AIR PRODUCTS

Knowledge Paper

Making solvent recovery more cost-effective

This paper is published to encourage the sharing and transfer of knowledge.

Charles Monroe works as an independent consultant for both Sapio in Italy and Air Products in the UK.

Making solvent recovery more cost-effective

Recovering solvents reduces emissions and cuts down raw materials costs. Charles Monroe describes an innovative recovery system that uses liquid nitrogen to condense solvents.

Using liquid nitrogen for solvent recovery and emission abatement languishes under the perception of being uneconomic and impractical: indeed, the technology appears to have been pushed to the sidelines. Yet there are benefits from using a condensation process so that implementation can be a proactive plan to enhance the manufacturing process and improve profitability. Meeting environmental legislation does not have to be an implementation programme of last resort to meet inspection requirements, but a programme to improve the profitability of a process.

Reducing solvent emission and meeting environmental standards is often viewed as an externally imposed cost that reduces profitability. This is often the case where abatement technologies shift the disposal problem, or merely treat the off gas to remove solvents, without offering any process advantages. In contrast, liquid nitrogen-based condensation technologies can improve the profitability of a process, with any or all of the following benefits:

- Complete recovery of the solvents with no contamination from any other intermediate fluids;
- Emission standards met for virtually every solvent;
- The liquid nitrogen used for the process cooling is uncontaminated and can be recovered and used in the factory for inerting, thus reducing operating costs;
- Process dynamics can be improved by, for example, utilising the powerful cryo-pumping action of liquid nitrogen or operating the process under the inert conditions of nitrogen gas.

Compare these advantages with the effects of other technologies. Incineration, for example, cuts solvent emission, but in a fundamentally negative manner by destroying the solvents, creating combustion products which in themselves are a source of environmental concern and subject to reduction programmes. The absorption technology of water or acid scrubbers merely shifts the problem: solvents are trapped in the working fluid of the scrubber which itself has to be treated as wastewater. Adsorption technologies can recover the solvents, but offer no additional advantages to the process.

To understand the benefits of solvent recovery using liquid nitrogen condensation, it is necessary first to examine the principles of this technology.

Emission abatement by liquid nitrogen relies on condensing and even freezing the solvents. In more specific terms, as the temperature of a gas is lowered, the quantity of solvents that can remain in the gas phase decreases. The excess solvents change phase into liquid droplets or even frozen solids and, as particles, they can be stripped from the gas stream by phase separators. The enthalpy for this cooling is supplied by liquid nitrogen undergoing the reverse process: boiling at -196°C and warming to ambient temperature. Heat transfer occurs in a heat exchanger designed for the process.

The low temperature of liquid nitrogen offers two significant advantages. It is possible to recover practically every known solvent at levels in excess of 99%, and reach discharge concentrations of the process gas in the region 10mg/m^3. It is clear to see the limitations of abatement using mechanical refrigeration, and the unlimited potential with liquid nitrogen. Additionally, the process gas cools very rapidly and, if there are large quantities of solvents condensing, the volume contraction of the solvents changing phase from liquid to gas on the liquid nitrogen surfaces gives a powerful pumping action – an effect commonly known as cryo-pumping – which can accelerate a process otherwise limited by the installed pump capacity.

Nitrogen has another significant property: it can be considered as inert for the majority of processes. Therefore the design need not consider flammability levels or Lower Explosive Limits which push up volume flow rates, as is the case with air, where ever higher flow rates give greater dilution and lower or safer concentrations.

In contrast, the nitrogen used for condensation can be returned to the process, thus avoiding contamination by oxygen or moisture in the air. The same quantities of solvents can be carried safely at high concentrations so the total process flow rates are lower. Not only is the process equipment smaller, but it also becomes easier to meet the discharge levels, and the running costs of the abatement system become more favourable.

Solvent recovery by condensation

The particular heat transfer characteristics of liquid nitrogen and the build-up of frozen solvents calls for careful design and selection of heat exchangers. In some applications, it is possible to design the heat exchanger so the surface in contact with the process gas remains above the freezing temperature of the solvents, even with the liquid nitrogen boiling at temperatures which may be 100º lower. Where this is not possible, additional surface area and clearance is built into the heat exchanger to allow for the fouling of frozen particles during the duration of the batch. If the process operates continuously, there are two heat transfer paths operating alternately between the cycles of condensation and regeneration.

The main objective in the design of the system is minimum nitrogen consumption, since this often becomes the dominant factor in the economics. As a norm, the heat exchangers are designed to use up to 95% of the available enthalpy. If there is a high flow rate of incondensable gases leaving the liquid nitrogen-cooled heat exchanger, a pre-cooler heat exchanger can be installed to recover this cooling power. Yet the most dramatic savings can come from reducing the air or incondensable flow rate by using a lower flow of nitrogen gas.

It is accepted that nitrogen systems have high running costs, but these should be offset against the following:

- Comparatively low investment costs;
- Effective reduction in costs when there is an existing secondary use for nitrogen gas in the factory;
- The benefit of recovering solvents in pure form for re-use;
- Hidden costs of secondary disposal of solvents;
- Higher plant maintenance costs for other technologies: the sum total of the moving parts for a nitrogen-based system is the valves;
- Higher power consumption for other technologies, sometimes exceeding supply thresholds in the factory. With such considerations, the total life cycle costs for a nitrogen system can easily be the lowest.

