

Chemstations Technology Symposium

Houston, January 2005

BATCH REACTORS

THERMAL DESIGN

AND

PERFORMANCE

by

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MNL 052 12/04

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1.0 INTRODUCTION

Batch processing techniques are used extensively in the manufacture of a wide variety of fine and speciality chemicals and involve the following general criteria:

- Low to medium capital investment
- Low to medium cost to rectify design errors
- Low volume high value products
- Low to medium production costs
- Limited savings from yield improvements and reduced cycle times
- Multi-purpose capability often required

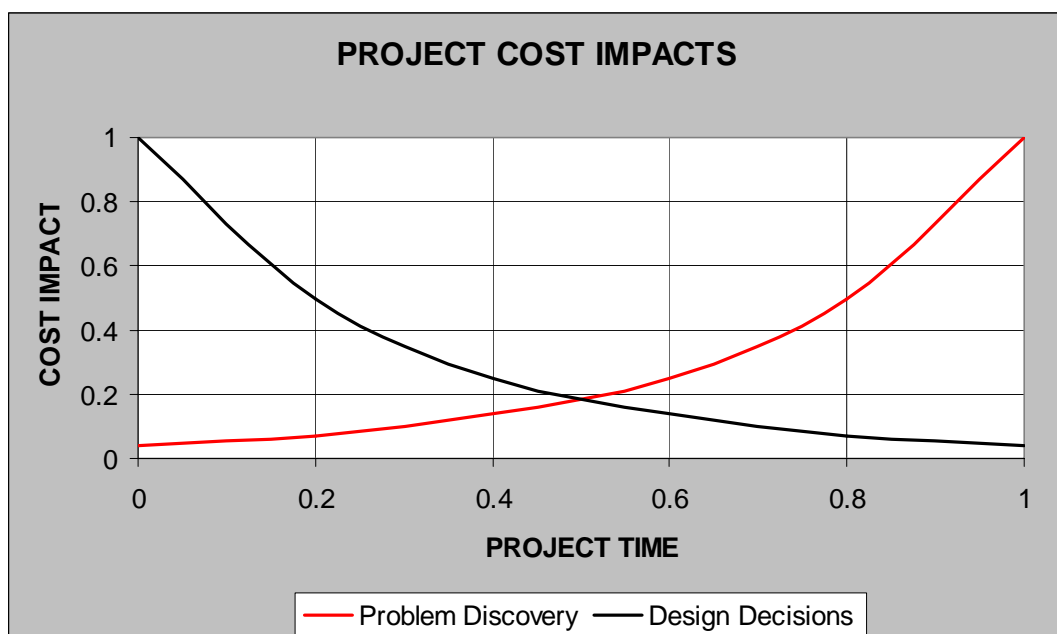
The central item of equipment is normally the stirred tank reactor. The capacity and thermal properties can be readily predicted to enable the operating performance and thermal behaviour to be characterised.

Batch processes are inherently transient in nature and to validate the equipment design the performance needs to be analysed dynamically using the physical properties of the process and service fluids under operating conditions.

The characteristic properties and design parameters are analysed dynamically using the CCReACS module from the CHEMCAD™ range of software by Chemstations of Houston. Dynamic modelling tests the capability to achieve stable and reproducible operating conditions to achieve the product purity, yield, cycle times to satisfy the commercial requirements and the safety and environmental issues for the regulatory authorities.

This article studies the fundamentals of stirred tank reactor dynamic behaviour to develop process design parameters for reactor sizing and jacket services configurations for temperature control.

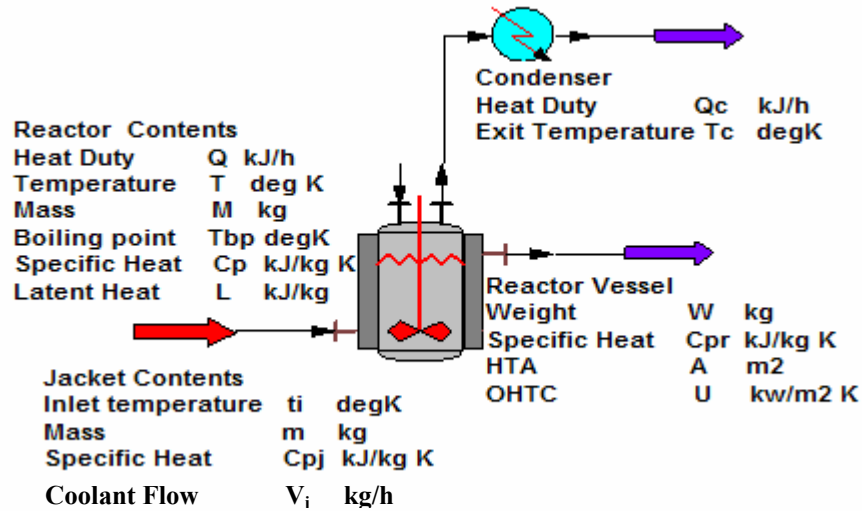
Dynamic simulation can achieve significant savings in the design process and identify errors at an early stage in the project life. The project cost impact curves show the benefits of testing design decisions to avoid the increased costs to rectify later in the project life cycle.



2.0 THERMAL DESIGN FUNDAMENTALS

2.1 Heat Transfer⁽⁹⁾

A typical batch reactor system is shown which defines the key variables for determining the basic thermal ratings.



The fundamental equation for heat transfer across a surface area A is given by:

$$Q = U A \Delta T_m = V_j C_{pj} (t_2 - t_1)$$

The mean temperature difference ΔT_m for a batch reactor with contents temperature T and services inlet and outlet temperatures t_1 and t_2 is approximated by:

$$\Delta T_m = T - \frac{(t_1 + t_2)}{2}$$

The overall heat transfer coefficient U is the sum of the individual resistances as follows:

$$\frac{1}{U} = \frac{1}{h_i} + \frac{1}{h_{fi}} + \frac{1}{k_w/x} + \frac{1}{h_o} + \frac{1}{h_{fo}}$$

When considering glass lined equipment the reactor wall thermal conductivity k_w includes the glass thickness x_g and metal wall thickness x_m with the thermal conductivities for glass k_g and metal k_m to give the following:

$$k_w = \frac{x_m + x_g}{x_g/k_g + x_m/k_m} \quad \text{where} \quad x = x_m + x_g$$

The inside fouling coefficient h_{fi} and outside fouling coefficient h_{fo} are determined by practical experience and there is extensive literature available on this subject. The combined fouling coefficient h_f is given by:

$$h_f = \frac{h_{fi} h_{fo}}{h_{fi} + h_{fo}}$$

2.1 Heat Transfer (Cont.)

The stirred batch reactor inside film coefficient h_i is predicted using the Seider-Tate equation defined as follows:

$$\text{Nu} = C \text{Re}^{0.667} \text{Pr}^p \left(\frac{\mu_b}{\mu_w} \right)^q$$

Nu Nusselt number

Re Reynolds number

Pr Prandtl number

μ_b process side bulk viscosity

μ_w process side viscosity at the wall

C, p, q empirical constants available from manufacturer

$$\text{Nu} = \frac{h_i D}{k_i}$$

D Reactor inside diameter

k_i Process fluid thermal conductivity

$$\text{Re} = \frac{\rho n d^2}{\mu_b}$$

n Impeller rotation rate

d Impeller diameter

ρ Process fluid density

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

C_p Process fluid specific heat

In low viscosity applications $\frac{\mu_b}{\mu_w}$ approaches unity and is ignored.

TYPICAL AGITATOR CONSTANTS				
AGITATOR TYPE	C	p	q	RANGE
IMPELLER (3 BLADES)	0.33	0.33	0.14	$2 \times 10^4 < \text{Re} < 2 \times 10^6$
ANCHOR	0.55	0.25	0.14	$5 \times 10^3 < \text{Re} < 4 \times 10^4$
TURBINE (6 BLADES)	0.44	0.33	0.24	$200 < \text{Re} < 1 \times 10^3$

The outside film coefficient h_o depends on the type of heat transfer surface, namely external jacket(s) with or without mixing nozzles, external half coil(s) or internal coil(s). For coils and half coils the outside film coefficient h_o is determined using the following:

$$\text{Nu} = \frac{h d_{\text{equ}}}{k_o} = 0.027 (\text{Re})^{0.8} (\text{Pr})^{0.33}$$

$$d_{\text{equ}} = \frac{4 \cdot X \cdot \text{Flow} \cdot \text{Cross} \cdot \text{Sectional} \cdot \text{Area}}{\text{Wetted} \cdot \text{Perimeter} \cdot \text{for} \cdot \text{Heat} \cdot \text{Transfer}}$$

Re is calculated using d_{equ} with h_o being evaluated under actual process conditions.

The outside film coefficient h_o for baffled jackets without mixing nozzles are calculated using the above relationships. For mixing nozzle applications, manufacturers' proprietary methods are used to calculate **Re**, raised to a modified coefficient, depending on whether turbulent or laminar flow conditions. CCRACS uses Pfaudler proprietary methods.

