Accelerated Scale-up

Reaction calorimetry and reactor simulation – combined techniques accelerate scale-up of a highly exothermic oxidation reaction.

Marc Bollyn, Armand von den Bergh, Allen Wright

Introduction

Temperature control of a process in which two thioethers were oxidized with hydrogen peroxide to the corresponding sulfones proved to be a major factor for safety and product quality. Preliminary reaction calorimetry (fig. 1) supplied data to calculate the cooling requirements for the vessel (1100 kJ/mole). However on scaling up to 400 l there remained an uncontrolled rise in reaction temperature which prohibited further increase in scale. The parameters which controlled the chemical conversion were already well established but no kinetic data were available.

This paper describes how a reaction model was build from reaction calorimetry data and converted into a process model. The feed sequence was optimized by running simulated experiments in which the heat production rate profile was evaluated. The outcome of these experiments was validated at pilot plant scale. Finally the data from the production vessel were included in the model which was used to optimize the production process.

This combined approach enabled fast scale-up and helped to convince a toll manufacturer to run the process. The result was a safe and economic process established with only a few experiments at pilot plant scale.

Problem Statement

The problem for this process was to establish operating conditions which would permit scale-up to the desired volume of 4 or 8 m³. The main problem which had to be addressed was the control of the high reaction exotherm of the multiple reaction steps. The overall reaction enthalpy of 1100 kJ/mole demands a high cooling capacity from the process vessel and good controllability of the process.

Suitable operating conditions had to be established within the previously imposed constraints on process operation. Most of these constraints were imposed on the variables which control the reaction exotherm. One of the most important limitations was temperature. It was experimentally proven that the operating temperature should not exceed 50°C. Higher temperatures did not affect the thermal stability of the reaction mixture, however product quality and yield were reduced. This constraint limits the cooling capacity of the vessel by restricting the acceptable temperature difference across the reactor wall. It also limits the reaction rate and hence the possibility to run the process under dosing controlled conditions. The catalyst ratio had to be kept as low as possible for reasons of economics and ecology.
Consequently increasing catalyst concentration was not an option if an increased reaction rate should be demanded. For reasons of handling safety, 35% hydrogen peroxide was chosen instead of a 50% solution. The increased dilution of the reaction mixture which arises from using 35% peroxide results in a reduction of the reaction rate.

Finally, in order to attain an acceptable price, it was necessary to run the process at ton scale in a 4 or 8 m³ vessel. This cost constraint also means that the batch cycle time (dosing time) should be as short as possible without neglecting the safety aspects.

Insufficient free production capacity at the AGFA plant made it necessary to approach a toll manufacturer to run the process. It was agreed that the process development up to 1.6 m³ would be done at AGFA and that the process would then be scaled-up to production in a 1000 gallon vessel. However, it was difficult to determine the optimum feed sequence and demonstrate that it would be possible to run the process safely. During several 400 l trials at AGFA a serious temperature control problem appeared. It was not apparent from the data collected in an automatic laboratory reactor that such a problem would arise. These experiments were done with different operating conditions (lower temperature and a catalyst ratio) and with technical grade starting material. The initial experiments in the RC1 did not reveal the real thermo-chemistry of the process, probably because of the short addition time.

**Project Target**

The aim of the project was to collect data and build a model to accurately simulate the thermal behavior of the process. It was hoped that the model would give an understanding of the temperature control problem and allow further scale-up conditions to be established. It was decided that only RC1 data could be used for this purpose. Several trials to establish concentration profiles of the reaction mixture failed because there was no suitable analytical technique. The problem was further complicated by the fact that the full process would be run with a technical grade of starting material 87% assay, (∼10% identified contaminants). The model would be established with data from pure starting material (> 97%) and subsequently the influence of the impurities (also oxidizable substances) would be incorporated. There was also a purification method for the starting material. About six months were available to finish this project.

**Solution Approach**

The approach to solve this problem consisted of several consecutive steps. The chemistry was already optimized for yield and product quality and a lot of knowledge about process conditions was available.