There are numerous systems for solvent recovery using condensation technologies. These have utilised mechanical refrigeration, liquid nitrogen condensation and membrane technologies to provide the optimum combination to meet customers' needs. The technical expertise in the supply of such systems has been developed by Air Products with its Italian partner Sapio, and in collaboration with an innovative German subcontractor, Herco Kiihltechnik. The systems are marketed worldwide under the name Cryo-Condap. Herco Kiihltechnik has brought the benefits of a small but flexible and innovative manufacturing company with an established reputation in refrigeration technology to the worldwide selling capabilities of Air Products.

All three companies worked together to develop software for the complex process analysis needed to design such equipment. In this way, the design capability can be distributed to sales engineers who can discuss a customer's requirements to achieve the desired balance between investment and running costs. Their ability to present a complex problem in a clear and concise manner can instil a great sense of confidence in the potential customer.

The technology has been applied across a diverse range of different industries. There are a number of recovery systems on drying ovens for coating lines. In the past, a large flow rate of air with low concentrations of solvents was discharged to an incinerator or even dumped directly into the atmosphere. This was replaced with a recirculating flow of nitrogen gas, carrying a higher concentration of solvents, which is treated in a recovery system which removes the solvents. Gas curtains are incorporated at the inlet and outlet of the drying oven so the product can pass through continuously. In such a way, the burden of increasing environmental legislation pointed the way to lower overall operating costs as the solvents were recycled.

Several systems have been sold to the medical sector for the recovery of ethylene oxide used to sterilise medical instruments. The very properties that make ethylene oxide effective for sterilisation also make it toxic and, if mishandled, liable to spontaneous explosion. Other technologies have used incineration or scrubbers (requiring wastewater disposal) to simply meet the emission standards. The Cryo-Condap system uses a combination of molecular sieves to remove moisture, and then condensation to recover ethylene oxide for reuse. The high value of the ethylene oxide provided rapid pay-back.

The technology also finds diverse applications in the chemical industry, where the flexibility of liquid nitrogen systems, low investment costs and the opportunity to reuse the nitrogen create significant opportunities.

5

Zanchetta Roto P processors

An example of a successful application can be found in pharmaceutical processing. Early in 1996, Zanchetta approached Sapio with a request to use the technology to improve their Roto P pharmaceutical processors. Customers were demanding a solution to meet ever-tightening environmental legislation, and some were following the route of water-based granulation. Zanchetta recognised the need for a standard range of equipment, and claimed effective solvent recovery with liquid nitrogen would have less environmental impact than water-based granulation.

The manufacturing process consists of four steps: mixing the ingredients; granulation; drying; and cooling. In the drying process, which can take up to 1 hr, the solvents are removed under vacuum. The existing process used a mechanical chiller to condense 40-60% of the solvents; the remainder were trapped in the working fluid of a liquid ring pump. There were no gas emissions, but at the cost of wastewater treatment.

Sapio replaced the water-cooled condenser with a purpose-built liquid nitrogen cooled condenser. In collaboration with Zanchetta, it undertook a development programme with the following objectives:

1. Recovering >99.5% of the solvents;
2. Replacing the liquid ring pump with a smaller dry pump;
3. Supplying nitrogen exhaust gas for reuse;
4. Reducing the drying time.

The equipment uses a single heat exchanger in a shell, the base of which includes the condensate tank. A programmable logic controller manages an automatic cooldown to the required operating temperature in 15min, and indicates when the plant is cold and ready to treat the process gas. The process is cooled on the heat exchanger to a temperature as low as –120°C, depending on the solvent in question. Solvents are stripped from the flow as liquid and solid. Small quantities of incondensable gases used to assist the drying process are allowed to pass through to the vacuum pump at 25mbar pressure absolute.

Trials have been undertaken with a prototype system on a range of different Zanchetta processes. Recovery levels of 99% have been reached with ethanol, and 95% with quantities of dichloromethane beyond the original design specification. Currently, a range of standard designs to match the range of Roto P processors are being prepared. The development programme is being continued to pursue the secondary advantages.

There is a common prejudice that the future lies in water-based processes. With the Zanchetta granulator, this creates a bigger environmental burden in terms of wastewater disposal and the energy needed to dry the solvents. On the other hand, liquid nitrogen-based condensation technologies can achieve recovery levels which create the smallest environmental impact, and can offer process advantages to the customers with low running costs, improving the overall profitability of the process.

Liquid nitrogen condensation technologies for solvent abatement are not confined to special applications. It is an established technology that can meet emission standards while bringing operating advantages to a process. The obvious benefits are the value of the recovered solvents but, additionally, there can be the reuse of nitrogen in the factory plus an enhanced process. Users have found the burden of meeting emission standards has, in reality, become a step to increased profitability.

AIR PRODUCTS

For more information

If you would like additional information, write or call Air Products at the location below, or visit our website.

UK and Europe
Air Products PLC
2 Millennium Gate
Westmere Drive
Crewe CW1 6AP
UK
Tel 0800 389 0202
Fax 01932 258 502

Americas
Air Products, Inc.
7201 Hamilton Boulevard
Allentown, PA 18195-1501
USA
Tel 610-481-4911
Fax 610-481-5900

Asia
Air Product Asia, Inc.
Suite 6505-7, Central Plaza
18 Harbour Road
Wanchai
Hong Kong
Tel 852-2527-1922
Fax 852-2527-1827

tell me more
www.airproducts.com

331-08-001-UK