SCI LECTURE PAPERS SERIES

PILOT HYDROGENATION PLANTS – DESIGN AND APPLICATION

R Cook
Whale Bridge Park, South Newbald, York YO43 4SU, UK

ISSN 1353-114X
LPS 0092/98

Key words *hydrogenation, pilot, reactor, loop, interesterification, eductor*

Background

Last year one of Ebortec's Malaysian clients asked us to assist them by designing and fabricating a miniature hydrogenation plant to his specific requirements.

Historically the client operated dead end batch equipment, but had just installed a new loop reactor. He needed a pilot plant that would accurately reproduce the performance of the loop reactor, which had a capacity of ten tons, as well as the smaller traditional units.

He looked at the equipment available on the market but decided that there was nothing that would fully meet his needs, hence his request to Ebortec. The specific requirements were as follows.

- Reliable reproduction of the performance of his industrial reactors.
- Independent operation.
- Variable batch size from 25-50 litres.
- Accurate hydrogen gas measurement.
- User friendliness.

Other aspects which were also taken into account were the potential benefits as a training tool, mobility of the unit, and, of course, safety in operation.

Design concept

Mixing of oil and gas was achieved using the eductor nozzle principle, whereby a high velocity stream of hot oil, containing catalyst generates a partial vacuum, draws in the head space of the reactor and creates very effective mixing.

In addition the collecting tube section was designed in such a way that it produces rotational agitation in the reactor.

When considering the performance of the plant the concentration of hydrogen gas on the surface of the catalyst is of primary concern. This in turn is a function of the following variables:

- The catalyst type

- Agitation (nozzle pressure)
- Absolute head pressure
- Partial pressure of hydrogen gas

All of the plant conditions had to be adjustable over an appropriate range to ensure full compatibility with the industrial plants. Therefore provision was made in the design for accurate adjustment of these parameters, including the use of nitrogen gas to reduce the partial pressure of the hydrogen in the head space, in exactly the same way as an industrial plant.

Elements of the pilot plant

Temperature control equipment

The equipment to control the operating temperature was carefully selected. We considered many options, including direct electric heating and steam heating, before finally selecting a separate electrically heated thermal unit. The one chosen was a modified version of equipment used in the blow moulding industry to control the temperature of the mould head. It has its own PLC control unit, and, with minor modifications, could monitor and control the temperature of the reactor using a signal from a resistance thermometer installed in the reactor, or the temperature of the thermal fluid itself. The unit was ideal because it could be used to cool as well as heat the reactor, allowing very accurate control of temperature and removing the exothermic heat of reaction in a similar way to industrial scale plants.

Reactor construction

The reactor was designed to BS5500 and constructed in stainless steel 304L. The unit was fitted with an external heating jacket, so that there would be no risk of heating fluid contamination and the inside of the reactor was left clean. Oil circulation through the reactor was achieved by a 1.85 kW centrifical pump, which was specifically selected for the duty, because of the low NPSH, the high temperature and abrasive nature of the catalyst. The circulation rate was designed for a mean of 50 litres/minute with a ± 50% range achieved by adjustment of pressure differentials across the nozzle.

Vacuum system

The vacuum system incorporated an industrial liquid ring pump providing a vacuum of 60 TORR with water at 32^{o}C (typical for Malaysia), lower pressures could be achieved if necessary, but would require the use of chilled water, or a two stage vacuum pump.

Filtration equipment

Although a simple bag filter would have been a suitable option for the unit, our client particularly wanted a traditional plate and frame filter press using paper and cloth filtration media. Johnson Progress were able to meet this requirement and supplied the smallest unit in their range, with a plate size of 250 mm^2. The units are available in cast iron, stainless steel or polypropylene.

Assembly

The whole plant was assembled on a stainless steel frame on castors, with stainless steel pipework and valves, and insulated to the same standard as an industrial plant.

Hydrogen gas measurement

When carrying out process development work, accurate end point control is essential. After considerable investigation we finally opted for one of the most reliable yet simplest methods i.e. a fixed volume gas accumulator with an accurate pressure gauge. This was coupled with pressure control equipment and a visual gas flow indicator. By reference to a calibration chart the client is able to control the gas usage to within ±1%. Higher accuracy can be achieved by use of an electronic gas pressure transmitter and digital readout.

Safety

Because hydrogen gas is highly explosive, both the skid mounted motor, and all the associated electrical systems, were designed to BS5345 (Zone 2 by Health & Safety definition). The thermal heating and cooling unit was designed to operate outside the Zone 2 envelope, pumping the heating fluid into the reactor using its integral pump unit.

Operation

This pilot plant has now been in operation for almost a year, and the client has expressed complete satisfaction especially with the following aspects:

(a) Its user friendliness, particularly for laboratory staff, who do not have plant experience.

(b) Its reliability. The unit was supplied with spare pump seals and other spares, but none has been used to date.

(c) Its reproducibility. From the first experiment the client found that he could produce products, which could then be accurately mimicked in his industrial plant.

From an operational perspective the last point is the key objective. To produce small parcels of products, to test out catalysts, make new products, modify existing products and to transfer the parameters to an industrial plant and produce the same products is a major benefit to the client. It avoids major plant downtime and serious problems in disposal of out of spec products.

Preliminary trials were conducted in the UK. Using standard catalyst levels and refined rapeseed oil, we found that typical rates of reaction were in line with those obtained on industrial loop reactors, with typically 30 minutes to reduce IV to ~ 60IV. If anything reaction rates were too fast, and the gassing rate had to be reduced by restricting the rate of circulation.

The client has reported similar performances in the field with excellent reproducibility on his industrial units including catalyst usage rates.

 ISSN 1353-114X

Why invest in a pilot plant

Quite simply it saves money, a lot of money. If we take a typical industrial unit producing 100 tons/day, the capital cost of the plant will be ~£2.0M.

Based on an annual use of 7000 hours, the cost of finance and depreciation = £30/hr. Add to this the cost of staff/management -£40/hr, and the cost of utilities/services -£10/hr. This gives a total of -£80/hr.

Experiments on a full size plant will be slow. The plant will need to be drained and cleaned. Laboratory control will lead to extra down time. It is easy to see how each trial on a full size unit can cost ~£1000 in plant downtime. Add to this the cost of disposal or downgrading of the products made, and one can immediately see how a pilot plant can pay for itself in a very short time.

Other developments

Ebortec is currently building a slightly larger plant for a client who requires a dual purpose plant for interesterification and hydrogenation experiments.

Another variation under review uses the suction created by the eductor nozzle to carry out other experiments involving condensation of gaseous compounds.

There is also a potential for directed interesterification reactions by selectively condensing volatiles such as methyl esters from the head space. An in-line condenser could also be used to produce methyl esters on a fractional basis, or can be used for other condensation and separation reactions.

Currently Ebortec is planning to produce a unit for hire for companies to conduct experiments in their own laboratories.

ISSN 1353-114X