PROCESS MODELLING AND CONTROL
OF
BATCH REACTORS IN THE FINE CHEMICALS INDUSTRY

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1.0 INTRODUCTION

Batch reactors are used extensively for the manufacture of small volume high value added products increasingly in production facilities intended for multipurpose use.

To achieve stable and reproducible operational conditions is increasingly of importance to achieve the required product purity, optimum yields and cycle times to satisfy the relevant regulatory authorities and commercial requirements.

Operators are also required to notify the appropriate Environment Agency of the peak and total emissions to the environment from their processes.

Batch processes are inherently transient in nature and the process engineer is being called upon to demonstrate dynamically the adequacy of the equipment design in this increasingly regulatory environment.

This paper reviews the basic techniques for process modelling and control of batch reaction systems under steady state and dynamic conditions.

The main aspects considered are:-

- Heat up and cool down performances.
- Sensitivity analysis for reactor parameters.
- Boil up rate predictions.
- Thermal stability under exothermic conditions.
- Control system design and loop tuning parameters.
- Condenser rating.
- Process emission predictions.

A further important consideration in the design and safe operation of batch reaction systems is the design of the emergency pressure relief and blowdown facilities which is beyond the scope of this paper. However, this topic is covered extensively elsewhere. \(^{(2)}\) \(^{(5)}\)

Several process modelling packages are available for the study of steady state conditions and the behaviour of batch processes. This paper is based on the Chemstations range of integrated packages which are linked to a considerable component physical property database complete with extensive thermodynamic options to allow a detailed study and understanding of all aspects of batch processing.
2.0 PROCESS CHARACTERISTICS

2.1 Reaction Chemistry

Endothermic reactions exhibit a marked degree of self regulation in regards to thermal stability and do not require further consideration.

Exothermic reactions require a detailed understanding of the reaction kinetics to provide reaction rate and heat of reaction which should be determined by screening tests using the appropriate calorimeter.\(^{(2)}\)

The heat removal requirement depends on the type of reaction. Vapour pressure systems with reaction at the reflux temperature are referred to as a tempered reaction system due to heat removal by latent heat reducing the heat removal requirement for the jacket/coil cooling system.

Gassy systems generate a permanent gas and require total heat evolved to be absorbed by the jacket/coil cooling system.

Hybrid systems are a combination of the previous two reaction types and can result in a significant emission of volatile organic compounds (VOC) from the overhead condensing system due to inerts.

The reaction rate \(^{(1)}\) depends on the reaction order where a first order reaction conversion varies with time and a second order reaction conversion varies with square of the reactant concentrations. First order reactions have the highest rate where the conversion is least i.e. at time zero.

The reaction rate can be limited by adding the reactant continuously at a controlled rate to ensure heat of reaction rate does not exceed the heat removal capability of the system.

Some reactions are carried out with all the reactants charged i.e. “all on board”. Their reactions are frequently carried out at total reflux which enhances the system heat removal capability.

Predicting the physical properties of the reaction mix during various steps in the process due to composition and temperature changes can have a significant effect on the reactor performance. ChemCAD includes an extensive component database with facilities for User added components which in conjunction with CCreacs provides a powerful modelling tool for predicting homogeneous reaction mixtures essential for thermal and emergency relief design.
2.2 Process Engineering Constraints

2.2.1 Thermal

An inherent characteristic of stirred batch reactors with coils or external jackets is the thermal lags associated with the heat capacities of the reactor, reaction mix, jacket contents and jacket service system and the dead time of the circulation system. Typically glass lined reactors vary in weight from 475 kg (100L) to 12200 kg (12500L) with equivalent jacket volumes of 46L and 1200L. The lags associated with the jacket services using thermal fluids are minimised by using forced circulation and plate heat exchangers for fast response. A reactor thermal rating spreadsheet is available as a design aid to establish overall plant utility requirements \(^{(6)}\) for typical systems (refer Figure 8).

The thermal conductivity of the reactor material of construction has a significant effect on wall temperatures, as demonstrated in Section 4, which can limit cycle times. Extreme temperature differences can result in product quality problems on certain processes.

Refer to Appendix I for a review of the heat transfer theory which is further covered in the literature.\(^{(7)}\) However, for poorly designed jacket services the jacket side film coefficient will become controlling if turbulent conditions are not established. The use of forced circulation and jacket inlet mixing nozzles on thermal fluid systems is essential.

The heat removal capability of the service system is a function of the resistances to heat transfer, the temperature difference between reaction temperature and jacket/coil side temperature and heat transfer area. A thermal runaway (increasing reaction temperature increases rate of reaction) will occur if the heat cannot be removed fast enough, e.g. reduction in heat transfer area due to reduction in reactor contents.

It may not always be possible to design for stability where enough heat transfer area is provided for the design temperature difference. However, stability will be assured if heat is removed by boiling one or more of the components since this tends to make the system isothermal.

The natural period of batch reactors can vary between 15 and 60 minutes (Refer Appendix III) and will increase on approach to instability making control more difficult.

There is extensive literature available on agitation systems with some manufacturer’s having proprietary arrangements. The Seider Tate correlation (Appendix I) is used to predict reactor inside film coefficients based on agitation system constants determined empirically.
2.2.2 Overheads

When processing flammable solvents it is standard practice to eliminate explosive mixtures by establishing and maintaining an inert atmosphere in the reactor. Inerting systems can be based on pressure balance or continuous flow. However, when carrying out reactions at reflux the inert flow should be reduced/eliminated to minimise volatile organic compounds (VOCs) emission to the environment. Some reactions involve the evolution of non condensable compounds which will become saturated with VOCs in proportion to the component vapour pressures at the condenser exit temperature and pressure.

