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AN INNOVATIVE APPROACH TO ROTARY EVAPORATOR DESIGN

INTRODUCTION

Electronic devices have changed chemistry. Just as home kitchen counters now feature machines—such as electric kettles and multicookers—that make food preparation more convenient, so modern laboratory benches have dedicated labor-saving devices. For many labs, especially within organic chemistry, one of the most commonly seen and frequently used pieces of equipment is the rotary evaporator. Next-generation, electronic rotary evaporators are beginning to offer tangible and significant performance benefits.

Usually known by users as a rotovap, these workhorses remove the solvent at the end of virtually any synthetic chemistry procedure and again after chromatography to leave the pure final product. In most pharmaceutical and organic chemistry labs, rotovaps are used so regularly that all lab members have their own machine. With such heavy use, any innovation that makes rotovaps faster, more convenient, or more energy efficient can have major payoffs for lab productivity and environmental sustainability.

ROTOVAP BASICS

At some point in most experimental chemical procedures, solvents in the reaction vessel or those enlisted during purification need to be removed. For most solvents, their relatively low boiling point can be exploited to evaporate off the solvent while leaving behind reaction products.

Chemists have long sought methods to accelerate solvent removal, such as applying a vacuum to lower the solvent's boiling point or warming the vessel. Rotovaps do both, while simultaneously spinning the sample to minimize "bumping." This is when superheated solvent erupts out of the reaction flask, carrying some of the precious reaction product.

Solvent vapor, which may contain hazardous substances, isn't simply vented into the environment, of course. The rotovap contains a condenser unit that cools the solvent vapor, condensing it back into a liquid that is then collected. The captured solvent drips into a receiving flask for safe disposal.

Until recently, the fundamentals of rotovap design and operation had changed little since the first commercial machine was released in 1957 by Swiss company Büchi. First-generation rotovaps, as well as those chemists may recall using just a decade ago, were heavy consumers of water.

"When I did my PhD, we used water aspirators and water running through the rotovap to cool the solvent distillate," says Richmond Sarpong, professor of organic chemistry at the University of California, Berkeley, who entered graduate school in 1995. Water aspirators, attached to a fast-running tap, generated the vacuum. The rotovap user would also continually run tap water through the condenser for cooling. All that water went directly down the drain.

Ecodyst's rotary evaporators incorporate condenser coils that are made from a polymer-coated metal rather than the traditional glass. Combining condenser and chiller into a single unit has proven to be a scalable approach.

Image credit: Ecodyst

One of the first steps in the nascent laboratory sustainability movement, beginning in the early 2000s, was to recognize that rotovaps' water consumption was unsustainable and introduce alternatives, says Kathryn Ramirez-Aguilar, who started the Green Labs program at the University of Colorado Boulder 10 years ago. "Prior to Green Labs, one of my supervisors worked with the chemistry labs on rotovaps," she says. Water aspirators and single-pass water condensers were replaced with vacuum pumps and recirculating chillers, which pump an antifreeze solution through the rotovap condenser's glass coils in a closed-loop system.

Other universities, companies, and research institutions opted to replace liquid coil condensers with "cold finger" attachments filled with dry ice. But each option has its drawbacks, says George Adjabeng, cofounder and CEO of Ecodyst, an instrument company founded in 2014. Adjabeng spent many years in the chemistry lab, including a decade at a major pharmaceutical company, using rotovaps almost daily.

"I grew frustrated with looking for dry ice," Adjabeng says. "Chillers were not an option, for two reasons," he explains. "One, they are heavy and bulky—we couldn't fit them in our fume hood. And second, they are very inefficient. It takes about 45 min to get the condenser down to –10 °C." These frustrations fueled Adjabeng's inventive spirit for a new type of rotovap.

FRUSTRATION-FREE TECHNOLOGY

Adjabeng's prototype had the chiller and condenser combined into a single, efficient, compact unit. "In 2015, I put our first condenser unit in a University of California, Berkeley, lab," he says. "That machine never came back; they liked it so much they bought it."

In conventional rotovap chillers, refrigerant cools a water-antifreeze mixture, which is then pumped to the glass coils of the rotovap condenser. The key advance Adjabeng made—which has gone on to underpin all of Ecodyst's innovations in the rotovap space—was not only to combine condenser and chiller but also to reassess the material used to make the condenser coil. "Everybody assumed glass is the only material chemically resistant enough to use in a rotovap," he says. "Nobody thought of making metallic coils and coating them with a chemically resistant polyfluoromer." The strong carbon-fluorine bonds in fluorine-rich polymers, such as Teflon, are resistant to chemical attack.