2.1 Heat Transfer (Cont)

To establish the thermal rating of the reactor a heat balance is made to establish the heat up and cool down rates based on agreed design parameters using the following:

$$Q = 3600 U A (T - t_i) = \frac{dT}{d\theta} (W C_{pr} + M C_p + m C_{pj}) 60 \quad \text{kJ/h}$$

where $\frac{dT}{d\theta}$ is the temperature change in °C / m.

The reactor duty is calculated from:

$$Q = 3600 U A (T - t_i) \quad \text{kJ/h}$$

The rate of temperature change is calculated from:

$$\frac{dT}{d\theta} = \frac{Q}{(W C_{pr} + M C_p + m C_{pj}) 60} \quad \text{°C/m}$$

The time θ for temperature change from T_1 to T_2 can be calculated^(3, Eq 18.7) using mean values for the specific heat and U in the temperature range under consideration:

$$\theta = \frac{(W C_{pr} + M C_p + m C_{pj})}{U A} \ln \frac{(T_1 - t_i)}{(T_2 - t_i)} \quad \text{m}$$

The boil up rate V without reflux is calculated from:

$$V = \frac{Q}{L} \quad \text{kg/h}$$

For total reflux the heat input is given by:

$$Q = V C_p (T_{bp} - T_c)$$

The total heat input⁽⁸⁾ to reduce the reactor contents from N_0 to N_1 mols at different operating conditions are given by :

For variable top composition (constant reflux ratio)

$$Q_T = L (R + 1) \int_{N_0}^{N_1} dN$$

For constant top composition (variable reflux ratio)

$$Q_T = L \int_{N_0}^{N_1} dN \int_{R_0}^{R_1} (R) dR$$

Both relationships indicate that the reflux ratio must be kept to a minimum, subject to satisfying the desired separation specification, to optimise the heat input.

2.2 Thermal Lag

Temperature control of batch reactor systems is inherently difficult due to the thermal lags associated with the heat transfer process. The thermal time constants for the different interfaces can be estimated ^(2, pages 75 to 76) as follows:

$$Q_r = h_i A (T - t_{wi}) + M C_p \frac{dT}{d\theta}$$

Q_r = rate of heat evolution T = reactor temperature
 h_i = inside film coefficient t_{wi} = inside wall temperature
 A = heat transfer area M = weight of reactants
 C_p = specific heat of reactants

Rearranging in the standard form we have

$$T + \frac{M C_p}{h_i A} \frac{dT}{d\theta} = t_{wi} + \frac{Q_r}{h_i A}$$

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

$$\Gamma_1 = \frac{M C_p}{h_i A} = \frac{M C_p}{Q_r} (T - t_{wi})$$

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

$$\Gamma_2 = \frac{W C_{pr} x}{k A} = \frac{W C_{pr}}{Q_r} (t_{wi} - t_{wo})$$

W = weight of wall x = wall thickness
 C_{pr} = specific heat of wall t_{wo} = outside wall temperature
 k = thermal conductivity of wall

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

$$\Gamma_3 = \frac{m C_{pj}}{h_o A} = \frac{m C_{pj}}{Q_r} (t_{wo} - t_c)$$

m = weight of jacket contents h_o = outside film coefficient
 C_{pj} = specific heat of jacket contents t_c = average coolant temperature

The reactor contents time constant based on overall heat transfer coefficient is given by:

$$\Gamma_o = \frac{M C_p}{U A}$$

Typical values are shown below for heating 1000kg toluene in a 1600L Hastelloy C reactor with Dowtherm J fluid.

Time Constant	Γ_1	Γ_2	Γ_3	Γ_o
Time (min)	15.4	3.1	2.6	21.7

2.3 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 procedure.⁽¹⁾

The reaction rate⁽²⁾ depends on the reaction order. First order reaction conversion varies with time and 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. time zero.

The kinetics of a reaction is determined from the Arrhenius rate law which states that the rate of a chemical reaction increases exponentially with absolute temperature and is given by:

$$k = A \exp \left(\frac{-E_a}{R T} \right)$$

Where **R** = universal gas constant = **8.314** J / °K mol
E_a = activation energy J / mol
A = frequency factor or pre-exponential factor consistent units

The values of **E_a** and **A** for a reaction can be determined experimentally by measuring the rate of reaction **k** at several temperatures and plotting **ln k** vs **1/T** noting:

$$\ln k = \ln A - \left(\frac{E_a}{R} \right) \left(\frac{1}{T} \right)$$

E_a is determined from the slope **E_a/R** and **A** from **ln A** the intercept of **1/T**.

In many applications the reaction kinetics will not be known. In these cases the overall heat of reaction **ΔH_r** and frequency factor **f** are required to establish reactor thermal design and stability. If the heat of reaction is not known it can be estimated from the standard heats of formation, **ΔH_f⁰**,⁽⁶⁾ and the stoichiometric coefficients **v_i** of the reactant and product species **i** involved, using Hess's law^(7, page 428) as follows:

$$\Delta H_r = \sum_{\text{Products}} v_i (\Delta H_f^0)_i - \sum_{\text{Reactants}} v_i (\Delta H_f^0)_i$$

The activation energy **E_a** cannot be derived logically from the heat of reaction **ΔH_r** but can be estimated using a thermodynamics analogy.^(5, pages 21-34)

For liquids and solids we have:

$$E_a = \Delta H_r - R T$$

and for gasses:

$$E_a = \Delta H_r - (\text{molecularity} - 1) R T$$

The units for **E_a** and **A** are used in various forms so caution is required in their use.

E_a is usually in the form energy/(mol reactant species) and **A** in mol/s referenced to reactant volume, depending on the units of **k**.

2.3 Reaction Chemistry (Cont.)

In a reaction, where the total mols of reactant N_r is converted in reaction time θ , the conversion rate r given by:

$$r = \frac{N_r}{\theta} \text{ mol/s}$$

Total heat of reaction H is given by:

$$H = N_r \Delta H_r \text{ kJ}$$

The frequency factor f in $\text{mol/m}^3\text{s}$ units is derived from the reactant mix volume V_r

$$f = \frac{r}{V_r} \text{ mol/m}^3\text{s}$$

The mean heat output from the reaction is given by:

$$Q_r = r \Delta H_r$$

For exothermic reactions to be carried out safely, the heat removal capability Q of the reactor system must exceed the maximum predicted heat output Q_r by an acceptable margin.

For a system U and design set ΔT_m there is an equilibrium heat transfer area where heat removal capability equals reaction heat output. This consideration determines an acceptable reactor size to ensure adequate heat transfer area under all reaction conditions.

Where reactions are carried out with all the reactants charged the implications of cooling failure, taking into account common mode failures, needs to be considered.

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

Tempered reactions, operating at boiling point, remove heat using the latent heat of vaporisation. This procedure is self regulating provided the overhead condenser is adequately sized to ensure material is not removed from the reaction resulting in a decrease in heat transfer area. In this case the reactor cooling system is only required to remove any excess heat from the reaction.

Gassy systems generate a permanent gas and require the 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 from the overhead condensing system due to inerts.

3.0 PROCESS DESIGN CONSIDERATIONS

The key determining factor for process design of batch reactor temperature control systems is the design operating temperature range. This is set taking into consideration the type of reactions and separation techniques, such as crystallisation, to be performed.

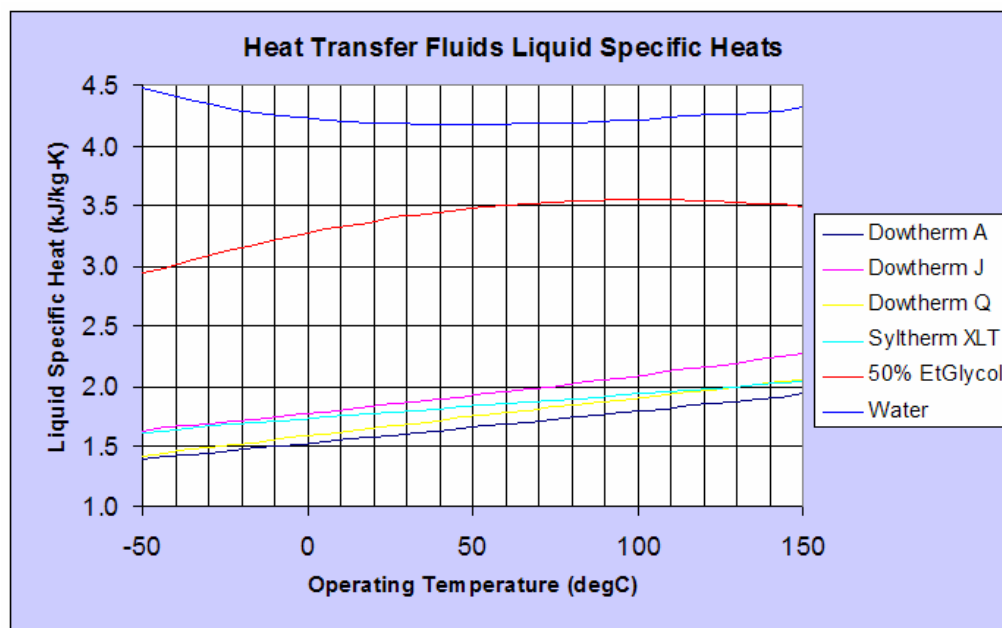
3.1 Jacket Service Fluid Selection

The relative merits of the heat transfer fluids, under consideration, are evaluated by study of key property data group component plots generated using CHEMCAD.