The following steps were taken to build a process model using different tools:

- Run experiments in RC1 at different conditions: RC1 equipment
- Extract reaction kinetics from experimental data: RATE (PC software)
- Develop chemistry mechanism and model: RATE/REACTION (PC software)
- Validate the model: RC1 SIMULATOR (PC software)
- Optimize ( thermo)chemistry at large scale (imposed temperature profile): REACTION (PC software)
- Include data from production vessel(s): REACTION
- Optimize process at production scale with simulation: REACTION
- Run the process at production scale for the first time under safe conditions: Plant
- Optimize the full process using the suggestions from the simulations and data of the previous runs: Plant

**Chemistry**

The chemistry of this process can best be explained with the reaction scheme in figure 2. The starting material contains, among others, two thioether functional groups which need to be converted to the corresponding sulfones over the sulfoxide stage. The catalyzed oxidation with hydrogen peroxide
through four steps. From TLC and qualitative HPLC data it was clear that the oxidation proceeded stepwise. The nature of the interaction between hydrogen peroxide and the catalyst was unknown for this system.

Development of the Chemistry Model

For the modelling of the reaction kinetics for the process, Qr data from one key RC1 experiment with pure starting material were taken. This was run at nearly isothermal conditions and yielded 94% product after workup.

The experimental Qr curve
The experimental Qr curve (figure 3) shows a number of features that the kinetics should reflect. The curve shows a flat region which would correspond to the first two steps of the reaction being very fast. Next there is an exponential rise in Qr. Because of the high heats of reaction of all the steps and the suddenness of this rise it seems essential that the model accounts for this behavior. The reaction exotherm falls before another sudden rise in Qr occurring at the end of the feed addition. This Qr peak is caused by the crystallization of the product at the end of the addition. By modifying the concentration this could be avoided and thus this phenomenon did not have to be explained by the model.

Model data
Data for the model were easily collected or calculated from the initial conditions. The reaction exotherm was determined using the RC1 evaluation software. As the process kinetics were to be estimated from the reaction heat data it was necessary to estimate the heats of reaction for each of the four steps. Since it was thought that the conversion proceeded stepwise an estimation of the reaction heat could be made from the calorimetry results together with the CHETAH package. The estimated values of the heats of reaction of each of the four reaction steps are summarized in table 1. The calculated values were corrected to match the experimental values and these were entered in the model.

Preliminary kinetic model
Initially a simple reaction scheme was used. The kinetic expressions were assumed to be simple power laws. The effect of the catalyst was not included at this stage (table 2). The rate constants k1, k2, k3, k4 together with the exponents α3, α4, β3 and β4 were fitted simultaneously using the kinetic fitting program RATE. A second order expression with respect to E and F were necessary to obtain an acceptable match over all the three regions of the Qr curve. This is illustrated in the figures 4 and 5.

Kinetic model including the catalyst
Next the formation of a peroxy complex was taken into account. This was assumed to be chemically reversible and may be represented as a series of fast equilibrium reactions. The concentrations of the peroxy species can be expressed in terms of [B] and [I] (table 3).

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 1</td>
<td>-231.29</td>
<td>-27.49</td>
<td>-223.10</td>
</tr>
<tr>
<td>Step 2</td>
<td>-231.29</td>
<td>-27.49</td>
<td>-223.10</td>
</tr>
<tr>
<td>Step 3</td>
<td>-336.99</td>
<td>-32.77</td>
<td>-326.40</td>
</tr>
<tr>
<td>Step 4</td>
<td>-336.99</td>
<td>-32.77</td>
<td>-326.40</td>
</tr>
<tr>
<td>Total</td>
<td>-1136</td>
<td>-120.52</td>
<td>-1098</td>
</tr>
</tbody>
</table>

Table 1: Estimated values of the heats of reaction of each of the four reaction steps.

<table>
<thead>
<tr>
<th>Reaction scheme</th>
<th>Kinetic expression</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 1</td>
<td>B + A = C + D</td>
</tr>
<tr>
<td></td>
<td>k1·[B]·[A]</td>
</tr>
<tr>
<td>Step 2</td>
<td>B + D = C + E</td>
</tr>
<tr>
<td></td>
<td>k2·[B]·[D]</td>
</tr>
<tr>
<td>Step 3</td>
<td>B + E = C + F</td>
</tr>
<tr>
<td></td>
<td>k3·[B]·β3·[E]·α3</td>
</tr>
<tr>
<td>Step 4</td>
<td>B + F = C + G</td>
</tr>
<tr>
<td></td>
<td>k4·[B]·β4·[F]·α4</td>
</tr>
</tbody>
</table>

Table 2: B = H2O2, C = H2O, A = starting material, G = final product, D, E, F are intermediates.