The presence of non condensables in the condenser results in a significant increase in the thermal resistance which can result in up to half the heat transfer area being required for the last zone alone.

ChemCAD Therm uses a method based on Silver (8) and Bell & Khaly (9) to account for non condensables with wet film and dry film condensing options.

Adopting holistic methods for condenser sizing, e.g. condenser area equal to reactor heat transfer area, can result in significant undersizing when applied to multipurpose plant operations.
3.0 CONTROL CONFIGURATIONS

3.1 Direct Jacket Heat / Cool (Scheme 1)

These systems involve the connection of the appropriate supply and return services directly to the reactor jacket/coils. Systems are in use providing operating ranges from −20°C to 180°C with ethylene/glycol mixes, water and steam as services. Ethylene glycol mixes should not be used above their boiling point.

There are many arrangements in use from total manual control to full automatic control involving forced circulation with steam/water mixing facilities. Ciba Geigy and Wiegand developed a steam jet heater providing a wide range of heating duties with a single phase in the operating range 5°C to 180°C. This arrangement requires careful design in relation to air removal/expansion/pressurisation facilities and service returns from high operating temperatures require special automatic blowdown facilities.

These systems provide excellent thermal response due to the elimination of heat transfer lags associated with heat exchangers. However, to provide combined heating/cooling facilities, with the exception of the Wiegand system, requires involved automatic valve sequencing and jacket/coil blowdown routines when changing services resulting in discontinuous control which can cause serious operational problems when handling exothermic reactions.

Operational problems associated with these systems include cross contamination of services, corrosion and the need for complex control routines when changing services from heat to cool involving time lags. These systems are not always cheaper than other alternatives.

It is now considered standard practice to use single fluid systems based on heat transfer fluids with external heat exchangers.
3.2 Direct Jacket Cool/Indirect Heat (Scheme 2)

This system is based on the use of a single appropriate heat transfer fluid for the desired operating range, typically –20°C to 180°C.

The coolant is directly injected into the reactor jacket/coil circulating loop and a heat exchanger with steam is provided for heating. Changeover between heating and cooling modes is seamless using control valves in split range.

Thermal response on cool is excellent due to direct injection and the use of a three way valve on the heat exchanger minimises thermal lags on heating.

Adopting a three way valve allows the steam to be applied continuously to the heat exchanger shell at full pressure which eliminates problems associated with condensate lift/return, prevents freezing when operating below 0°C and provides excellent linear control characteristics.

These systems require careful consideration to ensure thermal expansion is allowed throughout the loop.

Equipment in common use include sealless pumps and gasket free fully welded plate and shell heat exchangers.

On initial commissioning these systems have to be thoroughly dried out to prevent operational problems and equipment damage. Water breakthrough due to contamination or equipment failure will result in considerable downtime.

The key design parameters to be determined are circulation rate and heat exchanger sizing. The circulation rate is set depending on the number of mixing nozzles to be supplied and achieving the recommended nozzle pressure drop. The heat transfer fluid is on the plate side of the shell and plate heat exchanger, which provides a high film coefficient.

The heat transfer area is selected based on a reasonable LMTD at approach to service supply temperature and is sized to ensure that reactor heat transfer is limiting.

ChemCAD modelling of the system establishes the thermal response of the system selected.

3.3 Indirect Jacket Heat/Cool (Scheme 3)

The key features of this system are as for the previous Scheme 2. However, the use of an additional heat exchanger for cooling allows the selection of a less expensive fluid for the cooling service which may provide cost benefits with a centralised refrigeration facility involving the use of significant volumes of heat transfer fluid.

This system also allows for segregation of the reactor service system from other reactors which enables rapid identification of water breakthrough problems on a facility with several reactors.
3.4 Instrumentation and Control

Control systems for Schemes 2 and 3 only are considered.

The conventional control system uses cascade control with reactor contents temperature (master/primary) being cascaded to jacket/coil inlet/outlet temperature (slave/secondary). This system provides the following benefits:

1. Disturbances in the jacket/coil loop are corrected by the secondary controller before they can influence the primary controller. However, the secondary loop thermal time constant must be less than the primary loop thermal time constant, which is in fact the case (refer Appendix III).

2. Primary loop speed of response is enhanced due to reduction in phase lag.

3. Secondary loop gain variations are overcome within its own loop.

The secondary controller is ideally a Proportional only controller as Integral Action Time will slow the response. A proportional band of \( \leq 25\% \) is typical. However, when carrying out boil up control it is desired to set a temperature difference between jacket/coil and reactor contents by controlling jacket/coil inlet temperature. In this mode Integral Action is required to eliminate offset.

The primary controller is Proportional + Integral + Derivative and presents special problems for initial loop tuning and operation. Refer to Appendix III for a detailed discussion.

Temperature measurement is invariably by resistance sensor in conjunction with Smart transmitters which allows considerable flexibility when setting ranges. There is hardly any thermal lag associated with the sensor, however, there can be significant thermal lags associated with the thermowell if incorrectly designed or installed which can lead to an uncontrollable system. Designs are available which ensure fast response and should be adopted.