The operating temperature limits for a range of heat transfer fluids are summarised

Heat Transfer Fluid	Minimum °C	Atm BP °C
Dowtherm A	15	257
Dowtherm J	-80	181
Dowtherm Q	-35	267
Syltherm XLT	-100	172
50% EtGlycol	-30	100
Water	5	100

The liquid specific heat plots for common heat transfer fluids show a significant difference between water and organic based thermal fluids.



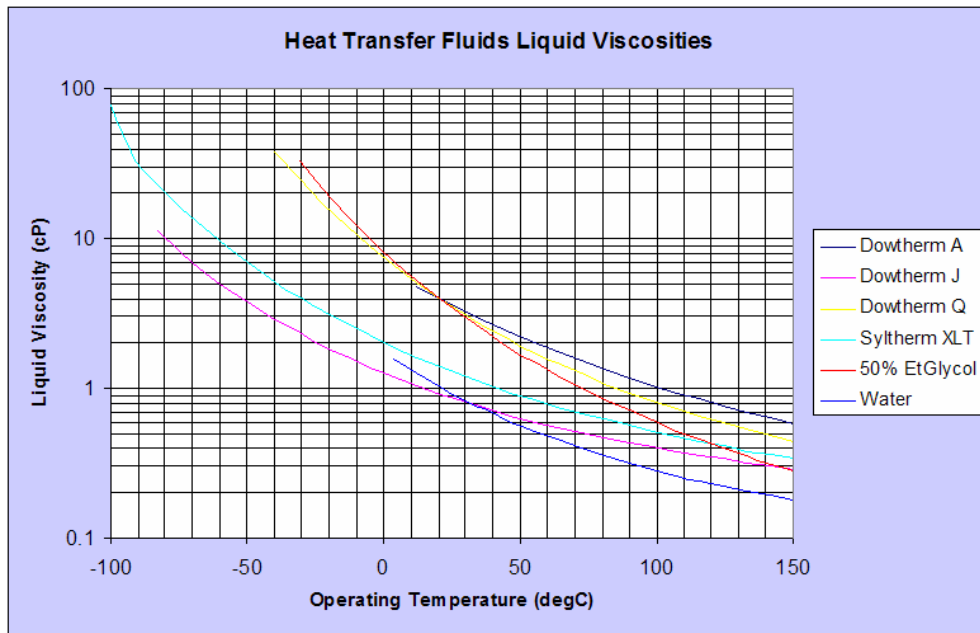
The heat balance across the jacket / coil is given by:

$$Q = V_j C_{pj} (t_2 - t_1)$$

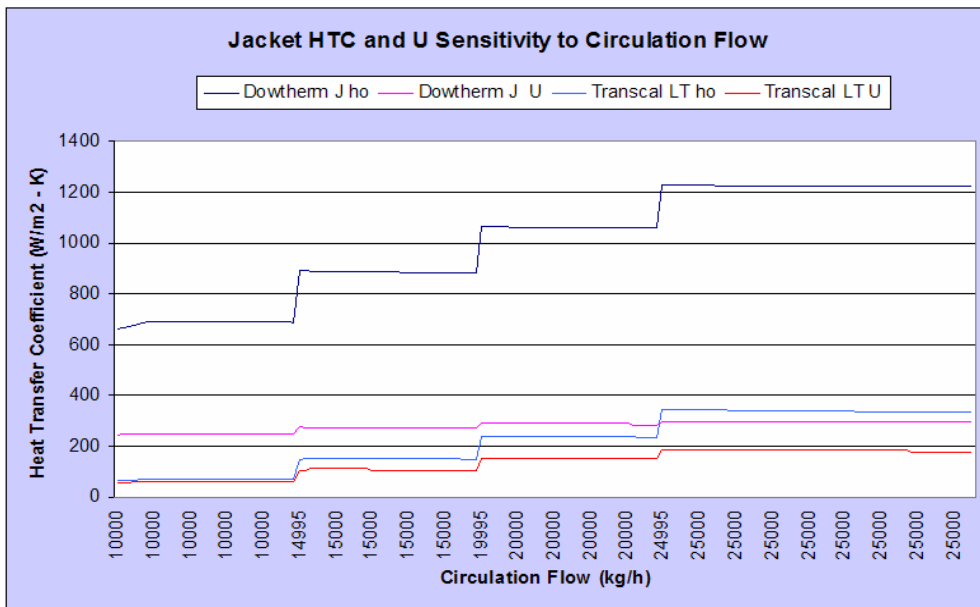
The heat removal capability using water, at constant temperature difference, is greater than that achieved with organic fluid flows in proportion to the ratio of the specific heats. In consequence, heat transfer fluid distribution systems are significantly more expensive to install, due to the increased pipe size and pumps required, over that for water systems. Also thermal fluid systems require a more rigorous specification for flange rating and jointing.

3.1 Jacket Service Fluid Selection (Cont.)

The liquid viscosity of the selected fluid throughout the operating temperature range is the key parameter in the selection. Viscous conditions can result in laminar flow in the cooling circuit with resulting poor heat removal capability leading to instability for exotherms.

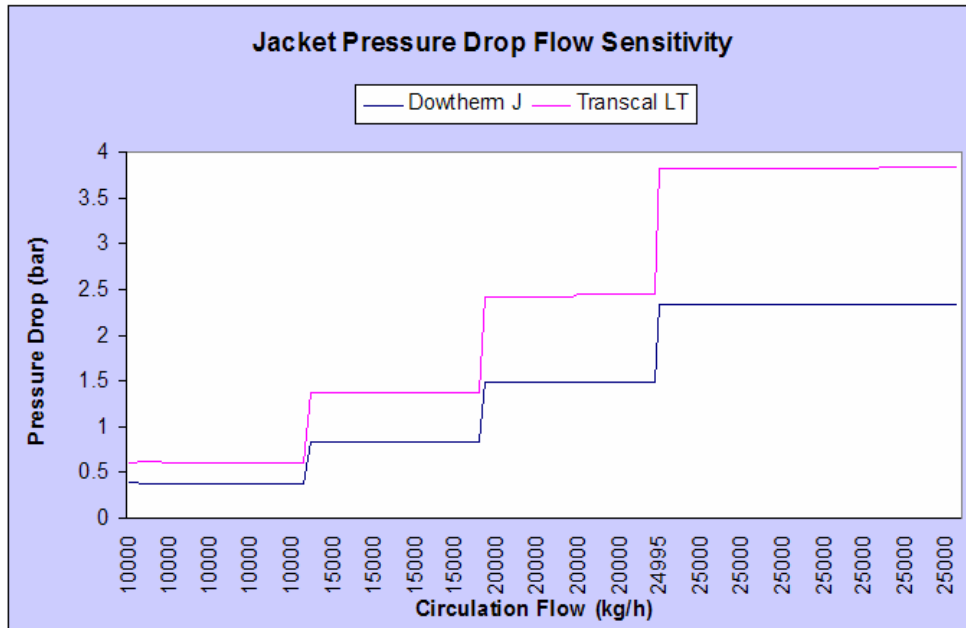


To demonstrate the implications of incorrect heat transfer fluid selection, an application using Dowtherm J or the more viscous Transcal LT is considered. Flow and temperature sensitivity curves have been generated using CHEMCAD for a 4.5 m³ Glass Lined jacketed reactor, with three mixing nozzles fitted, carrying out an exothermic reaction in the range 100 to 110 °C.



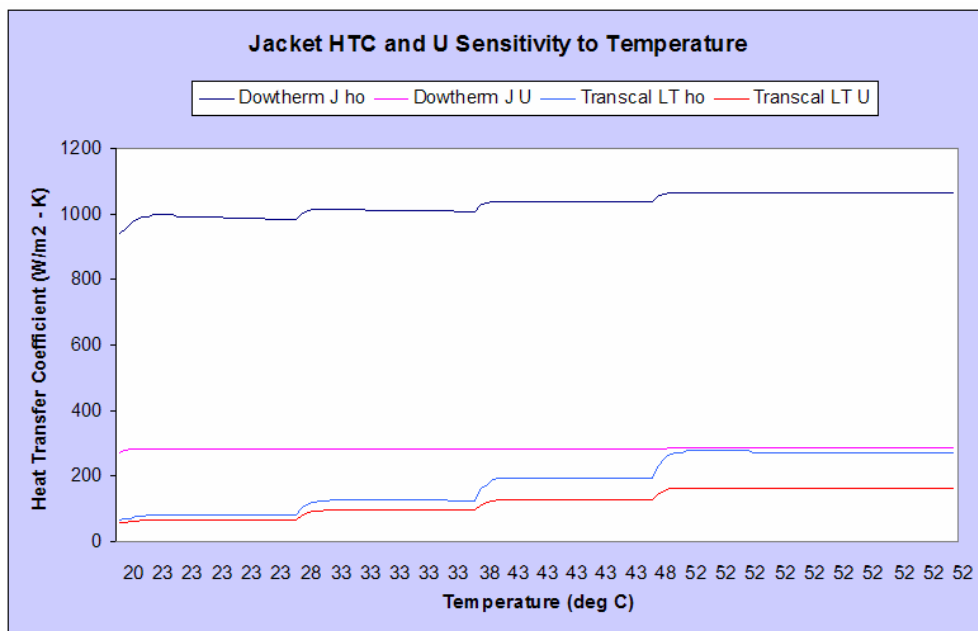
Circulation flow change results in the U for Dowtherm J varying from 252 to 295 W/m² °K and for Transcal LT varying from 62 to 181 W/m² °K.