The condenser's protective polymer coating has proved robust. "We have had units in industry for almost 5 years now, and they have not had any issues with corrosion," Adjabeng says. "I think we have established this is a better alternative to glass."

The cannabis industry is driving the transition toward stationary-flask rotovaps that have higher capacity than traditional rotating flask designs.

Image credit: Ecodyst

ACADEMIC RESEARCH SUSTAINABILITY

Many universities prioritize environmental sustainability today. The sustainability of university lab space came into question in around 2005, when universities began inventorying their greenhouse gas emissions.

“When those first greenhouse gas inventories were published, the footprint of research labs was a shock,” says Allen Doyle, a lab sustainability consultant in Atlanta who until 2018 was Green Labs program manager at the University of California, Davis. “The lab space at a major research university will typically account for 25–30% of the university’s square footage but 60–70% of their energy budget,” Doyle notes.

The electricity used by devices plugged into electrical sockets around the lab can be a significant contributor to laboratory energy consumption, according to Doyle. “Anything that involves heating is the biggest energy user, then freezing, then vacuum pumps,” he says. “Electric motors in labs are a small draw.” In addition to the direct impact on electricity consumption, appliances get hot during use, and may place more strain on building cooling if airflow is not adequate. The more electricity a device consumes, the more heat radiates into the room.

As scientists use rotovaps only intermittently during the day, the instrument’s impact on a lab’s energy consumption might be expected to be modest. When Ramirez-Aguilar installed electricity meters on rotovap equipment around the university, however, the results were surprising.

“We metered two chillers that were used for condensers on rotovaps,” Ramirez-Aguilar says. The chillers used 8.9 kW h/day and 9.6 kW h/day, about a third of the daily electricity consumption of the average US home. “That’s not a small amount of energy,” she says. The reason the chillers used so much electricity is that they were left running 24/7. “The scientists found they had to keep the chillers turned on to reach the temperatures that they need,” Ramirez-Aguilar says. Green Labs provided timers to automatically shut off chillers at night and restart them in the morning. Even so, a chiller is running for hours during a time the rotovap is not being used. “A condenser that gets down to temperature within a few minutes would be great,” Ramirez-Aguilar says. Ecodyst’s 1 min cool down eliminates that problem.

In Sarpong’s lab, most of the 30 members of his research group cool their rotovap condensers with a cold finger attachment, which runs on dry ice, rather than with chillers. But this approach also has drawbacks. “Dry ice prices are rising through the roof, and the supply is not fully reliable, especially on holidays and weekends when the dry ice delivery company cannot access our buildings,” Sarpong says. Not being able to run a rotovap is a major bottleneck in a synthetic organic chemistry lab, where the reaction solvent invariably needs to be removed to advance from one synthetic step to the next.

That’s where self-cooling units may come in handy. Sarpong’s team members use Ecodyst’s stand-alone EcoChyll condenser, which can be attached to any rotovap in place of a glass coil condenser or cold finger attachment.

The new condensers have been a popular addition to the lab, according to Sarpong. “The fact it cools down very quickly after you turn it on is definitely a convenience,” he says.

The all-in-one rotovap could replace traditional benchtop rotovaps.

Image credit: Ecodyst

THINKING BIG

The needs of the research laboratory may be different than an industry laboratory, but rotovaps are common in both settings. The burgeoning cannabis industry has a particular need for efficient extractions. Cannabis processors use solvent extraction to isolate the bioactive compounds from the rest of the plant material, then use rotovaps to obtain the purified oil. For people setting up small cannabis extraction facilities, dealing with dry ice or consuming large volumes of water to chill a conventional rotovap is not economically viable. As the cannabis industry grows, however, standard rotovap systems are becoming a bottleneck for scaling up operations. Many chemists in academia and industry have experienced frustration when they scale up a reaction and have to evaporate off a much larger volume of solvent than their rotovap was designed to handle.

Since introducing its first product—the EcoChyll condenser-chiller—Ecodyst has dramatically expanded its product offerings, with a focus on addressing the issue of scale. “We designed our cooling technology to be very scalable,” Adjabeng says. In 2017, the company introduced 22, 50, 72, and 100 L systems.