Figure 4: First order model.

Figure 5: Second order model.
It was also assumed that only one of the peroxy species was the oxidant and that this species only reacted with the sulfoxide to form the sulfone.

It was experimentally shown that the conversion from the first two steps could be easily performed without the catalyst. Since these steps were very fast with or without catalyst the model was simplified. In order to fit the exponential rise of the Qr curve the scheme requires a second order rate dependency on species E and F. This yields the kinetic scheme (table 4).

The program RATE was used to fit the kinetics for the different rate constants for values n=1 and n=2, while fixing $k_1 = k_2 = 0.2 \text{ m}^{-1}\text{mol}^{-1}\text{s}^{-1}$.

The reaction scheme with n=2 showed a closer agreement with the rise and fall of the exotherm. Next the values for $k_1$ and $k_2$ were fixed to a lower value ($0.05 \text{ m}^{-1}\text{mol}^{-1}\text{s}^{-2}$) and the fitting calculations were repeated for n=2 and n=3. The n=3 system also shows a good agreement between the model and experiment.

Refinement of the model

From the results of the preliminary model it appeared that it was necessary to have a second order rate dependency of both the species E and F.

This assumption was tested on the data from two experiments at different temperatures. The values of n were also varied. The following four mechanisms were tested against the experimental data. The statistical results of the fitting calculations are summarized in table 5. The strategy was repeated on a set of two experiments with a higher catalyst ratio. The conclusions of the first set were also applicable to these experiments.

Mechanism A (figure 6) gives the best fit both from a visual inspection of the graphs and from a statistical analysis of the results. The reaction 6 shows no significant variation in rate with temperature. The rate of this reaction probably increases with temperature similarly to reaction 5. This is however of minor importance in this study.

Mechanism B (figure 7) is almost as good as the mechanism A and the choice was made based on the graphs.

**Table 3.**

<table>
<thead>
<tr>
<th>Reaction scheme</th>
<th>Concentration expression</th>
<th>Concentration expression in terms of [I] and [B]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 1</td>
<td>B + I = J_1</td>
<td>$[J_1] = k_1 \cdot [B] \cdot [I]$</td>
</tr>
<tr>
<td>Step 2</td>
<td>B + J_1 = J_1</td>
<td>$[J_2] = k_2 \cdot [B] \cdot [J_1]$</td>
</tr>
<tr>
<td>Step 3</td>
<td>B + J_2 = J_3</td>
<td>$[J_3] = k_3 \cdot [B] \cdot [J_2]$</td>
</tr>
<tr>
<td>Step 4</td>
<td>B + J_3 = J_4</td>
<td>$[J_4] = k_4 \cdot [B] \cdot [J_3]$</td>
</tr>
</tbody>
</table>

**Table 4: B = H_2O_2, C = H_2O, A = starting material, G = final product, D,E,F are intermediates, I = catalyst and J = peroxy species.**

<table>
<thead>
<tr>
<th>Reaction scheme</th>
<th>Kinetic expression</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 1</td>
<td>B + A = C + D</td>
</tr>
<tr>
<td>Step 2</td>
<td>B + D = C + E</td>
</tr>
<tr>
<td>Step 3</td>
<td>I + nB = Jn</td>
</tr>
<tr>
<td>Step 4</td>
<td>Jn = I + B</td>
</tr>
<tr>
<td>Step 5</td>
<td>Jn + nE = nC + nF + I</td>
</tr>
<tr>
<td>Step 6</td>
<td>Jn + nF = nC + nG + I</td>
</tr>
</tbody>
</table>

**Caption for Mechanisms A to D**

(Figures 6 to 9)

B = H_2O_2, C = H_2O
A = starting material
G = final product
D, E, F are intermediates
I = catalyst and J = peroxy species

**Figure 6: Mechanism A.**

- calculated Qr
- measured Qr
- excluded Qr data
Accelerated Scale-up

From the reaction rates at the different temperatures for mechanism A the Arrhenius constants were calculated (table 6). These were used in the further development of the process model.