3.1 Jacket Service Fluid Selection (Cont.)



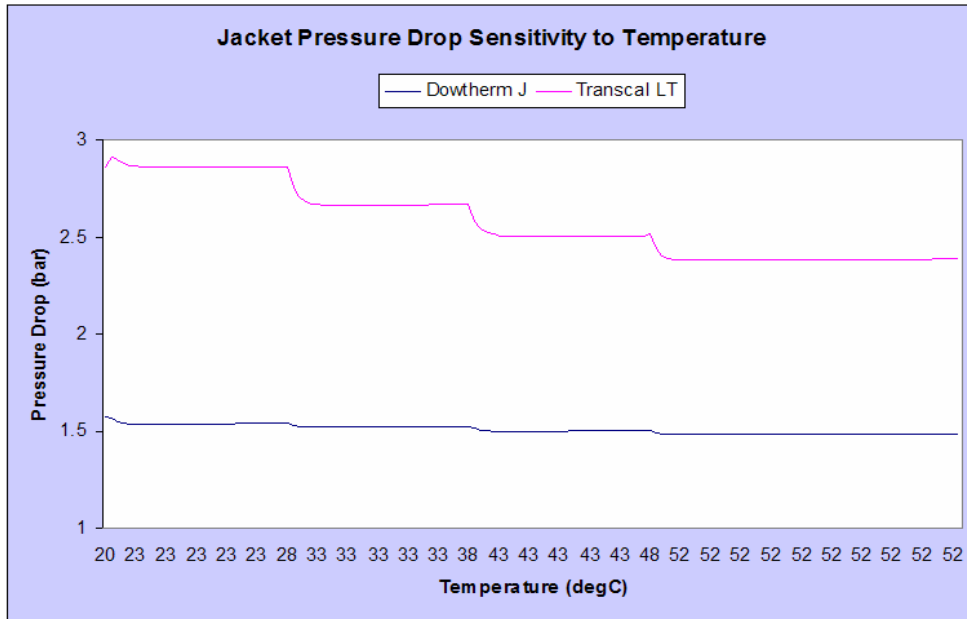
Circulation flow change results in the jacket pressure drop for Dowtherm J varying from 0.37 to 2.3 bar and for Transcal LT varying from 0.6 to 3.8 bar.

The sensitivity to flow change for Transcal LT is not acceptable for heat transfer and hydraulic considerations alone. Dowtherm J demonstrates a stable behaviour and will provide reproducible thermal properties under varying flow conditions.

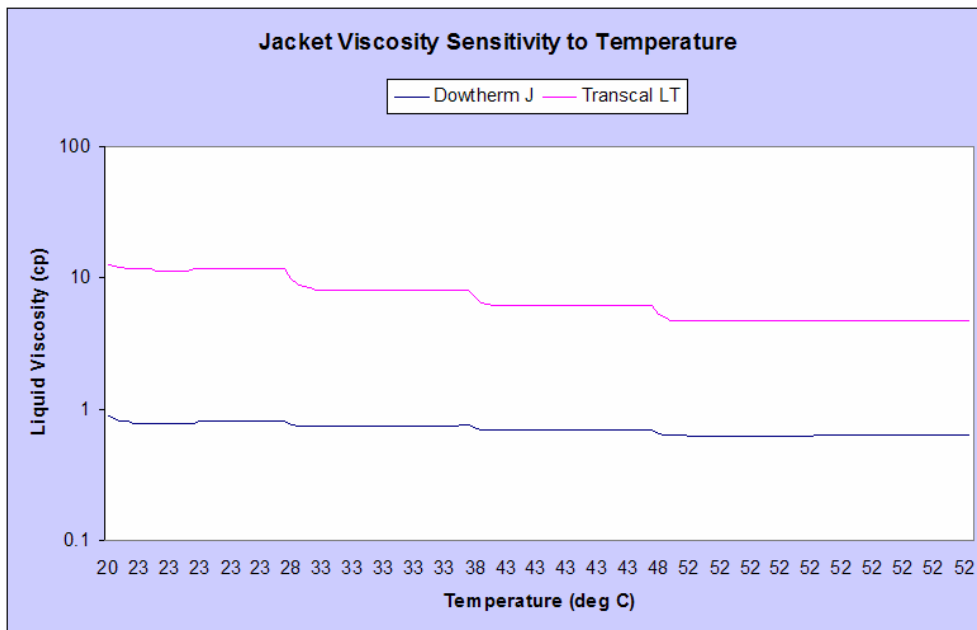


Jacket temperature change results in the U for Dowtherm J being constant at 283 W/m² °K and for Transcal LT varying from 66 to 161 W/m² °K.

3.1 Jacket Service Fluid Selection (Cont.)



Jacket temperature change results in the jacket pressure drop for Dowtherm J varying from 1.54 to 1.48 bar and for Transcal LT varying from 2.86 to 2.38 bar. The changes are not significant but the Transcal LT pressure drop is unacceptable for the circulation flow set for good heat transfer.



Jacket temperature change results in viscosity for Dowtherm J varying from 0.79 to 0.63 cp and for Transcal LT varying from 11.6 to 4.8 cp, demonstrating the unsuitability of Transcal LT for this application.

The advantages for using heat transfer fluids are summarised:

- Single phase state throughout operating temperature range provides simplification of the control system, equipment configuration and operation.
- Fluid properties are stable over a wide temperature operating range.
- Heat transfer surface corrosion and erosion minimised.
- Temperature differences controllable to minimise thermal shock effects.
- Provides future flexibility for multi-purpose plant operation.

The disadvantages for using heat transfer fluids are summarised:

- Lower thermal efficiency than systems based on water.
- Higher initial equipment and installation costs.
- High cost for initial fluid charge.
- Special commissioning, operational and maintenance procedures required.
- Longer downtime on equipment failure.
- Flammability, toxicity, odour and GMP issues to resolve.

These systems have to be thoroughly dried out during commissioning to prevent operational problems and equipment damage. This can take considerable time and needs to be done slowly to prevent equipment damage due to cavitation.

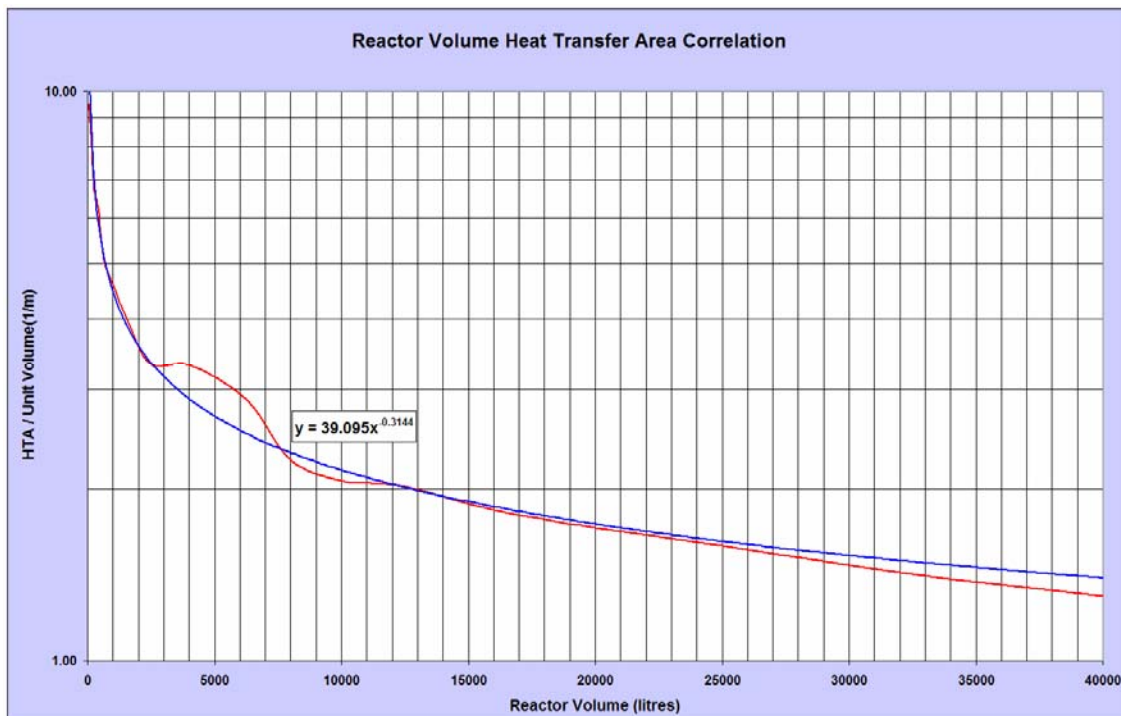
Water breakthrough, due to contamination or equipment failure, can result in considerable downtime to identify and rectify the problem. In low temperature operation water breakthrough will result in freezing leading to loss of circulation and possible further equipment damage

Heat transfer fluid selection is often driven by current site practice and to minimise the significant initial fill cost, which has frequently not been included in the capital budget.

