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BATCH REACTOR

MODULAR TEMPERATURE CONTROL UNITS

DESIGN FACTORS EVALUATION

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1.0 INTRODUCTION

The stirred batch reactor is used for carrying out reactions, solvent extraction, crystallisation and distillations, frequently in a multi-purpose environment, in the manufacture of a wide variety of fine and speciality chemicals. The temperature control unit (TCU) equipment, configuration and control is an essential component of a successful installation. Adopting a modular construction approach for the TCU provides significant savings in their design and build by standardisation of mechanical, instrumentation and construction techniques.

2.0 JACKET SERVICE FLUID SELECTION

The key determining factor for the process design of batch reactor temperature control systems is the design operating temperature range. This drives the selection of heat transfer fluid, subject to current site practice and minimising the initial fill cost.

Heat transfer fluids must not be used at a temperature above the manufacturers recommended maximum. It is considered good practice to select a fluid with temperature capabilities at least 20°C higher than the required maximum to safeguard against fluid breakdown. Temperature capability of some heat transfer fluids in common use are summarised below.

Common Heat Transfer Fluids Thermal Properties				
Heat Transfer Fluid	Freeze Pt °C	Minimum °C	Maximum °C	Atm BP °C
Dowtherm A	12	15	400	257
Dowtherm J	-81	-80	315	181
Dowtherm Q	-34	-35	330	271
Syltherm XLT	-111	-100	260	172
Therminol 55	-54(1)	-25	290	365
Therminol 59	-68(1)	-45	315	289
Therminol LT	-75	-75	180	181
Transcal LT	-39(1)	-30	250	340
50% EtGlycol	-38.4	-30	>100 with pressurisation	100
Water	0	5		100

Note 1: Pour Point, defined as the lowest temperature at which an oil will continue to flow.

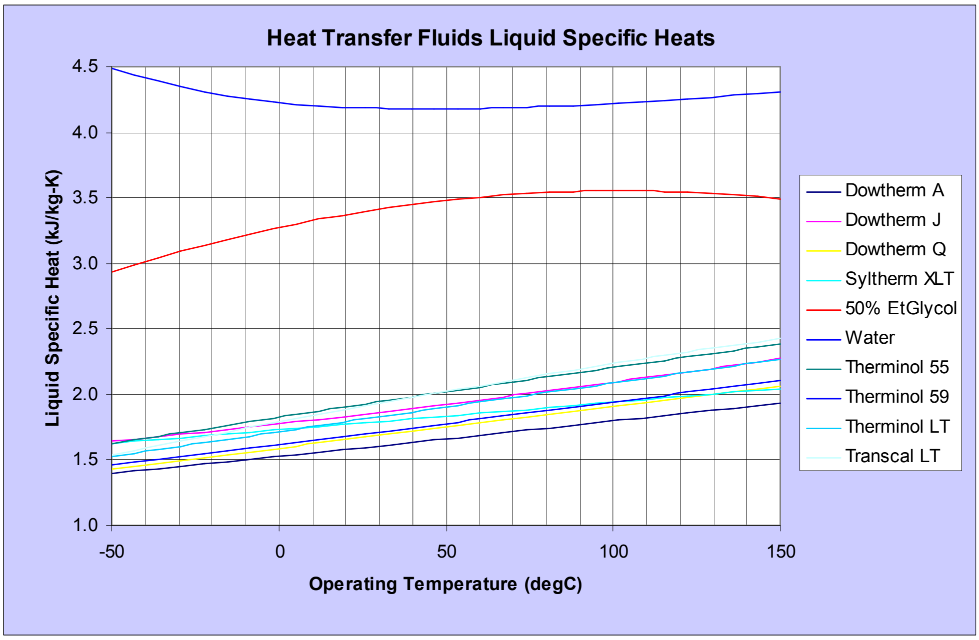
It should be noted that propylene glycol is preferred to ethylene glycol in the food industry due to its low oral toxicity. Since dissolved oxygen causes breakdown of glycol water based systems into organic acids, causing corrosion and fouling, inhibitors are required.

