**Deliverable 6.4**

EC's Framework Programme for Research and Innovation Horizon 2020 (2014-2020)
Grant agreement no. 636820

# Cross-sectorial real-time sensing, advanced control and optimisation of batch processes saving energy and raw materials (RECOBA)

Start of the project: Jan 1st, 2015
Duration: 36 month

## Report about the results of the comparison between conventional practices and new practices

Due date: Dec. 31st, 2017

Lead contractor for this deliverable: BASF


Author:

| Company | Name |
|---|---|
| BASF | Omar Naeem |
| TKSE | |
| ELKEM | |
| UCAM | |
| RWTH | |
| VSCHT | |
| PMAT | |
| BFI | |
| CYB | |
| MINKON | |


Dissimination level

| | | |
|---|---|---|
| PU | public | ☒ |
| PP | restricted to other programme participants (incl. the Commission Services) | ☐ |
| RE | restricted to a group specified by the consortium (incl. the Commission Services) | ☐ |
| CO | Confidential, only for members of the consortium (incl. the Commission Services) | ☐ |

# Content

# 1 Executive Summary

This deliverable summarizes the comparison between two types of process for polymerization case study – standard processes vs the optimized process operation.

The standard process is typically proceeded using fixed recipes – dosing of educts (monomers, initiator, additives etc.) and temperature. Thus the process operation is time based, and is inflexible towards any operation disturbances. These disturbances are inherent in process operations and must be taken into account during the development of recipes. As a result, the time based recipes are conservative to account for process disturbances and be within constraints. The result is a process, not running optimally thus resulting in wastage of asset effectiveness, sub-optimal process monitoring as well as higher probability of out of spec products.

The project, RECOBA addresses the suboptimal operations of the process using state of the art methods in, a) process monitoring using hard/soft sensors, b) novel models incorporating kinetics, product quality and reactor periphery, and, c) unique optimization and control framework.

During the course of the project, the novel models, control infrastructure and new sensors have been developed and reported in other deliverables. This deliverable specifically reports the comparison between the standard process operations to the optimal batch, after implementation of developed tools to pilot plant reactor in BASF SE.

The comparison shows the exclusive process operation using model based online optimization and control methods and sensors. The process operates optimal for given constraints and disturbances. The optimization has higher weights on the product quality (i.e., better product quality) rather than economic objective, thus the batch time is comparable to the standard batch. If the identical product quality as of standard batch would be the constraint, the batch time could be reduced considerably.

# 2 Standard & Optimal batch results

This section reports the results of the two batches, standard and optimal, in pilot plant, separately. Following experiments (Table 1) were carried out at pilot plant to successfully achieve the demonstration for polymerization case-study.

| Experiment | Objective | Result | Remarks |
|---|---|---|---|
| **08 Jan 2018** | Water run | Successful | • Software, communication between DCS and CENIT tested using water |
| **09 Jan 2018** | Reference batch – CENIT as soft-senor | Successful | Standard batch |
| **11 Jan 2018** | Temperature control | Majorly okay – issues with recipe scale-up | The reaction runs into trouble in the later stages of batch because the initiator is depleted. The ratio between the initiator and redox agent is unfit for the pilot reactor |
| **15 Jan 2018** | Demonstration – Temperature control | Majorly successful | Same issue as above in the later stages of the batch – rest |
| **22 Jan 2018** | Optimal trajectories for quality control – morphology control | Not up-to the mark – Problem with NMPC tuning | • Due to below-par controller tuning, CENIT tracked the dosing rate trajectories rather than the concentration trajectories.<br>• Issue with recipe at the later stage of the batch |
| **25 Jan 2018** | Optimal trajectories for quality control – morphology control | Fairly good tracking of optimal profiles | Controller tuning was rather satisfactory. Some further tuning could improve the batch further. |
| **29 Jan 2018** | Optimal trajectories for quality control – morphology control | Successful batch – closed loop control of monomer using RAMAN and CENIT | • The tracking of optimal concentration trajectories went well. The Raman measurements did not coincide with the model.<br>• CENIT used the Raman measurement to estimate the propagation of monomer on-line, successfully so. The Raman measurement confirms that we tracked the optimal concentrations. |

**Table 1: Experiments carried out at pilot plant in BASF**

## 2.1 Results of the Standard batch operations

The batch chosen and considered in this deliverable is the one which is operated with normal, standard recipe but the reactor temperature control is regulated at given set-point (from standard recipe) using CENIT (a tool for real time optimization & control).

