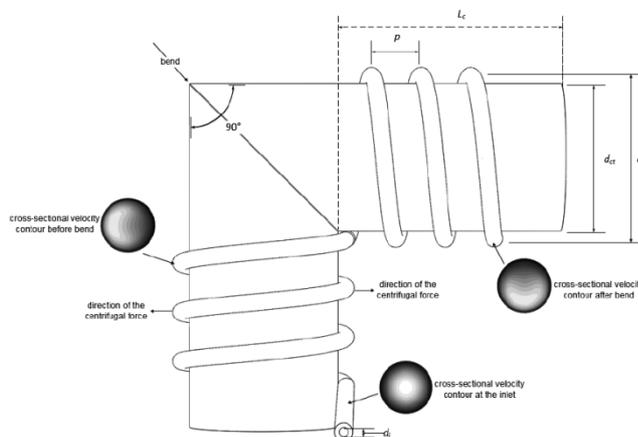
1. Mix the two monomers with water by continuous stirring
2. Adjust the pH value of the solution by adding an acid
3. Add the first initiator to the solution
4. Remove the oxygen from the solution by degassing for 1h
5. Add the second initiator to the reactor
6. Stop the stirrer after 2 minutes and wait until the polymerization is finished

This procedure at the end provides a high viscous gel that has to be removed mechanically from the reactor. During the experiments in the batch reactor and the analysis of the final product qualities in terms of intrinsic viscosity and residual monomer it became clear that step 4 can be also realized by degassing all components separately. Therefore the continuous process can be divided into several spatially separated tasks. The production concept is shown in the figure below



Figure 1 Concept for the continuous process

The main requirements for the first stage reactor are that it is suitable for the production of high viscous solutions and provides a narrow residence time distribution. The chosen reactor concept is based on a tubular reactor without any static mixers, as the static mixers would increase the probability of a blockage and are more difficult to clean afterwards. This was experienced as a key problem in the copolymerization case study of the F3 Factory Project. Thus a good mixing capability and a narrow residence time distribution have to be achieved without static mixers. A concept which is able to fulfil these requirements is a coiled flow inverter. The principle of the coiled flow inverter is the generation of dean vortices that are introduced by the winding of the tube and the change of the direction of the winding. The setup and the resulting flow profiles are shown in the figure below. This concept can be used to build up the reactor in a modular approach where each coil section without changes of the direction of winding represents an element of the reactor. The elements are interconnected at the 90° bends.



Taken from: Kurt, S. K. et al.; Modular Concept of a Smart Scale Helically Coiled Tubular Reactor for Continuous Operation of Multiphase Reaction Systems; *ASME-ICNMM 2016*, 2016, p. V001T13A001

Figure 2 Setup of a Coiled Flow Inverter

The possible measurements that can be installed at the CFI are temperatures, pressure differences, viscosity of the solution at the outlet, and the concentrations of the residual monomers at the outlet. The sensors belong to two groups, the group of currently available standard sensors for measuring temperatures, viscosity and pressure differences, and the PAT-Sensor for the concentration measurement based on the RAMAN spectroscopy. The different measurements were investigated for their applicability in the control of the process based on model predictions and experiments in the lab scale continuous pre reactor. The goal of the control is to maintain a constant quality (viscosity, conversion) of the product which is transferred to the kneader reactor. The process scheme which has been investigated is shown in Figure 3.

The scheme shows the four storage tanks for the feeds, which comprise the two monomers and the two initiators. The degassing of the feeds takes place in the storage tanks by the introduction of nitrogen. The feeds are pumped to the mixing section, while the pump rates are controlled via flow measurements in each feed stream. Additionally pressure sensors are located in the pumps to measure the pressure in the system. The mixing section includes an additional inlet of nitrogen which is used for cleaning or in the case of a possible blockage of the reactor. In both cases the nitrogen flow will push out the liquid from the reactor. After mixing the feeds, the solution is transferred to the CFI reactor that consists of a certain number of elements, depending on the specified residence time. The inlet temperature of each element is measured, while the residual concentration and the viscosity are measured at the outlet of the CFI reactor before the solution enters the kneader reactor. As the experimental work in this case study deals only with the first stage of the process, the kneader reactor is not included in the modelling.

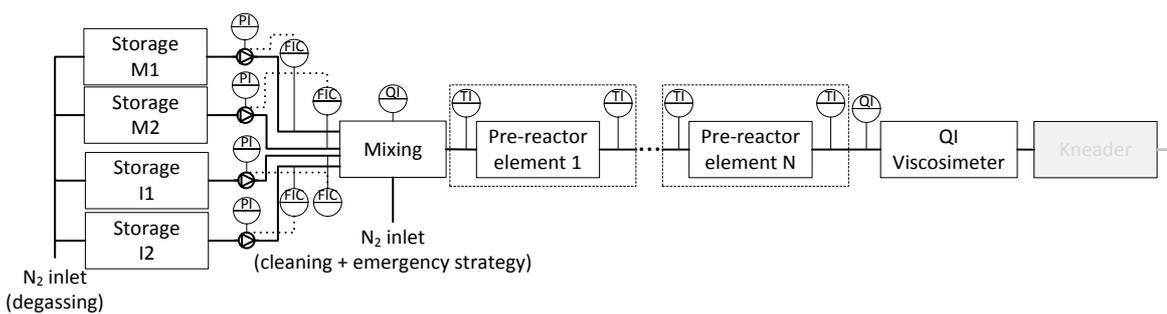


Figure 3 Scheme of the first stage of the continuous process for the production of high-viscous copolymers