Where possible pressurised systems should be avoided by selecting a fluid with acceptable vapour pressure at maximum operating temperature. This will simplify system design and operation.

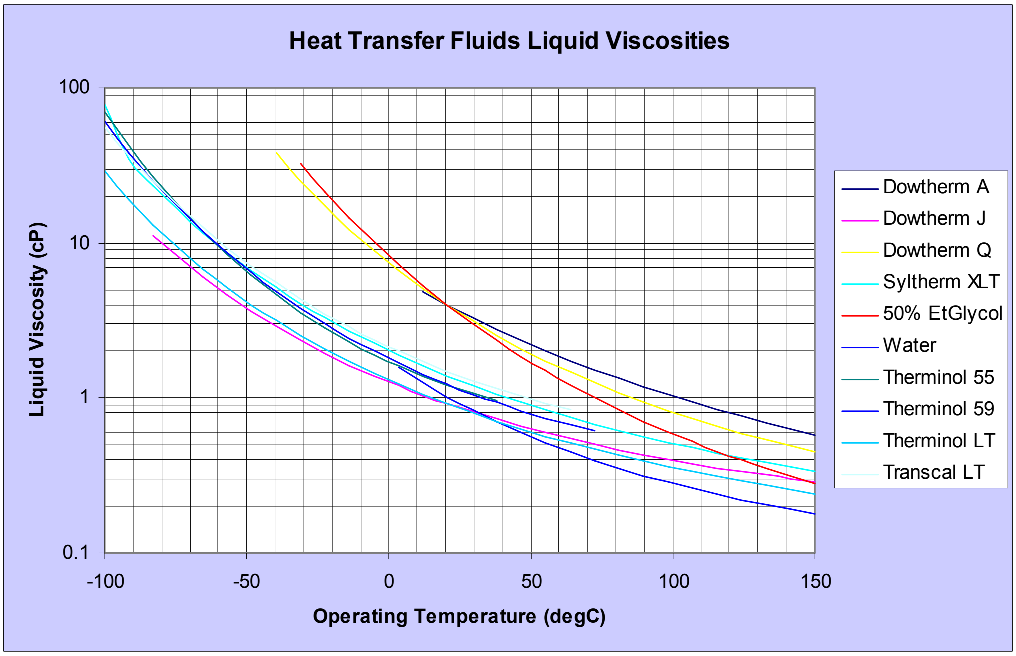
Key heat transfer fluid physical properties are evaluated for suitability over the operating temperature range.

The following plots have been created using CHEMCAD software.

Liquid specific heats for heat transfer fluids vary significantly for water based and organic thermal fluids. The heat removal capability for water is greater than that for organic fluids in proportion to the ratio of the specific heats.



The liquid viscosity throughout the operating temperature range is a key parameter. 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 and acceptable freeze point will allow operations down to -90°C.(5)



The advantages for using heat transfer fluids are:

- 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 of using heat transfer fluids are:

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

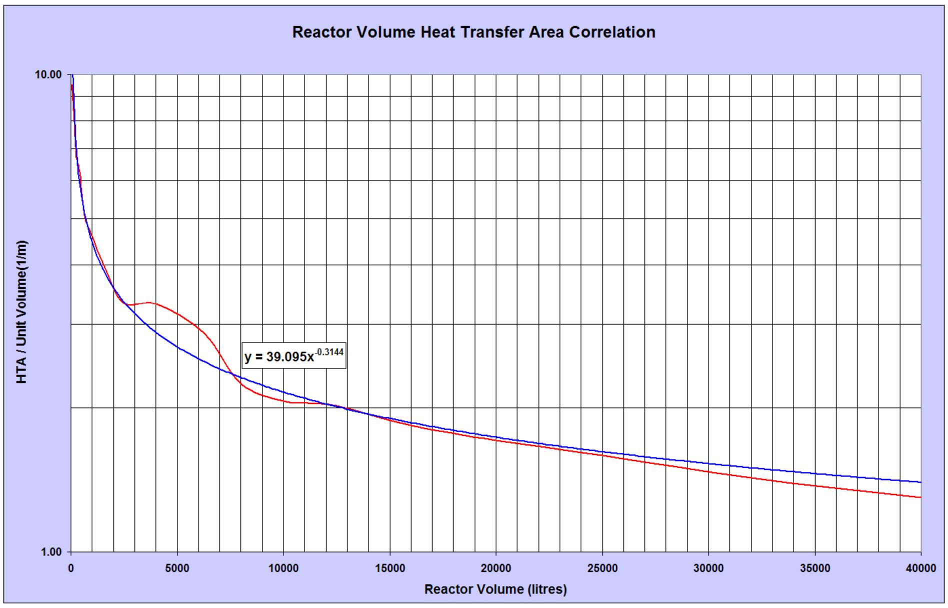
Heat transfer fluids are extremely searching so high integrity equipment and piping specifications are required. Sealless pumps should be used for heat transfer fluid circulation, a minimum flow being required to prevent bearing damage. This can be achieved by installing a restriction orifice in a spillback arrangement. The piping design should use 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.

These systems have to be thoroughly dried out during commissioning to prevent operational problems and equipment damage. This can take days on large installations 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 rectify the problem. At low temperatures water breakthrough will result in freezing leading to loss of circulation and possible equipment damage.

3.0 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. Incorrect selection will result in partially filled reactors reducing the heat transfer area, mixing problems and exothermic reaction instability.



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

For heat transfer the thermal conductivity differences are significant, whereas the density and specific heat differences are not.

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 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.

4.0 REACTOR HEAT TRANSFER (2,3,4)

Stirred batch reactors, with coils or external jackets, have inherent thermal lags (1, pages 75to76) 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.

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 thermal time constants for the different interfaces can be estimated. Typical values for heating 1000 kg toluene in a 1600L Hastelloy C reactor with Dowtherm J fluid are shown:

Resistance	Inside	Wall	Outside	Overall
Time (min)	15.4	3.1	2.6	21.7

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. 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.

When 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 should be 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.

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 will achieve optimum economic heat transfer. Service side circulating pumps are required to achieve acceptable velocities. Jacket inlet circulating nozzles induces a rotational movement similar to spiral baffles and significantly reduces the circulation flow required for efficient heat transfer. The reactor 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.

4.0 Reactor Heat Transfer (2, 3, 4) (Cont.)

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

The heat load on any refrigeration system is to be minimised by using the higher temperature cooling service and then switching to the lower temperature medium when appropriate. Excessive evaporator temperature in the refrigeration loop will result in compressor shutdown and ultimately failure. If any water is present in the system it will accumulate and freeze at the compressor suction.

Boil up and wall temperature may be excessive with direct steam and could require pressure control. Boil up can be limiting with indirect heat transfer fluid systems and can only be increased by increasing the jacket temperature, subject to maximum operating temperature constraints.

5.0 EXTERNAL HEAT EXCHANGERS

Rapid temperature cycling leads to severe thermal stresses in this application. The fully welded shell and plate heat exchanger is recommended, the less expensive brazed construction may experience stress failure. The thermal fluid is usually on the plate side and the service fluid on the shell side. For cryogenic applications the coiled tube heat exchanger is recommended, with liquid nitrogen on the tube side.(6)

The heat duty for heat exchanger sizing is based on the reactor heat transfer area available at maximum operating level.

Inlet and outlet temperature differences, are determined from the services supply and return temperatures and by selecting reasonable heat transfer fluid inlet and exit temperatures at the approach to the services inlet temperatures.