Control valve actuators are pneumatic with positioners fitted. The calibration for split range operation of the valves should be achieved at the positioners, not with scaled multiple controller outputs, to ensure loop integrity is maintained. Control valve characteristic used is normally equal % though situations may arise where a linear characteristic provides better control. Refer to Appendix II for further discussions.

ChemCAD provides the facilities to carry out extensive modelling of the control loop responses for all operational modes and establishes the system stability characteristics.
4.0 Performance Prediction

Reactor performance predictions are shown in the Attachments. The model used is essentially Scheme 3 (Figures 2, 3) with external heat exchangers.

The reactor studied was a 1600L Hastelloy C Reactor with a single jacket and reactor contents were based on 1000 kg toluene.

The heat transfer fluid selected was Dowtherm J with flowrates as indicated in Figure 3.

Figure 9 indicates the Plot Options available with ChemCAD Reacs.

The external heat exchangers UA are as indicated in Figures 2 and 3 and are based on Vahterus plate and shell type units.

The Graphs 1 – 5 demonstrate that a wide range of performance characteristics can be studied including:-

- Heat up and cool down rates (Graphs 1, 3).
- Heat transfer rates (Graphs 1, 3).
- Heat transfer coefficients (Graphs 1, 3).
- Boil up rates (Graph 2).
- Condenser emission rates (Graph 2).
- Reactor stability under exothermic conditions (Graph 3).
- Control system tuning (Graphs 4, 5).

Graphs 4 and 5 demonstrate the use of the Ziegler Nichols tuning method for the Condenser Model (Figures 4 and 5) as discussed in Appendix III.

CCReacs allows detailed Sensitivity analyses to be performed.

- Optimum heat exchanger sizing.
- Jacket temperature and heat up/cool down rates.
- Jacket temperature and boil up rates.
- VOC emission and inert flow rate.
- Condenser performance criteria.
- Heat removal capability and exotherm stability.
- Optimisation of control system parameters.

The possibilities available are extensive and depend very much on the initiative of the user and the particular problem being studied.
Appendix I

Acknowledgements to Pfaudler Reactor Systems for permission to reproduce this information

Summary of Heat Transfer in Glass Lined Reactors

Inside film coefficient $h_i$

For a three bladed impellor agitator the inside film coefficient $h_i$ is predicted by

$$\text{Nu} = 0.33 \ast (\text{Re}^{0.67} \text{Pr}^{0.33}) \left( \frac{\mu_{bulk}}{\mu_{wall}} \right)^{0.14}$$

Where

- $\text{Nu} = \text{Nusselt Number} = \frac{h_i D}{k}$
- Inside film coefficient $h_i$ Btu/ft$^2$°F
- Inside reactor diameter $D$ ft
- Thermal conductivity of process fluid $k$ Btu/ft$^2$°F/ft

$$\text{Re} = \text{Reynolds Number} = \frac{\rho n d^2}{\mu}$$

- Impellor rotation rate (normal 90 rpm) $n$ rpm
- Impellor diameter $d$ ft
- Process fluid density $\rho$ consistent units
- Process fluid viscosity $\mu$ consistent units

$$\text{Pr} = \text{Prandtl Number} = \frac{C_p \mu}{k}$$

- Process fluid specific heat $C_p$ consistent units

Normally the viscosity ratio term $\frac{\mu_{bulk}}{\mu_{wall}}$ is unity and can therefore be ignored.

Glass wall

For heat transfer through glass linings use

$$\frac{k_g}{L_g}$$

Where

- $k_g =$ thermal conductivity of glass = 6.9 Btu/ft$^2$°F/in
- $L_g =$ glass thickness (in)

Glass thickness can vary between 1 mm and 2 mm. Use 1.5mm(0.06in) thick as a good average.
Metal wall

Physical properties of common reactor materials are as follows

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (kg/m$^3$)</th>
<th>Specific Heat (kJ/kgK)</th>
<th>Thermal Conductivity (w/mK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hastelloy C</td>
<td>8690</td>
<td>0.423</td>
<td>11.1</td>
</tr>
<tr>
<td>Stainless steel</td>
<td>8000</td>
<td>0.5</td>
<td>16.2</td>
</tr>
<tr>
<td>Carbon steel</td>
<td>8000</td>
<td>0.4</td>
<td>52(360 Btu/ft$^2$°F/in)</td>
</tr>
</tbody>
</table>

$L_m$ = metal wall thickness (in)

Overall conductivity ($k_o$) for a glass lined vessel is given by (CCReacs allows only one value)

\[
k_o = \frac{L_m + L_g}{L_g / k_g + L_m / k_m}
\]

Outside film coefficient $h_o$

For steam on the jacket side take an outside film coefficient of 8517 w/m$^2$K (1500 Btu/ft$^2$°F)

For water or other fluid on the jacket side, use the Pfaudler equations or consult the circulating nozzle leaflets.

Fouling coefficients

- Fouling on inner vessel: allow 5678 w/m$^2$K (1000 Btu/ft$^2$°F) $f_i$
- Fouling on jacket side: allow 5678 w/m$^2$K (1000 Btu/ft$^2$°F) $f_o$

or substitute known values.

