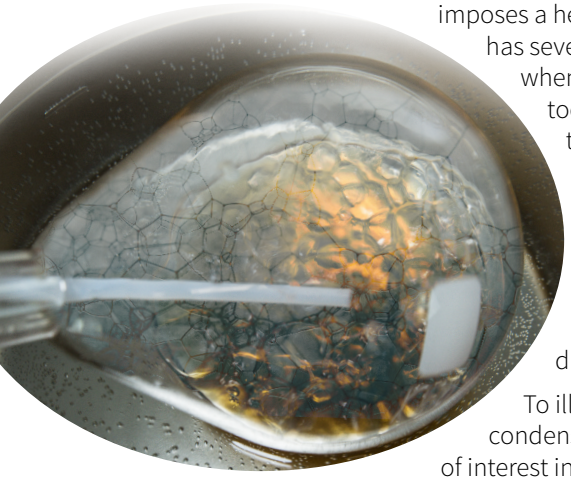
Optimizing Extractions with Vacuum:

Achieving High Yields, High Quality, and High

Rotary evaporators, or rotavaps, are a well-proven tool for extracting a substance of interest from a multi-component solution. Rotavaps separate solvents from the substance of interest by causing the solvents to evaporate. This leaves the substance of interest behind in a rotating flask. The solvent vapors flow through the rotavap until they reach the cold surface of the condenser. Once those vapors come in contact with the condenser, they recondense and flow down into a collection flask, where they can be collected and reused later. This process is a balancing act between the rate of evaporation in the rotating flask and the rate of condensation at the condenser.

In a production setting, the challenge is often how to achieve the balance that brings high yield, high product quality, and high throughput (i.e., short process times). The rates of evaporation and condensation are both dictated by a number of material-specific parameters, such as vapor pressure, and process parameters, like temperature, pressure (vacuum), and surface area. Of these process parameters, temperature and pressure have the greatest impact. However, temperature control brings about slow changes. In contrast, changes to the vacuum level inside the rotavap result in changes which are nearly instantaneous. For this reason, vacuum-based control is the preferred way to maintain the 'right' balance between evaporation and condensation to maximize yield, quality, and throughput.

Some begin process development focused solely on maximizing throughput by maximizing the rate of evaporation without regard to the impact on yield, quality, or cost. For example, the rotavap bath temperature is often set relatively high and the vacuum pump is used to provide the deepest vacuum possible. This approach imposes a heavy load on both the condenser and the vacuum pump, and consequently has several pitfalls. The compound of interest can degrade or breakdown entirely when exposed to high temperature, reducing product quality. Vacuum which is too deep can cause samples to bump and foam, lowering the process yield. And throughput, as measured in terms of sellable product produced per hour of operation, drops due to both low yield and the long process times. Process time increases significantly as the rates of evaporation and condensation become unbalanced. When the rate of condensation is lower than the rate of evaporation, the excess solvent vapor must flow through the vacuum pump. In turn, this requires the use of an oversized pump and results in lower solvent recovery. Oversized equipment and low solvent recovery both drive up costs.



To illustrate the importance of condenser efficiency and balancing the rate of condensation with the rate of evaporation, consider a simple mixture of a compound of interest in solution with ten liters of ethanol. Typical process parameters would include setting the water bath of the rotavap to 60°C (140°F), and targeting a process time of not more than 2 hours. Using a rule of thumb for rotavap operation, the temperature of the solution inside the rotating flask would be approximately 40°C (104°F). At this temperature, we can find that the vapor pressure of ethanol is 179 mbar (134 Torr). If the pressure is above 179 mbar, the ethanol will stay in the liquid state; if the pressure is at or below 179 mbar, ethanol will exist in the gaseous state. In our example, 10 liters of liquid ethanol will expand into approximately 26,700 liters of ethanol vapor at 40°C. In a case where the condenser recovers only 50% of the solvent vapor, the vacuum pump must be sized to accommodate this high vapor flow in the desired process time of two hours, or the process must be allowed more time to complete. In our example, the vacuum pump would need to provide at least 6.7 m^3/hr. (about 112 liters per minute) at 179 mbar to complete the extraction in 2 hours with a 50% solvent recovery rate. Alternatively, an efficient condenser would recover about 95% of the ethanol vapor, collecting 9.5 liters of liquid ethanol. This leaves only about 1335 liters of gaseous ethanol to flow through the pump. Given our nominal 2 hour process time, this means that the pump must be sized to provide a pumping speed of at least 0.7 m^3/hr. (about 11 liters per minute) at 179 mbar. This is well within the capabilities of a smaller, less expensive pump than would be required if the condenser efficiency is low. In addition, the 95% of the ethanol that recovered under this scenario can be reused later, drastically reducing the cost of solvents.

But keep in mind that these sizing estimates depend on holding the vacuum level stable at 179 mbar. If the vacuum level rises too far above 179 mbar, the rate of evaporation will slow and the process will take longer. If the vacuum level drops much below 179 mbar, the rate of evaporation in the flask can be too high and result in low yields and low product quality because of bumping and foaming. This also reduces the amount of solvent recovered. Precise, reliable vacuum control is needed in order to hold the right vacuum level which maintains the proper balance between the rate of evaporation and rate of condensation. Two different methods are commonly used to achieve the necessary vacuum control.

The first approach is referred to as two-point control. This approach can be used with any appropriately sized vacuum pump. A control module is combined with a solenoid valve and a vacuum sensor. Based on the sensor reading, the control module opens and closes the valve to hold the pressure level close to the user-defined set point. Commercially available controllers can offer additional, advanced capabilities. For example, VACUUBRAND's VACUU·SELECT® complete vacuum controller comes configured with many common lab vacuum applications that can be selected at the touch of a button. The VACUU·SELECT controller can be configured to hold a specific, user-adjustable vacuum level. Or, simply by selecting a different application, the controller can be set to automatically detect the first solvent boiling point and then hold the vacuum at that level.

Processes can be optimized with an even better, more precise approach to vacuum control which constantly adjusts the pump motor speed in order to control the vacuum level with exacting precision. Continuous adjustments to the pump motor speed allow the vacuum level to respond instantaneously to the ever-changing conditions inside the rotavap. This is the control scheme used on VACUUBRAND's VARIO® select pumps. The VARIO select pump line integrates the VACUU·SELECT controller and its broad array of preconfigured vacuum applications. And by combining the advanced capabilities of the VACUU·SELECT controller with the variable motor speed control, you can fully automate extraction processes at the touch of a button. The controller continuously adjusts the pumping speed and vacuum level in order to maintain the balance between solvent evaporation and condensation. This 'Automatic Evaporation' application ensures that the process is completed as fast as possible while eliminating sample loss from bumping and foaming and producing repeatable, reliable results.

By maintaining the balance between solvent evaporation and condensation, extraction processes are optimized. Achieving a high, but controlled, rate of evaporation maximizes your rate of production by maximizing both throughput and yield. The cost of equipment and consumables is at the same time driven down as the extraction process is brought into balance. The best and easiest way to realize this balance is to use vacuum control, since extractions and distillation processes are sensitive to changes in vacuum level, and therefore respond rapidly. Easy-to-use products such as VACUUBRAND's VACUU·SELECT complete controllers and VARIO select pumps make it easy and cost-effective to implement the necessary vacuum control, and so balance and optimize your extraction processes.