3 Modelling of the pre-reactor

The model of the polymerization process in the CFI reactor is based on a standard tubular reactor model. The model is described by partial differential equations. The model consists of 33 differential states, which describe the temperature in the reactor and the temperature of the reactor wall, the concentrations of the monomers and the initiators and the produced polymer in terms of its chain length and sequence length distribution. The calculation of the average chain length and the average sequence length of a polymer chain are performed by applying the method of moments. The information about chain length and sequence length distribution is needed to calculate the differences in the product quality due to the change from the discontinuous process to the continuous process operated in a CFI reactor. The general component balance in a tubular reactor is given by the equation below.

$$\frac{dn_i}{dt} = R_i + \left[-\frac{\partial}{\partial z} (vn_i) + D_{ax} \frac{\partial^2}{\partial z^2} \left(\frac{n_i}{dV} \right) \right] dV$$

This equation can be divided into three parts. The first part, represented by R_i , describes the source/sink term of the component due to the polymerization reaction, i.e. it is a reaction rate. The second part describes the convection of the component with the velocity v , while the third part represents the diffusion/dispersion of the component i leading to backmixing. n_i describes the molar amount of the component i in the corresponding reactor volume dV . In order to estimate the crucial model term R_i which depends on the full state vector of the model, several batch experiments for the copolymerization and the homopolymerizations of the two monomers were performed in the lab. For the evaluation of the experiments, a RAMAN spectroscopy instrument was calibrated and included to the batch reactor. Additionally the temperature and the viscosity of the solution in the batch reactor were measured. A standard model for a polymerization in a batch reactor was set up to perform the parameter estimation of the kinetic parameters. The temperature dependency was described by an Arrhenius approach. The discontinuous model consists of the same number of mass balances as the continuous reactor but the convective and diffusive/dispersive terms of the equation above are not necessary. The estimation is based on a least squares optimization formulation, using all data obtained during the discontinuous homopolymerization and copolymerization experiments. The comparison of the model predictions and the measured concentrations of the two monomers in a batch experiment are shown in the figure below. The grey areas indicate the confidence intervals of the estimation. The comparison is presented for a copolymerization experiment in the batch reactor.

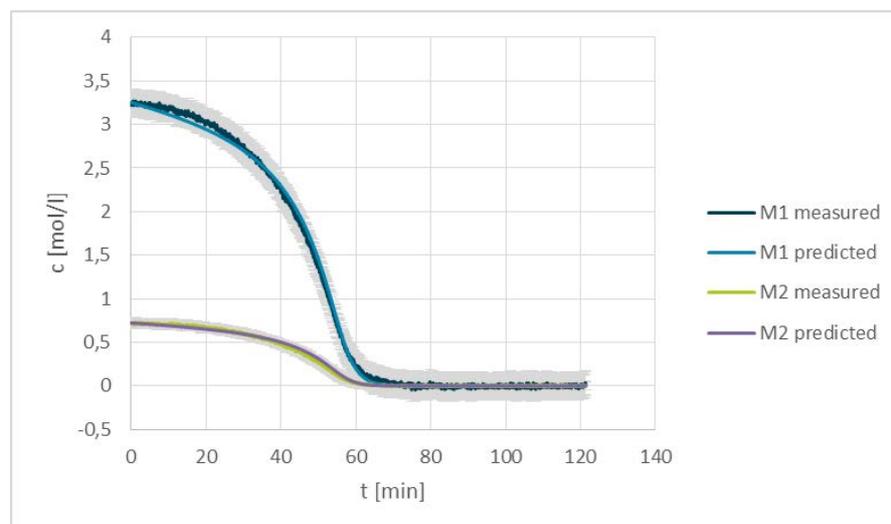


Figure 4 Comparison of measured monomer concentrations and estimated concentration trajectories in a batch copolymerization experiment

The resulting estimated kinetic parameters from the batch experimental data are assumed to be valid also in the continuous process. This defines the term R_i in the equation above.

The variable v which represents the velocity can easily be determined using the assumption of an incompressible fluid being fed to and pushed through the reactor, where the free diameter of the tube is constant. The equation to calculate v is shown below.

$$\frac{\dot{m}_{in}}{\rho_{fluid}A} = v$$

The variable \dot{m}_{in} describes the total mass flow that enters the reactor. The results from the evaluation of the batch experiments for the density of the solution show that the density of the solution ρ_{fluid} is not a function of time as shown in Figure 5. Therefore the density is assumed to be constant in the model.