Overall heat transfer coefficient $U$

\[
\frac{1}{U} = \frac{1}{h_i} + \frac{1}{f_i} + \frac{1}{k_g / L_g} + \frac{1}{k_m / L_m} + \frac{1}{h_o} + \frac{1}{f_f}
\]

Rate of Heat Flow

\[
Q = UA\Delta T
\]

Where

- $U$ = overall heat transfer coefficient Btu/ft$^2$°F
- $A$ = heat transfer area ft$^2$
- $\Delta T$ = log mean temperature difference °F
Heat transfer process side

Seider Tate correlation

\[ Nu = C Re^{0.667} Pr^p \left( \frac{\eta_p}{\eta_w} \right)^q \]

\[ Nu = \frac{h_i D}{k} \]
\[ Re = \frac{nd^2}{i} \]
\[ Pr = \frac{Cp i}{k} \]

\( C, p, q \) = constants for Agitator
\( Nu \) = Nusselt number
\( Re \) = Reynolds number
\( Pr \) = Prandtl number
\( D \) = Vessel internal diameter
\( d \) = Agitator diameter
\( n \) = RPM of agitator
\( \eta_p \) = process side dynamic viscosity
\( \eta_w \) = process side wall viscosity
\( k \) = process side thermal conductivity
\( h_i \) = heat transfer coefficient, product side
\( Cp \) = process fluid specific heat

Agitator Constants

<table>
<thead>
<tr>
<th>AGITATOR TYPE</th>
<th>C</th>
<th>p</th>
<th>q</th>
<th>RANGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>ONE PIECE</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>IMPELLER</td>
<td>0.33</td>
<td>1/3</td>
<td>0.14</td>
<td>( 2 \times 10^4 &lt; Re &lt; 2 \times 10^6 )</td>
</tr>
<tr>
<td>ANCHOR</td>
<td>0.55</td>
<td>1/4</td>
<td>0.14</td>
<td>( 5 \times 10^3 &lt; Re &lt; 4 \times 10^4 )</td>
</tr>
<tr>
<td>TURBINE (6 BLADES)</td>
<td>0.44</td>
<td>1/3</td>
<td>0.24</td>
<td>( 200 &lt; Re &lt; 1 \times 10^3 )</td>
</tr>
<tr>
<td>LOOP</td>
<td>0.36</td>
<td>1/3</td>
<td>0.14</td>
<td>( 300 &lt; Re &lt; 4 \times 10^4 )</td>
</tr>
<tr>
<td>CYRO LOCK</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CBT – TURBINE</td>
<td>0.48</td>
<td>1/3</td>
<td>0.14</td>
<td>( 2 \times 10^4 &lt; Re &lt; 2 \times 10^6 )</td>
</tr>
<tr>
<td>FBT – TURBINE</td>
<td>0.69</td>
<td>1/3</td>
<td>0.14</td>
<td>( 400 &lt; Re &lt; 3 \times 10^5 )</td>
</tr>
<tr>
<td>PBT – TURBINE</td>
<td>0.62</td>
<td>1/3</td>
<td>0.14</td>
<td>( 300 &lt; Re &lt; 3 \times 10^5 )</td>
</tr>
<tr>
<td>TURBOFOIL</td>
<td>0.55</td>
<td>1/3</td>
<td>0.14</td>
<td>( 1 \times 10^3 &lt; Re &lt; 1 \times 10^6 )</td>
</tr>
</tbody>
</table>
Typical overall heat transfer coefficients $U$ for steam heating

<table>
<thead>
<tr>
<th>Product</th>
<th>XL 3410(L)</th>
<th>RA60 1360(L)</th>
<th>RA60 2270(L)</th>
<th>RA84 9090(L)</th>
<th>RA96 13640(L)</th>
<th>$hi$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>70</td>
<td>68</td>
<td>68</td>
<td>68</td>
<td>67</td>
<td>659</td>
</tr>
<tr>
<td>Toluene</td>
<td>59</td>
<td>55</td>
<td>55</td>
<td>55</td>
<td>56</td>
<td>232</td>
</tr>
<tr>
<td>Chloroform</td>
<td>60</td>
<td>57</td>
<td>57</td>
<td>57</td>
<td>57</td>
<td>259</td>
</tr>
<tr>
<td>Ethyl alcohol</td>
<td>57</td>
<td>54</td>
<td>54</td>
<td>53</td>
<td>54</td>
<td>213</td>
</tr>
<tr>
<td>Methyl alcohol</td>
<td>62</td>
<td>58</td>
<td>58</td>
<td>59</td>
<td>59</td>
<td>302</td>
</tr>
<tr>
<td>Acetone</td>
<td>62</td>
<td>59</td>
<td>59</td>
<td>58</td>
<td>58</td>
<td>284</td>
</tr>
</tbody>
</table>

Overall heat transfer coefficients units Btu/h ft$^2.0$F

Assumptions:
- glass thickness: 1.5 mm
- fouling steam side: 3333 Btu/h ft$^2.0$F
- fouling process water: 1000 Btu/h ft$^2.0$F
- fouling organic solvents: 1000 Btu/h ft$^2.0$F
- impeller speed: 90 rpm

\[ h_i = \text{process side inside film coefficient (last column above)} \]
\[ h_o = \text{jacket side film coefficient} \]

\[ \frac{1}{U} = \frac{1}{h_i} + \frac{1}{1000} \frac{1}{k_g/L_g} + \frac{1}{k_m/L_m} + \frac{1}{h_o} + \frac{1}{3333} \]

\[ F_f = F_i \]

\[ \frac{1}{k_o/L_o} = \frac{1}{k_g/L_g} + \frac{1}{k_m/L_m} = \frac{1}{117} + \frac{1}{600} \quad \text{(symbols as above)} \]

Overall heat transfer for thermal fluid on the jacket side

Let $U_S = \text{overall heat transfer coefficient for steam}$ $h_o = 1500$ for steam

$U_O = \text{overall heat transfer coefficient for oil}$ $h_o = 250$ for oil

\[ \frac{1}{U_o} = \frac{1}{U_s} = \frac{1}{1500} + \frac{1}{250} \]
Appendix II

Control Valve Characteristic Selection\(^{(3)}\)

The selection of a control valve with the appropriate operating characteristics is essential for satisfactory control loop performance. A valve has two characteristics namely the inherent characteristic (relationship between flow and stroke at constant \(\Delta P\)) and the operational characteristic where the inherent characteristic is modified by the process pressure conditions.