Dynamic simulation demonstrates the importance of testing the selection being made and provides a basis for justifying a new selection.

3.2 Reactor Parameters

The heat transfer area / reactor volume ratio increases as the reactor size reduces. This needs to be considered carefully during scale up and emphasises the importance of correctly matching reactor size to the batch size. Using partially filled reactors reduces the heat transfer area as well as increasing the potential for minimum stir problems.



The thermal conductivities of the reactor materials of construction have a significant effect on the wall temperatures which can limit cycle times. Extreme temperature differences can result in product quality problems on certain processes.

Physical Properties of Common Reactor Materials			
Material of Construction	Density kg/m ³	Specific Heat kJ/kg°K	Thermal Conductivity Btu/ft ² h°F/in
Hastelloy C	8690	0.423	76.9
Stainless Steel	8000	0.5	112.3
Carbon Steel	8000	0.4	360
Glass			6.9

As can be seen from the table, the density and specific heat differences are not significant for heat transfer considerations but the thermal conductivity differences are. Glass lined carbon steel and Hastelloy C are similar with Stainless Steel a factor 1.5 higher and Carbon Steel a factor of 4.7 higher.

3.3 Reactor Heat Transfer ⁽¹⁰⁾

Stirred batch reactors, with coils or external jackets, have inherent thermal lags due to the heat capacities of the masses associated with the reactor, reaction mix, jacket contents and jacket services. These lags are minimised, where possible, by minimising jacket service volumes, thermal masses associated with external equipment and good thermal insulation.

The service side film transfer coefficient will be controlling if certain design techniques are not applied. It has been established empirically that by achieving a velocity of 1 m/s across the heat transfer surface provides optimum economic heat transfer.

As the vessel size increases the cross sectional area for fluid flow increases, being determined by the annulus width for jackets and the pipe diameter for coils. Unbaffled jackets result in laminar flow and result in very poor thermal performance. Several techniques have been developed to increase the velocity, namely baffling in the jacket annulus, dimple jackets, half coils and inlet agitating nozzles. The methods used to increase velocity are limited by mechanical design, construction and cost constraints.

Service side circulating pumps are required to achieve acceptable velocities. The installation of agitating nozzles on the jacket inlets induces a rotational movement similar to spiral baffles and significantly reduces the circulation flow required for efficient heat transfer.

The reactor size and nozzle pressure drop determines the number and size of mixing nozzles. Empirical curves are available from the manufacturers to establish the optimum circulation rate and pressure drop.

When using heat transfer fluids, that may have high viscosities within the operating temperature range, mixing nozzle pressure drops may become limiting and half pipe coil constructions may have to be considered.

Typical overall heat transfer coefficient and fouling factor data are shown below for steam heating of glass lined reactors:

Typical Process Side Heat Transfer Coefficients (Btu/h ft ² °F) Glass (1.5mm thick) Lined Vessel, 90 rpm Impeller, Steam Heating			
Product	U	h _i	h _f
Water	68	659	1000
Organic Solvents	55	232	1000

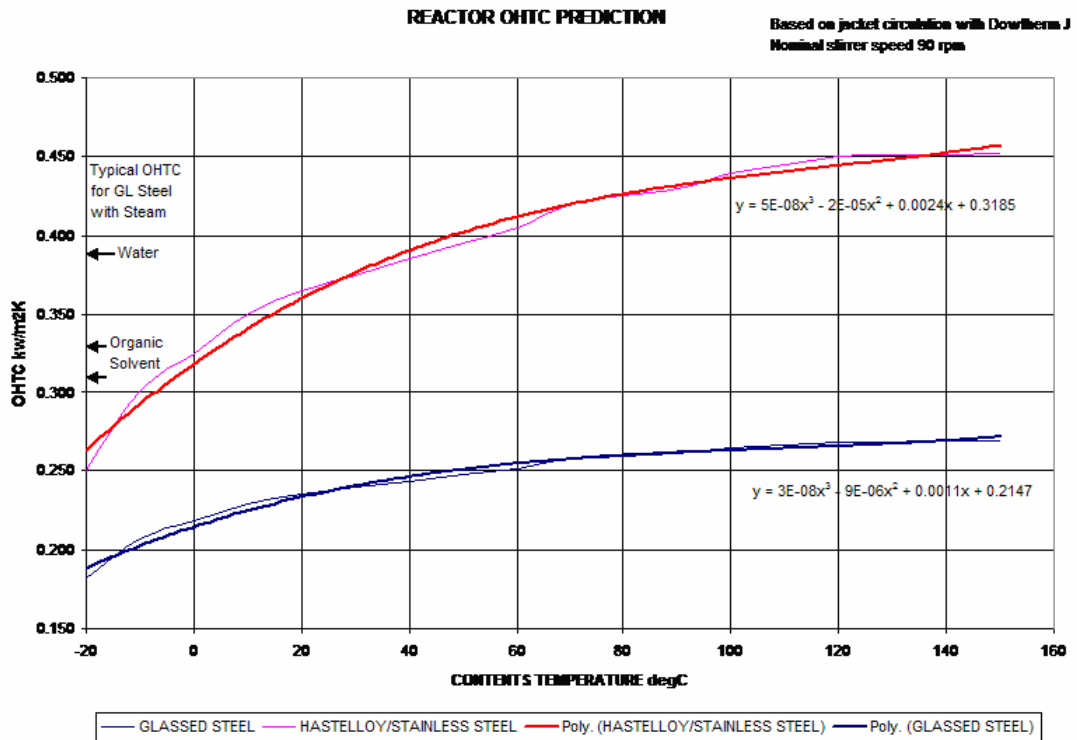
Typical Jacket Side Film Coefficients (Btu/h ft ² °F) Annular Jacket with Inlet Mixing Nozzles except for Steam		
Fluid	h _o	h _f
Steam	1500	3333
Water	500	1000
Glycol/Water Mix	30	500
Heat Transfer Fluid	250	500

A reasonable film coefficient for steam is 1500 Btu/h ft² °F and for heat transfer oil is 250 Btu/h ft² °F. The overall heat transfer coefficient using heat transfer oil can be estimated using the following:

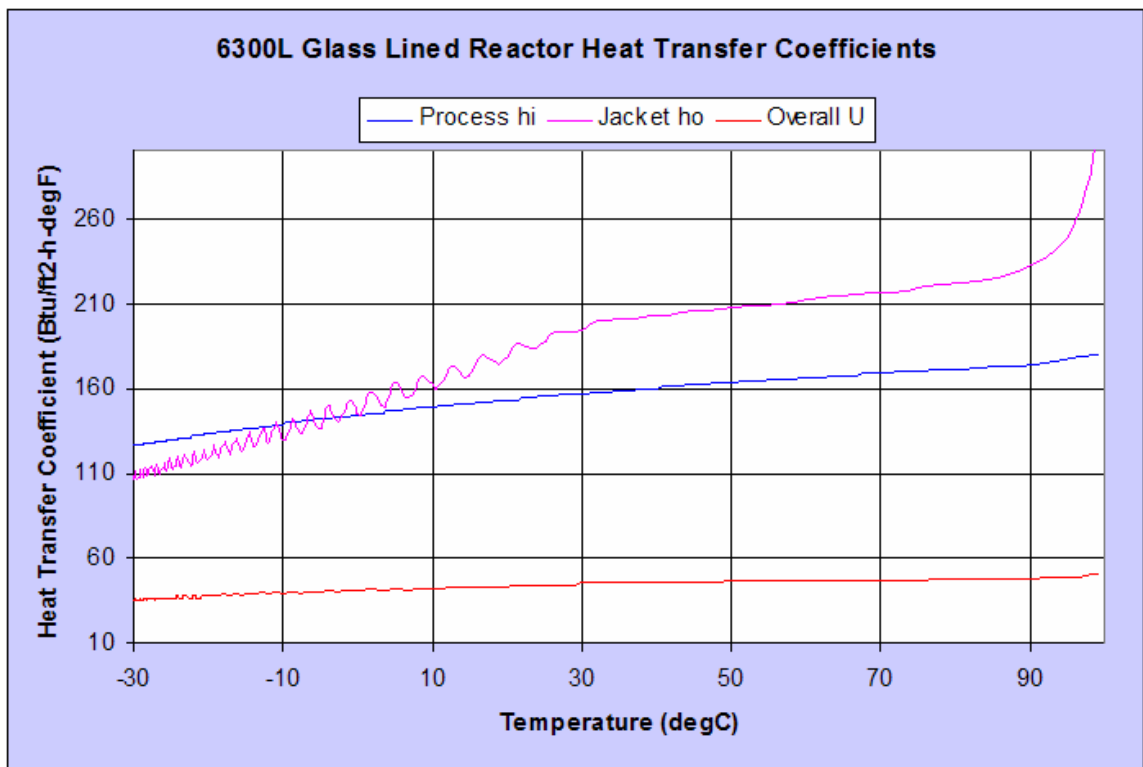
$$\frac{1}{U_{oil}} = \frac{1}{U_{steam}} - \frac{1}{1500} + \frac{1}{250}$$

3.3 Reactor Heat Transfer (Cont.)

The curves below are used in a short cut method for rating batch reactors with a software module BREAC by P&I Design. The overall heat transfer coefficient is modified as a function of reactor contents temperature, only, and is not a rigorous analysis.