For heating, with steam, the inlet and outlet temperature differences 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 selected to give an economic design subject to providing a heat transfer capability better than the reactor by a reasonable margin.

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 determined by the type of cooling system and its operation.

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.

6.0 TEMPERATURE CONTROL

As the reactor size increases the thermal lag from reactor contents to reactor wall increases and the heat transfer area per reactor volume decreases. Temperature control is characterised by sustained errors between set point and measurement during heat-up and cooldown and by varying thermal responses.

A typical control system uses cascade control with the reactor contents temperature primary controller output being cascaded to the jacket/coil temperature secondary controller set point.

The primary control modes are normally Proportional (P)+Integral (I)+Derivative (D) with P normally set in the range 35 to 50%, the I mode set slower than the overall time constant and the D mode set at I/4. The I mode should only be activated when the measurement is within the proportional band and be set conservatively such that energy is not driven into the process at a rate faster than the process can accept which will result in oscillation.

The secondary control mode is normally a P only controller, as the I mode slows down the response, with P set $\leq 25\%$.

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 I mode to be activated to eliminate offset.

High accuracy temperature measurement should use resistance sensors with Smart transmitters to provide flexibility when setting ranges. Thermal lag associated with the sensor is minimal. However there can be a significant thermal lag associated with the thermowell if incorrectly designed or installed and this can lead to an uncontrollable system. Fast response designs are available and should be used.

The selection of a control valve with the appropriate operating characteristics is essential for satisfactory performance. A valve has an inherent characteristic (relationship between flow and stroke at constant ΔP) and an operational characteristic where the inherent characteristic is modified by the process pressure conditions. An equal % valve operating characteristic tends towards a linear characteristic as ΔP max/ΔP min increases. A linear valve operating characteristic tends towards a quick opening characteristic as ΔP max/ΔP min increases.

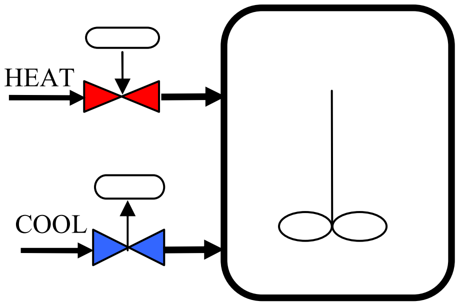
For temperature control the valve characteristic normally used is equal % though situations may arise where a linear characteristic provides better control. The operational characteristic of a valve can be modified by controller output signal characterisation.

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.

7.0 JACKET / COIL SERVICES MODULE CONFIGURATIONS

- **Direct Heat / Direct Cool**

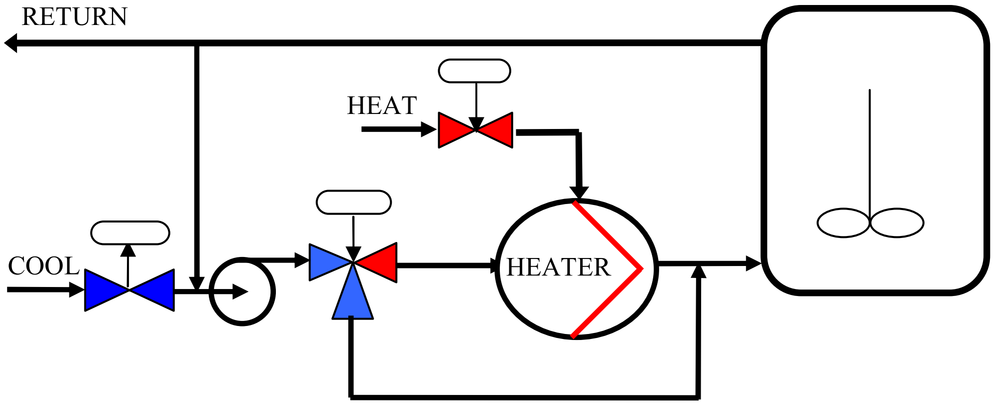
The appropriate supply and return services are connected directly to the reactor jacket/coils. Temperature ranges from –20°C to 180°C with water, steam or ethylene glycol/water can be used with pressurised systems. Arrangements vary from total manual to fully automatic and include forced circulation with steam/water mixing facilities. Combined heating/cooling facilities require automatic valve sequencing and jacket/coil blowdown routines when changing services. This configuration exhibits good thermal response. Operational problems include cross contamination of services, jacket fouling, corrosion, thermal shock problems with glass lined equipment and possible product problems with high wall temperatures.