An equal % valve operating characteristic tends towards a linear characteristic as \(\Delta P_{\text{max}}/\Delta P_{\text{min}}\) increases. A linear valve operating characteristic tends towards a quick opening characteristic as \(\Delta P_{\text{max}}/\Delta P_{\text{min}}\) increases.

The operational characteristic of a valve can also be modified by controller output signal characterisation using various techniques.

The graph of valve stem position vs flow demonstrates the range of valve characteristics available (refer Figure 6).

The following is a summary of selection criteria in good industrial practice:

<table>
<thead>
<tr>
<th>Process Variable</th>
<th>(\Delta P_{\text{max}}/\Delta P_{\text{min}})</th>
<th>Characteristic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>&lt; 2</td>
<td>Equal %</td>
</tr>
<tr>
<td>Pressure</td>
<td>Constant &lt; 2</td>
<td>Linear</td>
</tr>
<tr>
<td>Level</td>
<td>&lt; 2½</td>
<td>Linear</td>
</tr>
<tr>
<td></td>
<td>&gt; 2½</td>
<td>Equal %</td>
</tr>
<tr>
<td>Flow (linearised)</td>
<td>&lt; 2½(^{(\text{Note 1})})</td>
<td>Linear</td>
</tr>
<tr>
<td></td>
<td>&gt; 2½(^{(\text{Note 2})})</td>
<td>Equal %</td>
</tr>
</tbody>
</table>

Note 1 Value \(\Delta P\) at flow maximum > 40% system \(\Delta P\)

Note 2 Value \(\Delta P\) at flow maximum < 40% system \(\Delta P\)

\(\Delta P_{\text{max}}\) will be close to maximum shutoff \(\Delta P\) and \(\Delta P_{\text{min}}\) at full flow should not be underestimated which favours equal % characteristic.

ChemCAD allows dynamic analysis of the process enabling appropriate characteristic selection.
Appendix III

Control Loop Tuning

When tuning control loops it is important to understand the impact the tuning parameters have on the process.

The minimum effective values are:

- **Proportional Band** (PB%) highest value (minimum gain)
- **Integral Action Time** \( (T_i\) min) longest time
- **Derivative Action Time** \( (T_d\) min) shortest time (or off)

The following ground rules should always be considered:

1. **Proportional Band**
   - Wide PB (50 – 500%) on fast and noisy processes such as Flow, Liquid Pressure and Composition.
   - Narrow PB (1- 50%) on slow processes such as Gas Pressure, Temperature and Vapour Pressure.

2. **Integral Action Time** \( (T_i)\)
   - Integral action time changes the controller output at a rate proportional to the magnitude of the error \( (e)\) and should never be set so short such that the resulting load change imposed exceeds the load response characteristics of the process. (Consider approaching a corner too fast).
   - Short \( T_i \) (0.05 – 2 min) on fast and noisy processes such as Flow and Liquid Pressure.
   - Long \( T_i \) (2 – 120 min) on slow processes such as Temperature, Vapour Pressure and Composition.
   - \( T_i \) is unnecessary on Gas Pressure and is seldom used on Liquid Level as steady flow is usually the required condition.

3. **Derivative Action Time** \( (T_d)\)
   - Derivation Action Time changes the controller output at a rate proportional to the rate of change of error \( \frac{de}{dt}\) and as such should never be used on noisy processes such as Flow, Liquid Pressure and Liquid Level.
   - \( T_d \) is used on Temperature, Vapour Pressure and Composition and should be set conservatively (short \( T_d \)) to protect against control instability resulting from unanticipated load changes. \( T_d \) set in the range \( \frac{T_i}{4} < T_d < \frac{T_i}{2}\) is considered reasonable.
4. **Integral Action Time Saturation (Windup)**

When a sustained error is maintained between set point and measured variable on a controller with $T_i$ the output is driven off scale (valve full open or closed).

Control action does not commence until the measured variable crosses the set point resulting in overshoot.

This condition is typical of heating/cooling a batch reactor to a desired temperature and composition endpoint control.

Controllers incorporating Proportional and Derivative modes overcome this problem.

5. **Controller Loop Tuning Method**

Ziegler and Nichols (4) developed the Ultimate Sensitivity Method which provides a useful basis for control loop tuning as follows:-

1. Set the control modes to their minimum effective values.
   
   Maximum $PB$, maximum $T_i$, minimum $T_d$ (or off).

2. Reduce $PB$ whilst making small set point changes (both directions) until the measured variable begins to oscillate at constant amplitude.
   
   The proportional band $PB_u$ and the period $T_u$ are noted.