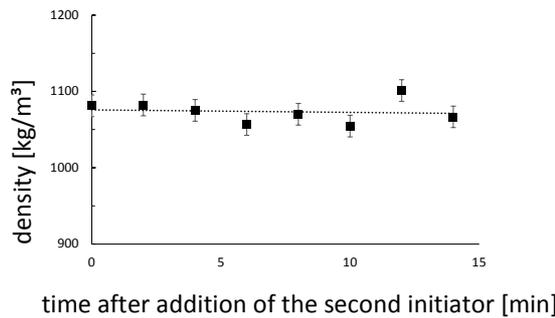


Figure 5 Evolution of the density of the solution in the reactor during a batch experiment

In order to estimate the axial diffusion coefficient D_{ax} the CFI concept was investigated by performing residence time distribution measurements. As the relation of D_{ax} and the Bodenstein number Bo is given by the equation

$$Bo = \frac{vL}{D_{ax}}$$

the Bodenstein number for different setups of the CFI was determined. The different setups differed by the number of turns per element and the total number of bends. Experimental investigations showed that the axial dispersion coefficient is independent of the length of the CFI reactor, while D_{ax} will increase considerably if a straight helix without bends is investigated. The result of this comparison is shown in the figure below. The explanation is that the 90° bends provide a better radial mixing. In consequence, the investigation of a single CFI element is sufficient to determine the axial dispersion coefficient.

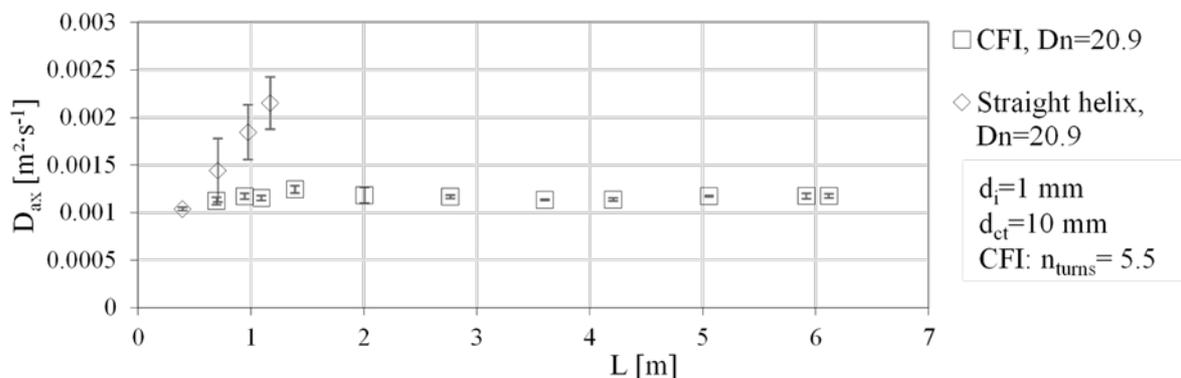


Figure 6 Comparison of the axial dispersion coefficient in a helical reactor and in a CFI

In order to set up a correlation for the axial dispersion coefficient in the CFI, different flow regimes and configurations were investigated by varying the Reynolds number Re and the Dean number Dn . The definitions of both numbers are shown in the equations below.

$$Re = \frac{v d_i \rho_{fluid}}{\eta} ; \quad Dn = Re \sqrt{\frac{d_i}{d_{beam}}}$$

η represents the dynamic viscosity, d_i the inner diameter of the CFI and d_{beam} represents the diameter of the beam which is used to coil the tube. The resulting correlation of the axial diffusion coefficient and the Dean number is a fourth order polynomial which is used in the model. Thus the influence of the reaction progress on the mixing characteristic, described by D_{ax} , due to the variation of the dynamic viscosity η is obtained.

Based on the concentration measurement, it is possible to calculate the amount of polymer produced during the reaction. This information can be used to correlate it with the viscosity measurements in order to derive a relation between the reaction progress, in terms of produced amount of polymer, and the dynamic viscosity of the solution. The evolution of the dynamic viscosity over the weight percentage of polymer in a batch experiment is shown in Figure 7. A polynomial approximation is used to describe the dependence and to integrate it into the model