- **Indirect Jacket Heat / Direct Cool**

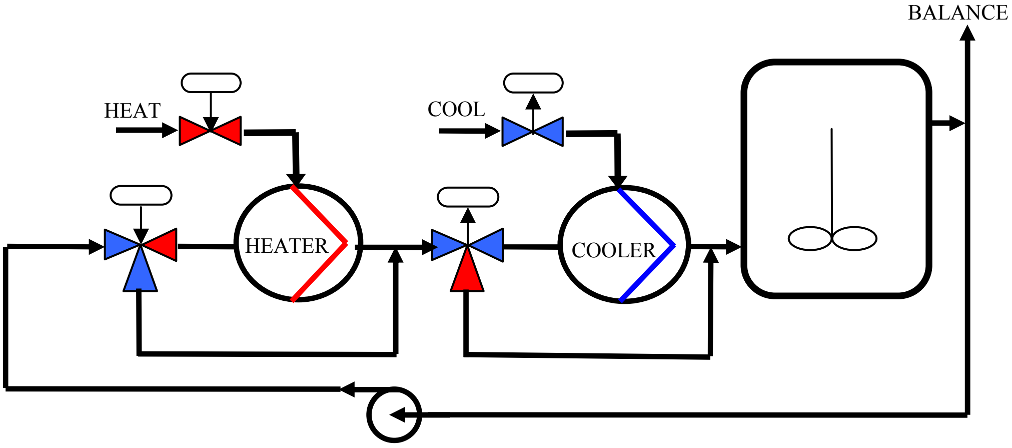
A single heat transfer fluid is used with the coolant being injected into the reactor circulating loop. Heating is provided by a heat exchanger with steam on the service side. Changeover between heating and cooling mode is seamless using control valves in split range.

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.



- **Indirect Jacket Heat / Indirect Cool**

This is probably the most common arrangement. The three way valve at the steam heat exchanger provides fast response bypass control by eliminating the thermal lag associated with the heat exchangers.(1) 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.



Thermal response on cool is slower than direct injection due to the added thermal lag of the cooling heat exchanger. The cooling heat exchanger allows for 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. 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.

8.0 DYNAMIC SIMULATION

The operation of reactor systems throughout the manufacturing process cycle involves a complex interaction of many fluid physical properties and process parameters. To evaluate system performance under all anticipated operating conditions dynamic simulations are required. The simulation results will validate the design, allow start-up validation protocols to be established and enable production scheduling to be carried out with some confidence. Several software packages are available. CC-ReACs simulation software by Chemstations of Houston is specifically designed for this application.

9.0 REFERENCES

1. F.G. Shinskey, “Process Control Systems”, M^c Graw Hill, 1967.
2. F. Bondy and S. Lippa, “Heat Transfer in Agitated Vessels”, Chemical Engineer, 1983.
3. J.E.Edwards, “Process Modelling & Control of Batch Reactors in the Fine Chemicals Industry”, UK CHEMCAD Seminar 2000.
4. Pfaudler Balfour, “Agitating Nozzles for Jacketed Reactors” Data Sheet DS65-700-1 and Bulletin 235-2e.
5. J.Lines, “How Low Can You Go?”, Chemical Processing, January 2003.
6. *R.E.Athey, L.M.Saupe, “Convective Flow in Coiled Tubes”, Graham Manufacturing.*

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