3. Initial controller settings proposed are:-

   - **P only controller**
     
     $PB = 2 \times PB_u \%$

   - **P + I controller**
     
     $PB = 2.2 \times PB_u \%$
     
     $T_i = \frac{T_u}{1.2}$ mins

   - **P + I + D controller**
     
     $PB = 1.6 \times PB_u \%$
     
     $T_i = \frac{T_u}{2}$ mins
     
     $T_d = \frac{T_u}{8} = \frac{T_i}{4}$

This procedure can be used as a ‘starting point’ for tuning control systems on batch processes, $T_u$ being established at a stable condition. Typical periods ($T_u$) for batch reactors vary in the range 15 to 60 minutes.

Fine tuning involves a trade off between the conflicting requirements of heat up/cool down to a set point, control of exothermic reactions at a set point, controlling on jacket temperature to achieve required boil up rates and constraints associated with wall temperature requirements.
6. **Reactor Time Constant Estimation**

\[ Q = h_1 A \left( T - T_1 \right) + W_1 C_1 \frac{dT}{dt} \]

**Batch Reactor Model Parameters**

- \( Q \) = rate of heat evolution
- \( h_1 \) = inside film coefficient
- \( A \) = heat transfer area
- \( T \) = reactor temperature
- \( T_1 \) = wall temperature
- \( W_1 \) = weight of reactants
- \( C_1 \) = specific heat of reactants

Rearranging in the standard form we have

\[ T + \frac{W_1 C_1}{h_1 A} \frac{dT}{dt} = T_1 + \frac{Q}{h_1 A} \]

The thermal time constant for reactor contents to reactor wall is given by

\[ \Gamma_1 = \frac{W_1 C_1}{h_1 A} \left( T - T_1 \right) = 15.4 \text{ min} \]

\[ \frac{1000 \times 0.478}{387.2 \times 4.82} = 0.25 \text{ h} \]

(For 12500L reactor with 25.2m² area the thermal time constant becomes 37 min full toluene charge).

The thermal time constant for reactor outside wall to reactor inside wall is given by

\[ \Gamma_2 = \frac{W_2 C_2 L}{k A} = \frac{W_2 C_2}{Q} \left( T_1 - T_2 \right) = 3.1 \text{ min} \]

\[ \frac{2500 \times 0.101 \times 0.00953}{9.55 \times 4.82} = 0.052 \text{ h} \]

- \( W_2 \) = weight of wall
- \( C_2 \) = specific heat of wall
- \( k \) = thermal conductivity of wall
- \( L \) = wall thickness
- \( T_2 \) = outside wall temperature

The thermal time constant for reactor outside wall to jacket contents is given by

\[ \Gamma_3 = \frac{W_3 C_3}{h_3 A} = \frac{W_3 C_3}{Q} \left( T_2 - T_c \right) = 2.6 \text{ min} \]

\[ \frac{453 \times 0.55}{1205 \times 4.82} = 0.043 \text{ h} \]

- \( W_3 \) = weight of jacket contents
- \( C_3 \) = specific heat of jacket contents
- \( h_3 \) = outside film coefficient
- \( T_c \) = average coolant temperature
The reactor contents time constant based on the overall heat transfer coefficient $U$ as defined in Appendix I is given by:

$$\Gamma_o = \frac{W_1 C_1}{U A}$$

21.7 min

- $W_1$ = weight of reactants = 1000 kg toluene
- $C_1$ = specific heat of reactants = 0.478 kcal/kg°C
- $A$ = area = 4.82 m$^2$
- $U$ = overall heat transfer coefficient = 274 kcal/h m$^2$°C

By studying the heat up and cool down curves from CCReacs dynamic simulations the thermal response can be characterised where we have:

<table>
<thead>
<tr>
<th>Time Constant</th>
<th>Temperature % $\Delta T$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>63.2</td>
</tr>
<tr>
<td>2</td>
<td>87.0</td>
</tr>
<tr>
<td>3</td>
<td>95.0</td>
</tr>
<tr>
<td>4</td>
<td>98.0</td>
</tr>
</tbody>
</table>

Graph 1 indicates a system $T_o$ of 24 min.
Appendix IV

ChemCAD User Guidance Notes

The following provides a summary of key points to achieve rapid results from the dynamic models.

PID Controller

The controller algorithm is representative of a model which does not exhibit interaction between control modes:-

\[
\text{Controller output } P_{\text{out}} = \frac{100}{\text{PB}} \left( e + \frac{1}{T_i} \int e dt + T_d \frac{de}{dt} \right) + P_o
\]

Where,
- \( PB \) = proportional band %
- \( T_i \) = integral action time, min
- \( T_d \) = derivative action time, min
- \( P_o \) = steady state controller output
- \( e \) = error between measured variable and set point

Note when \( e = 0 \), \( P_{\text{out}} = P_o \)

The measurement calibrated range in engineering units is converted to a 4 to 20 mA signal \( C_{\text{in}} \) as a linear or quadratic function. In modern control systems most inputs are now linearised.

Controller output direction in response to an error (\( \pm \)) is determined by the controller action. (Refer to Figure 7).

Direct action (increasing input / increasing output or decreasing input / decreasing output)

\[ e = C_{\text{in}} - C_{\text{set}} \quad \text{(level with fail closed valve)} \]

Reverse action (increasing input / decreasing output or decreasing input / increasing output)

\[ e = C_{\text{set}} - C_{\text{in}} \quad \text{(heating with fail closed valve / cooling with fail open valve)} \]

In completing the dialog box consider the following:-

The controller output can be directed to one valve or two valves for split range operation or another controller set point for cascade operation, assign UnitOp identifiers as appropriate.