The results of the experiment show that process temperature control is satisfactory as illustrated in Figure 1. The lower plot in the figure shows the activation time of the controller, and it is evident that the peaks and valleys in the reactor temperature are caused by base-layer control of the reactor.

The monomer dosing profiles are shown in Figure 2. It illustrates the standard recipe for monomer dosing. The lower plot depicts the agreement of model (solid blue line) for first

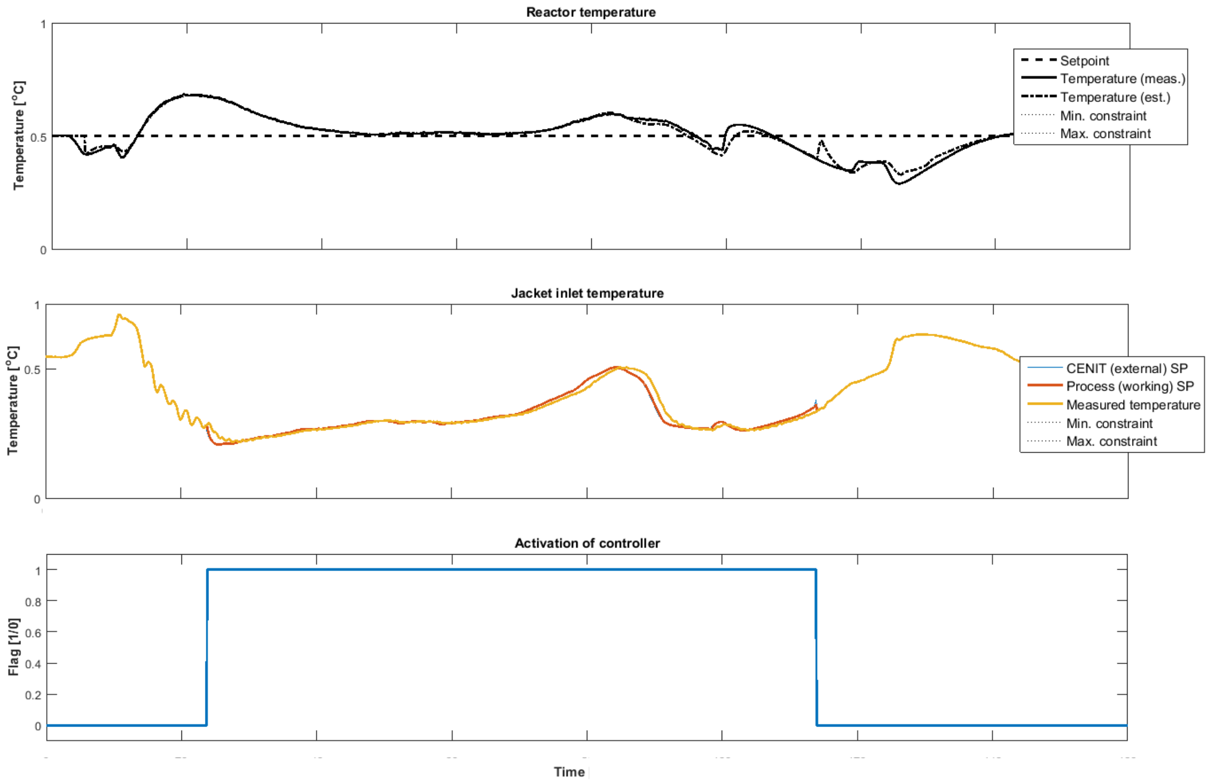


**Figure 1: Temperature control for standard batch**

monomer with inline RAMAN measurements. The agreement is rather satisfactory, considering the fact that this is one of the early experiments for RAMAN calibration at pilot plant. The measurements of RAMAN are in excellent agreement with offline HPLC samples (pink dots). The experiment was used to improve the model parameters and improve it.