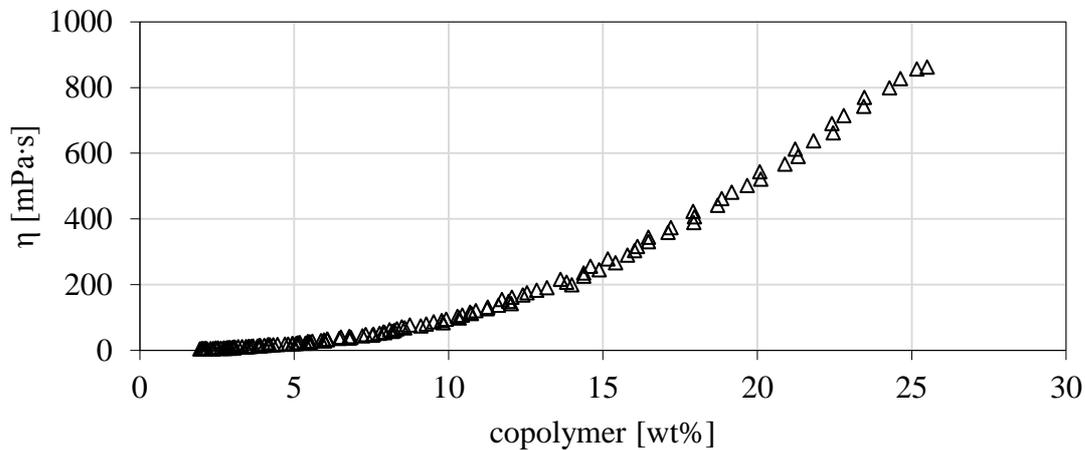


Figure 7 Correlation of the concentration of polymer in the batch reactor and the viscosity of the solution

In addition to the component balances the model consists of energy balances to simulate the temperatures of the reactor solution and the temperature of the reactor wall. The energy balance to calculate the temperature in the reactor T_r is shown below.

$$\frac{dT_r}{dt} = -\frac{\partial(vT_r)}{\partial z} + D_{t,fluid} \frac{\partial^2 T_r}{\partial z^2} + \frac{1}{\rho_{fluid} c_{p,fluid}} (\dot{Q}_r + kA_{e,r}(T_t - T_r))$$

The thermal diffusivity of the reaction solution is considered in the parameter $D_{t,fluid}$, $c_{p,fluid}$ represents the heat capacity of the fluid in the reactor, \dot{Q}_r describes the heat produced by the polymerization, $kA_{e,r}$ represents the heat transfer coefficient of the energy transport from the reactor to the reactor wall. While the parameters $D_{t,fluid}$ and $c_{p,fluid}$ could be obtained from the literature, \dot{Q}_r was estimated from batch experiments. The estimation was based on data from the concentration measurement and the temperature measurement and the assumption that \dot{Q}_r depends only on the propagation steps during the reaction. In order to calculate the heat transfer coefficients for the both energy transfers (reactor solution \leftrightarrow reactor wall \leftrightarrow environment) empirical equations have been implemented. As the energy transport to the environment is based on free convection, the Nusselt number is calculated from a relation including the Prandtl number and Rayleigh number. In the calculation of the heat transfer coefficient for the heat transport from the reactor solution to

the reactor wall, the Nusselt number is calculated from an empirical equation based on the Dean number and the Prandtl number as the transport is driven by a forced convection due to the flow in the reactor.

As the pressure drop in the reactor is also a measured variable in the process, it is calculated based on the following equation. The equation is based on the calculation of the pressure drop in a straight tube in a laminar flow regime including an extension which depends on the Dean number and thus on the coiling of the tubes.

$$\Delta p = \frac{\rho_{fluid} v^2}{2d_{tube,i}} \int_0^L z \frac{64}{Re(z)} (1 + 0.056Dn(z)^{0.603}) dz$$

4 Simulation results and sensitivity analysis

The dynamic model that was described in the previous chapter is used to simulate the trajectories of the process. Beside the simulation of the start-up/shut down of the process, the model enables the investigation of the sensitivity of the measured variables towards deviations from the steady state operation point and the investigation of possible control concepts. As an example for the simulation results of the start-up phase, Figure 8 shows the concentration profiles of monomer 1 depending on the position in the reactor and the time of operation. The initial state of the system is a reactor of 25m length filled with water and the temperatures are equal to the ambient temperature. The simulation is run at the nominal flow of 1 kg/h, which leads to a residence time of 28 s/m. The reaction starts immediately after start up, but as a certain amount of energy is consumed to heat up the reactor, the location of the full conversion shifts during the start up. The start-up period ends if the steady state is reached. The steady state profile is indicated by a red line in the figure below.