The measurement can be derived from a stream or equipment variable. For reaction temperature locate controller in jacket inlet stream and assign Calculated Temperature.

Consider Appendix III when selecting and setting control parameters.

For batch processes set the steady state controller output (\( P_o \)) at the simulation start value i.e. full open or full closed control valves.
Control Valve (CVAL)

It is beyond the scope of this note to cover all aspects of this module, refer to ChemCAD CCReacs for a detailed description. However, the following should be taken into consideration:-

If a value for valve flow coefficient $C_v$ is not known use the Equipment Sizing $C_v$ option which will provide a value at stated stream conditions.

Control valve rangeability based on the ratio of maximum to minimum controllable flows depends on the entire control loop. For simulation use 50 for equal % characteristic and 10 for linear characteristic.

For control valve characteristic selection refer to Appendix II.

The control valve action in relation to the controller output signal $P_{out}$ is set as follows:-

Valve open  $0 = A_v \cdot P_{out}^0 + B_v$

Valve closed $1 = A_v \cdot P_{out}^1 + B_v$

Where $P_{out}$ is the equivalent output signal in mA Refer to Figure 7 for typical settings.

The position of CVAL in the model is critical and the rules can be summarized:-

1. It requires inlet ($P_i$) and outlet ($P_o$) pressures to be specified where $P_i$ is taken from the inlet stream. $P_o$ can be specified directly or overtaken indirectly by an assigned UnitOp in the model.

2. A mass balance must be maintained around CVAL. To achieve this, locate immediately after a Feed Arrow (direct flow adjust) or after Stream Reference (SREF) or Divider (DIVI) (indirect flow regulation). Dynamic Vessel, Dynamic Reactor and Dynamic Column can also be assigned for indirect flow regulation. The optimal mass flowrate transfer is used to set up Indirect Flowrate Regulation.

Heat Exchanger (HTXR)

HTXR is a steady state UnitOp. To use in Dynamic simulation specify Overall Heat Transfer Coefficient (U) and Heat Transfer Area (A) only. Tubeside and shellside pressure drops can be added.

When rating an existing unit use CCTherm to determine the UA for the duty being considered. This enables confirmation of manufacturer’s design data. CCTherm option will not run in Dynamic simulations.
**Batch Reactor (BREA)**

The following should be considered when completing the dialog boxes:

**Initial Charge**

Temperature and pressure at initial state.
Temperature must be below component boiling point or reactor will be considered empty.

Enter one (1) reaction if proposing to enter User heat of reaction.
Enter Mixed phase, liquid phase reaction if considering boil up.
Thermal Mode 5 Specify control systems is the only mode allowing consideration of jackets/coils.
Enter operating pressure for boil up studies.
Complete Feed and Product streams as appropriate.
To enter User heat of reaction create a “dummy” reaction by using the same component for one mole (-1) of reactant to create one mole (1) of product. Enter a frequency factor of 0.01.

**Reactor Specification**

Enter wall temperature at initial state.
Enter heat transfer data as appropriate with model calculating process side heat transfer coefficient using Seider Tate correlation (refer Appendix 1).

**Jacket/Coil Specification**

Unbaffled jacket calculated outside film coefficient ($h_o$) does not assume turbulence. If mixing nozzles fitted enter appropriate $h_o$ from Appendix I.
Set initial conditions in jacket for pressure and temperature.
If jacket is full set initial liquid fraction at one (1).
If this liquid fraction is not set the model assumes the jacket is empty.

**Dynamic Menu**

Access Dynamic Menu from Run.
Set run time and step size based on batch time and number of integration steps set in BREA general.
Set the Streams and UnitOps to be studied, failure to do this will result in no data being retained.

**Plot**

Dynamic run curves for variables to be studied are obtained via Plot and Dynamic History options.
REACTOR CONTROL SYSTEMS

Figure 1

DIRECT JACKET

BATCH REACTOR

SCHEME 1

DIRECT JACKET COOL

BATCH REACTOR

SCHEME 2

INDIRECT JACKET

HEAT/COOL

BATCH REACTOR

SCHEME 3

Heating

Coolant

Cooling
INDIRECT JACKET HEATING SYSTEM SCHEME 3

Figure 2

BATCH REACTOR WITH EXTERNAL STEAM HEAT EXCHANGER

STEAM

OPEN @ 20mA
CLOSE @ 12mA

NITROGEN

U=1210 W/m²K
A=6.73 m²

SLAVE

MASTER

TC

HASTELLOY C
REACTOR 1.6m³

COOLANT

U=298.7 W/m²K
A=10.15m²

VENT EMISION

DOWTHERM J
RECIRCULATION 16m³/h
INDIRECT JACKET COOLING SYSTEM SCHEME 3

BATCH REACTOR WITH EXTERNAL COOLING HEAT EXCHANGER

HASTELLOY C REACTOR 1.6m3
CONDENSER CONTROL SYSTEM

Inlet temperature control prevents freezing

Figure 4
CONDENSER CONTROL SYSTEM MODEL

DIRECT COOLING CONDENSOR SERVICE INLET TEMPERATURE CONTROL

- Reactor Overheads
- Condenser (U=776 W/m²K, A=9.86 m²)
- Single Loop Control
- Dowtherm J Recirculation (20,000 kg/h)
- Coolant Supply
- Coolant Return

