Operating Cost Benefits of Oil-Free Vacuum
for Mini-Plants and Kilo Labs

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Introduction

Historically, mini-plants have utilized oil-sealed rotary vane vacuum pumps as a vacuum source because they provide relatively high flow rates and because they can reach relatively deep vacuum levels, such as 10-3 mbar. The same can be said of kilo labs. However, rotary vane pumps also include several critical disadvantages which make their use in pilot plants and kilo labs suboptimal. Most notably, these disadvantages include significant maintenance requirements in terms of both time and material related costs. By switching from an oil pump to an oil-free technology such as a diaphragm pump, there are significant benefits to be realized. These benefits include better process control, chemically inert wetted materials, and significantly reduced maintenance.

DSM Nutritional Products recently worked with VACUUBRAND GMBH to realize these benefits in its mini-plant technical center in Sisseln, Switzerland. DSM Nutritional Products converts pure ingredients into formulations suitable for commercial use as additives in food (ice cream, beverages), pharmaceutical products (tablets, capsules), feed (cube, premixes), and cosmetics. It falls to the researchers in Kaiseraugst to develop methodologies which ensure that the product has the necessary bioavailability, stability, and solubility characteristics. The team in Sisseln is then responsible for scaling this process from research-scale to pilot-scale while simultaneously optimizing the process.

DSM operates “mini-plants” to optimize process parameters. (Similar facilities may be called “pilot plants”or “kilo labs”in other parts of the world.) Plants of this scale may also be used to produce small quantities of product, though DSM does not use its Sisseln mini-plant technical center in this way. Instead, the technical team in Sisseln operates a pilot plant (what may be called a micro plant in other parts of the world), in parallel with its mini-plant facilities, to supply

product for application testing and further R&D work. In order to optimize processes to meet the quality and efficacy standards, DSM makes use of the latest production technologies in their mini-plant facilities. Consequently, DSM has updated their vacuum supply in recent years. In the current instance, DSM’s goal was to replace oil-sealed rotary vane pumps with an oil-free alternative in its mini-plant operations where technically feasible.

DSM’s Initial Analysis

The technicians at DSM worked with counterparts at VACUUBRAND to analyze if it would be feasible for DSM to convert to VACUUBRAND’s line of chemically resistant diaphragm pumps. DSM’s teams identified two critical factors for this project.

The operating vacuum for most applications is in the range of 1 mbar – 500 mbar. Diaphragm pumps are well suited to providing vacuum in this range, often referred to as the “rough vacuum range. ”Certain high vacuum processes like short path distillation require the deeper vacuum provided by rotary vane pumps.

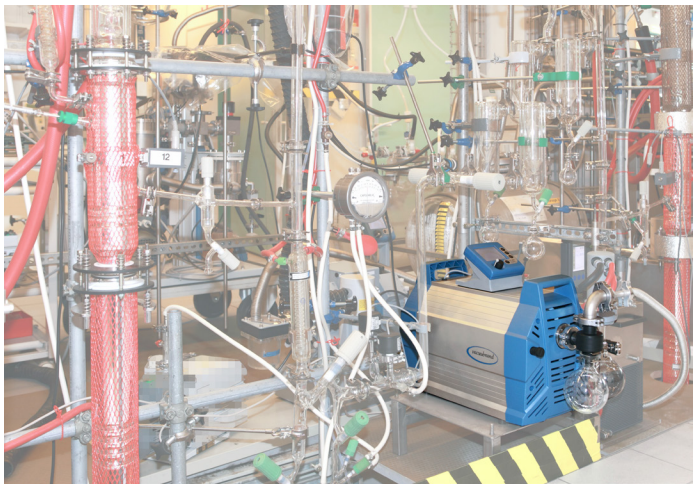
Once the mini-plant set-up has been evacuated down to the operating vacuum level, the expected pumping speed (i.e., flow) is expected to be minimal. Because most of the process vapors condense before reaching the pump, the pumping speed requirement is driven by residual process vapors and system leaks.

Both observations favored a switch from rotary vane pumps to oil free diaphragm pumps. Since the operating vacuum level is within the capabilities of a diaphragm pump, it was clearly possible to use a diaphragm pump to meet the operating vacuum specifications. On the other hand, the system leakage was found to be substantial enough that oversized (rotary vane) pumps were needed. DSM’s analysis of the pumping speed requirements showed that by reducing system leaks, the necessary flow capacity of the pump would

also be well within the rated pumping speeds of diaphragm pump technology. A diaphragm vacuum pump, with its low maintenance requirement and chemically-resistant construction, could be used.

Mini-Plant Optimization

The processes conducted in DSM's mini-plants are normally multi-step operations including rectifications, distillations, evaporations, synthesis steps, or reactions and recrystallizations. Solvent mixtures must be separated and purified, and energy balances must be determined. These plants consist largely of glass components, and around 10 process set-ups are used in a single plant. Equipment such as distillation columns with diameters up to 70mm and a height of 14m are used for these purposes. Similarly, rotary evaporators with flask sizes of up to 50 L and reactors in the range of 0.5 L to 25 L are used.