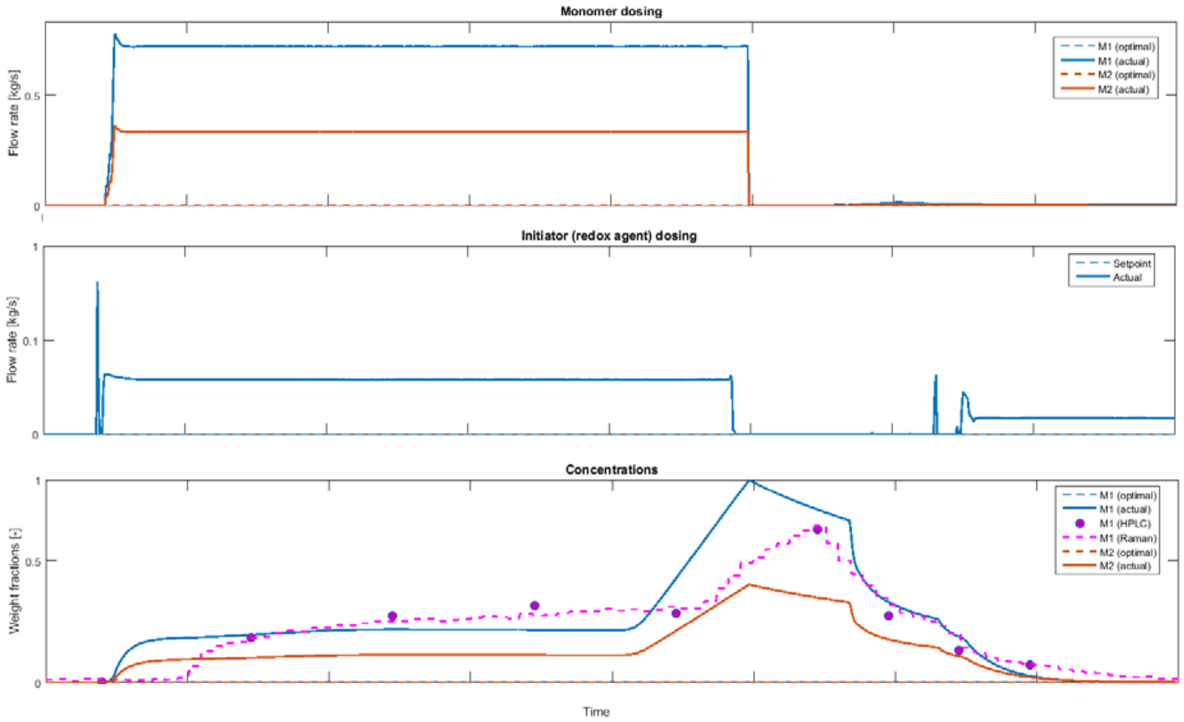


**Figure 2: Dosing of monomers and concentration profiles for standard batch**

## 2.2 Results of the Optimal batch operations

The batch selected this deliverable for comparison is the last batch of the demonstration campaign at pilot plant. The objective for the optimizer is to achieve the desired particle morphology of the emulsion using validated model (kinetics & morphology), hard sensors for energy measurements as well as RAMAN for closed loop monomer concentration tracking, and online controller to track the given concentration profile, to achieve intended morphology.

The temperature of the reactor is this not important control variable, rather it is constrainted between the upper and lower bounds to achieve desired morphology. Apprantly, the optimizer keep the reactor temperature at pervious set-point, which is a good signal (the initial guess to operate process at this SP is not bad). Anyhow, the weights of the optimizer to manipulate the temperature are not high. The rector temperature is depicted in Figure 3.