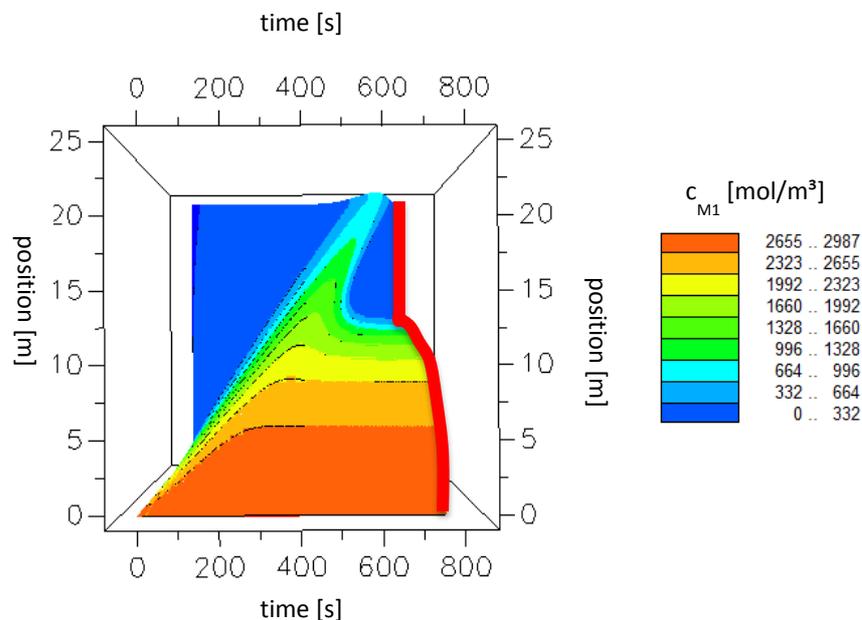


Figure 8 Concentration profile of monomer 1 during the start up procedure of the CFI reactor

Based on the simulation of both processes, the continuous and the discontinuous process, the differences of the product properties in terms of chain length and sequence length distribution can be analysed. The resulting simulated weight average chain length (WACL) and the number average chain length (NACL) in the continuous reactor with a length of 5 m (residence time 140 sec) and the discontinuous reactor with the same residence time are shown in the table below. Additionally the polydispersity index (PDI), which is defined as the ratio of WACL and NACL is presented.

Table 1 Comparison of the simulated chain length distribution in both processes at the same residence time

Parameters of the chain length distribution (simulated)	Batch process	Continuous Process
WACL	$4.22 \cdot 10^7$	$5.3 \cdot 10^7$
NACL	$2.25 \cdot 10^7$	$2.75 \cdot 10^7$
PDI	1.83	1.93

The control of the chain length distribution fully depends on model predictions as the chain length distribution cannot be measured online. Therefore model predictive control must be used here. For control of the chain length distribution in addition to conversion, additional degrees of freedom have to be used, e.g. the ratios of monomers and initiators, and the temperature of the environment. For a jacketed reactor, the jacket temperatures of the sections can be optimized such that desired product properties result.

In order to develop a suitable control concept, the steady state profiles of the continuous process were investigated first. The steady state profiles for the concentration of the first monomer and of the evolution of the viscosity in the reactor are shown in Figure 9. Depending on the product quality that has to be achieved for the transfer to the kneader reactor, two examples for possible operation points are shown. These represent extreme situations for the handover to the kneader reactor, at quite low and at quite high viscosities of the reaction solution. To shift from one operation point to another one and to stabilize the viscosity at the outlet, either the length of the reactor or the residence time in a reactor with a fixed length must be varied. As the first option is not suitable for control, the latter one is going to be investigated in the following sections. The inlet concentrations of the two monomers and of the two initiators are fixed based upon the batch recipe and as no additional side feeds are available, the variables that can be manipulated are the total mass flow entering the reactor and the temperature of the environment (which can be varied if a heating/cooling jacket is used).

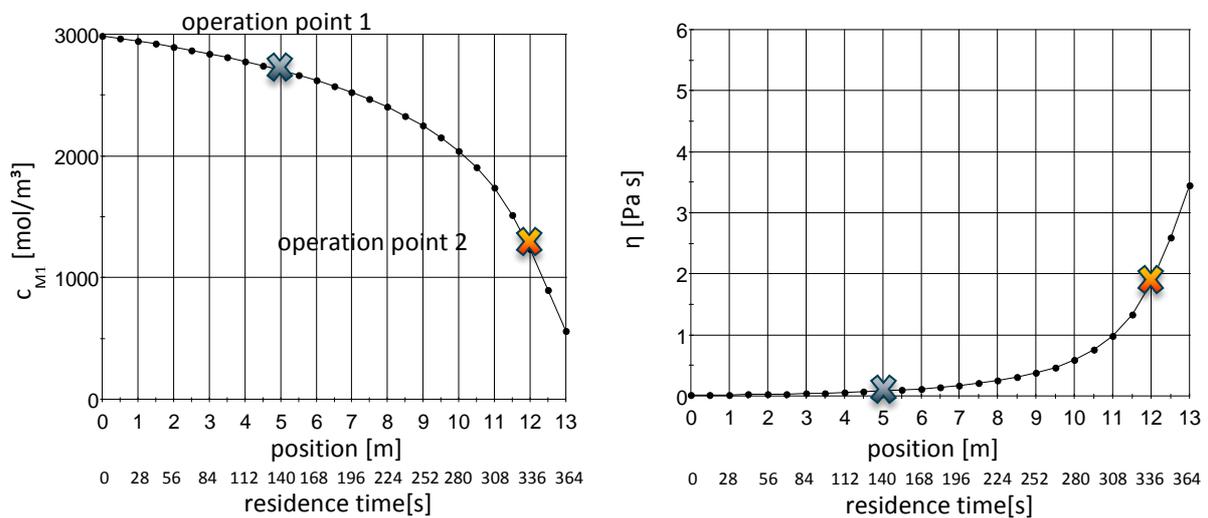


Figure 9 Steady state profiles of the concentration of monomer 1 and of the viscosity along the reactor and two characteristic operation points for a total mass flow of 1 kg/h

In order to control the total mass flow entering the reactor three different control structures are possible using the available instrumentation of the process. The first control structure uses the pressure drop as the controlled variable and is depicted in Figure 10. The control concept is based on a two layer controller. The upper level controller adapts the total mass flow according to the set point of the pressure drop in the system, while the lower level feedforward controller creates the set points for the flows of each stream as defined by the batch recipe.