Open @ 4mA, Closed @ 20mA
CONTROL VALVE CHARACTERISTICS

Figure 6
CONTROLLER & CONTROL VALVE ACTION

CONTROL ACTION REVERSE (Heating) (output 4 to 20 ma dc)
Increasing Measurement    Decreasing Output    (Error = Xset– X)

CONTROL ACTION DIRECT (Cooling)
Increasing Measurement    Increasing Output    (Error = X–Xset)

SINGLE VALVE ACTIONS   output position state  logic
FAIL CLOSED       4 (0%)    closed     0      0 = Av* 4 + Bv
Av=0.0625  Bv= -0.25  20(100%)  open     1      1 = Av*20 + Bv
FAIL OPEN        4 (0%)    open      1      1 = Av* 4 + Bv
Av= -0.0625 Bv= 1.25  20(100%)  closed   0      0 = Av*20 + Bv

SPLIT RANGE VALVE ACTIONS
COOLING         4 (0%)    open     1      1 = Av* 4 + Bv
Av= -0.125 Bv = 1.5  12 (50%)  closed   0      0 = Av*12+Bv
HEATING         12 (50%)  closed   0      0 = Av*12+Bv
Av = 0.125 Bv = -1.5  20(100%)  open     1      1 = Av*20 + Bv
# Reactor Thermal Rating Spreadsheet

## Reactor Jacket Fluid

<table>
<thead>
<tr>
<th>Species</th>
<th>( V_{\text{CF}} )</th>
<th>( T_{\text{MIN}} )</th>
<th>( T_{\text{MAX}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>0.422 m³</td>
<td>-20 °C</td>
<td>60 °C</td>
</tr>
</tbody>
</table>

## Reactor Operation

### Mode

<table>
<thead>
<tr>
<th>Mode</th>
<th>( T_{\text{IN}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heatup</td>
<td>0.00 °C</td>
</tr>
</tbody>
</table>

### Reactor Operation

<table>
<thead>
<tr>
<th>Mode</th>
<th>( T_{\text{IN}} )</th>
<th>Buffer Heat Balance Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heatup</td>
<td>0.00 °C</td>
<td>Adjust Unit Above Cell Cond.</td>
</tr>
</tbody>
</table>

## Reactor Contacts

<table>
<thead>
<tr>
<th>Species</th>
<th>( N_{\text{CF}} )</th>
<th>( Z_{\text{CF}} )</th>
<th>Resistance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>50.98</td>
<td>55.59</td>
<td>132.49</td>
</tr>
</tbody>
</table>

## Reactor Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Stainless Steel</td>
</tr>
</tbody>
</table>

## Reactor Vessel

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vessel Volume</td>
<td>1.361 m³</td>
</tr>
<tr>
<td>Heat Transfer Area</td>
<td>4.6 m²</td>
</tr>
<tr>
<td>Reactor Weight</td>
<td>200 kg</td>
</tr>
</tbody>
</table>

## Reactor Thermal Data

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Heat</td>
<td>0.416 kJ/kgK</td>
</tr>
<tr>
<td>Contact Temperature</td>
<td>-20 °C</td>
</tr>
<tr>
<td>Overall Heat Transfer Coefficient</td>
<td>0.052 m²/°C</td>
</tr>
</tbody>
</table>

## Reactor Thermal Rating

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat of Reaction</td>
<td>0.00 kJ</td>
</tr>
<tr>
<td>Max Time</td>
<td>0.00 h</td>
</tr>
<tr>
<td>Cool Down Time</td>
<td>0.00 h</td>
</tr>
<tr>
<td>Exit Therm</td>
<td>0.00 kJ</td>
</tr>
</tbody>
</table>

## Figure 8
BATCH REACTOR PLOT OPTIONS

Figure 9
1600L HASTELLOY REACTOR HEAT UP

Graph 1

Graph showing
- Contents
- Wall
- Jacket

Graph showing
- Heat Transfer Coefficient

Graph showing
- Heat (MJ/h)

Graph showing
- Contents hi
- Jacket ho
- Overall U
1600L REACTOR BOIL UP/EMISSION

Graph 2

- **Top Graph:**
  - **X-axis:** Time (h)
  - **Y-axis:** Mass Flow (kg/h)
  - Lines: Toluene and Total

- **Bottom Graph:**
  - **X-axis:** Time (h)
  - **Y-axis:** Boil Up Rate (kg/h)
  - Line: Total Flow
1600L HASTELLOY REACTOR COOLDOWN

Graph 3

- **Contents**
  - Wall
  - Jacket

- **Heat Transfer Coefficient (W/m²K)**
  - Contents hi
  - Jacket ho
  - Overall U

- **Heat Transfer Mcal/h**
  - Q to Reactor
  - Q to Jacket

- **Exotherm**
  - Contents
  - Wall
  - Jacket
CONDENSER MODEL CONTROLLER TUNING Tu

Graph 4

Condenser Model Period Tu Determination

PBu 40% Tu 7.3 m
CONDENSER MODEL CONTROLLER TUNING

Condenser Model Ziegler Nichols Tuning Method

PB 64% IAT 3.65m DAT 0.91m Based on Tu of 7.3m(0.122h)

Condenser Model Ziegler Nichols Tuning Method

PB 88% IAT 6.1m Based on Tu of 7.3m