The monomer dosing profiles are shown in Figure 4. The first observable point is, that the dosing profiles for the monomers and initiator are not block profiles anymore. The dashed lines are the offline optimal calculations (using model) and give the path towards optimal batch operation and the online controller should track closer to these profilesto achieve desired morphology. The lowest plot shows the concentration profiles (dashed lines) from offline model and these have to be tracked in order to achieve the objective, while the online controller is permitted to manipulate dosing profiles in order to track the concentration profiles. But the model and online controller have inline measurements for the monomer concentrations (closed loop) in form of RAMAN signal. This is used by the controller as the most important feedback and used to manipulate the dosings of monomers and initiator to track the measurements. The results show that controller is capable of tracking these measurements excellently. This depicts that the disturbances in the process operation are rather usual and the online corrections are necessary to achieve desired product quality (morphology in this case).

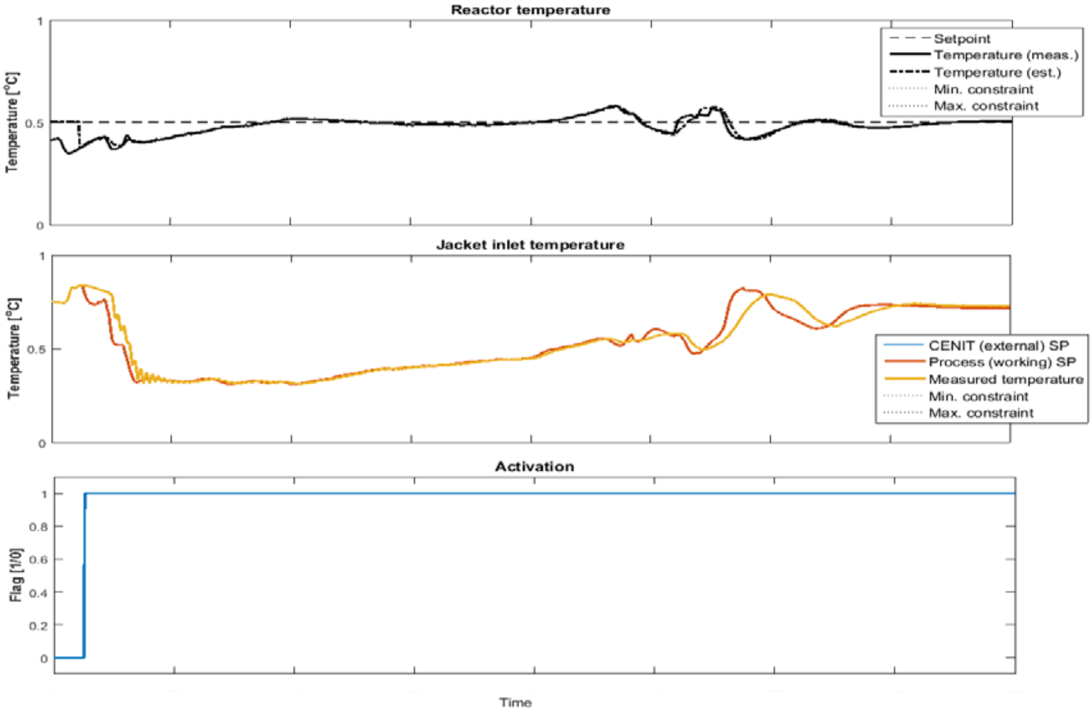


**Figure 3: Temperature control for Optimal batch**

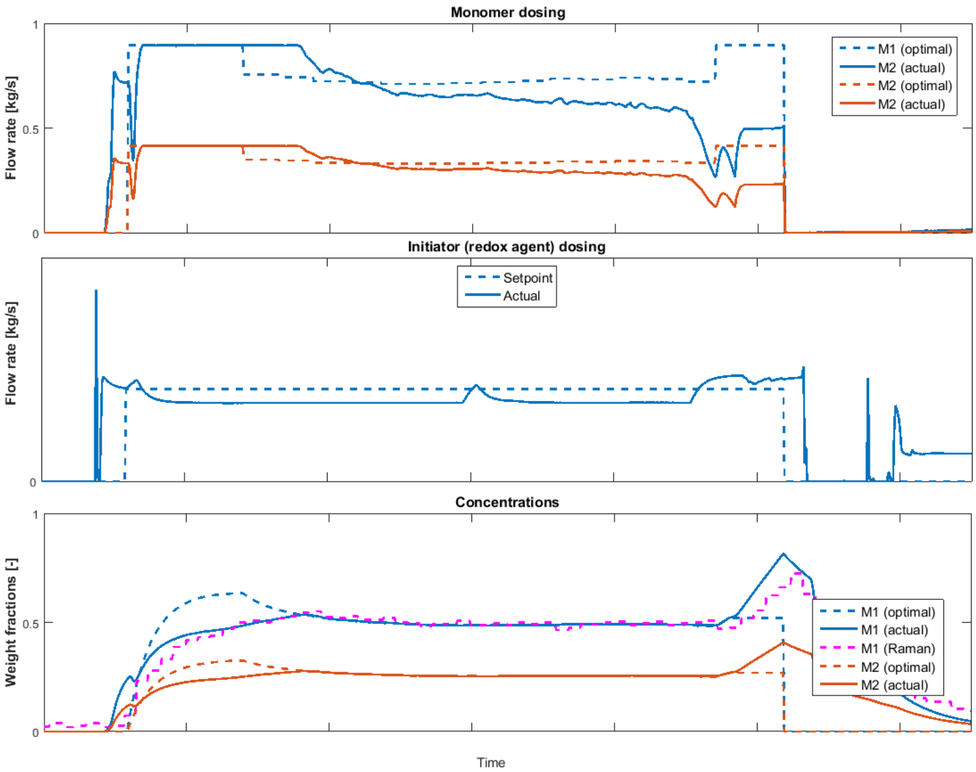


**Figure 4: Monomer dosing and concentration profiles for Optimal batch**

## 2.3 Comparison of standard vs optimal batch

This sub-section summarizes the comparison of the two chosen batches, standard recipe (CENIT temperature control) vs optimized batch.

Figure 5 shows the comparison of temperature control of reactor, and it is actually hard to differentate between the two, since CENIT is controlling the reactor temperature, actively.