Figures 7, 8, and 9 show the reaction schemes for mechanisms B, C, and D, respectively. The tables summarize the statistical results of the fitting calculations for each mechanism (O.F. = Objective Function; % VAR. = % variation explained by the model).

From the reaction rates at the different temperatures for mechanism A the Arrhenius constants were calculated (table 6). These were used in the further development of the process model.

**Table 5:** Summary of the statistical results of the fitting calculations (O.F. = Objective Function; % VAR. = % variation explained by the model).

<table>
<thead>
<tr>
<th>Mechanism</th>
<th>Reaction scheme</th>
<th>Kinetics</th>
<th>A</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 1</td>
<td>B + A = C + D</td>
<td>k1[B][A]</td>
<td>2.025\times10^8</td>
<td>56861</td>
</tr>
<tr>
<td>Step 2</td>
<td>B + D = C + E</td>
<td>k2[B][D]</td>
<td>2.025\times10^8</td>
<td>56861</td>
</tr>
<tr>
<td>Step 3</td>
<td>I + 2B = J</td>
<td>k3[B]^2[I]</td>
<td>6.79</td>
<td>0</td>
</tr>
<tr>
<td>Step 4</td>
<td>J = I + 2B</td>
<td>k4[J]</td>
<td>0.056</td>
<td>109</td>
</tr>
<tr>
<td>Step 5</td>
<td>J + 2E = 2C+2F+I</td>
<td>k5[J][E]^2</td>
<td>1.018\times10^{11}</td>
<td>75342</td>
</tr>
<tr>
<td>Step 6</td>
<td>J + 2F = 2C+2G+I</td>
<td>k6[J][F]^2</td>
<td>0.523</td>
<td>2709</td>
</tr>
</tbody>
</table>

**Table 6:** Arrhenius constants.
Validation of the Chemistry Model

The RC1 SIMULATOR was used for validation of the reaction model. The experimental data together with the Arrhenius coefficients and the values for the reaction heats were incorporated in a model file. The experimental conditions for several experiments were simulated and compared with the $Q_r$, $T_a$ and $T_r$ results of the actual experiments (figures 10 and 11). There was a good agreement between the models and the experiments especially using mechanism A.

For experiments with the technical grade of starting material, the values for the reaction heats were scaled and these experiments also showed good agreement. This model could therefore be used to simulate a real plant process with this grade of starting material. At a later stage more validation experiments were done.

Discussion

Through the model development the following understanding about the reaction was gained. The sharp rise in exotherm is caused by the simultaneous combination of the following factors:

- a high concentration of the intermediate bis-sulfoxide (as a result of the high selectivity of the sulfide to the sulfoxide oxidation), formed prior to the rate-limiting step,
- a higher reaction enthalpy for the sulfoxide to sulfone conversion (337 versus 231 kJ/mole) and
- the fact, that these reactions are 2nd order, which accelerates the heat release

Development of a Process Model

The validated RC1 model was then used for simulation experiments at large scale. The main concern was to find a feed sequence which would allow to control the reaction exotherm for the third oxidation stage within the following constraints: the feed time should be reasonable with regard to the cost of the product; secondly it was known that side reactions occurred above 50°C and since feed times longer than 24 hours had never been studied it was assumed that a too long feed time at 45°C could also induce these side reactions and hence lead to product losses.

Trial productions would be run in a 1.6 m³ vessel with 1100 moles of starting material to supply product and demonstrate the process to the toll manufacturer. These conditions were therefore used to develop a process model. It was estimated that the 1.6 m³ vessel had a cooling capacity of about 32 kW under the normal process conditions. This value was used to decide whether a proposal was acceptable or not.