For the multistep organic syntheses undertaken in Sarpong’s lab, bringing starting materials through the first few steps of a synthesis means running reactions at large scale, often above 100 g. Evaporating off the solvent at the end of such a reaction using a benchtop rotovap can be a slow and inefficient process. The weight of a solvent-filled flask can compromise the glass piece that goes in the rotovap and cause leaks.

Demand for larger-capacity rotovaps is particularly acute in the rapidly growing cannabis industry. For example, Tikun Olam, a large Israeli medicinal cannabis company started up 15 years ago, now has subsidiaries in the US and Australia. Until recently, like many cannabis companies, Tikum Olam was using standard industrial-scale, 20 L rotovaps to evaporate off the ethanol used to extract cannabis oils from the plant material. It was a major production bottleneck, even when the rotovaps were run 24/7, according to Oded Lahmish, who heads the company’s wet-production facility in Israel and handles all R&D related to production.

“We realized the cannabis industry needed larger industrial-scale machines,” Adjabeng says.

As with its innovative step to merge the chiller and condenser into a single unit, and to switch to a metal condenser material, Ecodyst has pioneered a path with its larger-scale machines. “We realized that there is no need to rotate the flask,” Adjabeng says. The flasks sit vertical and stationary inside a heating mantle and use high-speed overhead stirrers to minimize bumping. The flask can be filled to capacity because it’s upright, whereas a traditional, 20 L rotovap, which tilts the rotating flask on an angle, can be filled only halfway.

Thar Process, a tolling company, relies on Ecodyst's large-capacity machines to convert customers' cannabis plant biomass to cannabis oil. "Since we do very large-scale extractions, typically 1,000 kg/day of hemp biomass, we require the largest solutions available," says Jason Lupoi, project manager in Thar's chemistry department.

North Carolina hemp extraction firm Innovative AgriProducts is moving in the same direction, says Patrick Healy, the company's extraction lab manager. Healy can fit a whole extraction batch into each 72 L Ecodyst machine, which he says has a relatively compact footprint because of its integrated design. "They fit within our building without a whole lot of building modifications or drilling holes in walls for chillers and heaters."

In cannabis processing, removing the ethanol to isolate the extracted oil is usually the biggest bottleneck. "It's not just the evaporation but unloading the product out of the machine at the end" that slows the process, Healy says. The consistency of raw cannabis oil is similar to that of thick molasses, so removing it from the system can be a time-consuming and messy process involving unwanted machine downtime. Ecodyst's larger units feature valves in the bottom of the flask that speed this process.

"With these larger models, we can just drain [the oil] out of the bottom," Healy says. Ecodyst and the Innovative AgriProducts team have worked together to refine various aspects of the machine's design, including the valve.

Tikun Olam says its 50 L Ecodyst machine is now paying dividends. Rather than the 3–4 L/h evaporation rate it was seeing with a 20 L standard rotovap, the Ecodyst machine is removing 9–12 L/h. "In our protocol, it shortened a 4-day process to 1.5 days," Lahmish says. The system also saves energy. Ecodyst's 100 L machine consumes 10 kW of power—about the same as a traditional, 20 L rotovap—but has 10 times the capacity because the angled flask on the 20 L rotovap.

The large-scale machines have already caught the eye of researchers in other chemistry-related industries—even bringing Adjabeng full circle, back to the pharmaceutical industry where he had worked. "We recently set up a 100 L machine for a pharma company," he says. Previously, when faced with a large volume of solvent, the company's scientists would split it between four 20 L rotovaps. "Then they would wait and wait," he says. "On a conventional 20 L machine, for a solvent like ethyl acetate, they were removing 10 L an hour if they were lucky. In our system, they were getting 50 L an hour."

There are two factors at play behind the enhanced speed, Adjabeng says. With their very efficient metal condensers, filled directly with refrigerant, researchers can turn up the heat on the sample to evaporate solvent vapors faster, without the risk of overheating the condenser.

Ecodyst's stationary systems are particularly suited to liquid products because they feature drain valves at the bottom to collect the product at the end of the process. Solid products can be handled by the system, too. For convenient product isolation, however, chemists may prefer to use the 100 L machine to quickly get the solvent volume down to a manageable 10 L and then switch to a conventional rotovap to finish the task.

The environmental and productivity benefits of the new generation of rotovaps are only magnified at scale. However big they get, they remain the favorite labor-saving electronic device of most bench chemists.

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