Evaluation of Reactor Performance: Triple vs Double Walled jacketed reaction vessels.

Setting the scene:

Asynt ReactoMate jacketed reaction systems are regularly specified for low temperature chemistries by our customers. Often the highest cost for a reactor system that is going to sub-ambient temperatures is not the reactor and support system, but the circulator itself. The lower the desired minimum temperature of the reaction, then the more power that is needed to cool the reactor and therefore lower temperatures will require a more expensive circulator to be specified. Another issue encountered with single jacketed vessels at temperatures below 0° is the formation of ice which can prevent good visibility of the reactor contents. An option from Asynt is to specify our reaction vessels with a vacuum jacket, whereby an additional wall of glass allows us to insulate the oil jacket and vessel contents with a vacuum during manufacture.

Objective:

In these experiments we wanted to show the real difference between two identical sized reactors utilising the same circulator system. We chose the lowest powered Presto circulator system from JULABO, the A30, and used this in conjunction with our ReactoMate 5000ml vessels.

Equipment:

**JULABO Presto A30
M16 JULABO insulated hoses
Asynt no restriction, no leak, quick connect M16 vessel fittings.
*Asynt 5 litre jacketed reactor
*Asynt 5 litre vacuum jacketed reactor
ReactoMate Super Support system.
IKA RW60 Control overhead stirrer and PTFE turbine stirrer
PTFE Reactor lid.
PTFE PT100 Baffled temperature probe for solution temperature measurement/control
PTFE Baffles

Environmental conditions:

Ambient temperature:	26 °C
Humidity:	86%

Experimental:

Each reactor was set up with 3000ml of ethylene glycol as the reactor contents.

The JULABO Presto A30 and the reactor oil jackets were filled with HL60 heat transfer fluid and the vessels connected directly to the circulator and not through any additional connectors such as drain down manifolds to maximise performance. The Presto A30 was set in EXT (external control) to achieve the lowest possible temperature.

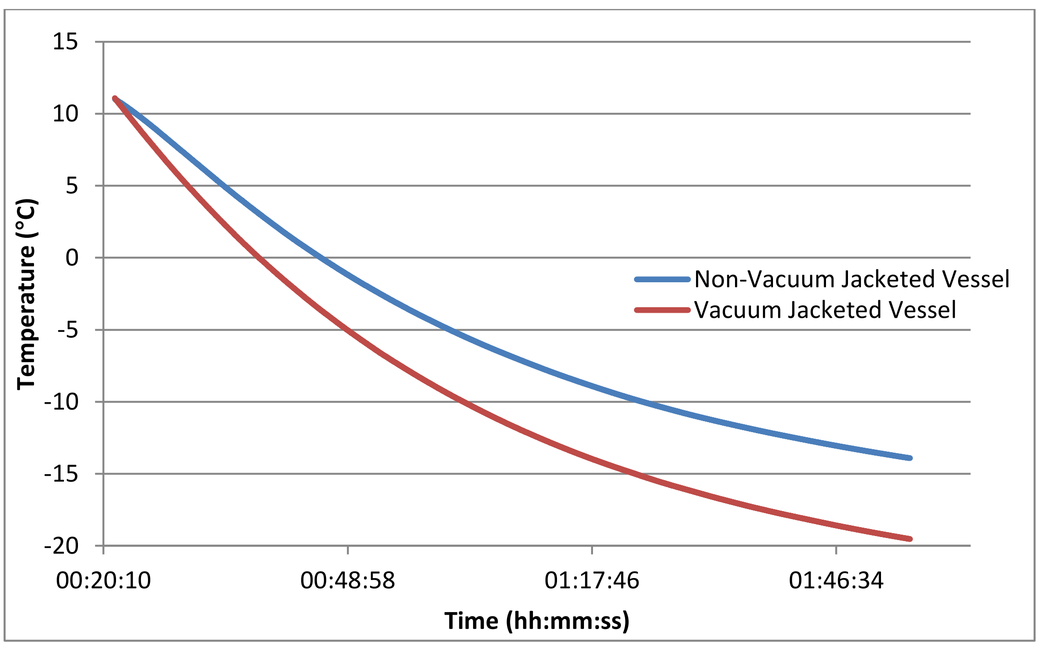
Data:

5 litre jacketed reactor:

The Presto A30 achieved the lowest solution temperature of -15.0 °C

5 litre vacuum jacketed reactor:

The Presto A30 achieved the lowest solution temperature of -20.0 °C



The graph above was made from the point when both solution temperatures were equal.

Further Observations:

Pictures 1-4 were taken at the conclusion of each experiment, with the reactors at their coolest, and show the degree of ice formation on the exposed surfaces.

Pictures 1-2 show the complete coverage of the jacketed reactor vessel in a layer of ice.

Pictures 3-4 show the vacuum jacketed vessel. Ice formation can be observed on the surface of the connector (as shown in picture 4) however only light condensation is seen on the reactor body due to the vacuum insulation inside.

Picture 1 : 5 litre jacketed reactor and Presto

Asynt Ltd 29 Hall Barn Road Industrial Estate Isleham Cambridgeshire United Kingdom CB7 5RJ

T: +44 (0)1638 781709 F:+44(0)1638 781706 sales@asynt.com www.asynt.com

Registered office: Eldo House, Kempson Way, Bury St Edmunds, Suffolk, IP32 7AR Registration No: 5160407

VAT No: GB 838 5592 82

Picture 2: 5 litre jacketed reactor

Picture 3: 5 litre vacuum jacketed reactor and Presto

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Picture 4: 5 litre vacuum jacketed reactor

Conclusions:

A vacuum jacketed reactor does indeed offer a significant performance advantage over the standard jacketed vessel, allowing for not just faster cool down times, but also lower temperatures in the order of 5 degrees Celsius.

Furthermore, the use of a vacuum jacketed reaction vessel offers another important benefit for the synthetic chemist, one that can clearly be seen in the four photographs - the ability to still observe the chemistry under test.

Finally, the JULABO Presto A30 offers a strong solution for sub-ambient synthetic chemistry on the 5 litre scale allowing for experiments to run down to -20 °C, depending on reactor configuration and setup.

*Both of the vessels used were of a squat design, each with a 200mm internal diameter fitted with Schott type flanges.

** The Presto A30 has the below cooling power.

Cooling capacity (Medium Ethanol)

°C	200	100	20	0	-10	-20	-30
kW	0.5	0.5	0.5	0.4	0.3	0.2	0.05