The following aspects were also taken into account. The feeding of hydrogen peroxide would start at room temperature and the reaction heat (first hour) used to raise the reaction mixture to the desired temperature. It would be desirable to fit the addition of hydrogen peroxide into two shifts of 8 hours. The model was simplified by using an imposed temperature profile. Since the temperature control of the production trials would be done manually information about the cooling and the control system were not relevant. This simplification also reduced the time required to complete the simulations. From the simulations performed, the following six models are most instructive of what can be achieved with reaction simulation.

Case 1: 15 h addition time (reference). After 6 h a sharp rise in heat production from 19 to 24 kW is observed. The vessel should be able to control this as there is enough cooling capacity (figure 12).

Case 2: 12 hours addition time. After 6 h a sharp rise in heat production from 24 to 32 kW occurs. The estimated maximum cooling capacity is required to control this (figure 13).
Case 3: 15 h addition time, change of flow rate ratio, 2:1.
After 5 h and feeding 2 moles/mole the feed rate is reduced. The next 2.4 moles/mole are fed over 10 h. The maximum cooling requirement is about 27 kW. This feed sequence is considered to be safer than case 1 since the highest heat production occurs when the process is addition controlled (figure 14).

Case 4: 12 h addition time, change of flow rate ratio, 2:1.
After 4 h and feeding 2 moles/mole the feed rate is reduced. The next 2.4 moles/mole are fed over 8 h. The maximum cooling requirement is 33 kW which exceeds the estimated capacity of the vessel. There is a 100% utilization of the cooling capacity for 4 hours, but after that only 80% is used. This process is not acceptable (figure 15).

Case 5: 15 h addition time, change of flow rate ratio, 2:1.
After 5 h and feeding 1.7 moles/mole the feed rate is reduced. The next 2.7 moles/mole are fed over 10 h. During nearly 10 hours the cooling requirement is about 24 kW (75% of the estimated maximum). Instead of a rise in heat production there is a sharp fall at the time the feed rate is reduced. About one hour later the initial value is obtained (figure 16).
Case 6: 12 h addition time, change of flow rate ratio, 2:1. After 4 h and feeding 1.7 moles/mole the feed rate is reduced. The next 2.7 moles/mole are fed over 8 h. During nearly 8 h the cooling requirement is about 29 kW (90% of the estimated maximum). The sharp fall in heat production is about the same as in case 5 (fig. 17).

In case 6 the feed time can safely be reduced by three hours (20%) with only a 4 kW higher cooling requirement compared to case 1. Instead of a heat production rise at an uncertain moment the cooling requirement is reduced at the same time the operator changes the feed rate.

The feed sequence of case 6 seemed to be the most satisfactory compromise between the different requirements. It gives the best heat production profile of all the simulations and can easily be transferred to other scales and other vessels with different cooling capacities.

The proposed feed sequence can further be optimized to achieve a shorter feed time by enhancing the feed rate at the time when the heat production falls. A temperature rise during this period also speeds up the chemical conversion and hence reduces the stirring time.

This feed sequence was applied in several production trials at 1100 moles. During those trials other parameters were also changed. For example the catalyst ratio was reduced stepwise and it was demonstrated that 50% of the initial amount yielded the same results. This was not obvious from the laboratory work that had been done, but it was predicted by the model. Modifications of the feed sequence and temperature profile were also evaluated. Changes to the temperature profile did not give acceptable results, probably due to side reactions which cause a reduction in yield and product quality. On the other hand the feed time was successfully reduced by another hour to 11 hours by adding the last portion (the excess) at a higher rate.

Development of the Production Process

Near to the end of this project the discussions with a possible toll manufacturer were finalized in an agreement to proceed with the introduction of the process. One of the elements in this decision was the success of the trial productions at 1.6 m³ scale at the AGFA plant which were witnessed by co-workers of the toll manufacturing company. Also most of the items of the toll manufacturers safety program could be completed. They were convinced that the process could be scaled-up and run in a safe way in their production facilities.

Next it was necessary to obtain data on the production vessel the toll manufacturer intended to use. This comprised a 4.5 m³ Pfaudler glass lined reactor with an oil recirculation system to which cooling is provided by a water heat exchanger and chilled oil by direct injection (figures 18 and 19).