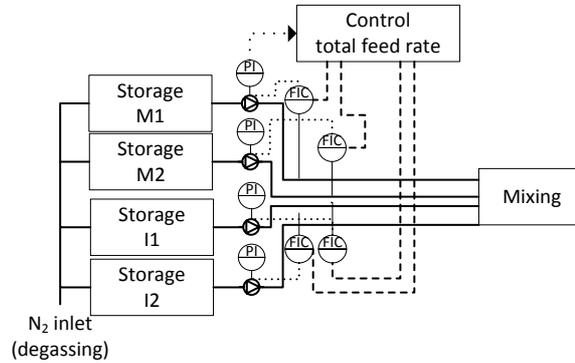


Figure 10 Control structure using the pressure drop as the controlled variable

The other two control concepts apply the same principle for the lower layer feedforward controller, while the controlled variable differs. In Figure 11 two different control structures are presented. The measured variables in alternative (1) are the concentrations of the monomers and in alternative (2) it is the viscosity of the reaction solution at the outlet of the CFI reactor.

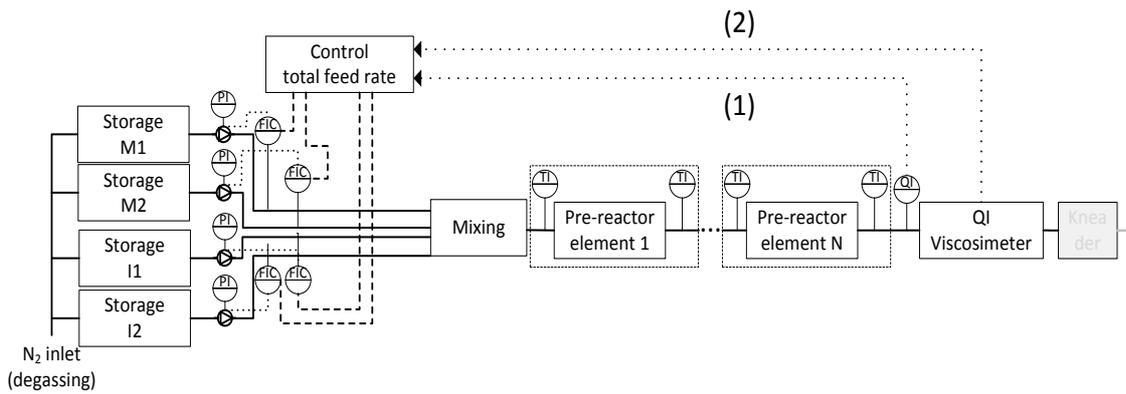


Figure 11 Control structures based on two different controlled variables: c_{Monomer} and viscosity

Based on simulations, a sensitivity analysis of the three different control schemes was performed to investigate the required accuracy of the sensors for the application in the control concept. For the control structure based on the pressure drop as the controlled variable, the resulting shift of the pressure drop if the process shifts from the original operation point is presented in Figure 12.

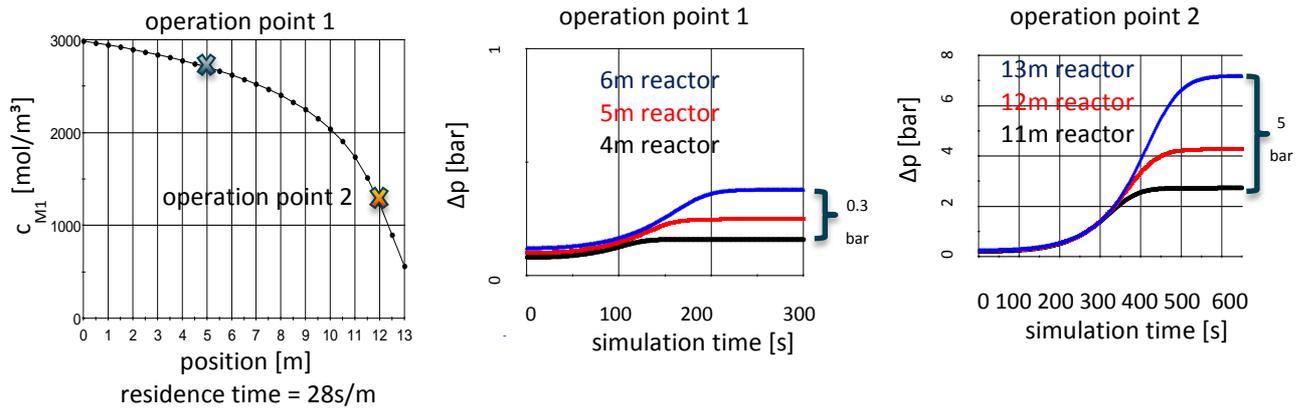


Figure 12 Sensitivity analysis of the pressure drop to shifts in the operation point