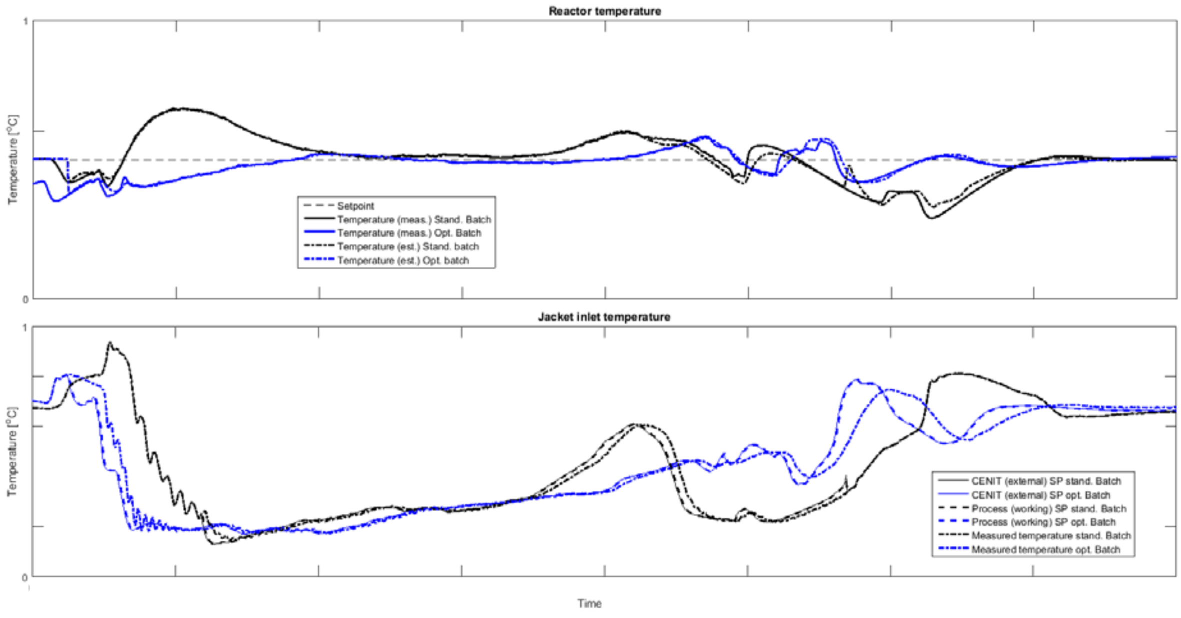


**Figure 5: Comparison of batch temperature control - standard batch vs Optimal Batch**

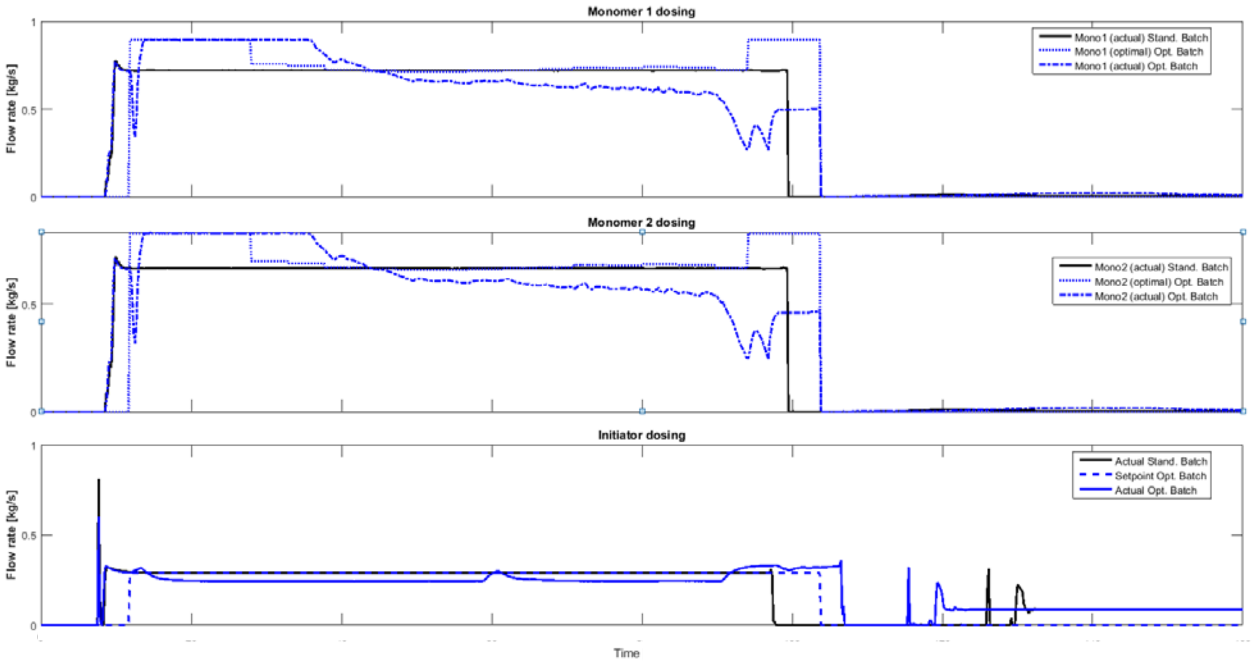


**Figure 6: Comparison of monomer/initiator dosing - standard batch vs Optimal Batch**

Figure 6 shows the dosing of educts to the reactor for both batches. As illustrated, the dosing profile in standard batch is block structure while for the optimal batch, there is no fixed dosing profile, though there is profile from offline optimization of the model, its used a guess by the online model. The actual objective for the online model is to trace the profile of concentrations achieved via closed loop, inline measurements (RAMAN). This is clearly depicted in Figure 7. The online model, controller and optimizer use the concentration profiles from offline model as initial key to achieve desired morphology. However, the offline model is not updated frequently, and thus the mismatch between the model and reality, as well as the process disturabnces (season's change, fouling on rector wall, monomer quality etc.) are the reasons for out of spec products.