These data were incorporated in the REACTION model. Some parameters on the cooling performance were not available. In first instance these were estimated using data from the literature and later obtained from a cooling experiment in the production vessel. The model was adapted for the technical grade of starting material by modifying the heats of reaction. This provided a model of the full production process which could be used for optimization and safety studies.

One of the requirements on the production process was that water of 10 to 15°C could be used to meet the cooling requirements. It was not acceptable for chilled fluid to be used for most of the addition time, since it is only available as a backup. To meet this requirement the heat production had to be as steady as possible. This could be achieved by modifying the feed sequence according to the results of the simulation experiments.
Accelerated Scale-up

A series of experiments were conducted after the introduction of the process at the toll manufacturers site. They needed to confirm the outcome of the simulation results and validate some assumptions made previously. The graphs below show the results of an experiment ran at production process conditions compared with the model used throughout this study (figures 22 and 23).

From the different simulations it became clear that the dynamic responses of the control/heat exchange system could be important. The steady state heat transfer capability of the reactor was estimated to be adequate, but when operated close to its maximum cooling capacity, the speed of response to the change in heat production could be poor. Both, the second exotherm during a linear addition and the fall in heat production at the time when the feed rate was reduced, would introduce a significant load disturbance to the control system. This is illustrated by the graphs above (figures 20 and 21). The temperature overshoot shown in the simulations would be acceptable in the process and would not influence the yield or the product quality. In neither of the simulations the chilled fluid was used to control the exotherm.

The first example shows the performance of the vessel with a linear feed of 20 hours (figure 20). The second one shows how the system would cope with the proposed feed sequence of 18 hours (figure 21).

Additional Validation of the Model

A series of experiments were conducted after the introduction of the process at the toll manufacturers site. They needed to confirm the outcome of the simulation results and validate some assumptions made previously. The graphs below show the results of an experiment ran at production process conditions compared with the model used throughout this study (figures 22 and 23).

Figure 20: The first example shows the performance of the vessel with a linear feed of 20 hours.

Figure 21: The second example shows the performance of the vessel with a linear feed of 18 hours.

Figure 22/23: Comparison of an experiment ran at production process conditions and the model used.
A «start-stop» experiment was also performed. This shows that the second part of the process is not dosing controlled (figure 24).

**Project Result**

Achievements of this study can be summarized as follows:

**Before:**
- 400 l scale • 8 hours of addition, no change in feed rate.
- 1000 l scale • 12 hours of addition, change in feed rate after 2 moles/mole at 4 h.

**After:**
- 1600 l scale • 15 hours of addition, change in feed rate after 1.7 moles/mole at 5 hours (initial trial).
- 12 hours of addition, change in feed rate after 1.7 moles/mole at 4 hours.
- 11 hours of addition, change in feed rate after 1.7 moles/mole at 4 hours. Catalyst ratio diminished in steps of 25% from 2 mole% to 1 mole%.
- 10 hours of addition, change in feed rate after 1.7 moles/mole at 4 hours. Catalyst ratio 1 mole%; higher feed rate near the end.
- 4000 l scale • 20 hours of addition, change in feed rate after 1.7 moles/mole at 7 hours (initial trial).
- 17 hours of addition, change in feed rate after 1.7 moles/mole at 6 hours.
- 20 hours of addition, change in feed rate after 1.7 moles/mole at 7 hours.
Use of 50% hydrogen peroxide.

**Further Developments**

If scale-up to 8 m³ were required, this could also be studied using the model with additional data for the vessel and its cooling system. For production at this scale, longer addition times should be evaluated experimentally to determine the influence of the side reactions.

Extending the model to include the crystallizations behavior could be useful. This would also require the known side reactions to be introduced in the model as these side products could influence the crystallization process.

The concentration profiles of the different chemicals throughout the addition of hydrogen peroxide could be validated quantitatively, possibly by using the FTIR technique. It would also be interesting to determine the hydrogen peroxide concentration throughout the batch.

The batch size could be increased by using 50% hydrogen peroxide. It is envisaged that this option will be pursued. This change will influence many parameters and these need to be studied carefully. A few experiments in the RC1 have already been performed and the model can predict the performance of these quite well.