For operation point 1 where low concentrations of polymer are present in the reactor, the sensitivity is low compared to the sensitivity at high conversions (operation point 2). Especially if high conversions should be reached, the higher probability of blocking the reactor has to be considered in the choice of the sensor. Thus the definition of the operation point with respect to viscosity is a trade-off between the risk of blocking the reactor and a preferable high conversion. The presence of noise will amplify the difficulty of control especially for high conversion operation points as the introduction of filters will add an additional delay to the response. The resulting limitation in the speed of control will cause a reduction of the possible desired viscosity at the outlet, as a blocking of the reactor has to be avoided.

The application of the three different types of sensors in control and the resulting viscosity at the outlet will be validated experimentally in the lab plant at TUDO.

5 Control concept

For the given process two different control concepts can be applied. The first concept relies on single-input-single-output controllers, like PI(D)-based controllers. In order to tune such controllers, the transfer functions from the manipulated variable (throughput) to the controlled variable were identified. For the control structure with the RAMAN instrument, the controlled output is the concentration of monomer 1. In order to obtain a mathematical description of the I/O behaviour, several changes of the total mass flow were applied and the corresponding concentration profiles were analysed. One of the applied sequences to identify the input/output behaviour is shown in Figure 13 below. In this figure, results for two different lengths of the CFI reactor are shown. The nonlinearity of the dependency is clearly visible. Low values of the inlet flow will result in large changes of the conversion, up to full conversion and hence very high viscosities. Such low mass flows must be avoided in order to prevent the reactor from blocking. In consequence, the range of the changes of the throughput must be limited.

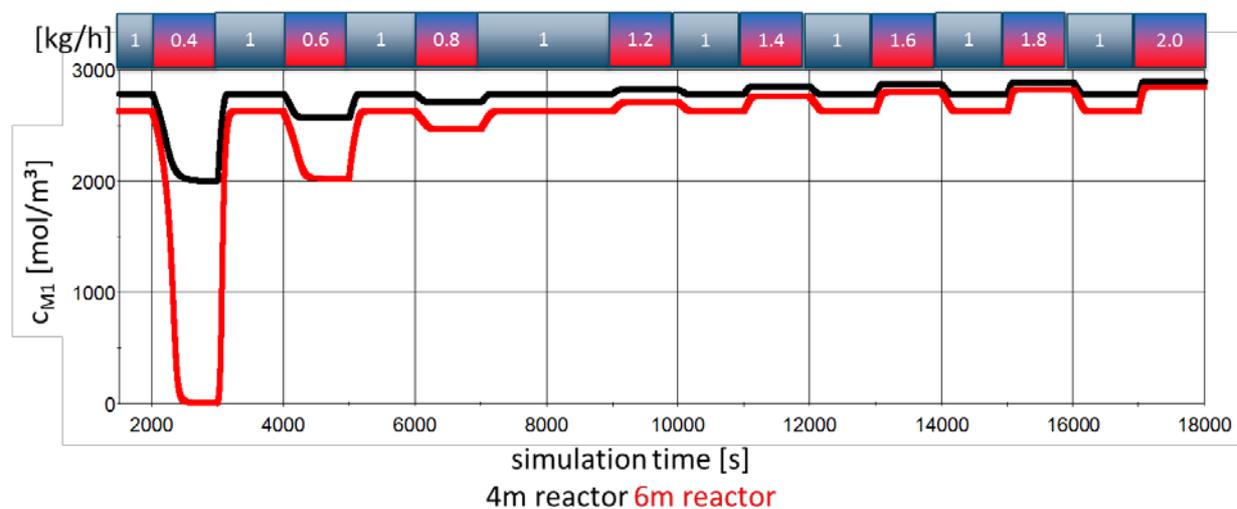


Figure 13 Step responses of the concentration of monomer 1 for a variation of the inlet mass flow (simulation)

For an analysis of the tuning of standard linear controllers, transfer functions of the system were estimated at different operating points. The generic form of the transfer functions is given below.

$$G(s) = \frac{K}{(1 + sT_1) \dots (1 + sT_n)} e^{-sT_d}$$

The first part describes a PT_n behavior of the system with n different time constants T_i , while the exponential part of the equation represents the pure time delay due to the fact that a shift of the input becomes detectable in the sensor only after passing through the reactor. The transfer functions were estimated assuming that the system can be described sufficiently accurately by a model with four different time constants T_i . The resulting parameters K and T_d of the transfer function for a reactor of 4 m length are shown in Figure 14.