A more rigorous analysis, using dynamic modelling, is shown for a system using Syltherm XLT heat transfer fluid, circulated at 23000 kg/h through a shell and plate heat exchanger, to cool down Toluene. It can be seen that the overall U reduces from 51 to 35 Btu/h ft²°F.



3.4 Jacket / Coil Services Heat Exchangers

The fully welded, gasketed shell and plate heat exchanger is recommended for this application, being able to handle the severe thermal stresses resulting from the rapid temperature cycling that occurs on jacket service systems. The thermal fluid is usually on the plate side with the service fluid on the shell side.

Sizing is based on the heat transfer relationship using the log mean temperature difference of the temperature differences at the heat exchanger inlet and outlet.

$$Q = (U A)_{\text{he}} \Delta T_{\text{lm}}$$

The heat duty for heat exchanger sizing is determined using the reactor clean overall heat transfer coefficient, the heat transfer area available at maximum operating level and maximum temperature differences during heating and cooling. Alternatively the heat duty could be determined from the type of reaction chemistry to be carried out.

The heat transfer fluid operating temperature range is determined from the reactor operating temperature range allowing for a reasonable temperature difference at the range limits.

The liquid service flow is determined by setting an acceptable temperature difference across the heat exchanger, typically 10°C. The maximum allowable return temperature is normally determined by the type of cooling system and its operation.

The heat exchanger inlet and outlet temperature differences, for sizing, are determined from the services supply and return temperatures and by setting reasonable heat transfer fluid inlet and exit temperatures at approach to the services inlet temperatures.

For heating, with steam, the inlet and outlet temperature differences are reasonable and are unlikely to be critical at the approach to maximum heat transfer fluid temperature.

For cooling, the inlet and outlet temperature differences can be critical at the approach to minimum heat transfer fluid temperature, particularly on low temperature applications. The design temperature difference is set to give an economic design.

The heat exchanger overall heat transfer coefficient is now calculated using manufacturers' proprietary software from the design flows, inlet and outlet temperature differences and the physical property data at inlet and outlet temperatures.

A characteristic of plate heat exchangers is that the cross sectional area for flow is small and the pressure drop, particularly at low temperatures, usually determines the number of plates and their geometric arrangement.

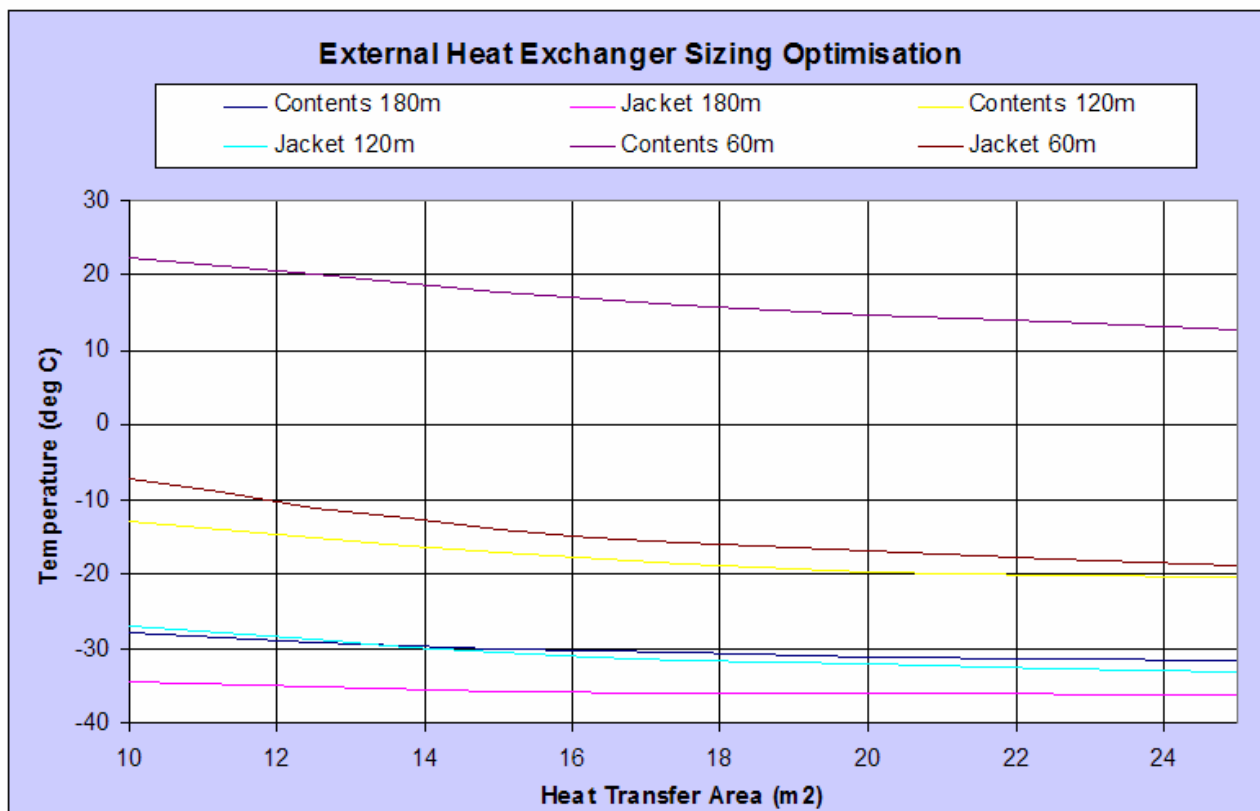
The heat transfer area is estimated thermally and the configuration is then adjusted to give an acceptable pressure drop. The plate area determined by pressure drop, usually on the circulating heat transfer fluid side, normally results in an increased design margin for heat transfer area.

For a given set of process conditions the reactor $(UA)_r$ is determined and the external heat exchanger $(UA)_{\text{he}}$ is checked to ensure sizing does not become limiting under operational conditions.

3.4 Jacket / Coil Services Heat Exchangers (Cont.)

Dynamic simulation is used to aid in the heat exchanger sizing selection. The reactor was a 6300L Glass Line Reactor fitted with an impeller agitator rotating at 90 rpm. The reactor charge, at the start of the simulation was 4900 kg of Toluene, giving a heat transfer area of 17.5 m². The heat transfer area at the end of the simulation was 15.5 m² due to the change in density.

A constant heat exchanger overall heat transfer coefficient of 800 W/m²°K was used in the simulation which is not rigorous. Variation in the heat exchanger performance can be applied to the dynamic simulation by mapping manufacturers' heat transfer coefficient data directly from an Excel™ spreadsheet.



Simulation demonstrates that an area increase from 20m² to 25m² provides a marginal increase in performance over the operating range. However the heat exchanger overall transfer coefficients will become controlling at lower temperatures so increase of the design margin should be considered.

Heat transfer fluids are extremely searching so high integrity equipment and piping specifications should be applied.

Sealless pumps are recommended for heat transfer fluid circulation. Precautions are required to ensure the minimum flow is maintained for the bearings by installing a restriction orifice in a spillback line round the pump.

The piping design should be based on the use of ANSI 300 flanges as a minimum to allow for high bolting torques. The gaskets should use an asbestos free filler reinforced with stainless steel spiral.

4.0 PERFORMANCE CHARACTERISTICS

Dynamic modelling provides the designer and the plant operator with the tools necessary to study the performance characteristics of a variety of process equipment and control system configurations. Batch reactor systems have the following inherent characteristics.

4.1 Jacket / Coil Services

The maximum heat input is achieved when using steam directly on the jacket/coil side, however this can lead to thermal shock when using glass lined equipment. Direct steam injected circulating liquid systems avoid the thermal lags associated with external heat exchangers and hence have superior heat input characteristics.

Single fluid systems using heat transfer fluids and external heat exchangers have the lowest heat input capability for a given temperature difference of the systems considered. The heat input is achieved by increasing the temperature difference at a rate consistent with thermal shock considerations.

The heat removal capability is a function of the resistances to heat transfer, the temperature difference and the heat transfer area. A thermal runaway (increasing reaction temperature increases rate of reaction) will occur if the heat cannot be removed fast enough further accelerated by a reduction in heat transfer area due to a decrease in reactor contents. It may not always be possible to design for stability where not enough heat transfer area is available 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.

Heat removal is normally achieved using single fluid liquid systems which will determine the UA achievable for a given reactor system. The heat removal is therefore determined by the operating temperature difference which can be enhanced by operating at higher reaction temperatures or under reflux conditions consistent with reaction kinetics.

The heat removal capability of a facility is limited by the temperature of the service fluids available. The heat load on any refrigeration system is to be optimised by using the higher temperature cooling service and then switching to the lower temperature medium when appropriate.

Heat transfer fluid viscosity effects at low temperatures can become limiting resulting in low jacket/coil side heat transfer coefficients and high pressure drops. Selection of a heat transfer fluid with reasonable viscosity characteristics will allow operation down to -90°C .