Thus the closed loop control, using hard sensors, updated models (model is updated every 1-5 minutes) and state of the art control and optimization methods are the answer to achieve in-spec product everytime, while accounting for process variations.

This implies that state based recipes are superior in all possible conditions to improve the process operations, by increasing the asset effectiveness, reduction of effort to blend bad/good batches, use of accurate amount of educts to operate the batch, as well as the lower energy consumption by operating at optimal temperature, thus positive influencing the sustainability of production processes.

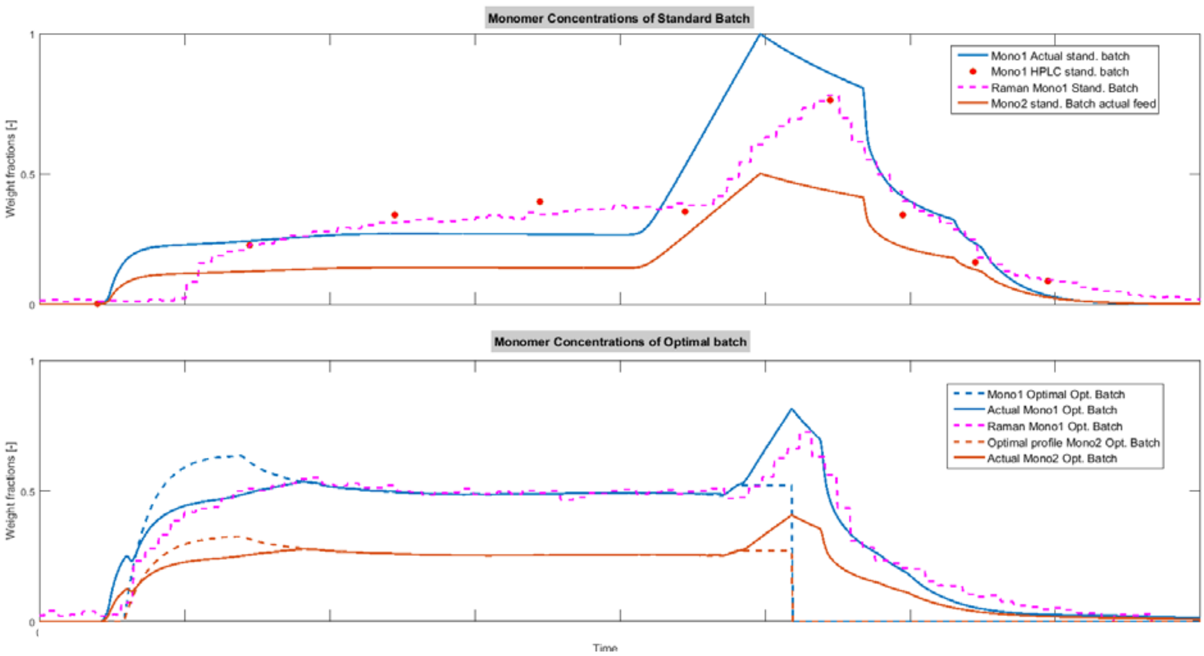


**Figure 7: Comparison of monomer concentration profiles - standard batch vs Optimal Batch**

# 3 Conclusions

This deliverable summarizes the comparison of standard batch vs optimized batch, for emulsion polymerization case study.

The report illustrated the superiority of state of the art hard sensors, models and control framework to operate a polymerization batch optimally. The results do show that improved process operation using novel tools and methods, result in improved product properties (through state based recipes), lower energy consumption (blending of batches, heating etc.), lower/minimal out of spec batches as well as improved social matters, such as lower stress levels for operators etc.

The results depicted in this report are limited to two batches for emsulsion polymerization processes , but the illustration is rather generic for similar processes, regardless of the chemistry involved.