

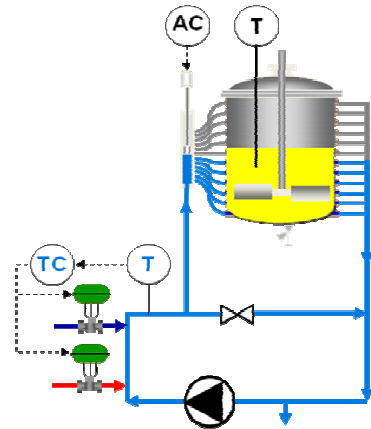
COFLUX Control

➡ Sensitive calorimetry and improved control

A conventional batch reactor relies on jacket temperature to regulate heating and cooling power.

Coflux batch reactors have segmented jackets and an additional valve to regulate the number of jacket segments in service.

This arrangement allows the user to dynamically change the heat transfer area.



The operator can use this facility to set the jacket height to a desired level or to vary the jacket area dynamically in order to optimise the reactor for control and process monitoring. Despite the simplicity of this concept, it transforms the capabilities of batch reactors in terms of temperature control, on line monitoring, energy efficiency and heating/cooling power.

➡ Prevent product damage from dry wall effects

Product splashing on to heated or cooled dry surfaces (above the liquid) can be subjected to rapid and extreme heating or cooling. This is particularly undesirable for temperature sensitive compounds or temperature sensitive processes like crystallisation. The traditional solution to this problem is to use a split jacket and switch off the upper jacket section when the vessel is less than full. Identifying the ideal jacket split point however is always the problem as batch reactors are used at different or even changing fill levels.

The Coflux jacket offers 20 or more separate operating levels. This allows the optimum jacket height to be selected irrespective of product level in the tank.

➡ Improve yield and quality with improved temperature control

Large conventional batch reactors suffer from sluggish temperature control. This is due to the high average residence time of the heat transfer fluid in the jacket (which can be anywhere between 30 seconds and 10 minutes).

The residence time of heat transfer fluid in the Coflux jacket is generally less than 3 seconds. This gives a 5000 litre Coflux reactor comparable control dynamics to a 1 litre lab reactor. This is not only desirable from a control perspective but also simplifies scale up.

► Protection from jacket hot/cold spots

The hottest (or coldest) zone in a reactor jacket exists where heat transfer fluid is injected. Conventional jackets rely on transient additions of 'over temperature' or 'under temperature' heat transfer fluid to improve control response. Although this reduces response delay it also creates transient hot or cold spots at the jacket inlet points. This is particularly undesirable for processes like crystallisation or where handling temperature sensitive materials. The Coflux jacket has a natural response speed which is up to 2 orders of magnitude faster than a conventional reactor jacket. This allows very fast response with negligible use of 'over temperature' or 'under temperature' heat transfer fluid. The operator can take this philosophy a step further by fixing inlet temperature to the jacket and using heat transfer area as the control parameter. Under these conditions, 'high gain' control strategies have no impact on hot or cold spot formation.

► Reduced energy consumption

Traditional batch reactors maintain high jacket flow irrespective of process heat load. High flows are necessary to prevent erratic distribution of heat transfer fluid in the jacket. This practice is not only wasteful in terms of pumping energy but the pumping energy has to be taken out by the chiller system when vessel is using cooling fluid. A second cause of inefficiency relates to cross mixing between hot and cold heat transfer loops. Conventional reactor jackets have large inventories of heat transfer fluid. This causes substantial cross mixing between the hot and cold loops when duty cycle switches between heating and cooling. Under some control conditions this can be extremely energy wasteful.

Coflux jackets use heat transfer fluid on demand. As demand falls, the jacket area is reduced and the flow of heat transfer fluid falls to a trickle without succumbing to erratic flow. Over a normal process cycle, the pumping energy is substantially reduced. The reduced jacket volume and improved control capabilities of the Coflux jacket also reduces cross mixing between hot and cold loops by between 90 and 99% on large vessels.

► Process Analytical Technology (PAT)

Heat balance measurements can be performed on a Coflux jacket with unprecedented accuracy and sensitivity.



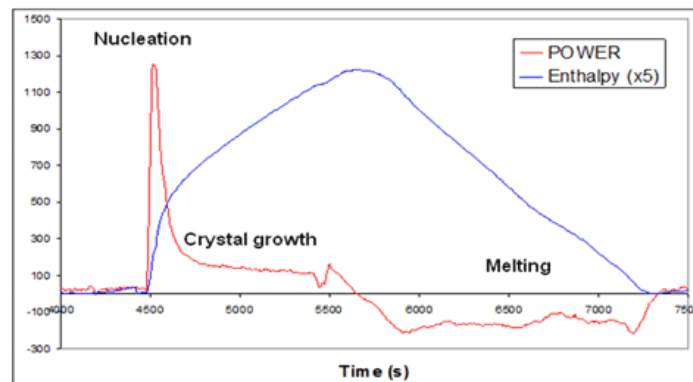
There are three reasons for this:

- Good temperature control is essential for accurate heat balance measurement. Coflux offers unmatched temperature control performance in terms of stability and speed of response.
- Coflux control eliminates the need for oscillations in Jacket temperature (since it offers the option of area control). This reduces noise to signal problems by orders of magnitude.