The boil up rates achievable with the various heating systems considered vary between being excessive with direct steam and being limiting with indirect heat transfer fluid systems. The only variable available to increase boil up is the jacket/coil temperature which will be limiting on water based systems. To control the boil up and wall temperature, if required, with steam requires pressure control. To achieve the boil up rates required with single fluid systems the temperature difference is controlled; the utility heating medium temperature needs to satisfy this requirement. The overhead condenser heat duty determines the maximum boil up rate achievable and the batch distillation process determines the minimum boil up rate to achieve a satisfactory separation by satisfying the minimum reflux ratio requirements. The inert flow should be reduced/eliminated to minimise volatile organic compounds (VOCs) emission to the environment.

4.2 Temperature Control

The primary thermal lag is from reactor contents to reactor wall which increases as reactor size increases, resulting in a decrease in heat transfer area per reactor volume.

Batch reactor temperature control is characterised by maintained errors between set point and measurement during heat-up and cooldown and by varying thermal responses. IAT should only be activated when the measurement is within the PB and set conservatively such that energy is not driven into the process at a rate faster than the process can accept.

A typical control system uses cascade control with the reactor contents temperature controller output being cascaded to the jacket/coil temperature controller set point. This arrangement ensures that disturbances in the faster jacket/coil loop are corrected by the secondary controller before they can influence the contents temperature.

The primary controller PB is normally be set in the range 35 to 50%. The IAT is set slower than the overall reactor time constant. The derivative time is normally set at IAT/4. For the case in Section 2.2, we would start an initial setting of IAT of 22 min and a derivative time of 5 min. An estimate of time constant can be obtained from a study of the heat-up and cooldown curves or responses to set point increase and decrease step changes.

The secondary controller is normally a Proportional only controller, as Integral Action Time (IAT) slows the response. A proportional band (PB) of $\leq 25\%$ is typical. The primary controller is normally three mode (Proportional + Integral + Derivative).

Distillation boil up is determined by the temperature difference between jacket/coil and reactor contents. This is achieved by controlling the jacket/coil inlet temperature. In this mode the secondary controller will require IAT to be activated to eliminate offset.

For high accuracy temperature measurement should use resistance sensor in conjunction with Smart transmitters to provide flexibility when setting ranges. Thermal lag associated with the sensor is minimal. There can be a significant thermal lag 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.

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 ΔP) 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 $\Delta P_{\max}/\Delta P_{\min}$ increases. A linear valve operating characteristic tends towards a quick opening characteristic as $\Delta P_{\max}/\Delta P_{\min}$ increases.

The control valve characteristic normally used is equal % though situations may arise where a linear characteristic provides better control. The operational characteristic of a valve can be modified by controller output signal characterisation.

Control valve actuators should be 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 under all failure modes.

4.3 Jacket / Coil Services Configurations ⁽⁹⁾

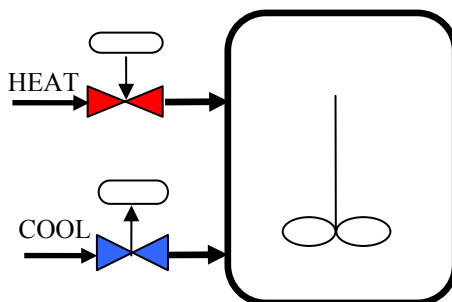
Jacket services system configurations in common use are presented. The dynamic simulation plots presented are all based on a 6300L Glass Line Reactor fitted with an impeller agitator rotating at 90 rpm. The reactor contents at the start of the simulation is 4000 kg of Toluene.

4.3.1 Direct Jacket Heat / Direct Cool

These systems involve the connection of the appropriate supply and return services directly to the reactor jacket/coils. There are many arrangements in use from total manual to fully automatic control involving forced circulation with steam/water mixing facilities.

Temperature ranges from -20°C to 180°C with water, steam or ethylene glycol/water can be used but ethylene glycol/water should not be used above its boiling point.

This arrangement provides good thermal response due there being no heat transfer lags associated with heat exchangers. However, to provide combined heating/cooling facilities requires automatic valve sequencing and jacket/coil blowdown routines when changing services. This results in discontinuous control, , with the exception of the Wiegand system, which can lead to operational problems if handling exothermic reactions.



The scheme above shows the basic system with automatic control of the steam and cooling water services. The changeover between heating and cooling modes can be done manually or automatically involving a timed sequence of valve switching operations. The changeover is not seamless as with a single fluid system and requires careful consideration if used on exothermic reactions.

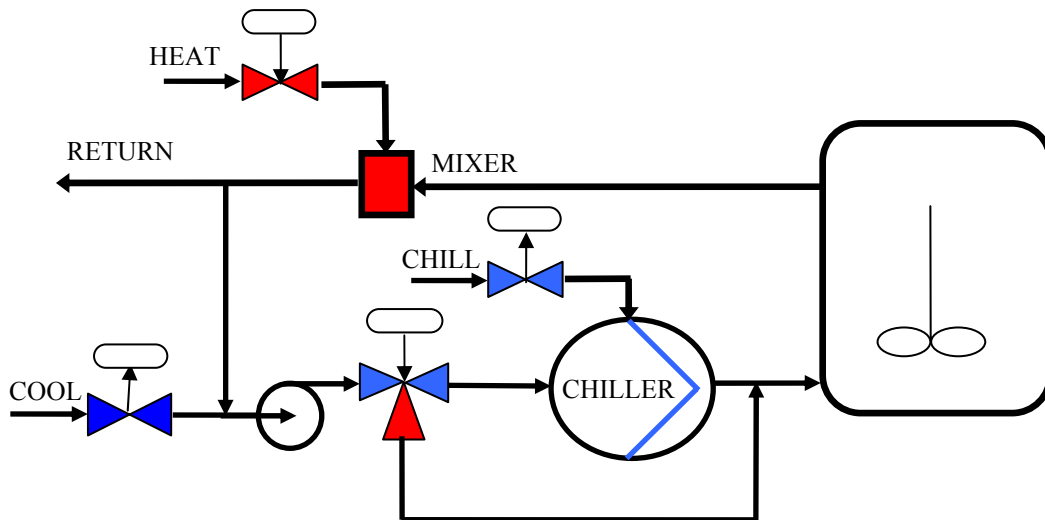
A study of the heat up and cool down curves demonstrate the fast response of these systems. However the rate of change of jacket wall temperature can lead to thermal shock problems with glass lined equipment and possibly product problems with the high wall temperatures that can be experienced.

The heat up curves for the direct system, shown below, have been modelled using 4 barg steam pressure.

Operational problems associated with this system and the mixing system, shown following, include cross contamination of services, jacket fouling and corrosion and the need for sequenced control routines when changing services from heat to cool.

These systems are not always cheaper than other alternatives.

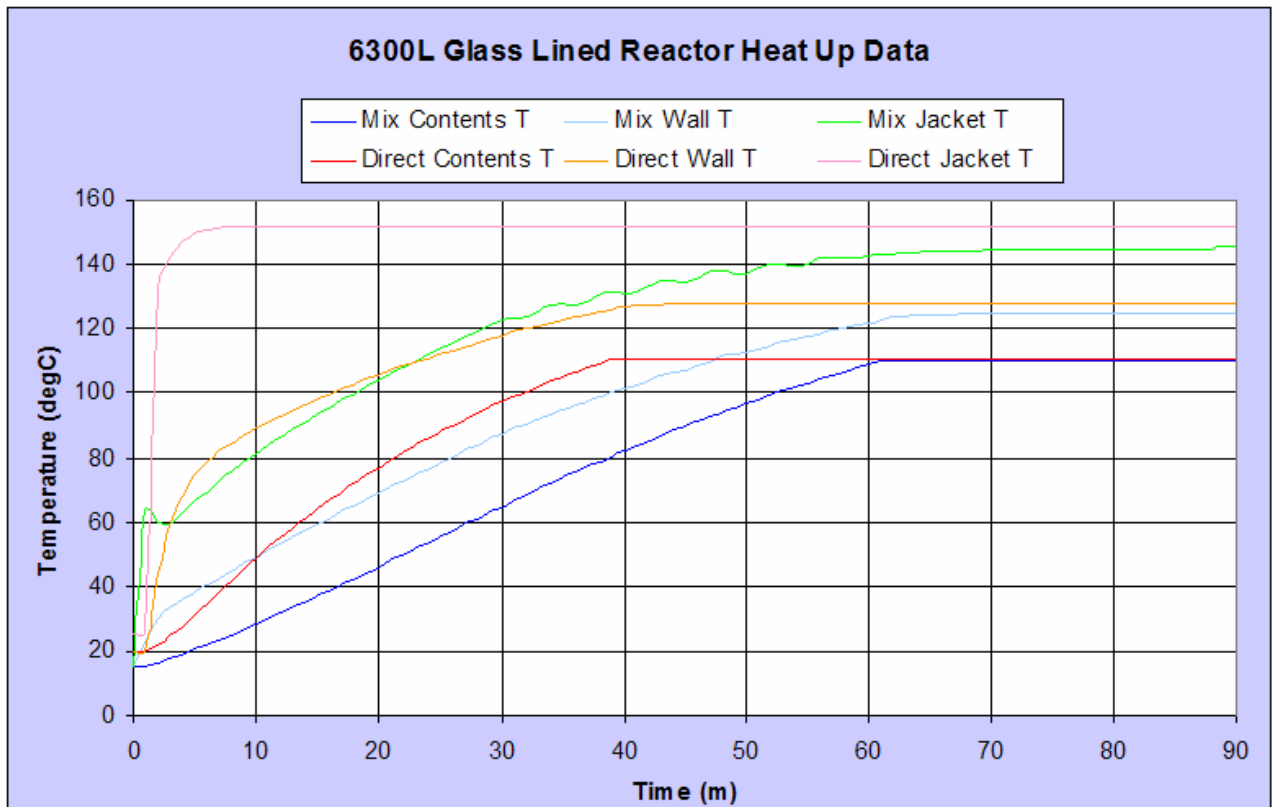
4.3.1 Direct Jacket Heat / Direct Cool (Cont.)