An external loop with heat exchanger would enhance the heat removal and could allow a shorter feed time. The conversion of the semi-batch process to continuous operation might be considered if the product volume would justify this. The REACTION model and the RC1 could be used for this purpose.

**Cost-Benefit Analysis**

By coincidence we had the chance to evaluate the benefits quantitatively. There was a price difference of 100% between the toll manufacturer who applied the model knowledge and the one who did not. The savings made by this approach on the product quantity of our first years order are of the magnitude of the total price of an RC1, the Batch-CAD software and all the project costs.

**Conclusion**

The combination of reaction calorimetry and reaction simulation accelerated the scale-up of this process and made us meet the deadlines. This approach also resulted in an optimized, economic, safe and well understood process. A lot of trials and risks were avoided by using computer simulated experiments.
The cooling capacity of the production vessel can be used close to the limit thus minimizing the feed time.

Without Reaction Calorimetry it would have been nearly impossible to run this process in a controlled manner and very difficult to optimize the feed sequence.

The combination of the RC1 and the BatchCAD software suite (REACTION, RATE & RC1 SIMULATOR) are interesting tools for process development. In those projects where scaling up proves to be difficult this more fundamental approach should be used.

The model can be used to perform a HAZOP study and give quantitative answers to the questions.

Acknowledgments

I would like to express my gratitude and respect to Allen Wright of BatchCAD Ltd. for helping to develop the kinetic model and the initial RC1 simulations. Later on he incorporated the data of the vessel from the toll manufacturer and discussed the results with their chemists.

I also would like to thank Armand Van den Bergh who is our RC1 specialist. He was responsible for the experiments in the RC1 and the RC1 evaluation. His cooperation in this project is gratefully acknowledged. The following co-workers also contributed to this project: Jan Venneman, Roger Van Bael, Dirk Van Dromme and Kurt Verstrepen.

The cooperation with the people of the toll manufacturing company were especially useful for the development of the full scale model.

I would also like to mention Mettler-Toledo for two things: developing nice instruments for a development chemist and also for the cooperation with BatchCAD Ltd. for developing the RC1 SIMULATOR which is in our development strategy a key tool for reaction simulation.

References

- «The Development and Application of a CAD package for the Batch Processing Industry»

- «The development of an Interface between the RC1 and the BatchCAD Reaction Program»

- «Kinetic fitting using RC1 data»

- «CHETAH – The ASTM Chemical Thermodynamic and Energy Release Evaluation Program»
Within the magnificent surroundings of the famous mountains Jungfrau, Mönch and Eiger, more than 150 chemical engineers from Europe, the US and Asia met, during five days, for an exchange of their experiences with the Automated Laboratory Reactors and Reaction Calorimeters «RC1» and «Contalab» from METTLER TOLEDO.

This event organized and sponsored by METTLER TOLEDO was held at the beginning of October 1995 at Interlaken. This kind of conferences take place regularly in Switzerland or in the US and in Japan each year. The RC User Forum is an ideal platform for knowhow and information transfer directly from user to user. The international user community presented 27 papers and attended different workshops on subjects as «Evaluation of Calorimetric Data», «Measurements under Reflux Conditions» and «Simulation».

The METTLER TOLEDO instruments used for chemical process optimizations, scale-up studies and chemical safety tests proofed again to be an irreplaceable piece of equipment for an increasing number of laboratories in the chemical industry all over the world. This year the audience selected democratically Dr. Marc Bollyn of AGFA Gevaert in Belgium to win the «Mettler-Toledo RC Award 1995» for the most interesting contribution during the Forum. His talk about reaction calorimetry combined with reactor simulation explained how a highly exothermic oxidation reaction was successfully scaled up using combined techniques.

The next RC User Forums will take place this year (October 27–30, 1996) in the US and in Japan (October 29–November 1, 1996). Switzerland will follow 1997 (November 2–7) in Lugano.

In accordance with this meeting, the first «ReactIR User Forum Europe» from Applied Systems Inc. & METTLER TOLEDO took place subsequently with two half day programs. A rapidly increasing number of RC1 users is utilizing the FT-Infrared in-line analytical technique («ReactIR 1000») together with their automated laboratory reactor and reaction calorimeter. The combined technique provides a more profound knowledge about process under investigation.