- The area of the Coflux jacket can be reduced to maintain a measurable heat balance temperature even with very low process heat outputs. This allows very sensitive heat balance measurements.

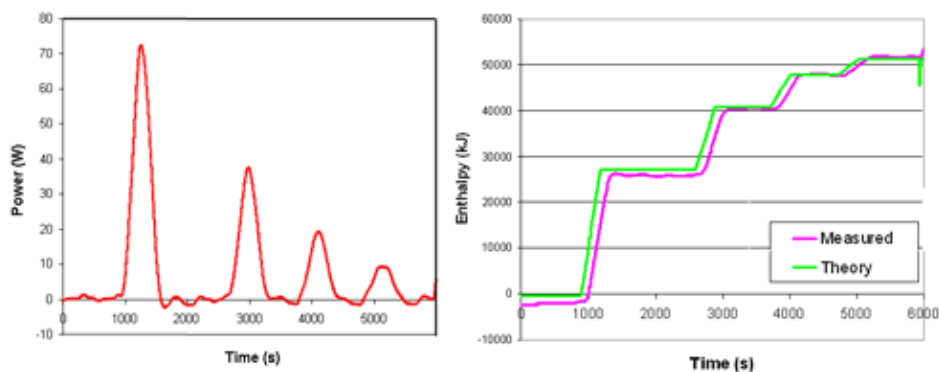
Heat balance measurement is a powerful process analytical technique that requires virtually no calibration or setup. The user only has to turn on the heating or cooling system to monitor the process.

➡ Improve crystallisation control with direct measurement



Ice formation and melting in a Coflux reactor. The rate and mass of crystals formed can be measured in real time throughout nucleation and crystal growth. No pre-calibration is required and any crystal deposits on the vessel wall have almost no impact on measurement. These measurements allow the operator to control the process within the meta stable zone by direct measurement. This eliminates guess work and delivers better quality and consistency of product. In the experiment shown above, ice crystals were re-melted at 5500 seconds. Coflux permitted the melting process to be tracked to completion.

➡ Improved control of chemical reactors



Heat measurement can be used to monitor and control chemical reactions. In the figure above, 4 different quantities of acetic anhydride were added to water. Heat

balance calorimetry was used to monitor the rate and progress of this of the reaction. This information gives the operator real time information on the progress of the reaction.

Achieving optimum yield and quality of batch chemical processes relies on using optimum process methods. The optimum process method however can be influenced by factors which can vary from one batch to the next. Coflux heat balance measurement provides a tool for more robust control methods. It can be used to control accumulation during addition reactions, for end point detection, pH control, catalyst addition, general process monitoring etc. The benefits of this are better process cycle times, improved quality and yield, reduced consumption of raw materials and safer process methods.

Improved heating and cooling capacity

It is generally assumed that the limitation for heating and cooling in a batch reactor is determined by the heat transfer coefficient. This is only true to the extent that a high process side film coefficient improves the product's tolerance to more extreme jacket temperatures. Such tolerance however is of no value unless the user is aware of it and can increase (or reduce) the jacket temperature to exploit this.

Ultimately, the maximum heating or cooling rate is determined by the product's tolerance to hot or cold heat transfer surfaces and this is heavily influenced by the prevailing process conditions. For example, a well mixed fluid with low viscosity and high thermal conductivity can tolerate jacket temperatures of 300C above the safe product temperature limit. A fluid with high viscosity and low thermal conductive by contrast can suffer damage with jacket temperatures of less than 30C above the safe product temperature limit. Thus the maximum heating or cooling power can vary by a factor of a 100 or more according to process conditions. In many processes, the prevailing conditions often change drastically with variations in viscosity, composition and level.

There are 3 key requirements to achieving the maximum heating or cooling power in a batch reactor:

1. The heat transfer conditions should be uniform. If not, localised zones will suffer too much or too little cooling. Uniform heat transfer requires uniform velocity and temperature of heat transfer fluid over the whole jacket area.
2. There should be no heating or cooling of dry walls. Product exposure to dry walls will cause severe product damage.
3. The user needs real time information about wall temperatures. Such measurements are not possible by direct measurement since any temperature sensor mounted in or on the heat transfer surface will change the local temperature conditions.

Coflux jackets solve all of these problems. The multi channel jacket delivers very uniform velocity and distribution of heat transfer fluid. The jacket height can be set to change dynamically in response to the changes in product level within the vessel.

Coflux® Batch Reactor System

The wall temperatures can be inferred from the rate of heat flux and the temperature difference between the product and the jacket. This allows the user to continuously monitor wall temperatures and constantly adapt the jacket temperature to give good heating/cooling without damaging the product.