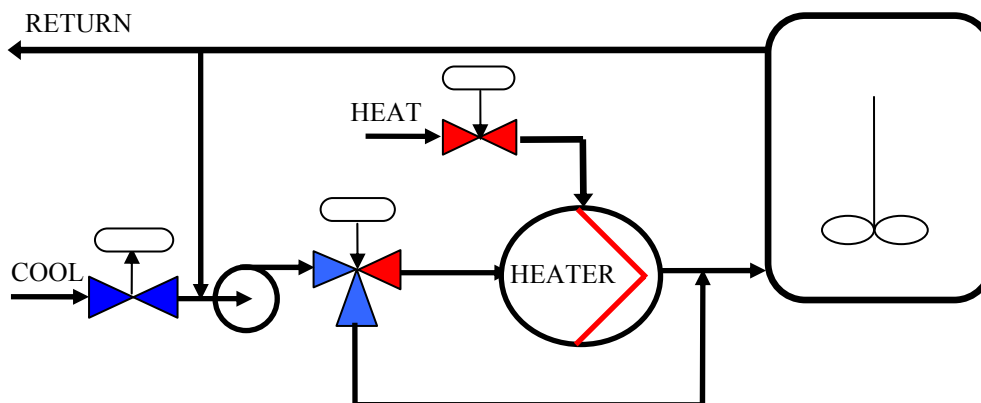
Ciba Geigy and Wiegand developed a steam jet water mixing heater which provides a wide range of heating duties using water as a single phase in the operating range 5°C to 180°C.

This arrangement requires careful design in relation to air removal, thermal expansion and pressurisation facilities, if operating above the boiling point, The service returns from high operating temperatures require special automatic blowdown facilities.

The heat up curves for the mix system, shown below, have been modelled using 9 barg steam pressure, an initial inlet temperature of 150°C and 20000 kg/h water circulation rate. It can be seen that the heat up time is increased by 50% due to the rate of jacket temperature increase being significantly slower. This type of system allows for better control of jacket temperature conditions but with the loss of heat input capability.



4.3.2 Indirect Jacket Heat / Direct Cool



This system is based on the use of a single heat transfer fluid suitable for the desired operating range.

The coolant is directly injected into the reactor services circulating loop. Heating is provided by a heat exchanger with steam on the service side. Changeover between heating and cooling modes is seamless using control valves in split range.

The heat transfer fluid is on the plate side of the shell and plate heat exchanger providing a high film coefficient.

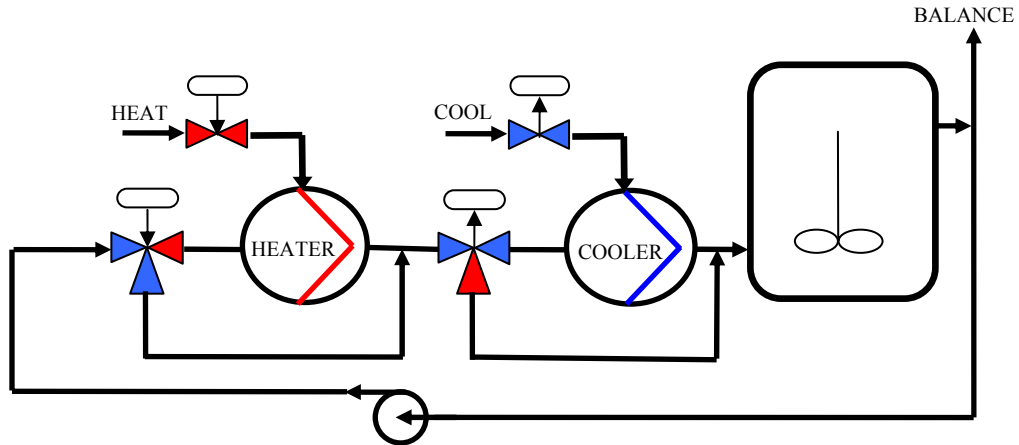
The three way valve at the steam heat exchanger provides fast response bypass control by eliminating the thermal lag. Steam can be applied continuously to the heat exchanger shell at full pressure eliminating problems associated with condensate lift and return, prevents freezing when operating below 0°C and provides excellent linear control characteristics.

These systems require careful consideration to ensure thermal expansion is provided throughout the loop under all operating conditions, eliminating the need for thermal relief valves.

The dynamic simulation plots below demonstrate the excellent thermal response on cooling due to direct injection and use of the three way valve on the heat exchanger. The heat transfer fluid is Syltherm XLT, with an inlet temperature of -40°C, circulating at 23000 kg/h

On a multiple reactor facility this system does not provide complete segregation of the reactor service system from the other reactors. This could result in an extended shutdown of the total facility in the event of water breakthrough due to a single heat exchanger failure. This arrangement requires a high start up cost for the heat transfer fluid charge.

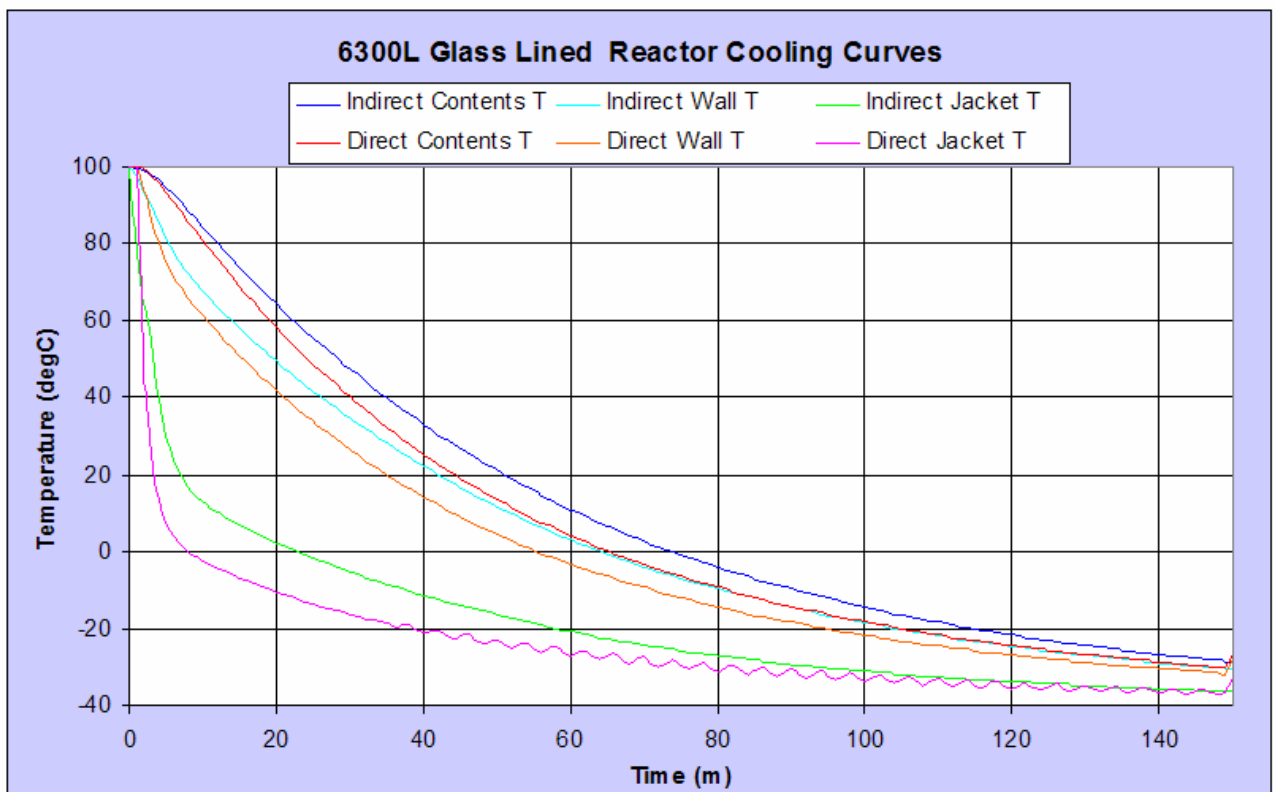
4.3.3 Indirect Jacket Heat / Indirect Cool



The key features of this system are as for the previous system. 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 over a centralised refrigeration facility involving the use of significant volumes of heat transfer fluid.

Thermal response on cool is slower than direct injection due to the added thermal lag of the cooling heat exchanger. These systems require careful consideration to ensure thermal expansion is allowed throughout the loop.

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.



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