The glass components that comprise the majority of these set-ups have been found to leak at the joints. These leaks are undesirable as they increase the pumping demand (i.e., flow requirements) so that unnecessarily large and less efficient pumps are required. The analysis performed by DSM showed that in many cases the required vacuum levels permitted a switch from rotary vane pumps to a chemical resistant diaphragm pump provided that the company could reduce flow demands on the pumps by minimizing leakage in the pilot plant equipment.

Historically, glass joints have been sealed using PTFE inserts. PTFE does offer chemical resistance but, as a seal, PTFE is often a poor material choice as it is does not conform well to the variations in the sealing surface. DSM's search for a better seal material led them to the perfluoroelastomer FFKM to replace their PTFE gaskets.

With the FFKM seals installed, the measured pressure rise of the set-up was 0.5 mbar/hr., which corresponds to a leak rate of 0.01 mbar-L/s. During subsequent leak tests, the set-up was found to have an ultimate vacuum of 1x10-2 mbar using a rotary vane pump. This is well beyond the 1 mbar – 500 mbar range identified as critical by DSM staff. Thus, with the change to FFKM seals, the switch to chemically resistant diaphragm pumps became a practical option in most instances.

The Benefits of VACUUBRAND's Diaphragm Pumps

By switching to from rotary vane pumps to VACUUBRAND's newly updated line of diaphragm pumps, DSM has realized a number of benefits, including more precise control, chemically inert wetted materials, and reductions in maintenance requirements.

The Benefits of VARIO® Control

Previously, process pressure was controlled by solenoid valves. These valves were used to meter in small amounts of air or nitrogen, or used to open and close the intake line to the pump in order to maintain the desired operating pressure. These valves were actuated to control pressure while the pump was run continuously at full speed. The so-called "two-point control" provided by the solenoid valves managed the drift of the actual process pressure around the desired process pressure. In addition to the hysteresis this approach introduces, it also reduces the efficiency of the process and reduces solvent recovery efficiency.

VACUUBRAND's variable speed pumps featuring VARIO® control allow for a substantially higher degree of precision than that afforded by simple "two-point control" with solenoid valves. By continuously varying pump speed, VARIO® control ensures the pump maintains the programmed vacuum level without the hysteresis associated with the "two-point control" approach while simplifying the control system at the same time. Furthermore, because the VARIO® control is achieved with a VACUUBRAND CVC 3000 vacuum controller that is integral to the pump, process data is readily available to the data acquisition system. The precise VARIO® control, combined with the ability to integrate with a data acquisition system, greatly helps to realize a pilot plants primary goal of process control and optimization. Finally, by adjusting motor speed to match vacuum demands, VARIO® control also yields significant power savings benefits. Savings of up to 90% can be achieved with VARIO® pump control when compared with a pump that operates continuously at a single speed.

The Benefits of Superior Chemical Resistance

Use of oil-sealed rotary vane pumps meant that harsh chemicals and solvent vapors had to be cooled before they could reach the pump so that these substances would condense and not contaminate the pump oil. The use of cold traps to remove these chemicals by condensing them out of the vapor stream protected the oil-sealed pumps from the damage caused by the incompatibility of the pump oil with the solvents. In spite of the best efforts to remove as much chemical vapor as possible through cold traps, some vapor inevitably reached the pump, necessitating oil changes 1 or 2 times per month under normal circumstances. Extreme circumstances required weekly oil changes or occasional pump rebuilds. These service interruptions required that a stock of standby pumps be maintained in order to avoid system downtime. Once the oil has been removed from the rotary vane pump, the contaminated oil must also be handled according to the appropriate hazardous waste regulations.

In contrast, VACUUBRAND's chemically resistant diaphragm pumps feature fluoropolymer wetted materials, making them a much better match to aggressive chemicals used in this pilot plant while simultaneously requiring less maintenance than rotary vane pumps. In the absence of oil, the residual chemical vapors not caught by the cold trap pass harmlessly through the pump and are either collected by a post-pump condenser or exhausted.

Summary

DSM's decision to work with VACUUBRAND was driven by a desire to reduce DSM's mini-plant operating costs and maintenance requirements. By improving the seals on its mini-plant set-ups, DSM was able to switch from rotary vane pumps to chemically resistant diaphragm vacuum pumps in many of these set ups. DSM was able to make this switch by working with VACUUBRAND to select and install VACUUBRAND's chemically resistant VARIO® pumping units. These pumps provide excellent resistance to aggressive chemicals, and the oil-free operation and variable pumping speed reduce operating costs, decrease maintenance requirements, and provide markedly improved process control.

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