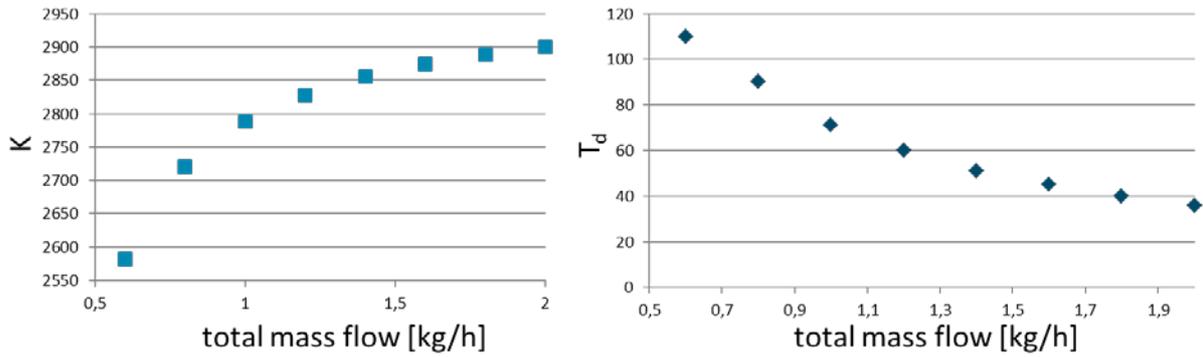


Figure 14 Gains K and delays T_d of the estimated transfer functions resulting from simulations

As the transfer functions vary depending on the desired operation point, the idea to implement the single-input-single-output controller is to adapt the controller parameters K_{PI} and T_{PI} in the PI controller, which is described by the following equation.

$$G_{PI}(s) = \frac{K_{PI}(1 + sT_{PI})}{s}$$

The parameters of the PI controller were computed for each single step response by solving an optimization problem which minimizes the settling time of the system, under the constraint of a 10% over-/undershoot of the closed loop system. The results of the parameter optimization are shown in the figure below.

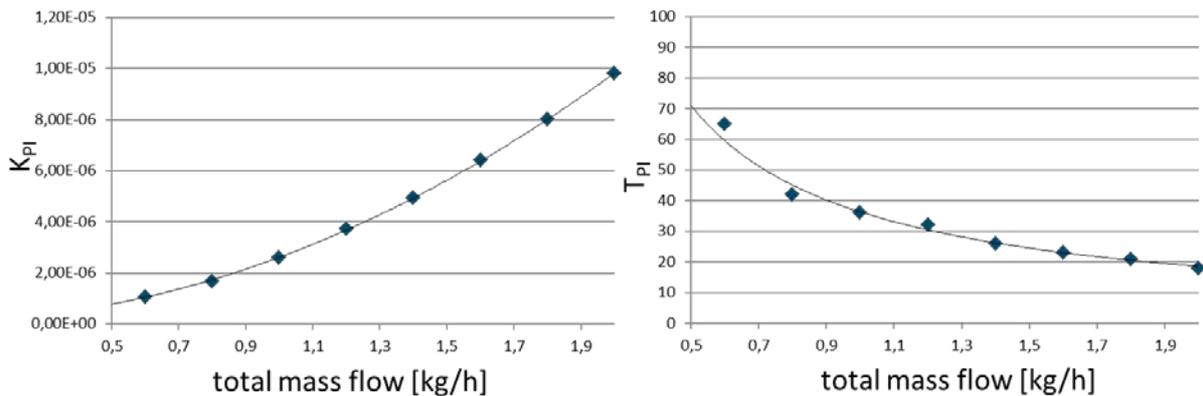


Figure 15 Controller parameters resulting from an optimization of the settling time of the closed loop

The controller implementation will use the controller parameters according to the desired setpoint of the total mass flow controller. Simulation tests of the controller are currently performed.

As the influence of changes in the throughput on the conversion and the viscosity at the reactor outlet is complex, due to the superposition of the immediate effect of a change of the flowrate which leads to a shift of the concentration profile in axial direction and the indirect influence due to a modification of the profile, more advanced control schemes should be preferred over simple linear SISO controllers. Model-predictive controllers can be based on precise nonlinear models of the process dynamics and in addition the satisfaction of constraints (e.g. a minimum flow rate) can directly and systematically be included. Also the use of the outside temperature as a second degree of freedom can be included. We will therefore also investigate the use of nonlinear model-predictive control of the conversion, similar to the work in Hashemi/Engell, *Macromolecular Reaction Engineering* 10(4), p. 415-434.

6 Future Steps

Based on the presented control concept the following steps will be performed until the end of the project:

- Validation of the model of the continuous plant, which was built up and is operational at TUDO
- Investigation of the application of the different types of sensors for the control of the continuous plant
- Testing of the presented linear SISO control mechanism based on scheduling of the controller gains and time constants in the lab scale plant
- Simulation studies and, if successful, experimental investigations of nonlinear model-predictive control for the control of conversion.
- Implementation of a state estimation scheme which uses the inlet and outlet temperatures of the reactor elements to estimate the monomer concentrations in the different sections
- Simulation studies of the control of the product properties in addition to conversion.